WOOD PROPERTIES OF NINE ACETYLATED TROPICAL HARDWOODS FROM FAST-GROWTH PLANTATIONS IN COSTA RICA

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Abstract. The treatment of acetylation on tropical woods is influenced by their different levels of permeability and how these affect the weight percentage gain (WPG) in acetylated wood. The objective of this study was to identify the effect of acetylation on physical properties, hygroscopic and dimensional stability, wetting rate, and durability of nine tropical species of hardwoods used for commercial reforestations in Costa Rica. The results showed that WPG varied from 2.2% to 16.8% among species. Positive significant correlations were observed between WPG and two parameters of dimensional and hygroscopic stability, whereas a negative correlation was observed with water absorption (WA). For species with a WPG of over 10% (*Vochysia ferruginea*, *Vochysia guatemalensis*, *Cordia alliodora*, and *Enterolobium cyclocarpum*) wetting rate, hygroscopic stability, and resistance to biological attack showed an increase while swelling, and WA decreased. For these species, the best behaviors were obtained with an acetylation time of 2.5 h. The same properties of wood in species with a WPG under 5% were found to be less affected by the different acetylation times and showed little difference in relation to untreated wood. Finally, the analysis showed that the dimensional stability obtained was attributed to the reduction of the absorptive capacity of the acetylated wood.

Keywords: Moisture absorption, wood properties, tropical wood, fast-growth plantation, dimensional stability.

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INTRODUCTION

Wood has several commercial and noncommercial uses, and as an industrial material, it is easily degraded or affected in presence of moisture (Adebawo et al 2016). Components of wood (ie lignin, cellulose, and hemicellulose) contain free hydroxyl radicals (Rowell 2012) that adsorb and release water depending on changes in temperature and RH conditions. This, in turn, causes the cell walls to adjust to the presence (or absence) of moisture, thereby, provoking changes in the dimensions of lumber (Giridhar et al 2017).

Recent research has studied chemical modifications to wood toward decreasing water absorption (WA) and, thus, to improve dimensional stability (Mantanis 2017), without altering other properties (Rowell 2006). Among these chemical treatments, most common is the use of acetic anhydride (Hill 2006; Rowell 2006), wherein the OH^- anion group of the wood components becomes chemically bound to a residue of the acetate (CH_3COO) of an acetic anhydride molecule ($(CH_3CO)_2$). This treatment is known as the acetylation process where the OH^- anion group is reduced, which decreases the wood hygroscopicity and increases its dimensional stability (Adebawo et al 2016). Other important wood properties take benefit from acetylation (Gérardin 2016; Mantanis 2017), notably: natural resistance to fungi and insects (Fojutowski et al 2014; Rowell 2016); resistance to marine conditions; reduced wettability (Bongers et al 2016) and wood swelling (Kozarić et al 2016); and increased wood hardness (Rowell 2006).

Polymer composition and distribution in hardwood species differ from those of softwood species (Engelund et al 2013), and, as such, the acetylation produces varied effects (Rowell 2016). In hardwood species, hydroxyl groups are reported to be present at proportions of 2.0% to 4.5%, whereas in softwoods these vary from 0.5% to 1.7% (Rowell 2016). This difference is attributed to the hemicellulose and lignin compositions, ie in hardwood species, hemicelluloses include glucuronoxylan, xyloglucan, and glucomannan, while softwood hemicellulose is primarily composed of xyloglucan, arabinoglucuronoxylan, and galactoglucomannan, as well as lignin in a lesser proportion (Wang et al 2017).

Studies have indicated that acetylated hardwoods achieve lesser weight percentage gain (WPG) compared with acetylated softwood species (Rowell 2016). Nonetheless, compared with softwoods, hardwoods contain a higher content of xylans, which do not contain a primary hydroxyl group in which to react (Rowell 2014). Moreover, softwood species contain a greater percentage of lignin, the component where the higher percentage of acetylation has been demonstrated (Rowell 2016).

In addition to the difference in the type and proportion of hemicellulose, the anatomical structure differs significantly between both wood species groups, eg hardwoods are characterized by the presence of conducting elements such as vessels, whereas softwoods are made of tracheids (Gibson 2012). This distinction causes the flow of liquid to vary between both groups (Gaitán-Álvarez et al 2020), therefore affecting the acetylation process, which is associated with liquid flow in wood (Kozarić et al 2016).

Despite these differences and the studies conducted on softwoods, relatively lesser research on hardwoods has been carried out, including Matsunaga et al (2016) and Gaitán-Álvarez et al (2021). WPG for tropical hardwoods ranges from 4% to 18%, values lower than those observed in softwood species (Gaitán-Álvarez et al 2021). This difference can be attributed mostly to the flow of liquid in wood, regulated by its permeability, which is determined by the anatomical elements involved in the flow of liquids (Emaminasab et al 2017). In these initial studies, the change in wood properties due to acetylation remains unknown, which has not allowed expanding the uses of hardwood species (Bollmus et al 2015).

In Central America, Costa Rica has implemented reforestation programs with fast-growth plantations that use a variety of hardwood species for lumber production (Adebawo et al 2019). In these programs, early-age tree harvesting yields juvenile wood, which is characterized by dimensional instability (Moya 2018). In line with this gap, a series of treatments have been implemented to improve lumber properties (Gaitán-Álvarez et al 2020, 2021; Tenorio and Moya 2021) and reduce the problems surrounding the durability and dimensional stability of these species (Moya et al 2017; Gaitán-Álvarez et al 2020). In such situations, acetylation provides an opportunity to improve dimensional stability, durability, and other wood properties of hardwood species (Mantanis and Young 1997; Rowell 2006, 2016; Adebawo et al 2016). Given that plantation species in tropical regions represent a great opportunity for production, it is very important to increase wood durability and dimensional stability to render added value to the products (Kojima et al 2009).

Therefore, the objective of the study was to evaluate the effect of three different acetylation times with acetic anhydride on nine hardwood species commonly used in commercial reforestation in Costa Rica. The effects of the acetylation on physical properties, hygroscopic stability, dimensional stability, wetting rate, and durability were thus evaluated.

METHODOLOGY

Materials

The species studied were *Cedrela odorata*, *Cordia alliodora*, *Enterolobium cyclocarpum*, *Gmelina arborea*, *Hieronyma alchorneoides*, *Samanea saman*, *Tectona grandis*, *Vochysia ferruginea*, and *Vochysia guatemalensis.* The wood of these species presents generally good permeability (Tenorio et al 2016) and has shown potential behavior for wood modification (Gaitán-Álvarez et al 2020). Gaitán-Álvarez et al (2020, 2021) presented the characteristics and origin of these woods in great detail. The wood samples were dried to achieve a MC ranging between 12% and 15% and comprised mostly of sapwood, to insure that juvenile wood is used. The reagents used were: acetic anhydride $(CH_3CO)_2O$ at 98% concentration, commercial brand J.T. Baker (Madrid, Spain) (https://www.fishersci.es/es/es/brands/IPF8MGDA/jt-baker.html); and glacial acetic acid CH_3COOH at 99% concentration, distributed by Químicos Holanda Costa Rica S.A. (Costa Rica) (https://www.brenntag.com/locations/en/brenntag-locations/loc-809-qu%C3%ADmicos-holanda-costa-rica-s-a.jsp).

Acetylation Process

For each species, three groups of 15 samples of 50 mm (R) × 50 mm (T) × 20 mm (L) in oven-dry condition (at 103°C for 24 h) were treated with different acetylation times per group. Another group of 15 samples were left untreated (control) to be compared with the acetylated material. A detailed description of the acetylation process is given in Gaitán-Álvarez et al (2020). The process consisted in applying vacuum for 15 min at −70 kPa (gauge mark), after which the solution of acetic anhydride and glacial acetic acid was introduced, in a 92:8 proportion, respectively. Once inside, the contents were subjected to a 690 kPa pressure for 30 min, after which, the excess liquid solution was extracted and nitrogen gas was injected to serve as the inert medium to control the internal temperature of wood samples. For the reaction, the temperature was fixed at 120°C and three different acetylation times were applied per species: 1.0, 2.5, and 4.0 h, labeled 1 h-acetylation-time, 2.5 h-acetylation-time, and 4 h-acetylation-time, respectively. Then, the acetylated samples were again oven-dry at 103°C for 24 h. Finally, the oven-dry weight of all samples was measured before placing them in a conditioning chamber at 20°C and 65% of RH until reaching a constant weight.

Evaluation of the Acetylation Process

The dimensions (ie length, width, and thickness) and oven-dry weight of the acetylated samples were measured before and after the treatment. The acetylation process was evaluated by determining the uptake of the solution (uptake) (Eq 1) and by the WPG (Eq 2) considering the oven-dry weight before acetylation as the initial weight.

$$\text{Uptake}\left(\frac{\text{liters}}{\text{m}^3}\right) = \left(\frac{\text{Weight}_{\text{after acetylation}}(\text{g}) - \text{Weight}_{\text{before acetylation}}(\text{g})}{\text{Volume of sample (cm}^3)}\right) \times \frac{1\ \text{liter}}{1000\text{g}} \quad (1)$$

$$\text{Weight percentage gain (WPG)} = \frac{\text{Weight}_{\text{after acetylation}}(\text{g}) - \text{Weight}_{\text{before acetylation}}(\text{g})}{\text{Weight}_{\text{before acetylation}}(\text{g})} \times 100 \quad (2)$$

Wettability Measurement

The contact angle was determined with an FTÅ ° D200 imaging goniometer (Folio Instruments Inc., Ontario, Canada) at 20°C. One drop (6 mL) of pure water was added to wood surfaces with an injection microsyringe. Measurements were carried out in the longitudinal direction. The contact angle was calculated as a mean of both sides of the drop to compensate for any horizontal variations. The procedure used by Cool and Hernández (2011) was followed in this study. Two contact angles were measured, ie the initial contact angle ($\theta_{initial}$) and the contact angle at 30 s (θ_{30s}). Afterward, the wetting rate was calculated as the variation of the contact angle ($\theta_{initial} - \theta_{30s}$) over the first 30 s of wetting to assess the spreading and penetration of pure water. This procedure was performed on five samples per treatment, for all wood species studied.

Physical Properties

Partial tangential swelling (TS) was determined by the changes in dimensions of samples occurring between 65% and 85% RH, at 22°C (Eq 3). Thus, the dimensions and weight of treated and untreated samples were first measured once conditioned at 65% RH. The samples were then equilibrated at 22°C and 85% RH according to ASTM D4933-16 standard (ASTM 2021). After conditioning, the dimensions and weight of samples were again measured. This hygrothermal condition was chosen because the Atlantic and some Pacific areas in Costa Rica present high RH, where wood can reach equilibrium moisture contents up to 18% MC. The sorption ratio (S) was used to evaluate the hygroscopic stability of wood. This ratio characterizes the sensitivity of changes in EMC (ΔEMC) to changes in RH (ΔRH) and is defined according to Eq 4 (Hernández 2007a). However, the differential TS ratio (G_T) was used to assess the dimensional stability of wood, which is defined according to Eq 5. These parameters (S and G_T) assume a linear relationship between EMC and RH and between TS and EMC, respectively (Hernández 2007b). Therefore, S and G_T values were calculated between 65% and 85% RH as follows:

$$\text{Partial tangential swelling (\%)} = \frac{\text{dimension at 85\% RH (mm)} - \text{dimension at 65\% RH (mm)}}{\text{dimension at 65\% RH (mm)}} * 100 \quad (3)$$

$$S\,\text{ratio factor} = \frac{\Delta\text{EMC}}{\Delta\text{RH}} = \frac{\text{EMC}_{\text{at }85\%} - \text{EMC}_{\text{at }65\%}}{85 - 65\%} \quad (4)$$

$$\text{Differential tangential swelling ratio (GT)} = \frac{\text{Partial tangential swelling from 65\% to 85\% (\%)}}{\text{MC}_{\text{at }85\%} - \text{MC}_{\text{at }65\%}} \quad (5)$$

Besides these wood properties, three other parameters were evaluated: 1) the difference in ΔMC between acetylated and untreated wood, 2) the difference in S ratio between acetylated and untreated wood, and 3) the difference in G_T between acetylated and untreated wood. These factors were determined according to Eq 6.

$$\text{Difference in } \Delta\text{MC}, \text{S ratio or GT } \Delta\text{MC}, \text{S ratio or GT}_{\text{untreated wood}} - \Delta\text{MC}, \text{S ratio or GT}_{\text{acetylated wood}} \quad (6)$$

(WA was also determined by immersion in cold distilled water for 24 h. For this, another set of 60 samples (ie 15 samples untreated and 15 samples for each time of acetylation, 1.0, 2.5, and 4.0 h) per species were weighed before and after the immersion in water. WA was calculated using Eq 7, following the ASTM D1037-12 standard (ASTM 2020).

$$\text{Water absorption} = \frac{\text{Weight}_{\text{atfer submersion for 24 h C}}(\text{g}) - \text{Weight}_{\text{oven-dry}}(\text{g})}{\text{Weight}_{\text{oven-dry}}(\text{g})} * 100 \quad (7)$$

Durability

The accelerated laboratory test of natural decay resistance was carried out according to the ASTM D-2017-81 standard (ASTM 2014). For each treatment and species, 30 samples measuring 2 cm wide, 2 cm long, and 2 cm thick were prepared. Two types of fungi were tested, namely, *Trametes versicolor* and *Lenzites acuta*, which correspond to white- and brown-rot fungi, respectively. For each type of fungus, 15 samples were exposed to fungal degradation per species and treatment.

Statistical Analysis

Data were tested for normality and homogeneity, and outliers of the variables evaluated were removed. For each species and treatment, the mean, standard deviation, and coefficient of variation were determined for each property studied. A variance analysis (ANOVA) was performed for each species, where wood properties were the dependent variables and time of acetylation was the source of variation, but for WGP was performed two-way ANOVA, where species, time of acetylation and the specie*time integrations were independent variables and WPG as dependent variable. The statistical significance level of $p < 0.05$ was applied to determine the effect of acetylation time. Moreover, Tukey's test was used to determine the statistical significance of the difference between the means of the properties. This analysis was performed with the SAS 9.4 program (SAS Institute Inc., Cary, NC). After, the effect of acetylation of wood, measured by WPG, on wood properties was established by Pearson correlation coefficient, where all properties were cataloged as a dependent variable and WPW as an independent variable.

RESULTS

Evaluation of the Acetylation Process

The evaluation of WPG by two-way ANOVA showed that species, time of acetylation and the interaction were statistically significant with F-values of 256.6, 6.9, and 4.8, all these valued statistically significant at *P*-value less than 0.05. The WPG varied between 2.2% and 16.8% (Fig 1). No statistical differences existed in the WPG among acetylation times for *V. ferruginea*, *V. guatemalensis*, *C. alliodora*, *C. odorata*, *G. arborea*, and *T. grandis*. However, significant statistical differences among acetylation times were observed in *E. cyclocarpum*, *S. saman*, and *H. alchorneoides*. Moreover, *E. cyclocarpum* and *S. saman* showed the highest WPG after 2.5 h-acetylation-time, whereas *H. alchorneoides* showed it after 1 h-acetylation-time (Fig 1). In relation to species, it was found that *V. ferruginea*, *V. guatemalensis*, *C. alliodora*, and *C. odorata* wood had the same performance of WPG in three times tested (Fig 2[a]-[c]), but the differences between species varied in the relation to time of acetylation for other species (*E. cyclocarpum*, *C. odorata*, *G. arborea*, *S. saman*, *H. alchorneoides*, and *T. grandis*), (Fig 2).

Wettability Measurement

The acetylation process decreased the wetting rate in *V. ferruginea* (Fig 3[a]), *V. guatemalensis* (Fig 3[b]), *S. saman* (Fig 3[e]), and *T. grandis* (Fig 3[i]). Untreated samples presented a significantly higher value compared with all acetylated samples and no statistical differences were observed among acetylation times, in these species. No statistical difference was found in *G. arborea* and *C. odorata* between untreated and acetylated samples and between acetylation times (Fig 3[g] and [h]). The acetylation did not affect the wetting rate in relation to the untreated samples and the 4.0 h-acetylation-time in *E. cyclocarpum* (Fig 3[c]), in 1.0 h-acetylation-time of *C. alliodora*, in 2.5 h- and 4.0 h-acetylation-time for *H. alchorneoides* (Fig 3[f]). A significant decrease in wetting rate was found for 2.5 h-acetylation-time for *E. cyclocarpum* (Fig 3[c]), for 2.5 h- and 4.0 h-acetylation-time for *C. alliodora* (Fig 3[d]), and 1.0 h-acetylation-time for *H. alchorneoides* (Fig 3[f]).

Physical Properties

The main physical properties for untreated and treated wood samples are presented in Table 1.

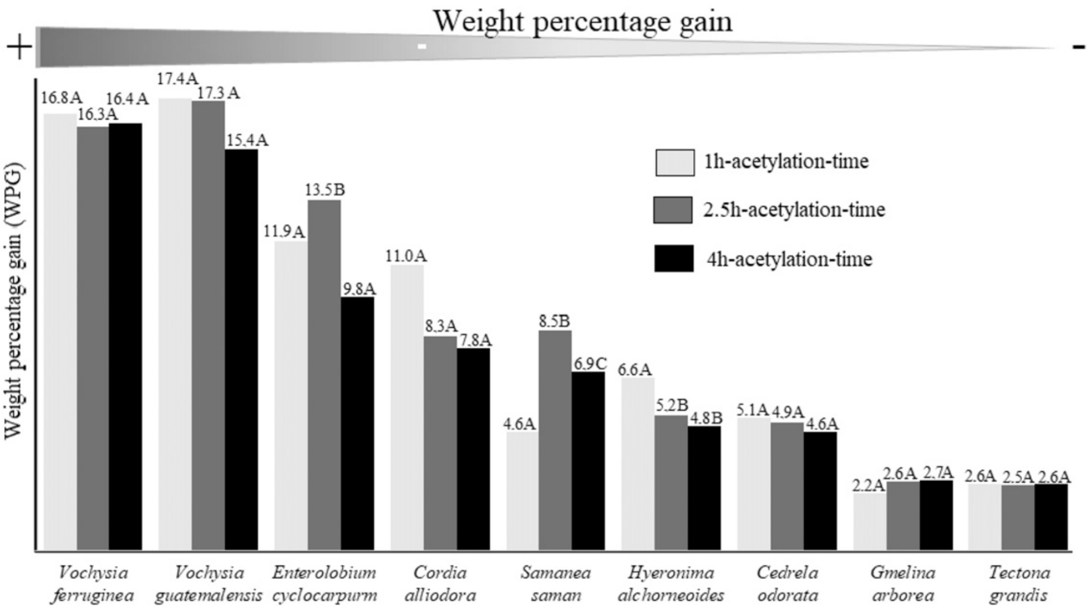

Figure 1. Weight percentage gain of nine fast-growth tropical species of Costa Rica for treated samples with three acetylation times. Legend: Different letters between acetylation times for a given parameter indicate statistical differences at 99%.

Wood density for original (untreated) woods at 65% RH varied between 349 kg/m^3 for *C. odorata* and 577 kg/m^3 for *H. alchorneoides.* Because this property is directly related to wood porosity, this could affect the WPG as discussed later. As expected, density generally increased after the acetylation process since that samples absorbed the chemical components. In some cases, density decreased after treatment, which however was attributed to differences in the initial density among the untreated group and the three treated groups of samples studied.

The MC at 65% RH for the untreated samples varied between 11.3% for *V. ferruginea* and 13.4% for *S. saman* (Table 1). These differences in EMC for a same RH could be due to the different amounts of extractives between the hardwoods studied (Hernández 2007a). As a result, comparisons on the effect of acetylation on hygroscopicity for the nine species becomes difficult. For this, comparisons of ΔMC and sorption ratio were more appropriate.

After wood samples were conditioned at 85%, the ΔMC values from 65% to 85% RH were all lower than those of untreated samples. The effect of the time of acetylation on ΔMC was generally not statistically significant, except for *S. saman*, where ΔMC means were different among different times of acetylation (Table 1). The sorption ratio (S) showed a similar behavior compared with ΔMC values, S decreased with the acetylation treatment, and some statistical differences were found among acetylation times (*S. saman*, *H. alchorneoides*, *G. arborea*, *C. odorata*; Table 1). Moreover, it was observed that the differences between the values of ΔMC and S factor of acetylated wood with untreated wood were lower in the species with lower WPG.

The partial TS significantly decreased after acetylation for all species (Table 1). There were no statistical differences between the acetylation times for *V. ferruginea*, *C. alliodora*, *H. alchorneoides*, *G. arborea*, and *C. odorata*. The highest partial TS among treatments was measured for 4.0 h-acetylation-time of *V. ferruginea*, *E. cyclocarpum*, *S. saman*, and *T. grandis* (Table 1). The differential TS ratio (G_T) was not affected by the acetylation in *V. ferruginea*, *V. guatemalensis*,

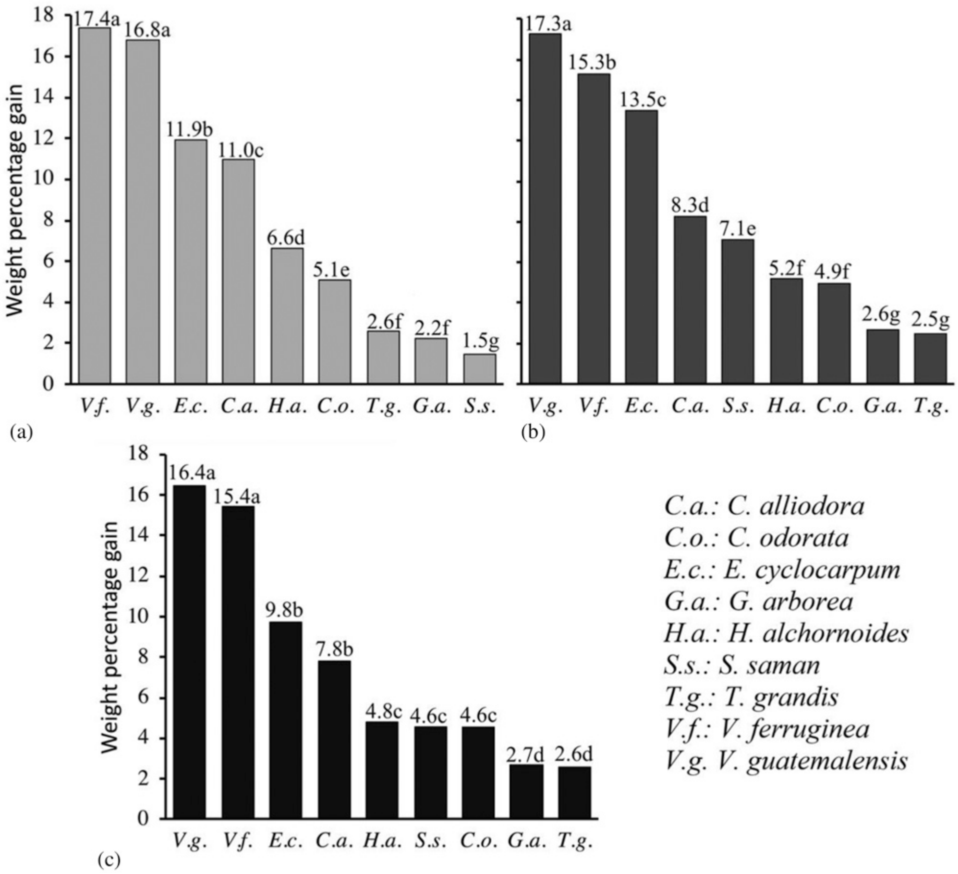

Figure 2. Weight percentage gain for (a) 1 h-acetylation, (b) 2.5 h-acetylation, and (c) 4 h-acetylation of nine fast-growth tropical species of Costa Rica. Legend: Different letters between acetylation times for a given parameter indicate statistical differences at 99%.

E. cyclocarpum, *C. alliodora*, *G. arborea*, *C. odorata*, and *T. grandis* but some acetylation time affected G_T parameter in *S. saman* and *H. alchorneoides*.

The acetylation did not affect the WA in *H. alchorneoides* and *G. arborea* in relation to untreated samples. However, WA decreased after acetylation in the three-time tested in relation to untreated samples for *V. guatemalensis*, *V. ferruginea*, *C. alliodora*, *S. saman*, and *T. grandis*. Also, this parameter (WA) decreased in 2.5 h-acetylation-time in *E. cyclocarpum* and 2.5 h- and 4.0 h-acetylation-time in *C. odorata*. In relation to different times of acetylation, no differences were found in *V. guatemalensis*, *H. alchorneoides*, and *G. arborea.* The lowest values of WA were found in 1.0 h-acetylation-time in *S. saman*, in 2.5 h-acetylation-time of *E. cyclocarpum* and *C. alliodora*, 2.5 h- and 4.0 h-acetylation-time of *C. alliodora*, in 4.0 h-acetylation-time of *V. ferruginea* and *T. grandis*, and 2.5 h- and 4.0 h-acetylation-time of *C. odorata* (Table 1).

Decay Resistance

In general, the acetylation process increased the resistance to decay, especially in species with higher values of WGP (Fig 4). For *L. acuta*, the acetylation treatments in all tested times did not

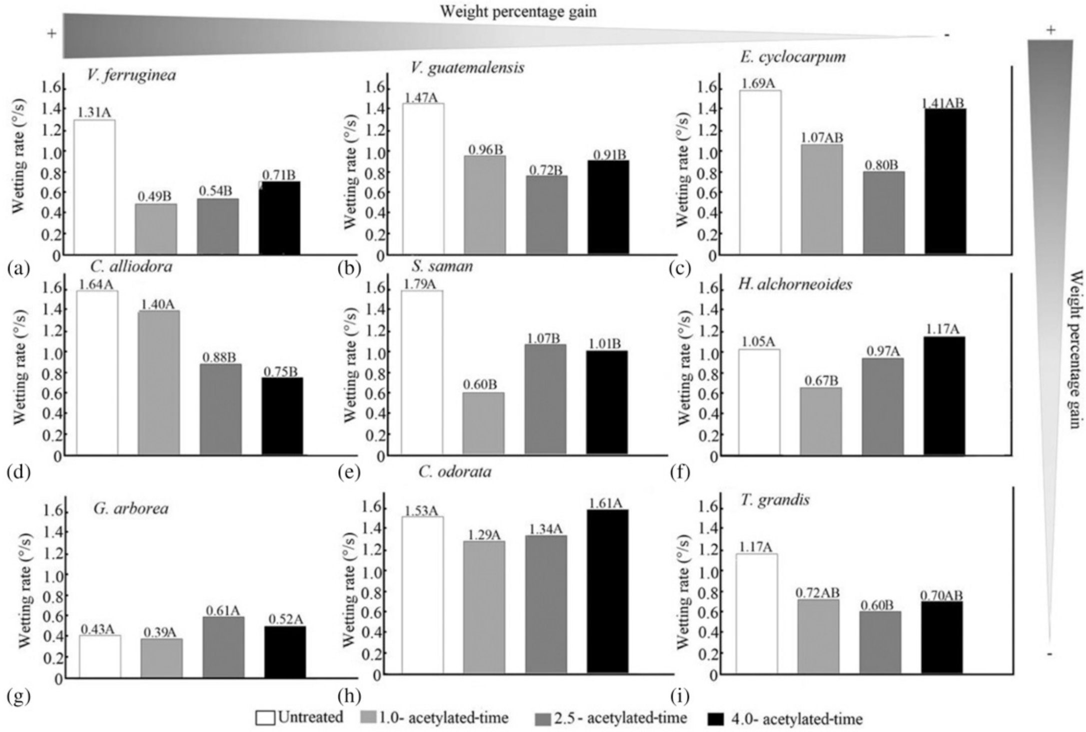

Figure 3. Wetting rate of nine fast-growth tropical species of Costa Rica for untreated and treated samples with three acetylation times. Legend: Different letters between acetylation times indicate statistical differences at 99% for each species.

affect weight loss in *H. alchorneoides* and *G. arborea*, in relation to the untreated samples. Weight loss decreased after acetylation three times in relation to untreated samples for *V. guatemalensis*, *V. ferruginea*, *C. odorata*, and *S. saman*. The lowest weight loss in three acetylation times was presented in *V. guatemalensis* and *S. saman*, in 2.5 h-acetylation-time in *V. ferruginea*, *E. cyclocarpum*, and *C. alliodora*, and in 4.0 h-acetylation-time in *T. grandis*. In relation to different times of acetylation, no differences were found in *V. guatemalensis*, *H. alchorneoides*, and *G. arborea.* The lowest values of weight loss of *L. acuta* fungus were found in 2.5 h-acetylation-time of *V. ferruginea*, *E. cyclocarpum*, *C. odorata*, and *C. alliodora*, 4.0 h-acetylation-time of *C. alliodora*, *T. grandis*, and *C. odorata* (Fig 4).

For *T. versicolor* (Fig 4), the acetylation process decreased the weight loss of this fungus in relation to untreated samples in *V. guatemalensis*, *V. ferruginea*, *S. saman*, *G. arborea*, and *C. odorata.* But this process affected *E. cyclocarpum*, *C. alliodora*, and *T. grandis* in 2.5 h-acetylation-time and for 4.0 h-acetylation-time of *C. alliodora*. For different time of acetylation, no affection was found in *V. guatemalensis*, *H. alchorneoides*, *G. arborea*, and *C. odorata*; also, the lowest weight loss was measured in 1.0 h- and 2.5 h-acetylation-time of *V. ferruginea*, in 2.5 h-acetylation-time of *E. cyclocarpum* and *T. grandis*, in 2.5 h- and 4.0 h-acetylation-time for *C. alliodora* and in 1.0 h-acetylation-time of *S. saman* (Fig 4).

Correlation Between WPG and Wood Properties of Acetylated Wood

A statistically positive correlation was observed between the WPGs, while a statistically negative correlation appeared in WPG with WA, Conversely, no correlation was found among wetting

Table 1. Density, MC at 65% RH, ΔMC, S ratio, partial tangential swelling (TS), differential TS, and water absorption (WA) of nine fast-growth tropical species of Costa Rica for untreated and treated samples with three acetylation times.

	Species	Acetylation time (h)	Density at 65% (kg/m^3)	MC at 65% RH	ΔMC from 65% to 85% RH	S ratio	Partial TS from 65% to 85% RH (%)	Differential TS (G_T)	WA after immersion for 24 h (%)
Weight percentage gain	*Vochysia ferruginea*	Untreated	317.6^{A}	11.3^{A}	6.91^{A}	0.35^{A}	1.79^{A}	0.26^{A}	67.2^{A}
		1.0	330.5^{A}	8.32^{B}	3.48^{B}	0.17^{B}	0.85^{B}	0.25^{A}	40.8^{B}
		2.5	340.4^{A}	8.31^{B}	3.35^{B}	0.16^{B}	0.85^{B}	0.25^{A}	40.4^{B}
		4.0	327.0^{A}	9.16^{B}	3.58^{B}	0.17^{B}	1.15^{C}	0.32^{A}	47.4^{C}
	Vochysia guatemalensis	Untreated	303.6^{A}	11.86^{A}	7.61^{A}	0.38^{A}	1.49^{A}	0.21^{A}	62.8^{A}
		1.0	317.1^{A}	8.5^{B}	4.32^{B}	0.22^{B}	0.88^{B}	0.21^{A}	48.8^{B}
		2.5	316.5^{A}	8.02^{C}	4.00^{B}	0.20^{B}	0.92^{B}	0.23^{A}	50.3^{B}
		4.0	355.7^{B}	9.12^{BC}	4.27^{B}	0.21^{B}	1.03^{B}	0.24^{A}	49.5^{B}
	Enterolobium cyclocarpum	Untreated	461.4^{A}	12.16^{A}	6.39^{A}	0.29^{A}	1.41^{A}	0.22^{A}	30.8^{A}
		1.0	466.5^{A}	9.09^{B}	3.57^{B}	0.18^{B}	0.67^{B}	0.19^{A}	29.9^{A}
		2.5	446.1^{A}	8.16^{C}	3.00^{B}	0.15^{B}	0.60^{B}	0.20^{A}	26.6^{B}
		4.0	479.9^{A}	9.56^{B}	3.74^{B}	0.19^{B}	0.90^{C}	0.24^{A}	30.9^{A}
	Cordia alliodora	Untreated	378.5^{A}	12.02^{A}	6.27^{A}	0.28^{A}	1.63^{A}	0.26^{A}	51.9^{A}
		1.0	388.4^{A}	9.86^{B}	3.67^{B}	0.17^{B}	0.81^{B}	0.24^{A}	35.8^{B}
		2.5	421.7^{B}	8.21^{C}	3.53^{B}	0.15^{B}	0.87^{B}	0.26^{A}	25.5^{C}
		4.0	381.6^{A}	8.70^{CD}	3.27^{B}	0.14^{B}	0.76^{B}	0.24^{A}	30.6^{B}
	Samanea saman	Untreated	522.9^{A}	13.36^{A}	6.47^{A}	0.32^{A}	1.74^{A}	0.27^{A}	35.1^{A}
		1.0	539.2^{A}	8.64^{B}	2.80^{B}	0.13^{B}	0.43^{B}	0.16^{B}	20.3^{C}
		2.5	546.8^{A}	10.77^{C}	3.89^{C}	0.19^{C}	0.99^{C}	0.25^{A}	39.4^{B}
		4.0	570.5^{B}	9.96^{D}	3.59^{D}	0.16^{B}	1.14^{C}	0.33^{C}	33.4^{B}
	Hieronyma alchorneoides	Untreated	556.7^{A}	12.52^{A}	6.20^{A}	0.31^{A}	1.93^{A}	0.31^{A}	28.1^{A}
		1.0	590.4^{B}	12.65^{A}	4.07^{B}	0.18^{B}	1.28^{B}	0.32^{A}	25.1^{A}
		2.5	526.3^{C}	12.60^{A}	4.33^{B}	0.22^{C}	1.19^{B}	0.27^{B}	26.1^{A}
		4.0	543.4^{C}	12.40^{A}	4.31^{B}	0.22^{C}	1.23^{B}	0.29^{AB}	25.4^{A}
	Gmelina arborea	Untreated	422.3^{A}	11.77^{A}	6.06^{A}	0.30^{A}	1.41^{A}	0.23^{A}	18.3^{A}
		1.0	397.7^{B}	10.96^{A}	4.42^{B}	0.22^{B}	1.00^{B}	0.23^{A}	24.1^{A}
		2.5	466.4^{C}	10.74^{A}	3.85^{B}	0.19^{C}	1.08^{B}	0.28^{A}	22.7^{A}
		4.0	409.1^{A}	9.71^{B}	4.37^{B}	0.22^{B}	1.07^{B}	0.24^{A}	25.9^{A}
	Cedrela odorata	Untreated	317.1^{A}	12.10^{A}	7.86^{A}	0.31^{A}	1.67^{A}	0.21^{A}	56.8^{A}
		1.0	286.2^{A}	10.79^{B}	5.98^{B}	0.30^{B}	1.14^{B}	0.19^{A}	53.3^{A}
		2.5	297.8^{A}	11.02^{B}	5.43^{B}	0.27^{C}	0.92^{B}	0.17^{A}	40.6^{B}
		4.0	282.7^{A}	10.50^{B}	5.44^{B}	0.25^{C}	0.90^{B}	0.17^{A}	41.2^{B}
	Tectona grandis	Untreated	495.4^{A}	11.88^{A}	5.96^{A}	0.27^{A}	1.00^{A}	0.17^{A}	35.6^{A}
		1.0	538.0^{AB}	9.23^{B}	4.75^{B}	0.24^{B}	0.68^{B}	0.14^{A}	22.6^{B}
		2.5	528.8^{AB}	8.58^{B}	4.50^{B}	0.23^{B}	0.87^{C}	0.19^{AB}	21.2^{B}
		4.0	549.2^{B}	9.3^{B}	4.45^{B}	0.22^{B}	1.03^{C}	0.23^{B}	17.1^{C}

Same letters are not statistically different at the 1% probability level for different acetylation time, for each species separately.

rate, wood density, MC, partial TS, ΔMC from 65% to 85% and S factor, differential TS ratio, and weight loss by *L. acuta* and *T. versicolor* (Table 2). In the relation to the new parameter derived of ΔMC, factor S, and GU, the difference of these parameters between acetylated and untreated wood, correlation coefficient showed that there was a positive correlation of these parameters with WPG for each species (Fig 5).

DISCUSSION

The absorption of liquids in hardwoods is related to the permeability of the wood species (Ahmed and Chun 2011), which varies as a function of their anatomical structure. In hardwoods, liquid flow occurs mainly along lumina vessels in the longitudinal axis of tree (Ahmed and Chun 2009), but this flow can be interrupted by the presence of tyloses or gum in the vessels (Ahmed and Chun

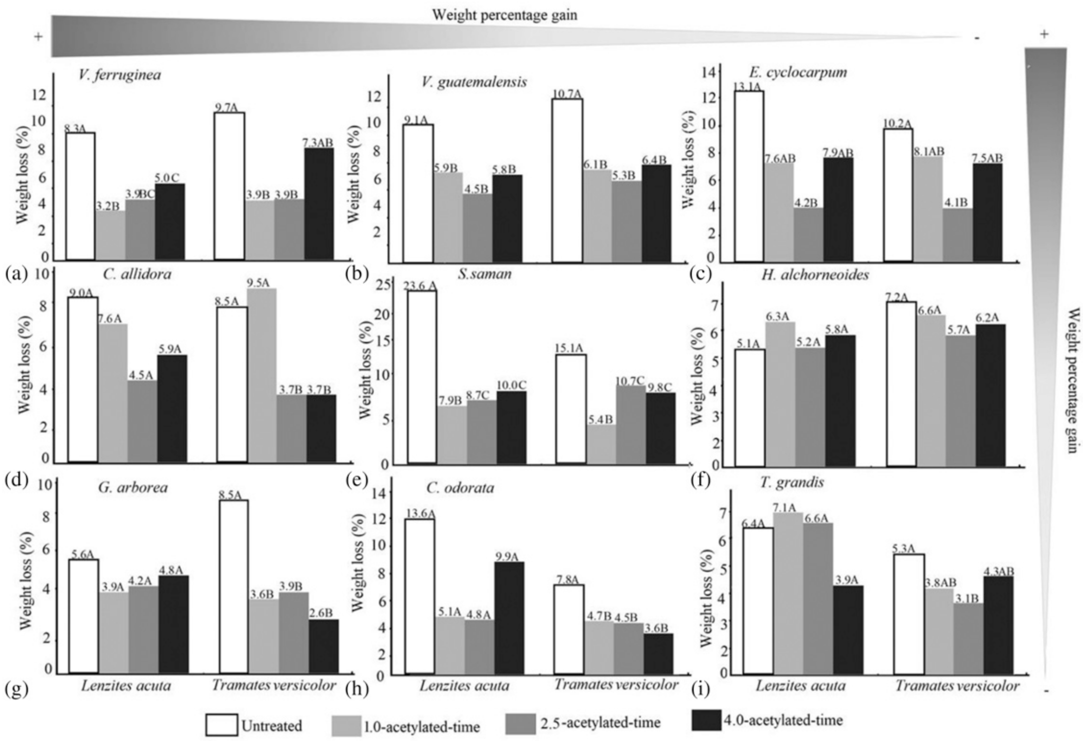

Figure 4. Weight loss due to fungal decay by *Lenzites acuta* and *Trametes versicolor* of nine fast-growth tropical species of Costa Rica for untreated and treated samples with three acetylation times. Legend: Different letters between acetylation times for a given fungal decay indicate statistical differences at 99%.

2011). Vessels connect longitudinal and radial parenchyma across wall pits, so liquids can subsequently flow through the ray lumina (Ahmed and Chun 2009). This flow is favored when the rays are composed of either over 3 series in width or a greater abundance of parenchyma is demonstrated (Ahmed and Chun 2011). Variations in the anatomy of the species tested in acetylation have been extensively discussed by Gaitán-Álvarez et al (2020). These authors reported that *E. cyclocarpum*, *H. alchorneoides*, *S. saman*, *V. ferruginea*, and *V. guatemalensis* present anatomical features of greater dimensions, specifically vessels' diameter (ie over 120 μm), rays' width (ie from 2 to 10 series of cells or over 252 μm) and their frequency of over 5 rays/mm^2, as well as various types of parenchyma (Table 3). These anatomical features are conducive to the flow of acetic anhydride and glacial acetic acid solutions, per the present study, in which these species present higher absorption and WPG values (Fig 1) and presented same variation of acetylation time (Fig 2). Conversely, species showing the lower absorption and WPG values were *C. odorata*, *G. arborea*, and

Table 2. Pearson correlation between weight percentage gain and the different properties of acetylated wood from nine fast-growth hardwoods in Costa Rica.

Wood properties	Correlation coefficient
Wetting rate	-0.04^{NS}
Wood density (kg/cm^3)	0.06^{NS}
MC at 65% of HR	-0.31^{NS}
ΔMC from 65% to 85%	-0.42^{NS}
S ratio	-0.38^{NS}
Differential TS ratio	0.19^{NS}
TS (%)	-0.27^{NS}
WA	-0.83^{**}
Weight loss by *Lenzites acuta*	0.04^{NS}
Weight loss by *Trametes versicolor*	0.16^{NS}

NS, not statistically significant.

** Statistically significant at 0.01 probability level.

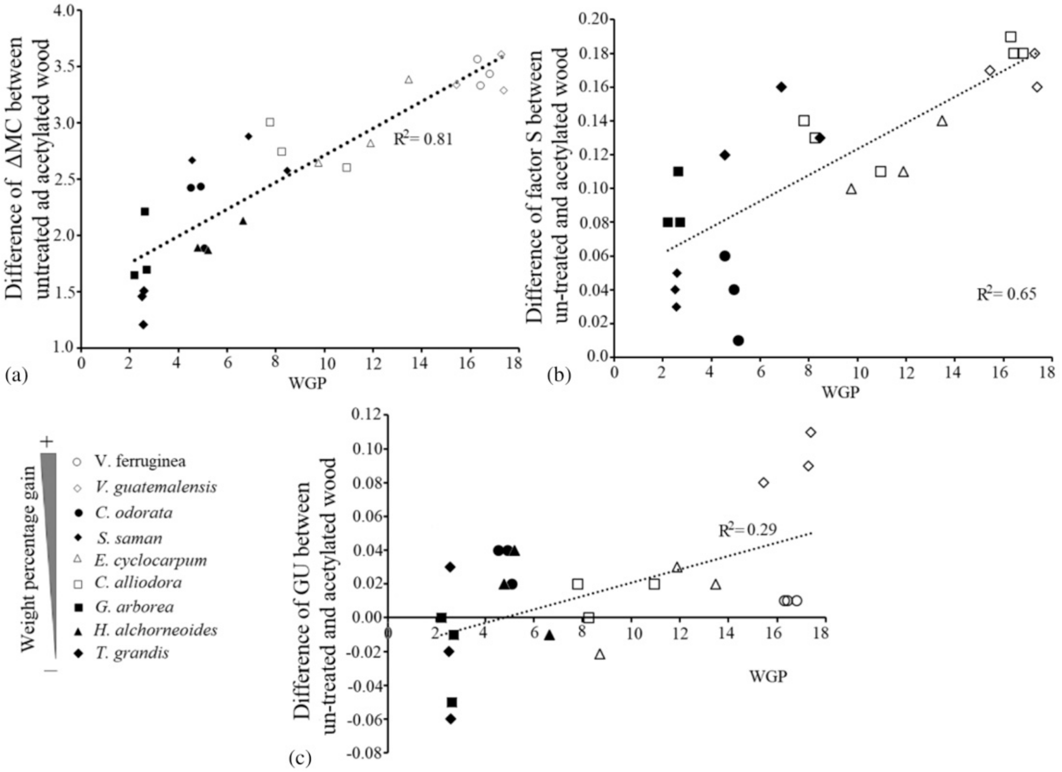

Figure 5. Relationship between weight percentage gain and the difference of ΔMC (a), S factor (b), and GU (c) of acetylated wood and untreated wood.

T. grandis (Fig 1), due to anatomical structure less favorable to liquid flow, such as smaller and less-frequent rays, deposits in vessels such as gum and tyloses, as well as few vessel-associated parenchyma (Table 3). Then the effects of acetylation varied with time, for some species, eg *T. grandis* presented higher WPG than *G. arborea* and *S. saman* at 1 h-acetylation-time (Fig 2[a]), but WPG was lowest values in 2.5 h-acetylation-time and 4 h-acetylation-time (Fig 2[b] and [c]).

In many of the species, the acetylation time did not have an effect on the absorption except for *E. cyclocarpum*, *S. saman*, and *H. alchorneoides* or partial TS, which the 4 h-acetylation-time treatment yielded a significantly lower absorption (Fig 1) or higher partial TS (Table 2). This decrease, for these species, may be attributed to the completion of the acetylation reaction well before the time limit. After some time, the reaction slows down and levels off, indicating that the reaction was "complete" (Rowell and Ibach 2018). After which, once acetylation is fulfilled, degradation of the acetic anhydride may have taken place, since the temperature used for the process (120°C) was close to the boiling temperature of this compound (139°C). As such, this result suggests that a longer acetylation time resulted appropriated for *E. cyclocarpum*, *S. saman*, and *H. alchorneoides*.

The wetting rate determines indirectly surface tension and the elation of the surface energy of the solid material (Gindl et al 2001) and, in the case of wood, these parameters are related to water adsorption (Collett 1972). In this way, in some species, namely *V. ferruginea* (Fig 3[a]), *V. guatemalensis* (Fig 3[b]), and *S. saman* (Fig 3[e]), these properties were influenced with acetylation in relation to untreated samples, because

Table 3. Anatomical characteristics of nine fast-growth tropical species in Costa Rica.

	Species	*C. odorata*	*C. alliodora*	*E. cyclocarpum*	*G. arborea*	*H. alchorneoides*	*S. saman*	*T. grandis*	*V. ferruginea*	*V. guatemalensis*
	FP (vessel mm^{-2})	9	7	6	5	17	4-5	4	2-3	3
	DV (μm)	125	166	167	189	116	152	150	145	169
	Deposits	G	T	G	T	G	G	G	—	—
	DPI	3	3	3	5	10	4	6	4	3
Radial parenchyma	Ray height	104	870	252	270	560	250	440	580	229
	Cells in ray width	4-10	2-6	1-3	1-3, 4-10	2-4	2-3	1-3, 4-10	1-3	1-3, 4-10
	Ray frequency	2	3	6-7	6	7	6-7	5	5	3
Axial parenchyma	PA	−	−	−	−	+	+	−	−	−
	PP	+	+	+	+	+	+	+	+	+
	PB	−	+	−	−	−	−	+	−	+

FP, pore frequency; LV, vessel length; DV, vessel diameter; T, tyloses; G, gums; DIP, intervascular punctuation diameter; PA, apotracheal parenchyma; PP, paratracheal parenchyma; PB, banded parenchyma.

wetting rate values showed a decrease, therefore these species were more affected by acetylation. In addition, the decrease in the wetting rate indicates a change in polarity of the surface for the different tropical wood species (Adebawo et al 2016). This is especially true in species with a high degree of acetylation (high WPG). Adebawo et al (2016) indicate that upon acetylation there comes incorporation of acetyl groups into the cell walls, thereby making the wood surface hydrophobic (Sandberg et al 2017). This performance was confirmed with the ΔMC values between 65% and 85% RH for the different species, where acetylated wood presented significantly lower ΔMC than untreated wood (Table 2). Another aspect of this relationship between wetting rate and WPG is that there are fewer differences between untreated and acetylated wood in species with low WPG, such as *G. arborea*, *C. odorata*, or *T. grandis* (Fig 3[g] and [h]). In contrast, no correlation was revealed between wetting rate and WPG (Table 3), probably due to fact that the wetting rate is related to other parameters of the wood that are not affected by the acetalization (Collett 1972).

Each acetylation time yields different values of WPG (Fig 1) and, of course, different surface effects which may or may not be compatible with water molecules (Gindl et al 2001). Among the different tropical species presented in this study, in those with a high WPG value, specifically *V. ferruginea*, *V. guatemalensis*, and *E. cyclocarpum* (Fig 3[a]-[c]), the 4 h-acetylation-time yields similar values of contact angles and T_{final} compared with untreated samples, suggesting better acetylation in 1 h-acetylation-time and 2.5 h-acetylation-time for those species. Meanwhile, in species with a lower WPG value, acetylation time from 1 to 4 h will not produce variation in contact angles and their final stabilization time (Fig 3[d]-[i]).

As expected, physical properties related to WA (MC, ΔMC, TS, S factor, GU, and WA) in most species presented lower values after acetylation treatment (Table 1). However, although the acetylation process affected wood properties in all species, the effect of this treatment is greater in species with higher WPG. The relationships between WPGs and the difference of ΔMC, TS, S ratio, G_T between acetylated and untreated wood is positive (Fig 5). Therefore, wood species with low WPG (such as *G. arborea* and *T. grandis*) showed less gain in dimensional and hygroscopic stabilities than those species with high WPGs, such as *V. guatemalensis* or *V. ferruginea* (Fig 5).

In fact, the lower acetylation and its effect on physical properties related to WA can also be observed in the correlation analysis shown for MC, TS, and WA, in which these presented a negative correlation with WPG (ie a decrease in each parameter with increasing WPG); however, only for WA, the physical properties showed statistical significance (Table 2). The presence and amount of hydroxyl groups, capable of forming hydrogen bonds with water molecules, is important to hygroscopic and dimensional stability and they are mainly present in hemicelluloses, followed by cellulose and lignin (Engelund et al 2013). During WA in these sites, the water molecule with two full-strength covalent bonds may become bound by two relatively strong H-bonds with a pair of nearby OH groups of the amorphous polysaccharide polymers in low MC. Meanwhile, the cooperativity in the H-bond network increases with increasing MC, gradually allowing the coalescence of water vapor molecules with already adsorbed water molecules to form a water dimer (Willems 2018). Thus, this gain in moisture makes wood dimensionally and hygroscopically unstable as the lumber gains weight. With acetylation, however, the OH^- anion group in wood components becomes chemically bound to a residue of the acetate (CH_3COO) of an acetic anhydride molecule $(CH_3CO)_2$ (Mantanis 2017). In this process, the OH^- anion group is reduced, decreasing the hygroscopicity of the wood and resultantly, increasing its dimensional stability (Adebawo et al 2016). Although the acetylation affected the hygroscopic S and dimensional stability of wood, the results showed that this treatment affected the former relatively more than the latter. The S ratio, which measures hygroscopic stability, decreased in all species (Table 1) and presented higher correlation coefficients (Fig 5[a] and [b]) than those obtained for G_T (dimensional stability, in Fig 5[c]). G_T in fact did not show a significant difference between acetylated wood and untreated wood (Table 1). Therefore, these results indicate that the changes in tangential dimensions for acetylated wood were caused by the reduction of the sorption capacity of wood.

However, according to Rowell (2016), acetylation of wood prevents its biological degradation by three possible mechanisms: 1) the first one consists in modifying the composition and physical configuration of the substrate where the specific enzymatic attack may take place, 2) the second one where the acetyl group forms a covalent bond, therefore it is no longer available for an enzymatic attack, and 3) the third theory based on the physical blockage of micropores of the cellular wall, rendering enzymatic penetration impossible. As confirmed in this study, susceptibility to fungal attack is related to WPG (Fojutowski et al 2014; Rowell 2014); however, this is a negative correlation, indicating that an increasing WPG decreases the loss of mass by the two fungi tested (Table 3). Therefore, differences between acetylated and untreated samples were found to be less or almost null in those species with lower WPG (Fig 4[b] and [f]-[h]). This effect can be attributed to low acetylation, which eases fungal enzymes' access to hydroxyl groups (Fojutowski et al 2014; Rowell 2014). There were no effects on resistance to decay related to the length of acetylation times, indicating that in shorter times acetyl groups have already been taken by the acetate group (CH_3COO), leaving little opportunity for fungal attack (Pawar et al. 2013).

CONCLUSIONS

The acetylation treatment of tropical hardwoods is achieved with varying levels of WPG, which is more directly related to the type of species and less to the length of the acetylation time. This clearly evidences that differences between the species are attributed to liquid flow inside the wood, or permeability, which depends on the anatomical features of the species. This variation in WPG per species produces various effects on the properties of wood. When species presented WPG values over 10%, eg *V. ferruginea*, *V. guatemalensis*, *C. alliodora*, or *E. cyclocarpum*, thermal stability and wetting rate, but the advantage that there was a decrease in the value of parameters related to water adsorption (ie swelling, MC, MC variation, ΔMC from 65% to 85%, S ratio, and G_T) and a favorable increase in resistance to biological degradation. Other important results were that species with low WPG values (ie low acetylation level), showed less gain in the dimensional or hygroscopic stability of the wood than those species with high WPG. It was also clear that gain in the dimensional stability was due to the better hygroscopic stability after acetylation. In addition, results showed that the 2.5 h time is an appropriate acetylation time.

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