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Evaluation of Thermally Modified Wood by Means of Stress Wave and Ultrasound Nondestructive Methods

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The paper aimed at studying the potential of two nondestructive methods to estimate the wood mechanical properties and mass loss due to thermal treatments. In this study, a low-density tropical hardwood species *Simarouba amara* (marupá) was used. Forty small beams with dimensions of (25 × 25 × 400) mm (width × thickness × length) were cut from this species. Initially, the beams were nondestructively tested using stress wave and ultrasound methods. Stress wave velocity (*Swv*), ultrasound velocity (V_{LL}), dynamic modulus of elasticity (E_d) and stiffness coefficient (C_{LL}) were longitudinally determined. Afterwards, the beams were thermally treated using a chamber without air circulation under atmospheric pressure. Two schedules were tested: 160 ºC for 180 minutes and 200 ºC for 70 minutes. Mass loss (*ML*) due to thermal treatment was calculated and the thermally treated material was again nondestructively evaluated. Afterwards, modulus of rupture (f_m), modulus of elasticity (E_M) and parallel compression strength ($f_{c,0}$) of treated material were assessed. Backward linear multiple regression analysis was run in order to estimate these properties. Parameters investigated through nondestructive testing (before and after treatment) and derivative variables were used as independent variables, totaling 12 variables. For both treatment schedules, all parameters related to nondestructive techniques were affected by the thermal treatment, thus acoustic velocities and stiffness values were improved. It was found that all evaluated properties of treated material could be modeled at a reasonable level (R^2 = 0.392 to 0.874) depending on the nondestructive method and treatment schedule used. Nevertheless, ultrasound method fitted the most suitable models for a large number of properties. The utilization of variables from both methods together yielded better models whose R^2 value ranged from 0.466 (f_m) to 0.941 (E_M). It was found that the most important nondestructive variables which entered into the models were: *Swv* before and after treatment, V_{LL} after treatment, E_d before treatment and C_{LL} after treatment. Finally, it could be concluded that stress wave and ultrasound nondestructive methods presented great potential to evaluate properties of thermally treated wood material.

Keywords: acoustic methods, multiple regressions, mechanical properties.

1. INTRODUCTION

Nondestructive evaluation (NDE) aims at obtaining material properties and using this information to make decisions regarding appropriate applications of the material [1]. For isotropic materials, NDE is used to detect voids, nonhomogeneous spots and other irregularities. However, these irregularities are common in wood products, so NDE is used to evaluate its effect on physical and mechanical properties. There are several nondestructive methods whose utilization depends on the kind of information required. Usually, these methods are employed to evaluate quality, strength, stiffness and degree of deterioration of lumber, wood structures or wood based-products. Stress wave, ultrasound and transverse vibration have been extensively employed to inspect the health of a wood structure and mainly to grade lumber according to its stiffness [2 – 3]. Although these methods can present different mathematical approaches, they generally require the mass of the material, which is expressed by density in the models.

Thermal treatments impart mass loss of the wood material due to carbohydrates degradation. When it happens, dimensional stability and biological durability are considerably improved since reduction in thickness swelling, water absorption and equilibrium moisture content are changed. Taking it into account, a useful way to evaluate the extension of the wood modification promoted by thermal treatments is to measure the mass loss of the treated material [4], once it has been found that the improvement on those properties is greater with the higher wood mass loss. Another way to evaluate these treatments effectiveness is to evaluate color changes in the treated material [5 – 6].

It is well known that acoustic wave velocity is highly affected by wood moisture content: the velocity is slower with higher moisture content [7 – 8]. Since thermal treatments change equilibrium moisture content it is expected that NDE methods could be sensitive to it. Nonetheless, information with respect to NDE utilization to evaluate thermally treated materials is relatively scarce. Moisture content, temperature and degradation are the factors that most affect acoustic wave velocity in wood [9].

The vibrational properties of treated wood were studied [10] and it was found that the longitudinal Young´s modulus determined through nondestructive testing was not a reliable variable to evaluate the treatment. The ultrasound method was employed for detecting internal checks in thermally treated wood [11]. The results showed that ultrasound wave velocity was reduced when checks

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were present. On the other hand, stress wave method was used to evaluate thermally treated oriented strandboards (OSB) and it was found that wave velocity was significantly higher as function of lower equilibrium moisture content [12]. The same method was recently employed to determine dynamic modulus of elasticity of eucalypt treated wood [13]. In another work, it was observed that ultrasound wave velocity was significantly improved in thermally treated bamboo [14].

In this context, the main objective of this present work was to evaluate the potential of two well-known acoustic nondestructive methods – stress wave and ultrasound – as assessment tools to predict mechanical properties – modulus of elasticity, modulus of rupture and parallel compression strength – and mass loss of thermally treated wood.

2. MATERIALS AND METHODS

Lumber from the tropical hardwood marupá (*Simarouba amara* Aubl.) was collected at trading companies and macroscopically identified through comparison with the standard samples deposited at the Wood Anatomy Section of the Forest Products Laboratory (*Index Xilarium* FPBw), Brazilian Forest Service. The initial apparent wood density was 0.457 g/cm^3 while the initial wood moisture content was 11.2 %. The lumber was stored in air-conditioned room (20 ºC; 65 % RH) for final moisture equalizing. Forty small beams measuring (25 × 25 × 400) mm (width × thickness × length) were cut from this material. Afterwards, the beams were thermally treated using a chamber without air circulation under atmospheric pressure. Two schedules were tested: 160 ºC for 180 minutes (T1) and 200 ºC for 70 minutes (T2). For each schedule 20 beams were thermally treated. Mass loss (*ML*, %) and density (ρ, g/cm^3) were calculated immediately after the treatment, whereas equilibrium moisture content (*EMC*, %) was calculated after treated beams air conditioning (20 ºC; 65 % RH).

Before and immediately after thermal treatment all 40 beams were nondestructively evaluated lengthwise (LL) by stress wave (Metriguard Stress Wave Timer model 239A) and ultrasound methods (Pundit Lab, 54 kHz). The first equipment has an impact pendulum that generates a stress wave which propagates through the beam. Two accelerometers are connected to the beam to measure the stress wave transit time (t, µs), which is the time required for the wave to travel between them. This value is used to determine the stress wave velocity (*Swv*, m/s) and then the stress wave dynamic modulus of elasticity (E_d, MPa), according to equations 1 and 2. The second equipment has two circular flat transducers, which are coupled to the beam using medicinal gel. Similarly, this equipment measures the ultrasound transit time (t, µs), which allows to determine ultrasound velocity (V_{LL}, m/s) to calculate the stiffness coefficient (C_{LL}, MPa), according to equation 3.

$$Swv \text{ (m/s)}; \; V_{LL} \text{ (m/s)} = L / (t \times 10^{-6}) , \quad (1)$$

$$E_d \text{ (MPa)} = \{(Swv^2 \times \rho) / g\} \times 10^{-5} , \quad (2)$$

$$C_{LL} \text{ (MPa)} = V_{LL}^2 \times \rho , \quad (3)$$

where *Swv* is the stress wave velocity (m/s); V_{LL} is the ultrasound velocity (m/s); L is the span (cm); t is the transit time (µs); g is the acceleration of gravity, 9.8 m/s^2; ρ is the density (kg/m³).

One-way analysis of variance (ANOVA) was run to compare ρ and *EMC* means between treatments. To evaluate the thermal treatment effect, the variables regarding density (ρ), stress wave (*Swv*, E_d) and ultrasound nondestructive methods (V_{LL}, C_{LL}) were compared before (b) and immediately after (a) treatment by the t-test for paired samples. Derivative variables (θ) were obtained calculating the relationship between variable values before and after treatment (b/a). Thus, 12 variables were determined in order to qualify the material (Table 1). These variables were employed as independent variables to estimate the mass loss (*ML*, %) imparted by the thermal treatment.

The following mechanical properties were assessed according to [15]: modulus of elasticity (E_M, MPa) modulus of rupture (f_m, MPa) and parallel compression strength ($f_{c,0}$, MPa). Backward multiple linear regressions were run to model *ML*, E_M, f_m and $f_{c,0}$ using the independent variables (Table 1). First, these analyses were run separately for each treatment (T1 and T2) according to the respective nondestructive method and, afterwards, using both methods together. Further analyses were done using only *Swv* and V_{LL} as independent variables.

Table 1. Identification of the 12 independent variables used to model the properties

Method	Independent variables		
	Before	After	Derivative
Stress wave	Swv_b; E_{db}	Swv_a; E_{da}	θSwv; θE_d
Ultrasound	V_{LLb}; C_{LLb}	V_{LLa}; C_{LLa}	θV_{LL}; θC_{LL}

3. RESULTS AND DISCUSSION

Table 2 presents the *ML*, *EMC*, ρ and acoustical properties for the material before and after treatment. According to the one-way ANOVA, T1 treatment (160 ºC/180´) promoted higher *ML* in comparison with T2 (200 ºC/70´).

Table 2. Density and acoustic variables of wood before and after the thermal treatments

Property	T1 (160 ºC, 180´)		T2 (200 ºC, 70´)	
	Before	After	Before	After
ML, %	–	15.4_A	–	13.3_B
EMC, %	–	8.51_A	–	8.15_B
ρ, g/cm^3	0.46_a	0.41_b	0.45_a	0.41_b
Swv, m/s	$4{,}206_a$	$4{,}319_b$	$4{,}216_a$	$5{,}714_b$
V_{LL}, m/s	$5{,}193_a$	$5{,}442_b$	$5{,}210_a$	$5{,}451_b$
E_d, MPa	$8{,}028_a$	$8{,}674_b$	$8{,}010_a$	$11{,}736_b$
C_{LL}, MPa	$12{,}177_a$	$13{,}903_b$	$12{,}141_a$	$12{,}017_a$

Different capital letters indicate that difference between treatment means is statistically significant at $\alpha = 0.05$, while different lower case ones indicate that difference before and after means, within each treatment, is statistically significant at same level.

It probably happened because despite the lower temperature, the longer time in T1 promoted more volatilization of chemical components and evaporation of water. However, the *EMC* values were not statistically significant. *ML* values can be considered relatively high compared to other authors. The effect of the thermal treatment (205 ºC/240´) on *Pinus sylvestris* and *Betula pendula* woods yielded *ML* near 5.7 % and 6.4 %, respectively [16]. In *Eucalyptus grandis* thermally treated wood (200 ºC/180´) a *ML* value of 5.3 % was found [17].

The *ML* referred in this work is that measured immediately after the thermal treatment, before the beams storage in air conditioned room for moisture equalizing. Since the initial *EMC* of the material was nearly 11.2 % and after the treatments was around 8 %, not only water was removed, but some polymer degradation happened as well. It can explain why the *EMC* of the treated material was reduced about 25.6 % compared to that untreated. Thermal treatments usually promote reduction of water adsorption sites due to the polymers structural reorganization, such as a cross-linking or by wood polymer degradation [18 – 19]. In addition, several authors [20 – 21] have observed that thermal treatments reduced the amorphous cellulose area affecting water adsorption. Hemicelluloses are the less thermally stable and the most hygroscopic wood polymer and they were severely degraded when OSB were thermally treated, as found by [4]. As a consequence of wood drying and degradation of its polymers, treated wood density was significantly reduced (≈ 9 %) after applying both thermal treatments.

Table 2 shows that the acoustic variables were also changed by the thermal treatment. All properties with respect to stress wave were changed immediately after thermal treatment. According to the paired *t*-test results, the stress wave velocity (*Swv*) was not significantly higher (≈ 2.7 %) after T1 treatment: 4,206 m/s × 4,319 m/s. This variation was much higher (≈ 35.5 %) for the T2-treated material: 4,216 m/s × 5,714 m/s. The improvement of stress wave velocity on treated material directly affected the dynamic modulus of elasticity (E_d), which was significantly higher despite the density reduction. Ultrasound velocity (V_{LL}) was similarly improved in both treatments (≈ 4.7 %), but imparted a significant effect on stiffness coefficient (C_{LL}) only for T1 treatment, which was improved from 12,177 MPa to 13,903 MPa (≈ 14.1 %). Differently, for the T2-treated material the C_{LL} after treatment was not changed and it presented almost the same value observed before the treatment.

Taking into account all polymer changes previously mentioned (cross-linking, degradation, crystallinity), they might be the responsible for changes observed on the acoustic nondestructive properties [12]. Nondestructive properties of 55 wood species were studied [22] and it was found that second layer (S2) microfibril angle (MFA) of the secondary cell wall presented major effect on the stress wave velocity: the wave is faster with the lower MFA. In the same way, it has been stated [10] that the nondestructive longitudinal modulus of elasticity strongly depends on the S2 MFA and less strongly on the crystallinity of cellulose and stiffness of the amorphous matrix. The authors concluded that these three elements could be slightly influenced by the heat treatment, this way explaining the observed improvement of the modulus. The results found here are in accordance with those observed by other authors.

Contact thermal treatment was used to improve dimensional stability of OSB [4, 12]. Two temperatures (190 ºC and 220 ºC) for 12, 16 and 20 minutes were tested. The boards were evaluated before and after treatment using the stress wave method. According to the results, for all treatment combinations *Swv* was significantly improved by the thermal treatment. In spite of this behavior, dynamic modulus of elasticity was reduced on treated boards. The authors explained that *Swv* was more sensitive to changes in equilibrium moisture content; whereas *ML* had more pronounced impact on E_d.

The effect of thermal treatment on *Dendrocalamus giganteus* bamboo properties was studied [14]. It was encountered that ultrasound velocity (V_{LL}) was consistently improved (≈ 32 % – 38 %) after the treatment up to 260 ºC. At 300 ºC, V_{LL} was drastically reduced from 4.354 m/s to 2.147 m/s. They also found that C_{LL} of treated bamboo was directly improved by the thermal treatment. Wood from *Eucalyptus grandis* was thermally treated at four temperatures (180, 200, 215 and 230 ºC) for 15, 60 and 240 minutes [13]. E_d was reduced by 13 % only for the most severe treatment (230 ºC, 240´), and other treatment combinations did not affect it. Figure 1 presents the mechanical properties of the treated material according to the treatment schedule. The untreated *Simarouba amara* wood presents the following values of mechanical properties according to [23]: f_m, 65.8 MPa; E_M, 8.2 GPa; and $f_{c,0}$ 35.3 MPa. The comparison between these data revealed that the thermal treatments did not affected significantly stiffness and compression strenght of the wood. On the other hand, bending strenght was severely affected and f_m values were reduced about 35 %.

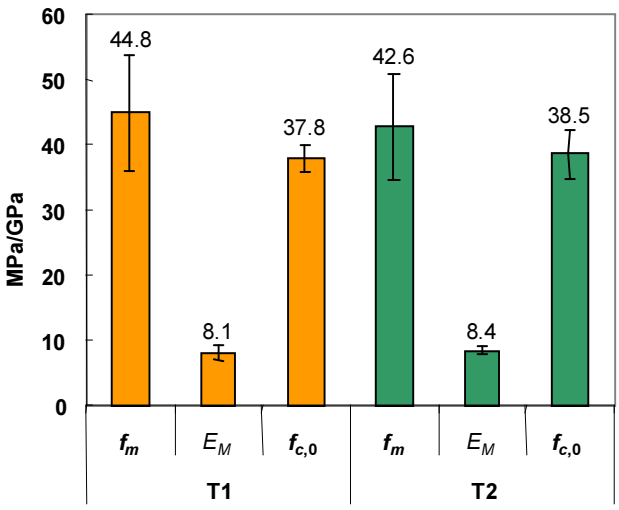

Fig. 1. Mechanical properties of the thermally treated wood according to the treatment schedule. (Note: E_M values are expressed as GPa; T1: 160 ºC, 180´; T2: 200 ºC, 70´)

Table 3 presents models fitted to estimate the properties of the thermally treated wood. Twelve models could be fitted to explain the variation of wood properties and all models and coefficients were statistically significant ($p < 0.05$) according to the ANOVA.

Table 3. Regression models and parameters to predict properties of thermally treated wood using all independent variables

Method	Property	Model	R^2	$F_{calc.}$	*SEE*
Treatment 1 (160 ºC, 180´)					
SW	*ML*	$-771.5 + 751.6\ \theta Swv + 0.047\ E_{db} - 0.043\ E_{da}$	0.652	4.98**	0.58
	$f_{c,0}$	$30.47 - 0.08\ V_{LLa} + 0.005\ E_{db}$	0.554	8.71**	1.57
	E_M	$672895.4 - 662873\ \theta Swv - 39.6\ Swv_b + 39.4\ Swv_a$	0.825	12.58**	512.4
US	*ML*	$-262.5 + 570.8\ \theta V_{LL} - 299.1\ \theta C_{LL}$	0.737	8.71**	0.56
	f_m	$351.6 - 0.082\ V_{LLa} + 0.01\ C_{LLa}$	0.466	4.36*	7.13
	E_M	$-5546.4 + 1.02\ C_{LLa}$	0.362	6.23*	850.1
Treatment 2 (200 ºC, 70´)					
SW	*ML*	$-17.24 + 22.23\ \theta Swv - 0.002\ E_{db}$	0.392	5.81**	0.59
	$f_{c,0}$	$74.6 - 58.72\ \theta Swv + 0.003\ E_{db}$	0.631	9.41**	2.36
US	*ML*	$469.61 + 0.041\ C_{LLb} - 0.041\ C_{Lla} + 490.3\ \theta C_{LL}$	0.793	11.47**	0.36
	$f_{c,0}$	$665.55 - 0.056\ C_{LLb} + 0.06\ C_{Lla} - 682.83\ \theta C_{LL}$	0.874	32.24**	1.44
	f_m	$3111.41 - 0.039\ V_{Lla} - 2910.4\ \theta V_{LL} - 0.101\ C_{LLb} + 0.117\ C_{LLa}$	0.742	5.74*	5.04
	E_M	$453052.5 - 10.19\ V_{Lla} - 32.7\ C_{LLb} + 34.57\ C_{Lla} - 411788.8\ \theta C_{LL}$	0.764	5.67**	357.19

**,* Significant at $\alpha = 0.01$ and $\alpha 0 = 0.05$ level, respectively; F_{calc}: calculated *F*; *SEE*: standard error of the estimate.

It can be observed that ultrasound (US) method could fit seven in possible eight models, while stress wave (SW) fitted five models. US based models also implied in higher R^2 values (≈ 0.677) than SW based ones (0.611). The models to predict the properties of wood treated under T2 conditions presented better predictability ($R^2 = 0.701$) than those fitted to wood treated under T1 conditions ($R^2 = 0.599$).

For the wood properties, four models could be fitted to predict *ML* using both nondestructive methods for both treatment schedules. It can be observed that *ML* imparted by T1 treatment could be moderately modeled ($R^2 = 0.652 - 0.737$) when variables from each method were considered separately. When variables from both methods were analyzed together, R^2 was statistically improved to 0.862 ($p < 0.015$). On the other hand, for the T2 treatment the models initially presented low-high predictability ($R^2 = 0.392$ to 0.874), but this value could not be improved ($R^2 = 0.764$; $p < 0.013$) when all nondestructive variables were analyzed together.

Three models were fitted to predict E_M and $f_{c,0}$. For E_M R^2 values ranged from 0.362 to 0.825 depending on the method used and treatment evaluated. Nevertheless, these values were highly improved (0.941; $p < 0.000$) when SW and US variables were combined. The variation of $f_{c,0}$ could be explained at a higher level, since R^2 values presented a narrower range: 0.554 to 0.872. However, these values could not be improved when the models considered variables from both nondestructive methods ($R^2 = 0.738$; $p < 0.016$). Only two models could be fitted to explain the f_m variation, both using US variables. The R^2 values for T1 and T2 were 0.466 and 0.742, respectively. Taking into account all models for each property, it was found that $f_{c,0}$ variation was better explained ($R^2 = 0.686$), followed by E_M ($R^2 = 0.651$), *ML* ($R^2 = 0.643$) and f_m ($R^2 = 0.604$).

Nevertheless, according to the results shown in Table 3, it can be inferred that most of E_M values could be predicted more accurately than $f_{c,0}$ and f_m. It is a very usual result widely found in the literature concerning nondestructive testing. The theory behind wood nondestructive testing is based on its elastic behavior. Therefore, in this case, the stiffness properties of the material usually present better relationship than those related to maximum strength, whose determination is beyond the elastic limit of the material. From the results presented in Table 3, it can be observed that the most important nondestructive variables that entered into the models were: *Swv* before and after treatment, V_{LL} after treatment, E_d before treatment and C_{LL} after treatment.

Table 4. Regression models and parameters to predict properties of thermally treated wood using only stress wave and ultrasound velocity

Method	Property	Model	R^2	F_{calc}	*SEE*
Treatment 1 (160 ºC, 180´)					
US	*ML*	$-8.035 + 0.005\ V_{LLb}$	0.383	6.84*	0.699
	f_m	$266.54 - 0.043\ V_{LLb}$	0.316	5.09*	7.69
Treatment 2 (200 ºC, 70´)					
SW	*ML*	$-12.16 + 18.47\ \theta Swv$	0.386	6.28*	0.58
	$f_{c,0}$	$-56.74 + 0.023\ Swv_b$	0.375	7.81*	2.83
US	*ML*	$44.31 - 30.06\ \theta Swv$	0.405	7.47*	0.553
	$f_{c,0}$	$-79.15 + 0.022\ V_{LLb}$	0.656	30.52**	2.21

**,* Significant at $\alpha = 0.01$ and $\alpha = 0.05$ level, respectively; F_{calc}: calculated *F*; *SEE*: standard error of the estimate.

Further analyses were run trying to model properties of the thermally treated material using only information with respect to wave velocity (Table 4). In this case, only six models could be fitted and the predictability was reduced and ranged from 0.383 to 0.656. Regardless of these values, it is a relevant finding since it means that only through wave velocity measuring it is possible to be aware of the some properties of the treated material. Therefore, the extra work required to determine wood density – a mandatory variable to calculate E_d and C_{LL} – can be avoided, which is very interesting because of the boards' dimensions at industrial level, for instance.

Nonetheless, for the stress wave method it was not possible to fit any statistically significant model to predict the property of wood treated under T1 conditions. From the comparison of nondestructive methods, it may be observed that V_{LL} yielded models whose R^2 values were slightly higher than those obtained using *Swv*. In fact, some authors have observed that using only wave velocity it is possible to model properties of wood and wood based materials [24].

The variation of the modulus of elasticity of laminated veneer lumber made from *Pinus kesiya* species could be explained at a reasonable level ($R^2 = 0.476$) using only stress wave velocity [24]. Similarly, the stiffness of the tropical hardwood *Sextonia rubra* could be predicted ($R^2 = 0.516$) using this variable [25].

4. CONCLUSIONS

The studied thermal treatments imparted significant mass loss on wood and, since heat induces to chemical changes and degradation of wood polymers; it reduced equilibrium moisture content and density of treated material. As a consequence all properties regarding stress wave and ultrasound were modified after applying the thermal treatments. It was found that stress wave and ultrasound velocities were improved as their respective modulus. These variations could be used to model at reasonable level the mass loss imparted by the thermal treatments and the mechanical properties of the treated material. Finally, it could be concluded that stress wave and ultrasound nondestructive methods presented great potential to evaluate wood properties and to estimate mass loss due to thermal treatment and research effort should be continued.

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