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Ultrasound to determinate the modulus of elasticity of heat-treated woods subjected to field test

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ABSTRACT: This study aimed to evaluate the modulus of elasticity of heat-treated wood subjected to decaying in field environment, by employing the ultrasound technique. For this, wood of *Eucalyptus grandis* and *Pinus taeda* from a TWBrazil plantation were used. From each species were selected five trees, which were then unfolded in boards that were dried and heat-treated at the temperatures of 140 and 160 ºC. Afterwards, 75 specimens were obtained for each species, which were then exposed to the weather elements during 200 days and evaluated every 40 days, in the completely randomized design with a double factorial arrangement for the heat treatment and exposure time. In this period were collected data of the fungal decay potential in the site, and apparent specific gravity and dynamic modulus of elasticity (*DMoE*) from the wood samples. We verified the fungal decay potential increase with the exposure time, which also caused a reduction of the apparent specific gravity and *DMoE* in all treatments, for both species. *E. grandis* had a low stiffness loss while *P. taeda* had a higher loss with the increasing of exposure time, with significant differences of the species *DMoE*.

Key words: *Eucalyptus*; heat treatment; mechanical strength; *Pinus*; wood durability

Ultrassom para determinação do módulo de elasticidade de madeiras termorretificadas submetidas a campo de apodrecimento

RESUMO: Esse estudo teve por objetivo avaliar o módulo de elasticidade de madeiras termorretificadas submetidas à deterioração em ambiente de campo, utilizando a técnica de ultrassom. Foram utilizadas madeiras de *Eucalyptus grandis* e *Pinus taeda* provenientes de um plantio da empresa TWBrazil. De cada espécie foram selecionadas 5 árvores, desdobradas em tábuas que foram secas e termorretificadas a temperaturas de 140 e 160 ºC. Em seguida, confeccionou-se 75 corpos de prova de cada espécie, que foram expostos às intempéries durante 200 dias, sendo avaliados a cada 40 dias num delineamento inteiramente ao acaso com arranjo fatorial duplo para a temperatura de termorretificação e tempo de exposição. Nesse período coletou-se dados do potencial de ataque fúngico do local, massa específica aparente e módulo de elasticidade dinâmico (*DMoE*) das madeiras. Verificou-se aumento do potencial de ataque fúngico com o tempo de exposição. O tempo de exposição ocasionou redução da massa específica aparente e do *DMoE* em todos os tratamentos para ambas as espécies. *E. grandis* apresentou baixa perda de rigidez enquanto o *P. taeda* teve maior perda de rigidez com o tempo de exposição, com diferenças significativas no *DMoE* das madeiras.

Palavras-chave: *Eucalyptus*; termorretificação; resistência mecânica; *Pinus*; durabilidade da madeira

Introduction

Wood from planted forests has become one of the main sources of raw material for the various sectors of the timber industry due to the rapid growth and versatility of its technological properties. However, most species planted in Brazil are of low natural resistance, making them subject to the action of several xylophagous organisms when they are used in direct contact with the soil.

Therefore, wood treatments are performed in order to both expand the economic potential and diversify even more the places of its use; among these, the heat treatment has been pointed out as a viable alternative, mainly because it has a lower impact on the environment.

Heat treatment consists of subjecting wood pieces to temperatures between 120 and 200 °C, in order to promote chemical changes in the cellulose, hemicellulose and lignin polymers, obtaining wood with a lower hygroscopicity, greater dimensional stability and greater biological resistance (Modes et al., 2017).

Heat treatment usage in Brazil has taken a prominent place in the strive for both sustainability and greater durability of the used wood without harming the environment, as it does not use chemicals that are harmful to it. Natural durability tests of wood in the field environment are important because it exposes the subject to various xylophagous organisms, as well as abiotic factors, emphasizing the visual health inspection of the service piece as an evaluation form (Stangerlin et al., 2015).

Nonetheless, the technique for evaluating mechanical properties in static bending tests can provide complementary answers to processes involving wood decay, as reported by Trevisan et al. (2008).

However, these tests, as well as static bending, require the material destruction; therefore, it makes unfeasible the further use of the wood after the test. Due to this fact, as an alternative to these tests, there are non-destructive tests such as the ultrasound through wave propagation in wood samples, which, according to Cunha & Matos (2010), allows the evaluation of the structural integrity of a workpiece, a faster analysis of a large population and versatility to fit itself into a standardized routine on a production line.

Calegari et al. (2008) state that several factors can influence the tests, limiting the propagation of waves in the wood due to the anatomical (fiber dimensions and porosity), physical (basic specific gravity and moisture content) and morphological (wood types and grain angle) characteristics, and the presence of defects (knots and cracks).

With this in mind, this study was developed in order to determine the mechanical resistance by means of the specific gravity and dynamic modulus of elasticity of heat-treated woods of *Pinus taeda* L. and *Eucalyptus grandis* Hill ex Maiden, subjected to decay in decay field.

Materials and Methods

In order to carry out the study, wood from five trees of *Eucalyptus grandis* and *Pinus taeda* species, from a commercial plantation of the TWBrazil Company, located in the Center North region of Paraná, were used. From this wood, boards with dimensions of 100 x 25 x 1200 mm (width x thickness x length) were produced, having been previously dried in a conventional drying chamber to a moisture content of around 12%, with a ± 2% variation.

Those dried boards were then submitted to the heat treatment process in the TWBrazil company by using the *VAP HolzSysteme®* method, which consists in the heat application at high temperatures (140 and 160 °C) through saturated steam, with efficient oxygen elimination in order to obtain the *Thermally Modified Timber* (*TMT*) product, as described by Batista et al. (2016).

Specimen preparation

Heat-treated and non-heat-treated pieces (control group) were randomly selected in order to obtain specimens with dimensions of 20 x 20 x 300 mm (width x thickness x length), thus achieving the conditions used in the experiment, as per Table 1.

All specimens were coded and placed in a climatic chamber (at 65% relative humidity and 20 ºC) to stabilize their mass at around 12% of moisture content. Afterwards, they were weighed and measured in order to determine the apparent specific gravity. Subsequently they were tested, using the Metriguard *Stress Wave Timer 239A* for stress wave propagation time readings at microsecond (µs), which was used to determine the stress velocity propagation velocity in the wood prior to exposure, as per Equation 1.

$$V = \frac{d}{t} \tag{1}$$

In which: V: velocity (cm s^{-1}); d: distance between transducers (cm); t: propagation time (s).

With this data, the dynamic modulus of elasticity of the specimens was determined before they were subjected to decay in the field test, with the aid of Equation 2.

$$\mathrm{DMoE} = \delta \cdot V^{-2} \cdot \frac{1}{g} \tag{2}$$

In which: *DMoE*: dynamic modulus of elasticity (Kgf.cm^{-2}); δ: specimen specific gravity (kg m^{-3}); g: acceleration of gravity (9.80665 m s^{-2}); V: longitudinal wave velocity (cm s^{-1}).

Subsequently, the specimens previously stabilized and tested for non-destructive technique were subjected to

Table 1. Design of the treatments applied to the woods of *E. grandis* and *P. taeda*.

Treatment	Replicates	SM	Total SM
Control	5	5	25
T. 140	5	5	25
T. 160	5	5	25

In which: T.140 = Heat treatment at 140 °C; T. 160 = Heat treatment at 160 °C; SM = specimen.

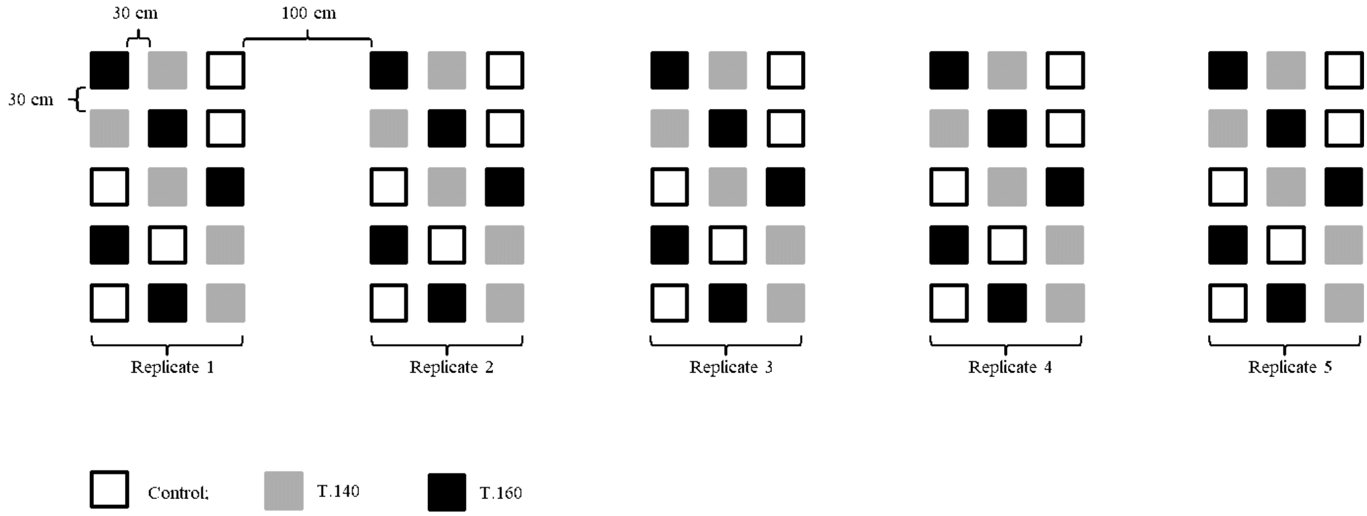

Figure 1. Illustrative sketch of specimen distribution in the field test for *E. grandis* and *P. taeda* wood.

field decaying tests from late October to early May, period equivalent to the wettest season in the region. For this, they were fixed to the ground to half-length (150 mm) in a field test with forest vegetation at coordinates 25°26’56.9” S and 49°14’16.3” W, as displayed in Figure 1.

In each replicate, observations were made by taking the soil samples at every 40 days and then performing five evaluations, corresponding to a total period of 200 days of exposure in the field test.

During this period, the observed climatic conditions at the experiment site (temperature, humidity and precipitation) were recorded in order to determine the fungal decay potential to which the species were subjected during the test, employing Equation 3, adapted by Martins et al. (2003).

$$\mathrm{FDP} = \sum \frac{\left[(\mathrm{T}-2)\cdot(\mathrm{D}-3)\right]}{16.7} \quad (3)$$

In which: FDP = Fungal decay potential of the species; T = average monthly temperature (ºC); D = Number of monthly days with rainfall equal or greater than 0.3 mm.

Specimens from each treatment were removed from the soil, cleaned and subjected to moisture stabilization (± 12%) in a climatic chamber after the field exposure. After conditioning, they were once again weighed and measured to determine the apparent specific gravity, then tested in the *Stress Wave Timer* for further readings and determination of new values from dynamic modulus of elasticity observed in each wood condition, of the two species exposed to decay in field environment.

Data analysis

In order to evaluate results, the analysis of variance was performed using the factorial design type 2 x 3 (specific gravity of two species and three treatment conditions), followed by a factorial type 3 x 5 (separately for the modulus of elasticity of each species for three treatment conditions and five periods of field exposure). Thus, the factors and interactions that showed significant differences by the analysis of variance had their means compared by Tukey test (p = 0.05). Mean values of apparent specific gravity and *DMoE* before and after exposure to the field test were compared by Student t-test.

Concomitantly, an electronic spreadsheet was used to develop wood stiffness loss curves and dynamic modulus of elasticity as a function of the fungal decay potential and exposure time of the field material.

Results and Discussion

Mean values of apparent specific gravity of the wood exposed in field environment (Table 2) showed that significant differences were observed for both *E. grandis* and *P. taeda* before and after subjecting to the field test, i.e. the period that the material was exposed resulted in a statistically significant alteration in this property.

Although not statistically different between treatments, it can be observed (Table 2) that there is an increase in the apparent specific gravity of heat-treated wood at 140 and 160 ºC in relation to the control treatment after exposure, which was also observed in the wood behavior before treatment, but with emphasis on T.140, differing statistically from the others. Despite this behavior, the lowest mass loss was observed for T.160 after the total exposure period, as reported by Carvalho et al. (2019), who used the same material to evaluate the mass loss.

Table 2. Apparent specific gravity of *E. grandis* wood before and after being subjected to the field test.

Species	Condition	Apparent specific gravity (g cm^{-3})		
		Control	T. 140	T. 160
E. grandis	Before	0.470*B	0.550*A	0.488*B
	After	0.447A	0.518A	0.471A
P. taeda	Before	0.584*A	0.446*B	0.442*B
	After	0.560A	0.417B	0.396B

* Significant difference in column by Student t-test. Means followed by the same uppercase letter in the row do not differ statistically from each other by Tukey test at 5% probability of error.

That was an expected situation because, in the case of exposed wood, the field conditions influenced the greater mass loss of the control and, consequently, less apparent specific gravity due to the attack by xylophagous microorganisms in relation to the heat-treated wood.

Regarding the *P. taeda* case, significant differences and reduction of apparent specific gravity were observed with the increasing temperature of the heat treatment. This situation may be related to the thermal treatments efficiency for degradation of cellulose and hemicellulose molecules that are most consumed by xylophagous organisms.

However, it can be emphasized that they were also affected by heat treatment temperatures, causing a greater reduction in the pine mass in relation to eucalyptus, where there was lesser temperature effect, as reported by Carvalho et al. (2019), similar to that observed by Cademartori et al. (2015) when evaluating four heat treatment temperatures for *Eucalyptus* sp.

These parameters may have influenced the behavior of the dynamic modulus of elasticity determined in the wood exposed in field environment, as can be observed in the analysis of variance presented in Table 3.

As shown in Table 3, the heat treatment of the species significantly influenced the wood dynamic modulus of elasticity determined by the *Stress Wave Timer*, similar to that observed by Icel et al. (2015) in a destructive essay. For Moura et al. (2012), during heat treatment there is carbohydrates elimination, and according to Boonstra et al. (2007), crystallization of cellulose molecules can be observed, a phenomena that directly influence the wood strength and stiffness.

Similar to the temperatures used in the analyzes of this study, Barcík et al. (2015), when evaluating colorimetric changes, state that the levels for carbohydrate reduction and crystallization of cellulose molecules start from 140 ºC onwards.

The local conditions of this experiment, a forest near a river and in a rainy season, causing soil saturation, are similar to those reported by Ribeiro et al. (2014), allowing the movement (and possible leaching) of extractives and other low molecular weight components to the wood surface, consequently to the propensity of biological degradation of their molecular constituents, altering the exposed wood stiffness, which can be observed by the statistical difference of all treatments, from both species before and after exposure to the field test (Table 4).

Table 3. Analysis of variance summary of the dynamic modulus of elasticity for *E. grandis* and *P. taeda* wood subjected to the field test.

Species	Variation sources	F significance
E. grandis	Heat treatment (A)	19.96*
	Exposure time (B)	0.72ns
	AxB	1.21ns
P. taeda	Heat treatment (A)	35.85*
	Exposure time (B)	1.02ns
	AxB	1.30ns

*Significant at 5% of probability ($p < 0.05$), nsNot significant ($p \geq 0.05$).

Table 4. Mean dynamic modulus of elasticity values of *E. grandis* and *P. taeda* subjected to the field test.

Species	Condition	Dynamic modulus of elasticity (kgf cm²)		
		Control	T.140	T.160
E. grandis	Before	122.425.6*A (16.41)	163.434.2*C (13.71)	145.218.8*B (19.31)
	After	107.041.4A (18.76)	145.625.8 C (13.11)	130.472.8B (19.74)
P. taeda	Before	119.928.4*A (24.94)	80.758.8*B (22.24)	73.538.7*B (19.27)
	After	104.829.3A (28.03)	70.574.4B (21.13)	58.963.9B (20.42)

* Significant difference in column by Student t-test. Means followed by the same capital letter in the row do not differ statistically from each other by the Tukey test at a 5% error probability level. Value in parentheses corresponds to the coefficient of variation from the samples.

Analyzing the condition before and after exposure, *E. grandis* control presented a higher percentage reduction, equivalent to 13.9% while the heat-treated wood (T.140 and T.160) had a reduction of around 10%. In other words, heat treatment proved to be efficient, minimizing the stiffness loss of the treated wood. For *P. taeda* though, the control displayed a reduction of 12.5%, lower than the treated wood, which had a reduction of 12.6% and 19.8% in T.140 and T.160, respectively, indicating that the temperature increase used in the heat treatment negatively affects this property.

We can observe that the dynamic modulus of elasticity from both species had different behaviors, and for *E. grandis* there are higher values of *DMoE* from heat-treated wood in relation to the control, contrary to what happened for *P. taeda*.

Table 4 shows that both species have in common the *DMoE* reduction in heat-treated wood at 160 °C in relation to the temperature of 140 °C. This situation is attributed to alterations in the properties that make the wood thermoplastic due to the crystallization of cellulose molecules and degradation of hemicelluloses. Schneid & Moraes (2017) cite these components sensitive to the temperature action, reporting that lignin is responsible for the cell wall stiffness, and that it undergoes changes at elevated temperatures, usually higher than 160 °C.

Considering that the field exposure time did not significantly influence *DMoE* variation, it can be inferred that the increase of this property, observed for *E. grandis*, is attributed to the protective action effect and efficiency of the heat treatment. In *P. taeda*, the *DMoE* gradual reduction is the result of carbohydrate degradation, as also reported by Carvalho et al. (2017) in *Pinus oocarpa* wood heat-treated at a temperature of 150 to 225 °C, causing the wood to loss both strength and stiffness.

Studies that analyzed MoE in heat-treated wood verified different behaviors, with Kacikova et al. (2013) observing a reduction of this property in *Eucalyptus regnans* wood from 140 °C onwards. However, Cademartori et al. (2015) did not observe MoE differences after the heat treatment process in *E. grandis* and *E. saligna*, with temperatures from 180 to 200 °C.

On the other hand, the observed differences in behavior after heat treatment between species may be associated with the wood anatomical structure (Boonstra et al., 2007), as well as to the food preference by the types of xylophagous microorganisms, due to the fungal decay potential.

Therefore, in Figure 2, are illustrated the variation of the dynamic modulus of elasticity and fungal decay potential as a function of the exposure time of *E. grandis* wood (A); *P. taeda* (B) in a field test environment.

Figure 2 displays that the fungal decay potential tends to increase as the wood exposure time increases to approximately 150 days, which is also the time period that we can begin to observe the reduction of wood attack potential by xylophages organisms. We can also observe that with increase in FDP there is a reduction of *DMoE* curves, with a contrary behavior occurring when there is a reduction in FDP, noticing a slight tendency to the slope of *DMoE* curves in the final period, for the control and for the T.160 in *E. grandis* wood, and for T.140 in *P. taeda* wood.

Figure 2-A displays the *E. grandis* behavior, with T.160 presenting a higher coefficient of determination in relation to the others, in which 76% of the *DMoE* variation is due to the exposure time and fungal decay potential. This said treatment also showed a relatively lower *DMoE* when compared to the others, at the beginning of the experiment, having a decreasing variation over time with a FDP increase, which kept gradually increasing until 160 days of wood exposure.

DMoE from the control and heat-treated wood at 140 °C had weak to moderate model fitting, with FDP increase over exposure time. This result emphasizes the possible efficiency of the T.160 treatment especially after 120 days of *E. grandis* wood exposure in the field environment, which coincides with the inflection region of the FDP curve.

For *P. taeda* (Figure 2-B), *DMoE* variations were also observed, however, these values showed greater interdependence in relation to FDP over the exposure time of the wood in the field. Regarding the control and the treatment at 160 °C cases, moderate coefficients of determination were verified with 53% and 40% respectively, particularly influenced by the *DMoE* values observed at the experiment beginning of the said treatments. As for the treatment at 140 ºC, it was verified the strong fit between the evaluated variables, with about 87% of the *DMoE* variations explained by the FDP variations at the field during the experiment.

Differences observed in *DMoE* between species subjected to the same decay condition and by the same heat treatments may be related to the differences in chemical composition, especially in the percentage of carbohydrates present in the species. Kollmann & Côté (1968) reported that the fraction of the percentage of cellulose, hemicellulose and lignin, among the evaluated species, results in the difference between the stiffness properties. Moreover, the anatomical difference is preponderant, since the hardwood species have thicker cells wall in relation to the softwood species, conferring a difference in strength and stiffness, as found by Braz et al. (2013) in *Toona ciliata* wood.

Regarding the stiffness loss of *E. grandis* in the tested treatments (Figure 3-A), with emphasis on the control and heat-treated wood at 140 °C, a loss of approximately 10% of the *DMoE* up to 120 days of exposure has been observed . However, the material analyzed after 120 days of exposure showed a proportional stability in stiffness loss of approximately 5%, increasing again after 160 and 200 days of exposure in the field test.

Variation of the stiffness loss over the exposure time probably happened due to the variation of the specific gravity of these species wood, according to the visual analysis, made by the loss of surface mass, possibly caused by fungal or xylophagous organisms attack.

Stiffness loss for eucalyptus was not the expected, as there is a tendency of xylophagous organisms activity increasing over time, which should lead to increased stiffness loss, i.e., eucalyptus wood requires a longer exposure time for observing changes, because, as reported by Carvalho et al. (2019), the total period mass loss of this material was less than 5%. This stiffness variation, according to Aprile et al. (1999), is explained by the fact that wood decomposition during the exposure is usually not continuous, therefore, during the exposure time it is represented a succession of phases with broad activity, and inhibition intervals, due to complete limitation of physical, chemical or biological processes in the wood decomposition.

As for the *P. taeda* (Figure 3 B), it can be observed that all treatments had the same behavior in stiffness loss, increasing progressively with the exposure time, which is reflected by the

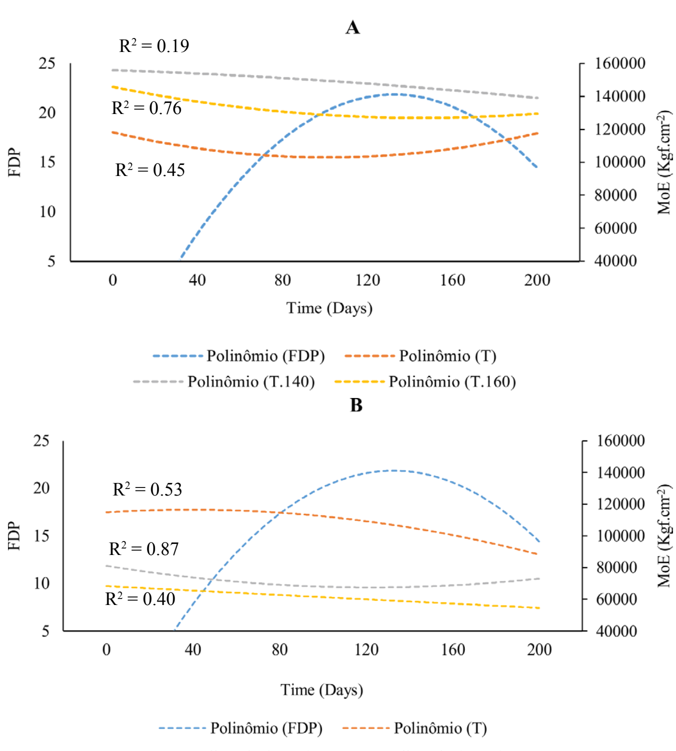

Figure 2. Wood modulus of elasticity variation from: A- *E. grandis*; B- *P. taeda* as a function of fungal decay potential and exposure time in the field environment.

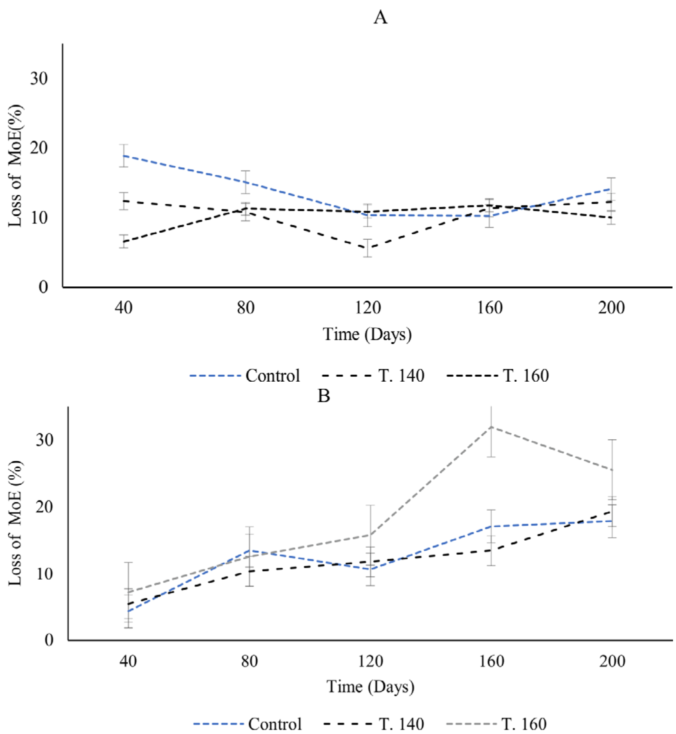

Figure 3. Stiffness loss variation of *E. grandis*-A and *P. taeda*-B wood as a function of exposure time in the field test.

gradual increase in mass loss of this species with the exposed period to the field (Carvalho et al., 2019).

For this species, it is worth mentioning that the T.160 treatment, after 120 days of exposure, showed increased stiffness loss, with reduction of about 30% in assays performed after 160 days of exposure, bearing greater losses than the observed both in T.140 and the control.

Therefore, it is possible to affirm that for *P. taeda*, heat treatment did not prevent the attack of xylophagous agents that caused the wood stiffness reduction, but quite on the contrary, when the wood was heat-treated at 160 °C, it had the highest percentage of stiffness loss, corroborating with Halabe & Reynold (1998), who state that when wood is decayed, it loses the cellulosic material compaction, becoming less elastic than healthy wood.

Conclusions

Exposure time in the field test reduced the apparent specific gravity or *DMoE* of all treatments from the evaluated species.

The heat treatments had influence on the wood *DMoE* variation of both species.

Loss of stiffness in *E. grandis* decreases in contrast to *P. taeda*, in which is verified an increased stiffness loss with the exposure time.

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