

Effect of thermal modification in the natural resistance of *Eucalyptus grandis* and *Pinus taeda* woods

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ABSTRACT: The aim of the study was to evaluate the natural durability of wood submitted to heat treatments and exposed in a field environment. Were used *Eucalyptus grandis* and *Pinus taeda* species, being selected five trees selected for each species studied, which were unfolded, dried in a conventional drying kiln, and later submitted to the heat treatments using temperatures of 140 and 160 °C. Subsequently 75 specimens of each species were prepared, which were tested in the field during six months of exposure, and the evaluations of the samples occurring every 40 days. The evaluation included the analysis of climatic variables, potential of fungal attack and the mass loss (%) of the woods, being the statistical analysis of the data by ANOVA for mass loss (%), in a completely randomized design with triple factorial arrangement, for the factor specie in two levels, factor thermal treatment in tree levels (control, 140 and 160 °C) and time of exposure in five levels (40, 80, 120, 160 and 200 days of exposure). *Eucalyptus* and pine treated at 160 °C presented respectively the highest and lowest resistance to mass loss. All woods are classified by the average values as very resistant.

Key words: biodeterioration of wood; mass loss; heat treatment

Efeito da modificação térmica na resistência natural da madeira *Eucalyptus grandis* e *Pinus taeda*

RESUMO: O objetivo do estudo foi avaliar a durabilidade natural de madeiras submetidas a tratamentos térmicos e expostas em ambiente de campo. Foram utilizadas madeiras das espécies *Eucalyptus grandis* e *Pinus taeda*, sendo selecionadas cinco árvores para cada espécie, as quais foram desdobradas, secas em câmara de secagem convencional e submetidas à modificação térmica utilizando temperaturas de 140 e 160 °C. Posteriormente foram confeccionados 75 corpos de prova de cada espécie, expostos em campo durante seis meses, e as avaliações ocorrendo a cada 40 dias. Procedeu-se com a análise de variáveis climáticas, potencial de ataque fúngico e a perda de massa (%) das madeiras, sendo a análise estatística dos dados em ANOVA para a perda de massa (%), em delineamento inteiramente ao acaso com arranjo fatorial triplo para o fator espécie em dois níveis, fator tratamento térmico em três níveis (testemunha, 140 e 160 °C) e fator tempo de exposição em cinco níveis (40; 80; 120; 160 e 200 dias de exposição). O eucalipto e o pinus tratados a 160 °C apresentaram, respectivamente, a maior e a menor resistência a perda de massa. Todas as madeiras são classificadas pelos valores médios como muito resistentes.

Palavras-chave: biodeterioração da madeira; perda de massa; tratamento térmico

Introduction

Natural durability is one of the factors that can limit usage of different wood species in various segments. Moreover, some physical, chemical and biological factors such as relative air humidity, temperature and precipitation can affect the natural resistance of the wood to the attack of xylophagous organisms, thus restricting their use and application in the forest sector.

Preservative treatments can be held by several methods, aiming to increase the natural durability of the wood, especially in the fast growing species.

The usage of chemicals products, harmful to the environment, has been a limiting factor due to the different applications of the wood, which in turn increases the interest in the heat treatment, where, according to Kocaefe et al. (2008), the wood is subjected to high temperatures (100 to 280 °C), thus causing modifications in its chemical composition in order to try to increase the useful life of the material, hence reducing the attack possibility by xylophagous organisms due to the degradation of celluloses, hemicelluloses and lignin molecules.

This technique has been claiming more and more space due to its practicality, low cost and low environmental impact, bringing some benefits for the wood while also stimulating the development of new researches and works aimed at the better use of wood products. This growth can also be explained by the value addition in the heat-treated woods, in addition to the cost-benefit ratio, allowing a longer useful life by reducing the susceptibility of attack by fungi and xylophagous insects (Paes et al., 2015).

Since thermal modification can also be conducted under the application of oxygen, nitrogen, steam and oils, the useful life of the wood can vary depending on the method and process; therefore, it is important to determine the natural durability for each used method. Moura et al. (2012) cite that the thermal modification process shows good performance regarding the resistance to the xylophagous agents that degrade wood, in addition to reducing the dimensional variations that can occur in the wood.

In this sense, Mattos et al. (2013) state that field deterioration tests are more reliable in characterizing the natural resistance of the wood as well as the efficiency of the preservative processes when compared to laboratory tests, since that in the field the wood is exposed to more severe conditions of deterioration, especially when it is in direct contact with the soil. Tied to this fact and thus justifying this type of study, Poubel et al. (2013), comment that studies related to wood durability are considered as problems to be solved by the wood technologists.

Therefore, this study was developed aiming to evaluate the natural durability of *Eucalyptus grandis* Hill ex Maiden and *Pinus taeda* L. species subjected to thermal treatments and then exposed in field environment.

Materials and Methods

Raw material

For the present research, it were used *Eucalyptus grandis* Hill ex Maiden and *Pinus taeda* L. woods from a commercial plantation of the TWBrazil Company. For the work, five trees of each species were selected according to their phytosanitary aspects, base diameter and bole shape, which were unfolded to obtain planks with dimensions of 100 x 25 x 1200 mm (width x thickness x length). These were also dried in a conventional drying chamber using a mild drying program until about 10% of humidity content, with a \pm 5% variation.

Heat treatment

Afterwards, the boards were subjected to the thermal modification process by applying heat at high temperatures using saturated steam in an oxygen elimination system (VAP HolzSysteme®) in order for obtaining the TMT or Thermally Modified Timber product.

For each of the evaluated species (*Eucalyptus grandis* and *Pinus taeda*), control pieces without any heat treatment applications were used, and pieces obtained from two thermal modification temperatures (140 and 160 °C) with a total cycle of eight hours, according to thermal modification program described by Batista et al. (2015).

Specimen preparation

After that, the control and thermally modified boards were reduced in specimens with dimensions of 20 x 20 x 300 mm (width x thickness x length), thus obtaining the following wood conditions used in the experiment:

Control group - oven-dried specimens at $10 \pm 5\%$ humidity;

T. 140 °C - Heat treatment specimens at 140 °C;

T. 160 °C - Heat treatment specimens at 160 °C;

Therefore, 75 specimens were prepared for the three wood conditions of each species, being then 25 for control samples, 25 for treatment at 140 °C and 25 for treatment at 160 °C, thus totaling 150 specimens from the two used species.

After obtaining the specimens, they were identified and set up for humidity stabilization in a climatic chamber and then subjected to a visual analysis for verifying the non-occurrence of flaws and defects prior to the exposure in a decay field test.

Deterioration tests

The exposure was done in a covered field, while in direct contact with the region clayey soil and near a superficial water table (25°26'56.9"S 49°14'16.3"W), where the specimens, previously identified and evaluated regarding absence of deformities that could affect their durability, were fixed up to half their length (150 mm), making then five replicates, each containing five test specimens representative of the conditions of control samples and the heat treated samples from *Eucalyptus grandis* and *Pinus taeda*.

The exposure was carried out in the period from September to March, during which at every 40 days the evaluations were made by withdrawing five specimens corresponding to

each replicate of the three wood treatment conditions, thus totaling five evaluations in an exposure period of 200 days of the two species.

In this period, the climatic conditions observed at the site of the experiment (temperature, humidity and precipitation) were recorded, and with these information the fungal attack potential (PAF) to which the species were subjected during the test was determined by using the Eq. 1, proposed by Scheffer (1971) and adapted by Martins et al. (2003).

$$PAF = \sum \left[\frac{(T-2) \times (D-3)}{16.7} \right] \quad (1)$$

In which PAF = Fungal attack potential of the species; T = average temperature of the period (°C); D = Number of days in the period with rainfall equal to or greater than 0.3 mm.

Afterwards, the specimens from each replicate collected at every 40 days were cleaned and once again submitted to humidity stabilization in climatic chamber. After the humidity stabilization, their natural resistance was evaluated in function of the mass loss (Table 1), as proposed by the ASTM D-2017 standard (2005).

Table 1. Resistance classes under the mass loss aspect (ASTM D-2017, 2005).

Resistance classes	Mass loss	Residual mass
	(%)	
Very resistant	0-10	90-00
Resistant	11-24	76-89
Moderately resistant	25-44	56-75
Not resistant	> 45	< 55

Statistical analysis

For the results evaluation, an electronic spreadsheet was used to develop curves of the climatic variables and the fungal attack potential, as well as variation trends of the mass loss in function of the fungal attack potential. Concomitantly, a statistical analysis of the ANOVA data for the mass loss was carried out, using a completely randomized design in a factorial experiment of the 2 x 3 x 5 type (two species x three conditions x five collection periods), and the evaluated factors which showed significant difference by the analysis of variance had their means compared by the Tukey test ($p = 0.05$).

Results and Discussion

We can observe that the rainy days, relative humidity and average temperature factors illustrated in Figure 1 follow a similar trend of variability. Such factors are primordial for the development of xylophagous agents that affect the cell wall components of the wood. The PAF follows the same trend as the factors mentioned, indicating the susceptibility of the exposed wood to organisms attack, potentiating the wood mass loss according to the exposure period, and as cited by Moreschi (2013), among biological agents, fungi cause

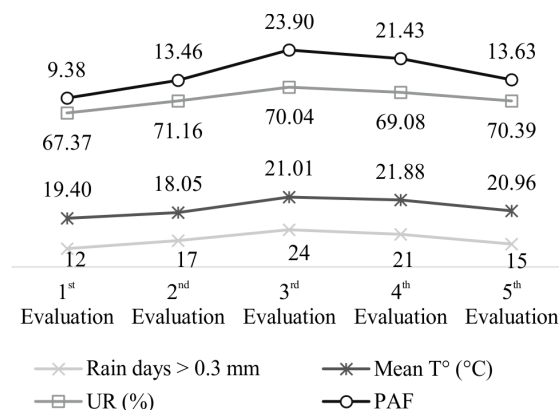


Figure 1. Fungal attack potential (PAF), relative humidity (UR), mean temperature (°C) and rainy days recorded during the exposure of the *Eucalyptus grandis* and *Pinus taeda* woods to the field environment.

the greatest incidence of problems for wood, when used in adverse situations.

Relative humidity and temperature are predominant factors for the fungal infestation, and the higher they are, the higher the fungus development will intensify. For Scheffer (1971) the occurrence and low temperatures in humid climates disfavor the occurrence of biodegradation organisms and Sedlbauer (2001) affirms that relative humidity rates above 65% coupled with elevated temperatures are ideal conditions for fungi appearance. In this study, the relative humidity ranged from 67.37 to 71.16%, fitting itself above the range cited by the author, which may provide the development of xylophagous fungi in the material in study.

As it can be observed, the higher the temperature, the higher the incidence of rainy days, the air relative humidity and the potentiality of a fungal attack will all be. With this, we can affirm that the potential can vary according to the microclimate where the wood will be exposed. Corroborating with such a statement, Casavecchia et al. (2016) when evaluating the PAF in Mato Grosso, concluded that in the state a variation occurs between the dry and rainy seasons, being the higher attack potential in the rainy season, which demands a greater attention to techniques that protect the wood.

Therefore, an effective protection of the wood is indicated when it is exposed to environments where external effects (rain, temperature and relative humidity) may increase the occurrence of organisms that degrade the wood. In this case, the use of wood thermoreftication processes can have the positive effect of protecting it.

Carvalho et al. (2016), when evaluating the PAF for more than one year, obtained mean temperature values of 21.8 °C and relative humidity (UR) of 75.3%, which resulted in a mean PAF of 10.1. For this work, the mean temperature was 20.5 °C and UR of 70.5%, similar to the result of the cited author; however, the mean PAF obtained for the present work was of 18.1, which can be explained by the higher number of rainy days, evidencing that the precipitation factor shows great influence on the probability of fungi occurrence. Another

factor that contributes to the obtained values is the evaluation period of the present study, which occurred in the months with the highest precipitation for the site.

Evaluation of the mass loss variation vs PAF

For all the figures and tables we observed the relation between mass loss and fungal attack potential of the three treatments applied for the two studied species (A – *Pinus taeda* and B – *Eucalyptus grandis*).

In Figure 2A, it is noted an increase in mass loss with the evolution of the evaluation time, with small oscillations. The T140 treatment was the one that presented the highest values of mass loss, similar to the behavior of the control group curve.

The interesting fact for this species is in the T160, which maintained itself with relatively homogeneous values of mass loss with the time progress of the evaluations. This behavior can be explained by the protective surface layer created in the wood from the thermoretification treatment, inhibiting the humidity penetration into the wood, in addition to the possible superficial occurrence of toxic substances and degrading agents of the wood cellular composition.

In a similar work, Modes et al. (2017) concluded that the heat treatment applied to *Pinus taeda* wood reduced its biological degradation caused by the fungus *Trametes versicolor*.

It is possible to observe this fact by relating the PAF in the third evaluation, where the potential for fungi occurrence was

the highest, but the mass loss was lower than other periods of evaluation where the PAF was higher, especially for the treatment with higher temperature.

In Figure 2B, the mass loss had similar behavior to the previous species, increasing its loss in accordance to the progress of the material exposure period, with a small oscillation for the T160 in the second evaluation. Subsequently to this evaluation the mass loss of this treatment increased dramatically just as in the T140, presenting a greater mass loss than the control group, which can be explained by the temperature and the time used in the treatment, which may have affected the cell wall composition of the species and consequently increased mass loss.

Modes et al. (2017) mention that this mass reduction behavior in some species can occur by the rupture and deterioration of the cell wall, mostly the polysaccharides and hemicelluloses, because it is considered sensitive to the elevated temperature action.

The obtained behavior of this species in the present work resembles that which was found by Menezes et al. (2014) when evaluating woods from *Corymbia citriodora* and *Eucalyptus saligna*, where the authors observed an increase in the mass loss in the woods thermoretificated with temperatures between 140 and 180 °C.

It is possible to observe in Table 2 that, in the different used species, the pre-treatments made for thermal modification and the exposure months of the wood in the field environment had a significant effect for the observed mass loss.

In respect of the interactions between the evaluated factors during the experiment, only the combination between the species and the pre-treatments, as well as the combination between the species and the exposure period, had significant effects on the wood mass loss. The remaining combinations were not significant in the mass loss of the two species.

The understanding of the observed phenomena between the two species evaluated during the exposure period can be explained by the observed differences in their wood formation. Hardwoods, such as eucalyptus, have in their structure a higher percentage of molecules of cellulose and hemicelluloses in relation to softwoods, such as the pine. This caused the two species to present different mass losses, since according to Castro et al. (1993), most of the agents prefer to consume the xylans which are in the highest percentage in

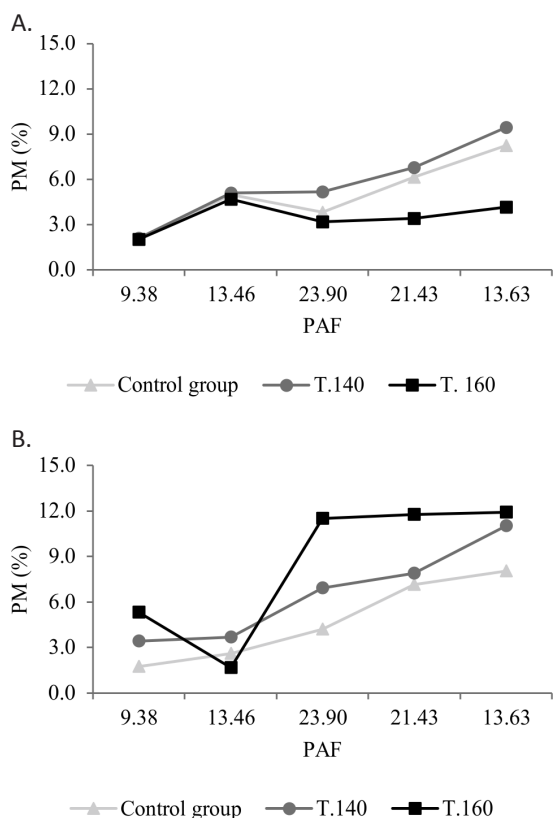


Figure 2. Mass loss variation in function of the PAF from *Pinus taeda* (A) and *Eucalyptus grandis* (B) woods in field environment.

Table 2. Analysis of variance overview of the mass loss from *Eucalyptus grandis* and *Pinus taeda* woods subjected to field environment.

Variable	Variation source	Significance of F
Mass loss	Species (A)	17.11**
	Pre-treatment (B)	3.54*
	Exposure months (C)	15.04**
	AxB	13.47**
	AxC	2.86*
	BXC	0.70 ^{ns}
	AxBxC	0.68 ^{ns}

** Significant at the 1% probability level ($p < 0.01$); * Significant at the 5% probability level ($p < 0.05$); ^{ns} Not significant ($p \geq 0.05$).

eucalyptus, in relation to the mannans present in the highest percentage in pine.

Another important aspect is the existing difference in the consumption ease of these substances, since the xylans are easily removable, since they do not require too much energy for their consumption by the biodeteriorating agents in relation to the mannans present in the hemicellulose molecules, as it was reported by Eriksson (1990), highlighting the statement by Castro et al. (1993) that the type of molecular elements is a preponderant factor for facilitating the attack by xylophagous organisms.

On the other hand, pre-treatments with different applied temperatures resulted in different mass losses during the exposure time, most probably because of the difference in desirable sugars by deteriorating agents in the exposed wood, due to the degradation of cellulose and hemicellulose molecules with the heat treatments application.

According to Otjen & Blanchette (1982), the lignin decomposition by heat treatments may have inhibited the action of biodeteriorating agents, especially in the initial stage, resulting in the difference of the wood consumption and deterioration between treated and untreated samples.

For Lazarotto et al. (2001), the heat treatments cause the hemicelluloses degradation, increasing the proportion of crystalline cellulose, thus promoting, according to Tjeerdma & Militz (2005), increases in the cross-links of the lignin network and the generation of new extractable compounds, which for Cademartori et al. (2014), may act on the wood as fungicides.

Therefore, the observed difference in the applied temperatures in the wood resulted in different consumption of starch and proteins, causing some pre-treatments to be consumed more than the others were.

Moreover, it is possible to observe in Table 2 the significant effect of the exposure time, remembering that the experiment had its start in the middle of the dry and cold periods, and having ended in the middle of the rainy and humid periods. This situation provided different ecological conditions at the site, which according to Melo et al. (2010) lead to differences in the wood deterioration agents activities for the consumption of sugars and consequent difference in the mass loss.

Allied to this, Melo et al. (2010) and Ribeiro et al. (2014) emphasize that the lower wood natural durability is related to the greater humidity existing in the environment, favoring the

diversity and performance of the fungi except in cases of soil saturation and less insolation of the place.

The unfolding of these results are explained by the observed mean values of mass loss during the experiment (Table 3) for the *Eucalyptus grandis* and *Pinus taeda* species, as well as for the different treatments (control group, thermoretification at 140 °C and 160 °C).

Regarding the evaluated treatments, it is possible to observe in Table 3 a greater emphasis for the *Pinus taeda* and *Eucalyptus grandis* thermoretificated at 160 °C, with the highest and smallest mass loss, respectively.

It is also observed that the thermoretification has shown itself as inefficient for *Pinus taeda*, considering that the control group presented a lower mass loss when compared to the treatments that received thermoretification, unlike of what was observed by Modes et al. (2017) when using the heat treatment to evaluate the accelerated degradation of *Pinus taeda*.

This fact may have occurred due to the inefficiency of the treatment on the evaluated species or, according to Doi et al. (2005), due to the production of low molecular weight sugar fragments, when there is degradation of the hemicelluloses during the high temperature processes.

Another aspect to be considered is the ecological characteristics of the site and the period in which the experiment was developed, besides the type of soil and the exposure site of the material near the water table are conditions that, according to Melo et al. (2010), Vivian et al. (2014) and Ribeiro et al. (2014), favored soil saturation during the rainy season, also favoring anaerobic conditions for fungi action.

Notwithstanding, Ribeiro et al. (2014) maintain that under conditions of soil saturation, one can observe the leaching of the low molecular weight chemical constituents that are the triggers of the deterioration process, favoring the higher resistance observed in Table 3, for the untreated samples from *Pinus taeda*

For the *Eucalyptus grandis* species, the heat treatment at 160 °C was efficient, reducing the mass loss when compared to the other treatments, except for the pinus control group. The low percentage of mass loss may have occurred in function of the used temperature in the thermoretification and also from the deposition of substances on the wood surface that prevented the attack of xylophagous organisms and humidity absorption.

Table 3. Observed mean values of mass loss (g) in *Eucalyptus grandis* and *Pinus taeda* woods during the exposure time.

Species	Treatment	Evaluations (days)					Means
		40	80	120	160	200	
<i>Eucalyptus grandis</i>	Control group	2.00	4.98	3.80	6.14	8.24	5.03b
	140 °C	2.10	5.09	5.16	6.78	9.44	5.72b
	160 °C	2.02	4.67	3.18	3.41	4.15	3.49c
	Mean	2.04Bc	4.91Bc	4.05Bc	5.44ABb	7.28Ab	
<i>Pinus taeda</i>	Control group	1.73	2.61	4.18	7.14	8.04	4.74bc
	140 °C	3.43	3.67	6.92	7.88	11.00	6.58b
	160 °C	5.32	5.52	12.67	13.22	12.13	9.77a
	Mean	3.49Cc	3.93Cc	7.92Bb	9.41Aa	10.39Aa	

Means followed by the same lowercase letter in the column, and by the same capital letter in the line, do not differ among themselves statistically by the Tukey test at 5% probability.

Table 4. Fitted models for prediction of mass loss from *Eucalyptus grandis* and *Pinus taeda* woods subjected to decay field.

Species	Treatment	B_0	B_1	B_2	R^2_{aj}	F
<i>Eucalyptus grandis</i>	Control group	13.1424	-27.2369	0.0390	25.26	5.06*
	140 °C	9.6269	-19.6930	0.0445	24.25	4.83*
	160 °C	2.6006	-0.0355	0.0075	-4.04	0.54 ^{ns}
<i>Pinus taeda</i>	Control group	9.6699	-16.4354	0.0393	64.68	22.97*
	140 °C	4.2490	-7.4564	0.0474	41.71	9.58*
	160 °C	-5.3677	19.6304	0.0526	39.32	7.15*

B_0 -Fitted model constant for prediction of mass loss (%); B_1 -Model coefficient for apparent specific mass of the wood (g cm^{-3}); B_2 -Model coefficient for the exposure time of the wood (days); R^2_{aj} = Fitted determination coefficient of the model; F- Calculated significance factor; *-significant at 95% probability; ^{ns}-Not significant.

As for the two species analyzed, it is observed that *Pinus taeda* showed a mass loss of 2.29% higher than the observed for *Eucalyptus grandis*, corroborating with Modes et al. (2017) when they evaluated the mass loss of pine and eucalyptus after thermoreification, where they also observed greater mass loss for the softwood.

The cell composition and density of the eucalyptus, besides the fungus species that attacked the specimens may have influenced the greater resistance to the xylophagous organisms attack. Modes et al. (2017) found a greater resistance to the attack of the *Trametes versicolor* fungus for *Pinus taeda* species and a greater resistance to the attack of the *Gloeophyllum trabeum* fungus for the *Eucalyptus grandis*.

As for the evaluation period, it was observed for the *Eucalyptus grandis* species that there was a gradual increase of the mass loss of the specimens, as it was expected, since the samples remain in contact with the soil longer, what in turn makes the wood humidity increases and it ends up providing the ideal conditions for the attack of deteriorating agents.

For *Pinus taeda*, it is observed that the mass loss also increased with the passing evaluations, however, it can be emphasized that in the third evaluation (7.92%) there was a drastic increase, which may have occurred in virtue of the analysis period being rainy, also tied to the temperature and relative humidity both ideal for fungi development, which propitiated the increase of the biodeteriorating agents attack.

In Table 4 it can be observed the fitted equations for estimating the mass loss of the studied wood, in function of the density and time in which the samples remained exposed to the environments.

In the mass loss prediction models generated for the evaluated species, only the 160 °C treatment equation for *Eucalyptus grandis* was not significant, not representing good fittings between the dependent and independent variables, besides also presenting poor model adaptation, contrasting with the other equations that presented good fittings.

However, the variation of the wood mass loss from the *Eucalyptus grandis* control group and the treated at 140 °C, despite being significant, is explained by a 25% reduction of the specific mass and increase of exposure time in field environment.

In general, it can be noted that the generated equations for the *Pinus taeda* species presented higher coefficients of determination in comparison to the *Eucalyptus grandis* species, which are considered efficient for the prediction of mass loss, as well as in the equations generated for the control group and treatment at 140 °C of *Eucalyptus grandis*.

Conclusions

There was an increase in the fungal attack potential with the mass loss for the treated and untreated woods of *Eucalyptus grandis* and *Pinus taeda* exposed in the field environment;

Eucalyptus grandis woods presented a lower mass loss in relation to the *Pinus taeda* woods exposed in the field environment;

Heat treatments were efficient in reducing the mass loss only for *Eucalyptus grandis*, when compared to *Pinus taeda*.

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