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ANALYSIS OF RIGIDITY LOSS AND DETERIORATION FROM EXPOSURE IN A DECAY TEST FIELD OF THERMORECTIFICATED Eucalyptus grandis WOOD♣

Henrique Trevisan [1,♠], João Vicente de Figueiredo Latorraca[2], Angelo Luíz Pacheco dos Santos[3], Juliana Grilo Teixeira[4], Acacio Geraldo de Carvalho[2]

In memoriam of Dr. Manfred SCHWANNINGER

ABSTRACT

The objective was to evaluate the elasticity dynamic modulus reduction (MOEd) and deterioration of *E. grandis* thermorectificated wood by exposure to environmental weathering. Six trees were used to obtain 14 logs of 2,4m, with seven from external (sapwood) and the others from internal (heartwood) part of the trunk. A total of 84 wood samples of 2,5x 5x 50cm were made, with half from the inner and the other from the external trunk portion. 14 treatments were evaluated with six replicates composed of thermorectificated wood submitted two differents times (2 and 4 hours) and three temperatures (200, 215 and 230˚C). An ultrasound Stress Wave Timer device was used to evaluate MOEd and the mass measured by weighing. Samples remained in the decay test field for ten months, and monthly inspected and recorded to xylophogous organism's occurrence. At the end of this period MOEd and mass were revaluated. The central portion of wood samples were more deteriorated, with mass losses and stiffness losses ranging from (8-56%) and (18-91%), respectively. Lower values were observed in the wood coming from the external portion, with mass losses and stiffness ranging from (3-10%) and (8-20%), respectively. The thermorectificated wood samples were more damaged by action the termites and less by the action of fungi, compared to controls.

Keywords: Xylophogous, term treatment, protection of wood.

INTRODUCTION

Environmental pressure on the use of native wood species and the enforcement of increasingly stricter laws on the use of chemicals traditionally used in wood preservation have entailed the development of research on commercial plantation wood submitted to a preservation treatment that has low toxicity to humans and the environment. In this sense, thermal treatment is a viable alternative.

This process can add to the wood desirable characteristics such as reduced equilibrium moisture content (EMC) improved dimensional stability and biological durability, without the need to use chemicals.

Thermal rectification is a process in which heat is applied to the wood at temperatures below those that trigger the degradation of its key chemical components, especially hemicelluloses, which are more sensitive to the action of heat (Pessoa *et al.* 2006). According to Guedira (1988) and Vovelle and Mellottee (1982), these temperatures would be between 100 and 250º C. For Pessoa *et al.* (2006), in turn, the thermal rectification or thermorectification process is that conducted at temperatures below those used in roasting (temperature ranging between 200 and 280º C).

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[1] Forest Engineer Doctor Post/ Forest Institute, Federal Rural University of Rio de Janeiro. Seropédica, Brazil.
[2] Professor, Department of Forest Products / Forest Institute, Federal Rural University of Rio de Janeiro. Seropédica, Brazil.
[3] Forest Engineer/Independent professional.
[4] Forest Engineer PhD student / Forest Institute, Federal Rural University of Rio de Janeiro. Seropédica, Brazil.
♠Corresponding author: hentrevisan@gmail.com.
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The result is a solid product with differentiated characteristics in comparison to natural wood.

Most thermal treatment methods are based on reduced accessibility of hydroxyl groups, which are found mainly in cellulose and hemicelluloses and are primarily responsible for the wood hygroscopicity. By blocking these groups, mainly those present in hemicelluloses and which are more accessible, wood reduces its ability to absorb water, with an impact on various properties of treated wood (Esteves and Pereira 2009).

In turn, the use of high temperatures can cause negative effects on wood, such as degradation of the internal structure, which consequently promotes loss of mechanical strength.

Therefore, the objective of this study was to evaluate the elasticity dynamic modulus reduction (MOEd) and deterioration of *E. grandis* thermorectified wood by exposure to environmental conditions.

MATERIAL AND METHODS

The experiment was conducted at the Forest Institute, Federal Rural University of Rio de Janeiro, in partnership with the Federal University of Paraná.

Six 23 year-old *Eucalyptus grandis* trees from a commercial plantation were used, from which three logs measuring 2,40 m in length from the DBH (diameter at breast height) were taken. Subsequently, the central, intermediate and external parts of each log were highlighted using different colors (Figure 1).

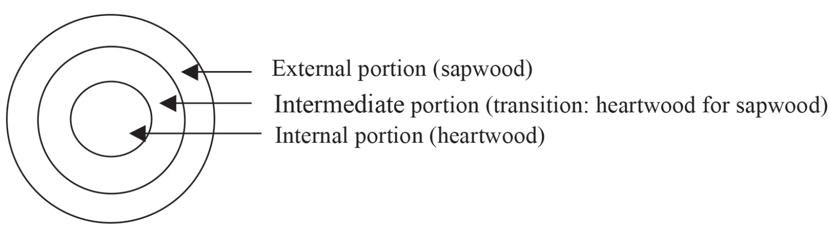

Figure 1. Schematic cross section of *Eucalyptus grandis* log identifying the portions of the trunk from which wood was taken to make up the samples.

In a wood shop two slabs were taken from each log using a band saw. The slabs were then transformed into semi-blocks approximately 15 cm high, which were processed using a multiple 2-axle circular saw, in order to obtain boards with a nominal thickness of 2,5 cm. Simultaneous cuts were made to split the block into tangential boards. These were identified by tree and portion according to the color painted on top of the pieces. This material was stacked with stickers and submitted to a gentle drying program in a kiln, which resulted in an average final moisture content of 8% in the wood.

The samples were produced using wood taken in a systematized way from the boards so that 14 samples could be obtained, of which seven were from the internal portion (heartwood) and seven from the external portion (sapwood) of the logs, with due care to select material free from the action of xylophogous organisms. Wood from the intermediate portion was discarded. The wood samples were standardized at 2,5 x 5 x 50 cm and placed in a climate-controlled chamber at 20±5°C and 65%±5% relative humidity, until a moisture content of approximately 12% was reached.

The thermal treatment was performed in the electric kiln model Linn Elektro therm, and the wood samples were submitted to three different temperatures and two exposure times, resulting, through the variation and combination of these parameters, in 12 treatments (Table 1). Treatments 13 and 14 corresponded to the controls, which were made of thermally untreated wood from the external and central portions.

Table 1. Exposure time and temperature used for wood thermorretification *Eucalyptus grandis* in 12 treatments.

Treatments	Time (h)	Temperature (ºC)	Position
T1	2	200	central
T2	2	215	central
T3	2	230	central
T4	2	200	external
T5	2	215	external
T6	2	230	external
T7	4	200	central
T8	4	215	central
T9	4	230	central
T10	4	200	external
T11	4	215	external
T12	4	230	external
T13	0	environment	central
T14	0	environment	external

After the thermal treatment, the wood samples were taken to the Federal University of Paraná, where the elasticity dynamic modulus reduction (MOEd) was measured, with the samples properly acclimated (relative humidity = ± 65%; Temperature = ± 27 º C). At the time, the mass of the wood samples was also recorded after conditioning, for the purpose of assessing the influence of deterioration on this parameter. The MOEd was determined using the Stress Wave Timer device. A wave was induced by positioning two transducers in the wood sample, and when the wave reached the start sensor, the instrument began counting time in microseconds and stopped when the wave reached the stop sensor. Thus, the device recorded and showed the transit time of tension through the wood sample. These values were used to obtain the MOEd applying the following formula:

$$\mathbf{MOEd = \rho \times V^2 \times 1/G \times 10^{-5}}$$

MOEd = elasticity dynamic modulus reduction (kgf /cm^2); ρ = wood density (kg/m^3); V = velocity of longitudinal wave (cm/s); G = gravity acceleration (9,804 m/s^2)

The velocity of wave propagation is obtained by the following equation:

$$V = d/t$$

V = velocity of longitudinal wave (cm/s); d=distance between transducers (cm); t = propagation time (μ s).

Once the MOEd had been measured, the wood samples were arranged in a decay test field, with the aim of evaluating deterioration and its effect on the stiffness of wood. A test field was set up with six wood samples, where these were arranged side by side with a 50 cm space between them and 20 cm burial depth. It was sought to establish that each sample came from a different tree in the different treatments.

The deterioration process was followed for three months through monthly evaluations. The occurrence of xylophogous fungi and termites was observed. The analysis parameters expressed in ASTM D-2278-06 (Table 2) were used for this first group of organisms, whereas the protocol suggested by the Institute of Technological Research (IPT) was used for the second group o organisms (Table 3).

Table 2. Parameters used to assess the occurrence and action the termites in the Eucalyptus grandis wood thermorectified (ASTM D-2278-06).

Description	Grade
Sound	10
Trace of attack	9
Moderate attack	7
Heavy attack	4
Failure by termite attack	0

Table 3. Parameters used to assess the occurrence and action the xylophogous fungi in the Eucalyptus grandis wood thermorectified (Lopes and Milano 1986).

Description	Grade
- Wood healthy without rotting.	1
- Fungi present, observed holes in the cell walls, wood structure in good general.	2
- Fungal moderate action, numerous holes, mold spores and hyphae present; moderately degraded wood structure with wood looks healthy.	3
- Intense action of fungi, numerous holes, hyphae and spores, very cellular structure altered by the fungi action.	4

Rate of ten is assigned to the sample when no termite action is observed. Rate of nine, in turn, is considered when a slight but positive termite action is observed. Rate of seven is assigned when heavy termite action is observed in the outer layers. Rate of four is assigned to wood heavily colonized by termites, but that does not break when a slight force is applied to it.

After ten months of exposure to field conditions, the samples were taken to the laboratory where they were cleaned, conditioned, weighed and the MOEd estimated again.

Arithmetic averages were obtained from the grades attributed to the wood samples, generating an average deterioration index. The values were assessed using the Kruskal-Wallis test at 5% significance level, and the average positions were compared using the Student-Newman-Keuls test; and differences were expressed in arithmetic averages.

RESULTS AND DISCUSSION

Regarding the occurrence and action of xylophogous fungi, the higher average deterioration rates observed in wood from the central region of the trunk of *E. grandis,* compared with the values corresponding to wood from the external region indicates that the latter is more susceptible to these organisms in both thermorectified and natural wood (Figure 2).

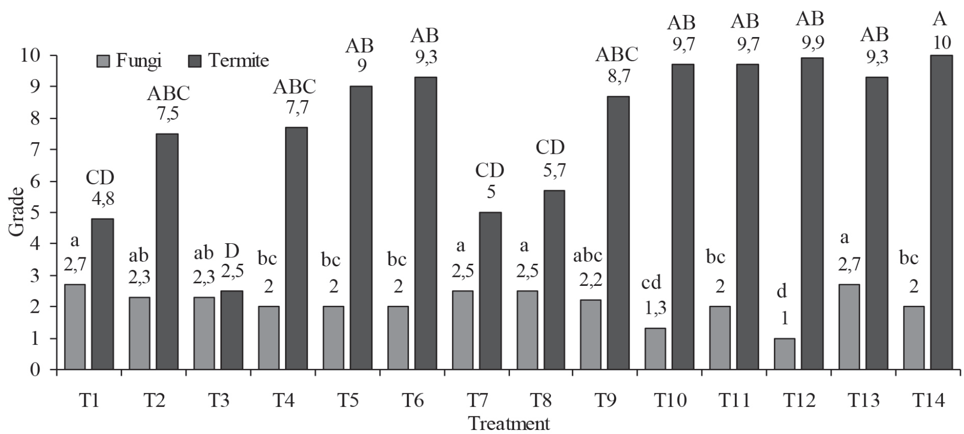

Figure 2. Average deterioration rates, assigned on the basis of the analysis of the occurrence and action of fungi and termites in wood thermorectified *Eucalyptus grandis,* exposed in the test field after ten months.

Different lowercase letters between columns express statistical differences for the evaluations carried out with fungi, whereas uppercase letters refer to termites.

However, with respect to thermorectification vis-à-vis the aggregation of resistance to these organisms, it was observed that treatment 12 (230°C for 4 hours) applied to the material coming from the external portion of the trunk produced the most significant results. In this sense, the average value of the deterioration rate corresponding to this thermorectification condition was significantly lower, revealing less action by these microorganisms as compared to the other average rate (Figure 2). It is worth pointing out that this protocol produced the best results as regards wood immunization against fungi, and no colonization by these microorganisms was observed in these samples during the ten months of exposure in the test field.

This observation can be interpreted as consistent when correlated with the information reported by Alén *et al.* (2002), Rousset *et al.* (2004), Metsä-Kortelainen *et al.* (2005), that one of the objectives of wood thermorectification is to confer higher biological resistance against the action of xylophogous agents. In the case of this study, it was demonstrated in relation to xylophogous fungi. It is also concurs with Momohara *et al.* (2003), who report that the greater the time and temperature of treatment, the better the resistance of wood to decay fungi.

As for other times and temperatures used to treat wood from the external and central parts of the trunk, it can be inferred that they also led to an increase in resistance against the action of these xylophogous organisms. However, the average deterioration rates generated by these other thermorectification conditions, although indicating that the action of these organisms tended to be inhibited, are statistically equal to the average value of the corresponding control (Fugure 2). This observation indicates that different treatment times and temperatures, although suggesting a trend, did not result in a significant increase in wood immunization. This observation is in contradiction to what was observed in wood taken from the external portion of the trunk treated at 230 º C for 4 hours (Figure 2).

In this context, when studying *E. grandis* wood thermorectified at 220º C relative to the action of *Pycnoporus sanguineus* fungus, Calonego (2009) concluded that this process resulted in a material 82% more resistant to mass loss than natural wood. It further explains that thermorectification alters the chemical composition of wood, promotes the unavailability of food (hemicellulose) to fungi, reduces the content of moisture balance, promotes the creation of new free molecules, which act as fungicide, in addition to affecting the lignin network, thus impairing recognition of the substrate by the fungus.

On the other hand, what was detected in relation to fungi was not observed in relation to the action of termites. In this case, the analysis performed suggests the opposite. Wood submitted to thermorectification was more susceptible to these xylophogous organisms, and the central portion (heartwood), as observed in the case of xylophogous fungi, was the trunk region that provided less resistant wood as compared to the external portion (sapwood).

Thus, the analysis of the comparisons of average deterioration rates in relation to the occurrence of termites in thermorectified wood shows that in both cases the amount of termites was lower in wood from the two radial positions (sapwood and heartwood) of the trunk when compared with the values corresponding to their control, showing a higher action of termites in these samples. However, statistical differences in relation to the corresponding control were observed only in rates related to wood samples taken from the central part. As for wood from the external part, their average rates, while also showing more intense action by these insects in thermorectified wood, were statistically equal when compared to the value observed in the corresponding control (Figure 1). This finding therefore reinforces the suggestion that wood from the central part can be more susceptible not only to fungi but to termites as well, and that thermal treatment can increase this susceptibility. (Figure 2).

On this issue, Pessoa *et al.* (2006) when assessing in the laboratory the action of *Cryptotermes brevis* on thermorectified *E. grandis* wood concluded that this process was not capable of protecting this material from the action of this termite. According to Silva *et al.* (2004), wood from this eucalyptus species is naturally susceptible to this termite. In this regard, the work of Pessoa *et al.* (2006) corroborates the findings of this study. However, these authors observed a reduction in insect attack levels and higher insect mortality as a result of the increase in wood treatment temperature as compared to the control. This differs from what was found in this study for comparison with the control relating to the level of attack. Although a similar trend was observed in this study for wood from the external region of the trunk, which is less resistant compared to the control, it was observed that at higher treatment temperatures the action of termites seemed to be more inhibited. But this pattern was not observed in wood from the central region of the trunk (Figure 2).

Pessoa *et al.* (2006), when explaining their findings also report that organic compounds derived from thermal degradation, such as phenolic compounds that can remain as residue in the obtained product could be toxic to insects. However, it should be taken into account that the tests conducted by Pessoa *et al.* (2006) occurred in a controlled environment which differs from the protocol used in this study. Thus, in the field these supposed volatile compounds might not present the action reported by Pessoa *et al.* (2006), thereby justifying the greater susceptibility of thermorectified wood to termites in comparison to natural wood, as found in this study. Therefore, studies that evaluate the effectiveness or even the negative impact of the thermorectification process vis-à-vis the aggregation of resistance in *E. grandis* wood to termites in field conditions, should be considered relevant.

As for mass and stiffness reduction, it was found that wood from the central portion of the trunk, whether thermorectified or not, showed values higher than those of wood from the external portion (Table 4). This corroborates what was found in relation to fungi and termites, therefore suggesting that this reduction is associated with more intense xylophogous action. It also confirms the suggestion that this wood is less durable than wood from the external portion.

In analyses of X-ray densitometry carried out with wood from 23-year-old *E. grandis* trees, Ramos *et al.* (2011) found that the zone of transition from juvenile wood to mature wood occurs between the 5^{th} and 11th growth ring (years 5 and 11). The wood used in this experiment was also obtained from 23-year-old trees; thus, the external portion of the trunk (sapwood) is made up of mature wood and the internal portion of juvenile wood (heartwood). In this context, the density of mature wood, which comes from the external portion, is higher in relation to the internal portion of the trunk (Oliveira *et al.* 2012, Ramos *et al.* 2011, Vidaure *et al.* 2011). Data also recorded in this experiment. Also with regard to mature wood, Vidaure *et al.* (2011) adds that tracheids length, cell wall thickness, cellulose content, and strength and stiffness are greater in wood with this characteristic. Thus, these records suggest that the different properties of juvenile wood and mature wood of *E. grandis* may have contributed to increase sapwood resistance to the action of wood-decay organisms compared with heartwood, as observed in this study. Bhat and Florence (2003), Dunish *et al.* (2010), when evaluating the natural resistance of juvenile wood and mature wood of Tectona grandis and Robinia pseudoacacia, respectively, to wood-decay fungi, concluded that mature wood was more resistant to the action of these organisms as compared with juvenile wood, a pattern similar to that observed in *E. grandis* in this study. Dunish *et al.* (2010) also observed, through chemical analysis, that juvenile wood had a lower content of extractable phenolics and flavonoid compounds, which may be one of the reasons why it was less resistant to the action of fungi.

Regarding the influence of the thermorectification in mass and stiffness reduction, it was found that the conditions adopted in treatments 11 and 12 conducted in material from the external portion of the trunk entailed the two smallest reductions in stiffness, as compared with the other thermorectification conditions. However these values are higher than those observed in the control (Table 4). In the evaluations performed in samples made of wood from the central part of the trunk for the analysis of loss of stiffness, the results were significantly different, with higher percentage reductions in the conditions of treatments 1, 3 and 7, and decreases lower than that of the corresponding control under conditions 2 and 9. In these last two, it can be understood that some resistance to deterioration has been given to wood, since in addition to lower stiffness reduction values in relation to the control it also presented lower rates of mass loss in thermally treated wood of this origin (Table 4).

Table 4. Mean values of mass (g) and dynamic elasticity modulus (kg/cm^2) measured before (condition I) and after (condition II) exposure for 10 months in decay test field, and respective percentage reductions in these parameters vis-à-vis the deterioration process, of samples made from *Eucalyptus grandis* wood thermorectified at different treatment temperatures and times.

Treatment	Condition I		Condition II		Reduction (%)	
	Mass	**Moed**	**Mass**	**Moed**	**Mass**	**Moed**
T1	247,80	112,058	120,38	33,473	51,42	70,13
T2	258,11	117,804	233,69	92,297	9,46	21,65
T3	233,85	114,804	104,77	11,563	55,19	90,92
T4	328,43	160,801	295,85	119,580	9,91	25,63
T5	364,95	174,009	348,00	145,195	4,64	16,56
T6	351,9	167,075	336,73	129,512	4,31	22,48
T7	259,25	125,102	163,15	30,835	37,06	75,35
T8	232,96	164,325	152,40	116,572	34,58	29,06
T9	313,53	152,968	274.00	127,413	12,60	18,32
T10	300,75	149,752	278,93	118,050	7,25	18,33
T11	337,21	164,905	321,80	142,269	4,57	12,46
T12	293,00	148,073	282,00	123,374	3,75	15,19
T13	290,9	120,314	266,90	87,305	8,25	29,00
T14	393,01	161,607	369,23	146,419	6,05	8,90

It is known that thermally treated wood normally shows an increase in MOEd and mass loss compared to natural wood depending on treatment time and temperature, as found by Moura *et al.* (2012), Calonego (2009), Santos (2000) in *E. grandis* wood. In this study, a greater MOEd increase was observed only in wood from the central part subjected to 4 hours of thermal treatment at the three temperatures used. In wood from the external part of the trunk, increases higher than those of the corresponding control were observed only-when the wood was treated for two hours at 215°C and 230°C and for four hours at 215°C.

CONCLUSIONS

Eucalyptus grandis wood from the external region of the trunk is more resistant to deterioration than wood from the central portion.

Thermally treated *Eucalyptus grandis* wood from the external region of the trunk is more resistant to the action of xylophogous fungi than natural wood.

Thermorectification applied to wood from the external region of the trunk of *Eucalyptus grandis* for four hours at 230°C immunized this material against the action of xylophogous fungi for a period of 10 months in field conditions.

Eucalyptus grandis wood from the central region of the trunk, when thermally treated, is more susceptible to the action of termites.

Thermal treatment applied at 215 and 230ºC on *Eucalyptus grandis* wood from the external region promotes a decrease in mass loss in relation to deterioration.

Thermorectification applied on wood from the external region of the trunk of *Eucalyptus grandis* increased the loss of stiffness in relation to the deterioration process.

The characteristics aggregated to *Eucalyptus grandis* wood as regards the thermorectification process differ according to the part of the trunk from where the sample is taken.

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