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The Effect of the Thermal Modification Temperature in the Resistance to the Parallel Compression of Fiber for Eucalyptus Grandis, Pinustaeda and Tectonagrandis Wood

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ABSTRACT

Background: Thermal modification of wood has been scientifically studied for nearly a century, yet industrially in Europe was consolidated only from the years 1990. However only from 2006, the Brazilian industry started its research on thermal modification, and currently has been developing an industrial process, called VAP HolzSysteme®. The evaluation of the mechanical resistance of wood is required because all its uses in solid demand this knowledge to enable the proper sizing of the products.In the case of those from thermally modified wood, the determination of these properties is critical, because the reduction in mechanical strength is known as the greatest prejudice given to process wood. **Objetive:** Therefore, this study aimed to evaluate the mechanical resistance to compression parallel to the fibers of the wood of Eucalyptus grandis, Pinustaeda and Tectonagrandis thermally modified by the VAP HolzSysteme ® process. **Methodology:** Thermal modification was performed in the company TWBrazil, which consisted of applying heat at high temperatures using saturated steam, combined with an efficient system of elimination of oxygen. For each evaluated species (Eucalyptus grandis, Pinustaeda and Tectonagrandis) were used two exposure temperatures (140 and 160° C), with a total cycle of eight hours. The mechanical tests of compression parallel to the fibers, were conducted in accordance with the recommendations of the Comisión Panamericana Technical Standards(COPANT). **Results:** The woods of Eucalyptus grandis, Pinustaeda and Tectonagrandis, due to the thermal gradients exposed, had their mechanical resistance affected, being the wood of Eucalyptus grandis species that lost resistance, followed by themadeiras de Pinustaedaand Tectonagrandis. The temperature displayed by the thermal modification of 160° C, was the temperature that most affect the mechanical properties of parallel compression to the fibers, to the three species evaluated. **Conclusions:** The wood exposed to thermal gradients showed reduction in compressive strength parallel to the fibers, being the wood of Eucalyptus grandis species evaluated which had the highest loss resistance and the wood of Tectonagrandis, presented the smallest loss of its compressive strength parallel to the fibers, which can be justified on the basis of the high content of extractives presented by this species.

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INTRODUCTION

The mechanical properties of wood are affected by its chemical constitution (cellulose, hemicellulose and lignin) as well as its physical and anatomical properties. However, some woods are not used in certain service requests due to its high dimensional instability and organoleptic characteristics such as their coloration.

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To minimize these effects, several studies have occurred, amongst them the thermal modification of wood, (STAMM, 1956; KUBOJIMA *et al*., 2000; KAMDEM *et al*., 2002; BEKHTA; NIEMZ, 2003; BATISTA; KLITZKE, 2010).

Thermal modification causes chemical, physical and mechanical changes in wood (RODRIGUES, 2009) and brings some advantages to the technological properties, such as the reduction of hygroscopicity of wood, resistance to xylophagous organisms and aesthetic changes of wood WEILAND; GUYONNET, 2003; ROUSSET *et al*., 2004; HAKKOU *et al*., 2006).

However, in the degradation of the chemical constituents of the wood, the loss of the material mass occurs, therefore the mechanical properties of wood are affected by thermal treatment (BODIG; JAYNE, 1982; THIAM *et al*., 2002; HAYGREEN; BOWYER, 1996; FOREST PRODUCTS LABORATORY, 1999) as some discrepancies between the results have being observed and reported by some authors (YILGOR *et al*., 2001; MORAES *et al*., 2005; SEVERO; TOMASELLI, 2000).Araújo *et al*. (2012) also adds that these differences lead to believing that these changes vary among species of woods beyond the parameters used in the thermal treatment. The effect of thermalrectification varies depending on the final temperature process, treatment time, the heating rate and the initial properties of wood.

Thereby, based on the suggestions of Santos (2000) and Unsal; Ayrilmis (2005) it is shown that the development of a process of thermalrectification and specific studies on its effect on the quality of the wood is important for the consolidation of the use of this wood by industry. Therefore, this study aims to evaluate the mechanical resistance to the parallel compression of fiber for *Eucalyptus grandis*, *Pinustaeda* and *Tectonagrandis* thermalrectification by the VAP HolzSysteme® process.

MATERIALS AND METHODS

Location, gathering and preparation of samples:

The wood used was from a commercial plantation of *Eucalyptus grandis*, *Pinustaeda* and *Tectonagrandis* located in the Northern Central region of Paraná. Five trees from each species according to their plant health, base diameter, stem form and concentric marrow were selected.

The material for this study was provided by the company *TWBrazil*, located in Ponta Grossa in Paraná state south of Brazil. The samples were prepared in the following dimensions; 80 x 30 x 800 mm (width x thickness x length). The samples had been previously dried in conventional drying kiln in a moisture content of around 10%, with a variation of ± 2%.

Thermal modification of wood:

The thermal modification was made in the company *TWBrazil*. It consists in applying heat to high temperatures using saturated steam, combined with an efficient system of elimination of oxygen, nominating the process *VAP HolzSysteme®* and adopting the terminology Thermally Modified Timber (TMT) to the product.

Two temperatures (140 and 160°C) with a total cycle of eight hours each treatment, totalizing nine treatments (Table 1) were used for each species studied (*Eucalyptus grandis*, *Pinustaeda* and *Tectonagrandis*).

Table 1: Treatments evaluated.

Espécie	Testemunha	Temperatura (ºC)	
Eucalyptus grandis	Controle	140	160
Pinustaeda	Controle	140	160
Tectonagrandis	Controle	140	160

The temperatures evaluated correspond to those commonly used by the company in generating their products. It was decided to make no changes in the variables and methodology of the process so that the search results correspond to the maximum to the reality of the products that are marketed by the company.

The equipment used for the process of thermal wood modification consists in a cylinder (pressure vessel) of 125 cm diameter, 850 cm long with a nominal capacity of 6 m³ of wood per cycle; Instant steam generator capable of producing 216 kgv.h-1; water tank; Programmable Logic Control, trolley rails (Figure 1).

The process of thermal modification VAP HolzSysteme® is characterized as hygrothermal (HILL,2006) being conducted in five steps, which consists of an initial heating at a given heating rate ($°C.min^{-1}$). Subsequently the temperature is kept constant at 110 °C for a period of 25 minutes. After this time, the second stage of heating starts, in wich there is a certain rate. The length varies according to the heating rate used and extends up to the device reaches the maximum preset temperature (140 or 160 °C). Reached the preset temperature, which corresponds to the thermal modification temperature, remaining for a time of 45 minutes in this step. To finalize the process which corresponds to cooling, it occurs naturally within the equipment until the temperature is around 30 °C (Figure 2).

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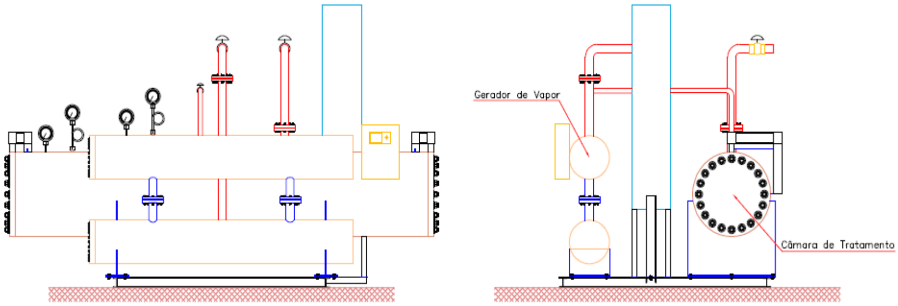

Fig. 1: Basic scheme set of the steam generator/sterilizer. Source: TWBrazil, 2012.

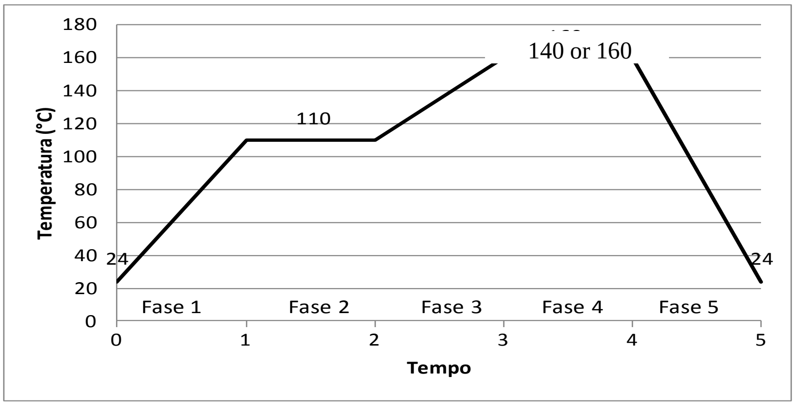

Fig. 2: Program of thermal modification process VAP HolzSysteme® Source: Batista (2012).

Mechanical characterization:

The mechanical characterization of wood was determined by the essay of compression parallel to the fiber, based on the recommendation of technical standard - COPANT 464 (1972). Samples containing cross section of 25mm by 25mm and the longitudinal fiber direction of 100 mm.

Resistance to compression parallel to the fibers (fc) and the elasticity modulus (MOE) was determined according to equations 1 and 2, respectively.

$$f_C = \frac{P}{A} \tag{1}$$

$$MOE = \frac{P'.L}{A.D'} \tag{2}$$

fc: resistance to compression parallel to the fiber (MPa);
MOE: modulus of elasticity in compression parallel to the fiber (MPa);
P: load rupture (N);
P: load at proportional limit (N);
A: cross sectional area of the test body (mm ²);
L: length of the transducer (mm);
D ': strain at proportional limit (mm).

Prior to the tests, it was determined the apparent specific gravity of the specimens by stereometric method after the samples reached the humidity of hygroscopic equilibrium with the climate-controlled chamber (20 ± 2 ° C and 65 ± 3% RH). The tests were performed in a universal testing machine in the different treatments, 10 samples being held for treatment.

Analysis of results:

To evaluate the mechanical resistance to compression parallel to the fiber for *Eucalyptus grandis*, *Pinustaeda* and *Tectonagrandis* subjected to thermal gradients, 10 replicates per treatment were used, resulting in a completely randomized design with factorial arrangement where was analyzed the effect of temperature of the Thermal modification of the method VAP HolzSysteme® in the mechanical properties of wood.

Statistical analysis was processed and it was applied the Tukey test at 5% significance level, for the factors and interaction identified as significant by the F test and as the need for homogeneity of variances of the average

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mechanical strength have been transformed into arcsen [square root (mass loss/100)] as recommended by Steel; Torrie (1988).

RESULTS AND DISCUSSIONS

The equilibrium of moisture content (Table 2) is reduced with temperature of thermal modification submitted to the woods (CHARANI *et al*., 2007, ESTEVES; PEREIRA, 2009; BATISTA; KLITZKE, 2010; MODES *et al*., 2013), as a fact occurring for the woods evaluated in this study (Table 2), where the wood of *E. grandis*, *P. taeda* and *T. grandis* exposed to temperatures of 160 °C had the lowest values of equilibrium of moisture content (8.19%; 7.55% and 5.34%), respectively. For the temperature of 140 °C and the control treatment for equilibrium moisture of the woods the results were close to 12%, except for the *Pinustaeda*.

Table 2: Average values of specific gravity at 12% of equilibrium moisture of woods.

Species	Treatment	Specific apparent gravity 12% ($g.cm^{-3}$)	Moistureequilibrium (%)
Eucalyptusgrandis	Control	0,52 (0,05)	11,26 (0,63)
	140	0,45 (0,03)	10,45 (1,29)
	160	0,47 (0,04)	7,73 (1,87)
Pinus taeda	Control	0,51 (0,04)	11,94 (1,25)
	140	0,38 (0,05)	9,17 (0,99)
	160	0,38 (0,05)	7,55 (0,39)
Tectonagrandis	Control	0,57 (0,01)	10,31 (1,02)
	140	0,51 (0,03)	10,10 (0,97)
	160	0,48 (0,01)	5,96 (0,80)

Values in parenthesis correspond to standard deviation.

Bodig; Jayne (1982), Haygreen; Bowyer (1996); Forest Products Laboratory - LPF (2010); Thiam *et al*. (2002); Oliveira (2007) reported that the decrease in moisture of the material positively influences its mechanical properties.

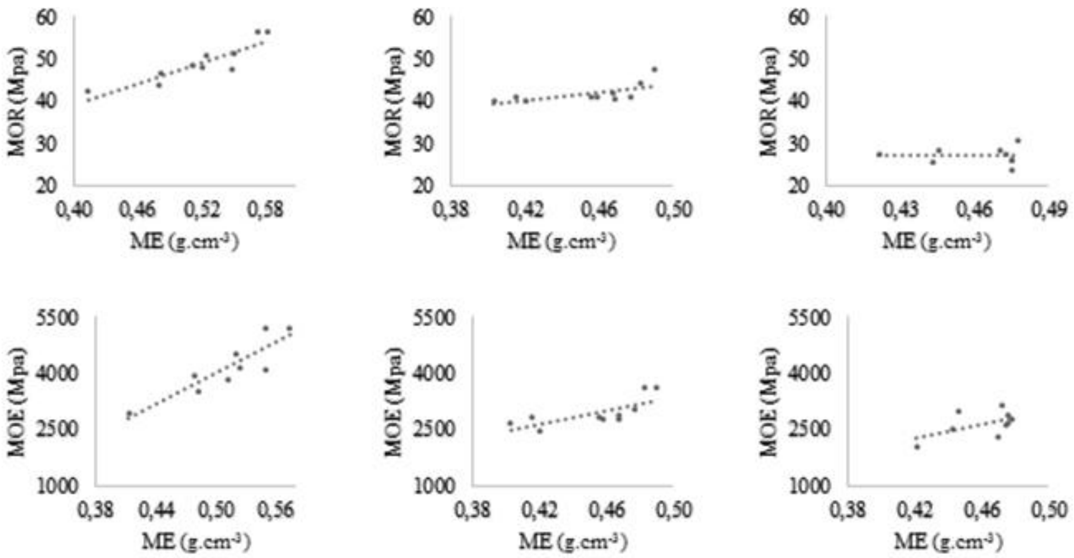
The wood of *Eucalyptus grandis*, *Pinustaeda* and *Tectonagrandis* showed apparent specific mass at 12% (0.52; 0.51 and 0.57 ($g.cm^{-3}$), respectively, for the samples that were not exposed to thermal modification (140 and 160 °C).

Evaluating the effect of thermal gradient (Table 3, Figure 3) the mechanical properties for *Eucalyptus grandis*,it is observed that there was no statistical difference between the temperatures evaluated (control, 140 and 160 °C) at 5% significance level. It is noted that there was a decrease of the mechanical resistance for both the Modulus of rupture - MOR (MPa) and to the modulus of elasticity - MOE (MPa) by increasing the exposure temperature, being the values 49.05 and 4258; 41.72 and 2936; 26.87 and 2614 respectively for the control wood and exposed to temperatures of 140 and 160 °C.

Table 3: Effect of temperature of thermal modification in modulus of rupture - MOR and modulus of elasticity - MOE for the woods of *E. grandis*, *T. grandis* and *P. taeda* to compression parallel to the fiber.

Species	MOR (MPa)			F
	Control	140	160	
Eucalyptus grandis	49,05 aA (9,54)*	41,72 aB (5,56)	26,87 bC (7,18)	26,57**
Pinustaeda	42,29 bA (10,65)	25,73 bB (22,41)	29,35 bB (15,87)	
Tectonagrandis	48,24 aA (8,26)	45,14 aA (5,76)	37,74 aB (8,35)	
	MOE (MPa)			
Eucalyptus grandis	4258 aA (18,07)	2936 bB (13,11)	2614 bB (20,53)	13,90**
Pinustaeda	3161 bA (20,93)	1836 cC (24,64)	2521 bB (19,97)	
Tectonagrandis	4588 aA (13,03)	3987 aB (8,88)	3560 aB (12,11)	

The average followed by the same uppercase letter (horizontal) and lowercase (vertical) do not differ significantly by the Tukey test ($p < 0.05$). **significant at 1%. *Values in parentheses correspond to the coefficient of variation (%).



Note: MOR: Modulus of Rupture. ME: apparent specific gravity at 12%.

Fig. 3: Ratio of modulus of rupture (MOR) and modulus of elasticity (MOE) as a function of apparent specific gravity at 12% for *Eucalyptus grandis* for the different temperatures of exposure (control, 140 and 160 °C).

It is observed that the linearly adjusted models (Table 4) to estimate the MOR and MOE, respectively for different exposure temperatures, as a function of apparent specific gravity at 12%, with the best correlation coefficients for both the MOR as MOE, were for the control treatments and as it increases the temperature of thermal modification, the correlation coefficients are not so significant, however, the standard error the estimation is low, concurrently, the adjusted models correspond to its purpose.

Table 4: Estimation of modulus of rupture (MOR) and modulus of elasticity (MOE) in MPa as a function of apparent specific gravity at 12% for *Eucalyptus grandis* for each temperature of thermal modification.

Species	Treatment	Adjusted Models	R_{aj}^2 (%)	S_{yx} (%)	F
Eucalyptus grandis	Control	MOR= 7,39943 + 80,5560* $ME_{apparent(12\%)}$	73,56	2,40	26,04**
	140 ºC	MOR = 21,246 + 45,0000* $ME_{apparent(12\%)}$	54,61	1,85	14,24*
	160 ºC	MOR= 27,7993 - 1,96270* $ME_{apparent(12\%)}$	32,45	2,04	8,99**
	Control	MOE = -2838,51 + 13720,90* $ME_{apparent(12\%)}$	79,90	3,45	36,78**
	140 ºC	MOE = -1006,44 + 8660,41* $ME_{apparent(12\%)}$	45,83	2,83	7,26**
	160 ºC	MOE = -944,127 + 7472,45* $ME_{apparent(12\%)}$	39,33	4,43	5,19*

Note: ME: apparent specific gravity at 12%; Raj2: adjusted coefficient of determination; Syx (%): standard error of the estimate; F: Significance factors; ** significant at 1%; * significant at 5%.

It is noted that Haygreen; Bowyer (1996); FPL (2010); Calonego (2009); Figueroa (2012);Loiola *et al*. (2012) and Batista (2013) evaluating the effect of temperature gradient on the mechanical properties of *Eucalyptus* have also found that thermal degradation of the chemical constituents of wood provided by the thermal gradient affects the mechanical strength properties of wood. But in certain exposure temperatures, the heat treatment did not affect the mechanical characteristics of the material, Severus; Tomaselli (2000) performing the heat treatment in logs and lumber of *Eucalyptus dunni* at 100 ºC of temperature concluded that the stiffness and resistance to compression parallel to the fiber were not changed.

For *Pinustaeda* (Table 3, Figure 4) there was statistical difference for the woods that were not exposed to the temperature gradient (control), with values of MOR and MOE (MPa) of 42.29; 3161 and the resistance values for the tested temperatures (140 and 160 °C) of 25.73; 1636 and 29.35; 2521 MPa, respectively. It is observed that the wood exposed to a temperature of 140 °C had lower resistance values than those exposed to a superior temperature (160 °C).

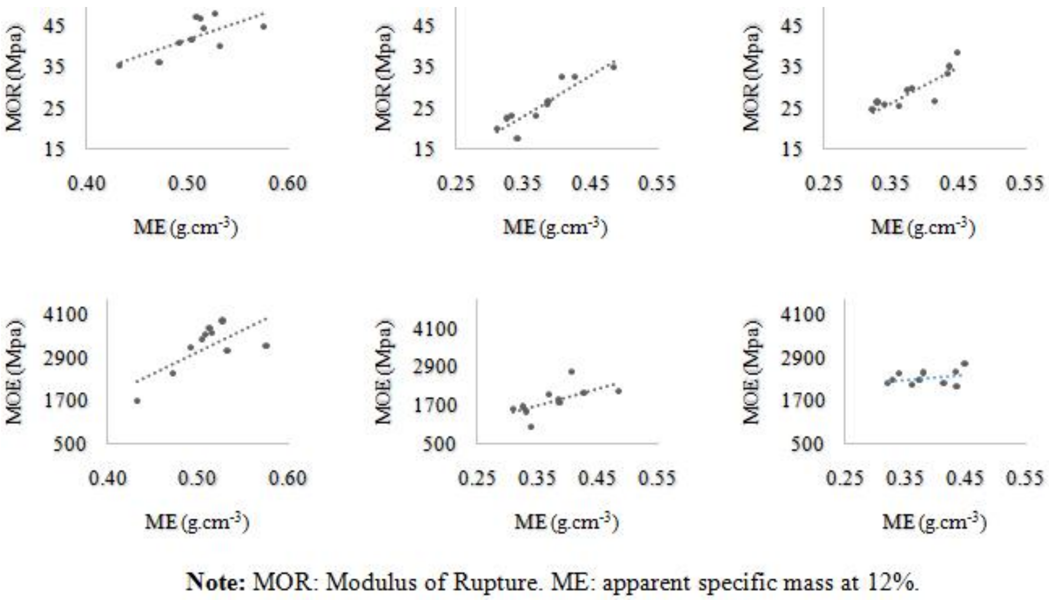


Fig. 4: Ratio of modulus of rupture (MOR) and modulus of elasticity (MOE) as a function of apparent specific mass at 12% for *Pinustaeda* for different exposure temperatures (control, 140 °C and 160 °C).

Linearly models were adjusted to estimate the MOR and MOE (Table 5), respectively for different exposure temperatures, as a function of apparent specific mass at 12%, with the best correlation coefficients for both the MOR and the MOE, for the control treatments, however, the correlation coefficients are not so significant, but the standard error of the estimate is low, concurrently, the adjusted models correspond to its purpose.

Table 5: Estimation of modulus of rupture (MOR) and modulus of elasticity (MOE) in MPa as a function of apparent specific mass at 12% for *Pinustaeda* for each temperature of thermal modification.

Species	Treatment	Adjusted Models	R_{aj}^2 (%)	S_{yx} (%)	F
Pinus taeda	Control	MOR= 2,26483 + 78,9550*$ME_{apparent(12\%)}$	48,48	3,43	7,53*
	140 ºC	MOR = -10,3029 + 95,0711* $ME_{apparent(12\%)}$	80,30	2,56	37,68**
	160 ºC	MOR= -3,80956 + 86,5837* $ME_{apparent(12\%)}$	73,95	2,38	26,54**
	Control	MOE = -2733,54 + 11621,90* $ME_{apparent(12\%)}$	48,77	5,01	7,62*
	140 ºC	MOE = -172,152 + 5296,27* $ME_{apparent(12\%)}$	41,58	3,67	5,69*
	160 ºC	MOE = -402,337 + 7629,53* $ME_{apparent(12\%)}$	40,03	3,73	8,37*

Note: ME: apparent specific gravity at 12%; Raj2: adjusted coefficient of determination; Syx (%): standard error of the estimate; F: Significance factors; **significant at 1%; *significant at 5%.

This fact can be explained as a function of position on the trunk of trees, as well as by the thermal rectification process used. As an industrial process was used, thus, with high volume production, not always is obtained control of all process variables. However, this result cannot be considered as mismatches occurred during the experimental process, Boonstra (2007) evaluating the *Pinussylvestris*, obtained values of resistance to compression parallel to the fiber exposed to a temperature of 180 °C higher than the control wood . Loiola *et al*. (2012) in studies with the wood of *Corymbiacitriodora* also obtained higher resistance values than their control samples resistance, and this author, exposing to temperatures of 200°C. The scientific basis for these results is the approach of the structural elements of the cellular wall and between anatomical components due to shrinkage of wood with the rise of temperature and moisture reduction of fibers, Schaffer (1973); Figueroa; Moraes (2009) reported that the wood exposed to a temperature of 160 °C, lignin begins the solidification process after the process in which softening starts at a temperature of 110 °C.

According to Figueroa and Moraes (2009), the influence of temperature on the wood polymers, especially about its softening characterized by the glass transition temperature, which is the passage of a rigid disordered state (glassy) polymer to further disordered state, giving the polymer chains for greater mobility. This mobility in the case of certain classes of natural polymers such as hemicellulose, cellulose and lignin, leads to a less rigid behavior. The hemicellulose and lignin are amorphous and essentially thermoplastic polymers for which the main point is the softening point of the glass transition. Glass transitions are connected to wood temperature, moisture content, molecular weight of the polymer, the nature of the material, in isolated form or shape of wood and in crystalline or amorphous form.

Analyzing the behavior of the wood of *Tectonagrandis* (Table 2, Figure 5) exposed to the thermal gradient, it is observed that the wood exposed to temperatures of 140 °C, obtained values of MOR and MOE (48.24; 4588

MPa, respectively) higher than the control samples (45.14, 3987 MPa, respectively), which at 5% significance level there was no statistical difference by Tukey test for the MOE, as well as for the MOR. For a temperature of 160 °C it was expected to decrease the mechanical resistance of the wood of *T. grandis*, with MOR and MOE (MPa) values of 37.74 and 3560 respectively, and this temperature was statistically equal to the exposure level of 5% significance for the other temperatures evaluated.

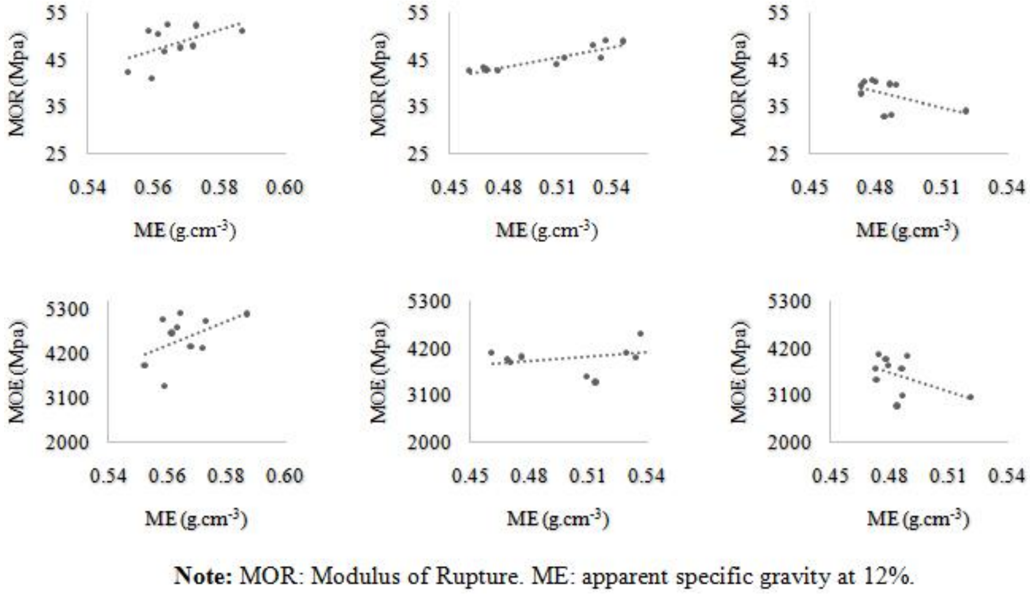


Fig. 5: Ratio of modulus of rupture (MOR) and modulus of elasticity (MOE) as a function of apparent specific mass at 12% for wood *Tectonagrandis* for different exposure temperatures (control, 140 and 160 °C).

It is observed that the adjusted models (Table 6) to the wood of *Tectonagrandis*, to estimate the MOR and MOE, respectively for different exposure temperatures, as a function of apparent specific gravity at 12%, were not satisfactory as the coefficients correlation, as well as the F Fisher, were low although significant.

Table 6: Estimation of modulus of rupture (MOR) and modulus of elasticity (MOE) in MPa as a function of apparent specific gravity at 12% for wood *Tectonagrandis* for each temperature of thermal modification.

Species	Treatment	Adjusted Models	R_{aj}^2 (%)	S_{yx} (%)	F
Tectonagrandis	Control	MOR= -44,8286 + 164,7140* $ME_{apparent(12\%)}$	19,97	3,78	$2,00^*$
	140 ºC	MOR = 8,49256 + 72,5751* $ME_{apparent(12\%)}$	81,48	1,19	$40,60^{**}$
	160 ºC	MOR= 84,3898 - 96,3922* $ME_{apparent(12\%)}$	21,24	2,96	$2,16^{**}$
	Control	MOE = -2838,51 + 13720,90* $ME_{apparent(12\%)}$	16,54	5,79	$1,59^*$
	140 ºC	MOE = -8134,78 + 22519,80* $ME_{apparent(12\%)}$	15,48	3,45	$1,47^*$
	160 ºC	MOE = 9342,67 - 11946,30* $ME_{apparent(12\%)}$	17,41	4,15	$1,69^*$

Note: ME: apparent specific gravity at 12%; Raj2: adjusted coefficient of determination; Syx (%): standard error of the estimate; F: Significance factors; **significant at 1%; *significant at 5%.

The woods that were not exposed to thermal treatments showed values of 49.05 MOR; 42.29 and 45.14 (*E. grandis*, *P. taeda* and *T. grandis*) respectively, in which only the wood of P. taeda differ statistically at 5% significance level for the other species.

Resistance values found for the wood of E. grandis in this study were higher than those found by Calonego (2009); Modes *et al*. (2013); Instituto de Pesquisa Tecnológica - IPT (2010); Batista (2013), while Serpa *et al*. (2003); Hein *et al*. (2009); Lima; Garcia (2011); found values of superior strength. For the wood of P. taeda resistance values found were higher than those described by Oliveira *et al*. (2006), Figueroa (2012). For the wood T. grandis resistance values encountered by FPL (2010); IPT (2010);Mota (2011) were higher than the values found in this work.

Conclusions:

The thermal rectification gives the wood of *E. grandis*, *P. taeda* and *T. grandis* lower equilibrium moisture content being the wood from *T. grandis* exposed to a temperature of 160 °C the wood that reached the lower equilibrium moisture.

The wood exposed to thermal gradients, decreased resistance to compression parallel to the fiber, being the wood of *E. grandis* evaluated as the species that had the highest loss resistance and the wood of *T. grandis* showed the smallest loss of its resistance to compression parallel the fibers, which can be justified due to the high extractives content presented by this species.

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