

## THE EFFECT OF THERMAL TREATMENT ON PHYSICAL AND MECHANICAL PROPERTIES OF *Luehea divaricata* HARDWOOD

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### ABSTRACT

This study aimed to characterise the effect of thermal treatment on physical and mechanical properties of açoita-cavalo (*Luehea divaricata*) wood. To achieve this, samples were treated at 160, 180 and 200°C for 2 h in an oven (dry conditions). Physical and mechanical characterisation was performed by weight loss, dry specific gravity at 12%, equilibrium moisture content, volumetric swelling and shrinkage, water absorption, water repellence, static bending and brittleness tests. Roughness and colour tests were performed in order to evaluate the modified surface. Main findings showed that dimensional stability of thermally modified wood increased, while mechanical strength of wood decreased as a function of temperature. A darkening of wood and a decrease in roughness were observed.

**Keywords:** Brittleness, dimensional stability, modulus of elasticity, modulus of rupture, roughness.

### INTRODUCTION

Currently, wood from native species in Brazil does not have the same importance as raw material for timber products due to the increase of fast-growing forests over the last years. Even so, wood presents limitations when in service because it is highly hygroscopic and anisotropic.

Açoita-cavalo (*Luehea divaricata*) is present in Brazil from the states of Minas Gerais and Rio de Janeiro to Rio Grande do Sul, as well as in other countries: Argentina, Uruguay and Paraguay. Açoita-cavalo wood has many uses, such as for furniture production, slats, moulding and skirting. However, wood from açoita-cavalo cannot be exposed to environments with a high level of biological degradation because it has low natural durability (Rizzini 1995).

Thermal modification was developed as an alternative to traditional wood treatments such as impregnation with chemical products (toxic products) that aims to improve wood properties. Thermal treatment is a process of modification of different materials at high temperatures, which can be performed between 180 and 260°C (Hill 2006). Nevertheless, thermal treatments also could be performed in lower temperatures (140 and 160°C, for example) as observed in previous studies (Pincelli 2012, Bal 2013).

Thermal treatment causes changes on wood, mainly related to their physical and mechanical properties. Amongst physical properties, emphasis is on weight loss, increase of dimensional stability and colour changes. Sundqvist *et al.* (2006) reported that weight loss of white birch wood treated at 160-200°C for 1-4 hours was directly related to the temperature of treatment. Colour changes on wood occur due to the modification of hemicelluloses structure, migration of extractives to surface and formation of oxidative products. Changes in chemical compounds induce darkening of thermally modified wood (Bekhta and Niemz 2003, Esteves *et al.* 2008, Akyildiz *et al.* 2009).

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Received: 20.02.2013 Accepted: 18.12.2013

However, mechanical properties are often negatively affected by thermal treatments. Bekhta and Niemz (2003) observed a decrease between 4 and 9% of modulus of elasticity at static bending in Norway spruce wood thermally modified at 200°C for 2 and 24 hours. Likewise, Militz and Tjeerdsma (2001) affirmed that flexural strength of *Fagus sylvatica* (European beech), *Betula pendula* (silver birch), *Pseudotsuga menziesii* (Douglas-fir), *Pinus sylvestris* (Scots pine) and *Pinus radiata* (Radiata pine) decreased between 5 and 18% when treated by “Plato-Process®”.

Considering aforementioned characteristics of thermal treatments and wood from *Luehea divaricata* specie, thermal modification could be a great alternative to improve their physical properties and to increase their aesthetic uses, since that açoita-cavalo wood is widely used for furniture production and other non-structural products, mainly in South American market.

In this context, this study aimed to evaluate physical and mechanical properties of açoita-cavalo (*Luehea divaricata*) thermally modified wood. Physical properties were characterised using several tests related to weight loss, dimensional stability, surface roughness and colour changes, while mechanical properties were evaluated using static bending tests to characterise stiffness, strength and brittleness of wood.

## MATERIAL AND METHODS

Ten açoita-cavalo (*Luehea divaricata* Mart. et Zucc Mart.) trees were randomly selected and harvested (licensing process by the State Ministry of Environment) from two regions located in Southern Brazil (29°14'30.91”S, 52°18'47.65”O), namely Central Depression and Upper Northeast Slopes.

The first log measuring 2500 mm length of each tree was cut in order to obtain 80 mm thick boards. Fifty-two samples measuring 25 x 25 x 410 mm (radial x tangential x longitudinal) were prepared, all of them flawless and with straight grain orientations.

Thermal treatments were performed in an electric oven without forced air circulation and heating rate of 2°C/min. Samples were treated at 160, 180 and 200°C for 2 h. After the thermal treatments, the samples were put in a desiccator until to reach room temperature. Control samples (untreated) were kept in a climate chamber at 25°C and 65% of relative humidity until to perform the physical and mechanical tests.

### Physical properties

Weight loss was measured by the difference of dry weight before and after thermal treatments (Equation 1). Dry specific gravity at 12% of moisture content was measured by equation 2.

$$WL = \frac{M_{BH} - M_{AH}}{M_{BH}} \times 100 \quad (1)$$

$$\rho_{12\%} = \frac{M_0}{V_{12\%}} \times 100 \quad (2)$$

WL = weight loss (%);  $M_{BH}$  = dry weight of samples before thermal treatment (g);  $M_{AH}$  = dry weight of samples after thermal treatment (g);  $\rho_{12\%}$  = dry specific gravity at 12% (g/cm<sup>3</sup>);  $M_0$  = dry weight of samples (g);  $V_{12\%}$  = volume at 12% of samples (cm<sup>3</sup>).

Samples measuring 25 x 25 x 50 mm (radial x tangential x longitudinal) were cut from original samples in order to evaluate dimensional stability after fully saturation. Volumetric swelling, volumetric shrinkage, water absorption and water repellence were measured by equations 3, 4, 5 and 6.

$$\alpha_v = \left( \frac{V_u - V_o}{V_o} \right) \times 100 \quad (3)$$

$$\beta_v = \left( \frac{V_u - V_o}{V_u} \right) \times 100 \quad (4)$$

$$WA = \left( \frac{M_u - M_s}{M_s} \right) \times 100 \quad (5)$$

$$WRE = \left( \frac{TR_{H_2O} - TT_{H_2O}}{TR_{H_2O}} \right) \times 100 \quad (6)$$

$\alpha_v$ = volumetric swelling (%);  $\beta_v$ = volumetric shrinkage (%);  $V_u$ = saturated volume of samples ( $\text{cm}^3$ );  $V_o$ = volume of dried samples ( $\text{cm}^3$ );  $WA$ = water absorption (%);  $M_u$ = saturated weight of samples;  $M_s$ = dry weight of samples;  $WRE$ = water repellence (%);  $TR_{H_2O}$ = mean water absorption of untreated samples (%);  $TT_{H_2O}$ = mean water absorption of treated samples (%).

### Mechanical properties

Mechanical tests were performed in an universal machine (EMIC) with capacity of 300 kN. Static bending tests were performed in a three-point bending apparatus with a span length of 360 mm and a loading speed of 1,30 mm/min, according to the American Society for Testing and Materials - ASTM D 143-94 (ASTM 2000).

After the mechanical tests, the load-deflection curves were analysed in order to estimate the brittleness of untreated and thermally treated wood according to the methodology described by Phuong *et al.* (2007).

### Colorimetric evaluation

Changes on colour of thermally modified wood were performed by a colorimeter (Konica Minolta CR-400). The equipment was configured to use a D65 illuminant and 10° standard observer (*CIE-Lab standard*).

Two measurements for each sample in both radial and tangential section were performed in order to obtain the parameters  $L^*$  (lightness),  $a^*$  (red-green chromatic coordinate) and  $b^*$  (yellow-blue chromatic coordinate).

$\Delta E$  (colour difference),  $C^*$  (saturation) and  $h$  (hue angle) were measured by equations 7, 8 and 9.

$$\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (7)$$

$$C^* = (a^{*2} + b^{*2})^{1/2} \quad (8)$$

$$h = \text{tang}^{-1}(b^*/a^*) \quad (9)$$

$\Delta E$ = colour difference;  $\Delta L^{*2}$ ,  $\Delta a^{*2}$  and  $\Delta b^{*2}$ = variation of lightness, red-green chromatic coordinate and blue-yellow chromatic coordinate between untreated and treated samples;  $C^*$ = saturation;  $h$ = hue angle;  $a^*$ = red-green chromatic coordinate;  $b^*$ = yellow-blue chromatic coordinate.

### Surface roughness evaluation

Surface roughness was carried out by a profilometer based on stylus device standard (Homis model 899), which can detect variations on height of wood surface using a probe in a predetermined distance. The profilometer was configured with a cut-off length of 2,5 mm, tracing line of 5 mm, pin diameter of 5 mm and pin top angle of 90°.

Measurements were performed in three distinct points of surface samples (perpendicular to the fibres) in radial section of each treatment in order to obtain four parameters, Ra (mean arithmetic deviation of profile), Rz (mean peak to-valley height), Rq (root mean square roughness) and Rt (maximum roughness).

### Statistical analysis

The collected data was analysed using descriptive statistic and analysis of variance ( $p < 0.05$ ). When the null hypothesis was rejected, the average values were compared with Tukey Test at 5% level of probability of error.

## RESULTS AND DISCUSSION

Mean values of  $\rho_{12\%}$  did not show statistical difference (Table 1), however a slight reduction as a function of treatment temperature was observed. On the other hand, EMC significant varied in relation to the control treatment (untreated samples). Mean values of EMC (Table 1) vary between 6 and 7%, in which the highest reduction was found in the treatment at 200°C (~35%). Günduz and Aydemir (2009) also found decrease of EMC in *Carpinus betulus* wood after thermal treatment at 170-210°C for 4-12h.

**Table 1.** Mean values of dry specific gravity at 12% ( $\rho_{12\%}$ ), equilibrium moisture content (EMC) and weight loss (WL) of *Luehea divaricata* (açoita-cavalo) untreated and thermally treated.

Treatment (°C)	$\rho_{12\%}$ (g/cm <sup>3</sup> )	EMC 65% (%)	WL (%)
Control (20)	0,61 (0,03) a	10,28 (0,55) a	-
160	0,59 (0,03) a	7,45 (1,53) b	1,09 a (17,36)
180	0,59 (0,04) a	7,19 (0,72) b	1,81 a (11,70)
200	0,57 (0,04) a	6,63 (0,46) b	5,49 b (28,08)
ANOVA	F = 1,85 p = 0,1513	F = 41,12 ** p < 0,01	F = 60,81 ** p < 0,01

Mean values in the same column followed by the same letter are not statistically different at level of 5% by the Tukey Test; p= probability; F= calculated value of F; \*\* Significant at 5% of probability of error; Value between parenthesis corresponding to coefficient of variation (%).

WL was directly proportional to the temperature of treatment (Table 1). However, significant weight loss was observed only at 200°C. According to Sundqvist *et al.* (2006), hemicelluloses presents great changes in its chemical structure when treated at 180-200°C for 2,5 and 4 h, which generating significant weight loss of wood.

The increase of dimensional stability of wood is one of the most important changes due to thermal treatments. The behaviour of the parameters related to the dimensional stability of untreated and thermally treated samples is verified in table 2.

**Table 2.** Mean values of volumetric swelling ( $\alpha_v$ ), volumetric shrinkage ( $\beta_v$ ), water absorption (WA) and water repellence (WRE) of *Luehea divaricata* (açoita-cavalo) untreated and thermally treated.

Treatment (°C)	$\alpha_v$ (%)	$\beta_v$ (%)	WA (%)	WRE (%)
Control (20)	12,55 a (0,99)	11,45 a (1,63)	137,28 a (11,18)	-
160	12,11 a (1,56)	10,61 a (1,25)	134,91 a (12,49)	1,72
180	11,59 a (1,05)	10,46 a (0,82)	136,36 a (6,18)	0,67
200	10,12 b (1,10)	9,15 b (0,91)	129,49 a (8,66)	5,67
ANOVA	F = 7,78** p < 0,0001	F = 6,96** p < 0,0001	F = 0,31 p = 0,82	- -

Statistical prerequisites as in Table 1.

$\alpha_v$  and  $\beta_v$  showed similar behaviour, in which both parameters decreased as a function of temperature of treatment. This reduction was significant only at 200°C.

Thermally modified wood largely increases its dimensional stability and reduced its hygroscopicity due to the degradation of hemicelluloses, polymer reticulation and breaking of hydroxyl groups from amorphous zone of cellulose (Weiland and Guyonnet 2003, Rousset *et al.* 2004, Wikberg and Maunu 2004).

On the other hand, WA did not present significant changes after thermal treatments. Nevertheless, a tendency of reduction as a function of treatment temperature was observed. The behaviour of WRE confirmed a significant reduction of hygroscopicity of wood at 200°C.

Differently from properties of dimensional stability, significant changes in the mechanical properties (Table 3) of thermally modified wood are negative.

**Table 3.** Mean values of modulus of elasticity (MOE) and modulus of rupture (MOR) of *Luehea divaricata* (açoita-cavalo) untreated and thermally treated wood.

Treatment (°C)	MOE (MPa)	MOR (MPa)
Control (20)	8462,29 a (1766,00)	81,77 a (10,39)
160	9022,82 a (1508,27)	87,50 a (6,67)
180	8443,37 a (1077,65)	83,79 a (9,05)
200	8940,47 a (1079,42)	63,04 b (13,01)
ANOVA	F= 0,64 p = 0,5956	F= 14,39** p > 0,0001

Statistical prerequisites as in Table 1.

Açoita-cavalo wood was susceptible to a significant reduction of MOR at 200°C. MOR decreased ~23%. Likewise, a significant and negative correlation between temperature of treatment and MOR ( $r = -0,70$ ) was observed.

Moura *et al.* 2012 reported that MOR of rose gum thermally modified wood presented a negative and significant correlation ( $r = -0,56$ ) with the temperature of treatment, while MOR of Caribbean pinewood was not affected. The authors stated that MOR of rose gum wood was affected after thermal treatments at 180°C.

Winandy and Lebow (2001) obtained a coefficient of determination of 0,75 for estimating mechanical strength loss of thermally modified wood. Strength loss occurred due to the degradation of carbohydrates during thermal treatments. Treatments between 200 and 260°C can cause significant degradation in hemicelluloses content of wood, which release a great content of acetic acid (Weiland *et al.* 1998).

MOE did not present a statistical variation as a function of temperature of treatment. Nevertheless, stiffness of açoita-cavalo thermally modified wood showed a tendency to increase with increasing temperature of treatment. Mean values of control and thermally treated samples hold between 8000 and 9000 MPa.

Similar results were found by Calonego *et al.* (2012), who did not observe a significant variation of MOE of rose gum thermally modified wood after tests of static bending and compression parallel to fibres. Bhuiyan *et al.* (2000) reported that non-modification of MOE is related to the increase of cellulose crystallisation and thickness of crystallites.

The thermal treatments significantly changed the brittleness of açoita-cavalo wood (Table 4). However, only exposure at 200°C was able to change significantly the brittleness, i.e, thermal treatments at 140 and 160°C did not significantly modify this property when compared to the control treatment.

**Table 4.** Brittleness changes of *Luehea divaricata* (açoita-cavalo) untreated and thermally treated wood.

Treatment (°C)	Brittleness (%)	CV (%)
Control (20)	16,87 a	6,65
160	22,21 a	30,33
180	23,67 a	19,11
200	59,49 b	20,51
ANOVA	F = 72,46** p < 0,0001	

Statistical prerequisites as in Table 1.

The highest decrease of brittleness was 71,64% for the treatment at 200°C. This behaviour was expected because high temperatures tend to significantly reduce the mechanical strength of wood. Moreover, a Pearson correlation presented a high correlation of brittleness with WL (0,86), MOR (-0,87) and *L\** (-0,86 in radial and -0,81 in tangential). Phuong *et al.* (2007) observed a similar behaviour in *Styrax tonkinensis* thermally modified wood at 160-200°C.

Thermal treatments present wide utilisation due to the possibility of colour modifications of wood, mainly for aesthetic uses. Visual analysis confirms absence of perceptible darkening (unaided eye) between control treatment and treatment at 160 and 180°C. A significant darkening (unaided eye) in both radial and tangential sections is observed only at 200°C (Table 5).

**Table 5.** Colour changes in *Luehea divaricata* (açoita-cavalo) untreated and thermally treated wood.

	T (°C)	<i>L</i> *	<i>a</i> *	<i>b</i> *	<i>C</i> *	<i>h</i> (°)	$\Delta E$
Radial	Control	54,80 a (6,95)	4,40 ab (17,26)	17,98 a (9,73)	18,52 a (9,81)	76,25 a (2,32)	-
	160	54,27 a (9,20)	4,78 a (14,50)	18,96 a (9,56)	19,95 a (9,76)	75,77 a (2,39)	1,18
	180	53,01 a (5,22)	4,21 b (11,37)	18,73 a (3,52)	19,41 a (7,35)	76,93 a (1,49)	1,95
	200	41,06 b (13,69)	6,60 c (8,07)	17,63 a (17,44)	18,97 a (15,45)	68,23 b (5,95)	13,92
	ANOVA	F = 52,31** p < 0,0001	F = 63,57** p < 0,0001	F = 1,87 p = 0,14	F = 1,88 p = 0,1396	F = 59,89** p < 0,0001	- -
Tangential	Control	51,93 a (9,79)	5,15 a (34,06)	18,89 a (15,17)	19,74 a (17,04)	74,09 a (5,06)	-
	160	53,74 a (7,53)	4,96 a (18,71)	19,23 a (7,20)	19,89 a (7,66)	75,50 a (2,39)	1,85
	180	50,48 a (8,43)	4,60 a (19,23)	18,70 a (3,24)	19,70 a (5,86)	76,03 a (2,72)	1,56
	200	39,57 b (15,90)	6,70 b (3,88)	17,30 a (19,33)	18,71 a (17,00)	67,38 b (5,06)	12,56
	ANOVA	F = 39,01** p < 0,0001	F = 11,70** p < 0,0001	F = 2,73 p = 0,05	F = 0,97 p = 0,41	F = 35,13** p < 0,0001	- -

Statistical prerequisites as in Table 1.

The parameter  $L^*$  was inversely proportional to the temperature of treatment in both anatomical sections. However,  $L^*$  significantly decreased only for treatment at 200°C, which was ~25% in radial section and ~24% in tangential section. According to Bekhta and Niemz (2003), thermal treatments cause darkening of wood mainly in the first 4 hours of treatment and increase at 200°C or higher.

A slight increase of  $a^*$  as a function of the temperature of treatment in both anatomical sections was observed. On the other hand,  $b^*$  values remained between 17 and 19 and not showed a tendency as a function of temperature of treatment.

Naturally, açoita-cavalo wood presents  $C^*$  referred to grey tones (relationship  $L^* \times C^*$ ). Despite thermal modification of wood, parameters  $b^*$  and  $C^*$  were constant as a function of temperature of treatment, i.e., thermally modified wood did not increase their grey tones (decrease of  $C^*$ ) or vivacity (increase of  $C^*$ ).

The parameter  $h$  showed slight changes only at 200°C, representing small loss of yellow colour (10% in radial section and 8% in tangential section) and increase of red colour.

The parameter  $\Delta E$  was inversely proportional to the temperature of treatment and confirmed occurrence of changes on colour of açoita-cavalo thermally modified wood. Values of  $\Delta E$  higher than 2-3 indicate that the human eye is able to distinguish the colour of materials (Hon and Minemura 1991, Sundqvist 2002).

All parameters of surface roughness of treated samples decreased significantly when compared to untreated samples (Table 6). The highest reduction of  $R_a$  was 25,62% for treatment at 200°C. However, mean values of surface roughness of wood thermally treated at 160-200°C did not show statistical difference, in which treatment at 160°C was sufficient to modify significantly the surface roughness of açoita-cavalo wood.

**Table 6.** Surface roughness changes in radial direction of *Luehea divaricata* (açoita-cavalo) untreated and thermally treated wood.

Treatment (°C)	Ra (µm)	Rz (µm)	Rq (µm)	Rt (µm)
Control (20)	10,77 a (15,94)	30,47 a (15,97)	11,61 a (15,69)	30,77 a (15,97)
160	8,43 b (24,66)	23,82 b (24,68)	8,98 b (24,80)	24,05 b (24,68)
180	8,98 b (25,67)	25,41 b (25,68)	9,51 b (25,11)	25,36 b (26,47)
200	8,01 b (11,84)	22,16 b (14,24)	8,21 b (14,66)	22,38 b (14,23)
ANOVA	F = 7,00** p = 0,004	F = 7,63** p = 0,0002	F = 9,02** p = 0,0001	F = 7,66** p = 0,0002

Mean values in the same column followed by the same letter are not statistically different at level of 5% by the Tukey Test; p= probability; F= calculated value of F; \*\* Significant at 5% of probability of error; Value between parenthesis corresponding to coefficient of variation (%).

These results presented similar behaviour of other studies (Gündüz *et al.* 2008, Korkut and Guller 2008) and confirmed the increase of quality of wood surface. According to Gündüz *et al.* (2008), decrease of roughness is important for many applications of solid wood because can reduce the losses in planning machine and can increase the quality of wood surface.

## CONCLUSIONS

This study investigated the changes on physical and mechanical properties of *Luehea divaricata* (açoita-cavalo) wood thermally modified at 160-200°C for 2 hours. In general, physical and mechanical properties of açoita-cavalo thermally modified wood showed significant modifications at 200°C. Dimensional stability increased and equilibrium moisture content decreased as a function of temperature. Mechanical strength of thermally modified wood significantly decreased and, consequently, brittleness increased at 200°C. Wood colour was influenced by the reduction of lightness ( $L^*$ ) and decrease of surface roughness was observed, which enable new practical uses of açoita-cavalo wood when aesthetic factors are considered.

## ACKNOWLEDGMENTS

The authors wish to thank CNPq (National Counsel of Technological and Scientific Development) and CAPES (Coordination for the Improvement of Higher Level Personnel) for the financial support.



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