Physical and Mechanical Properties of Fast-Growing Wood Subjected to Freeze-Heat Treatments

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The physical and mechanical performance of wood from the rose gum (*Eucalyptus grandis*) and the Gympie messmate (*Eucalyptus cloeziana*) species were investigated. The wood samples were treated with a two-stage freeze-heat thermal process. Fast-growing trees were used for preparing test samples, which were subjected to thermal treatments. The freezing stage had the treatment temperature fixed at -22 °C for 72 h, while the temperature of the heat stage ranged from 180 to 200 °C for 3.5 h. The measurements of mass loss, density, and equilibrium moisture content were determined to better understand the mechanical properties. Static bending, compression parallel to grain, Janka hardness, and impact tests were applied to reveal changes in the mechanical behavior of the treated wood. In general, the freezing stage decreased the mass loss and increased the moisture content of wood (when combined with the heating stage), which showed the opposite trend for the heating stage. Modulus of elasticity and compression strength were increased only after the heating stage, while decrements were found for modulus of rupture, impact strength, and Janka hardness. The two-stage treatments did not prevent a decrease in the mechanical properties; however, they were helpful in preventing higher mechanical resistance losses in hardness (the Gympie messmate) and impact resistance (the rose gum).

Keywords: Combined treatment; Wood freezing; Heat treatment; Wood technology

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INTRODUCTION

Wood is commonly used worldwide, but it has some limitations due to its intrinsic characteristics. Gradually, the use of raw materials from fast-growing forest plantations is increasing, especially for species from the *Eucalyptus* genus. According to ABRAF (2013), Brazilian forest plantations were 6.6 million ha in 2012, of which *Eucalyptus* species composed 76% (5.10 million ha).

Some *Eucalyptus* species, such as *Eucalyptus grandis* and *Eucalyptus cloeziana* are disadvantageous as a raw material due to low dimensional stability, high hygroscopicity, and high drying times. Furthermore, wood from *Eucalyptus* species can present different degrees of decay resistance (Delucis *et al*. 2016). Consequently, they have low value added in the market for purposes that need high dimensional stability. Thus, enhancing the added

value of these renewable materials becomes a market strategy, especially by using eco-friendly treatments with low environmental impacts.

Various thermal treatments are applied to wood to improve the biological and hygroscopic properties. However, the results from these works do not corroborate each other. In general, the mechanical properties of the treated wood do not follow any pattern, as they can increase (Boonstra *et al.* 2007; Pfriem *et al.* 2010; Todorovic *et al.* 2012), decrease (Cademartori *et al.* 2012, 2014, 2015; Bal and Bektaş 2013; Missio *et al.* 2016) or remain unchanged (Calonego *et al.* 2012; Cademartori *et al.* 2015). The effect of thermal treatments in these properties depends on the parameters of the treatment and the wood species (Hill 2006).

Thermal treatments at high temperatures can lead to permanent changes in the chemical composition of wood (Missio *et al.* 2015). Hemicelluloses are degraded at 160 °C, especially because of their low molecular weight (Fengel and Wegener 2003). The absence of hemicelluloses in the lignin/cellulose interface considerably changes the mechanical behavior of wood (Esteves and Pereira 2009; Gunduz *et al.* 2009). The crystalline fraction of the cellulose in the cell wall increases after the heat treatment because of the degradation and/or crystallization of the amorphous cellulose, which indirectly increases the axial compression strength of the wood cell wall (Boonstra *et al.* 2007). Furthermore, the high temperatures may create new crosslinked bonds between lignin moieties with improved mechanical resistance (Boonstra *et al.* 2007). According to the same authors, these two specific modifications in the wood microstructure are responsible for the decrease of their elastic characteristics.

On the other hand, wood freezing has been used to reduce the shrinkage and the wood drying defects (Ilic 1995; Awoyemi 2006), to increase the diffusion or the permeability of wood (Glossop 1994; Ilic 1995), to reduce the wood collapse (Chen and Cooper 1974; Ilic 1995) and reduce wood cracks (Chen and Cooper 1974; Ilic 1999). However, this technique has not been fully explored.

As an alternative, combined treatments have been used to change wood properties while avoiding extensive side effects. Such treatments include a two-stage impregnation and heat treatment (Perçin *et al.* 2015; Lahtela and Kärki 2016); magnetization followed by furfurylation (Dong *et al.* 2016); boron impregnation and heat treatment (Kartal *et al.* 2008); densification followed by oil heat treatment (Fang *et al.* 2011, 2012), as well as freezing and heating treatment (Awoyemi *et al.* 2010; Missio *et al.* 2015, 2016).

Interesting results were observed after treating tropical wood with a combination of freezing and heating (Awoyemi *et al.* 2010; Missio *et al.* 2015). The two-stage freeze-heat treatment was more efficient than just the heat treatment for water absorption, water repellence, and maximum impact strength (Missio *et al.* 2016). These results were attributed to a selective and partial degradation of hemicelluloses, which can be a good alternative to change the physical and mechanical properties of eucalyptus wood from fast-growth plantations. Thus, the use of combined treatments could reduce the effect caused by the high temperatures and make the loss of water easier due to the increase of wood porosity by the freezing.

This study investigated the influence of two-stage freeze-heat treatments in physical and mechanical properties of the rose gum (*Eucalyptus grandis* Hill ex Maiden) and the Gympie messmate (*Eucalyptus cloeziana* F. Muell.) wood.

EXPERIMENTAL

Materials

Twelve 21-year-old rose gum (*Eucalyptus grandis* Hill ex Maiden) and Gympie messmate (*Eucalyptus cloeziana* F. Muell.) trees were randomly selected from an experimental forest located in Southern Brazil (29°43'0.39"S, 53°43'46.03"N), according to the ASTM D5536-94 (2010). As described in the ASTM D143-94 (2000), NBR 7190 (ABNT 1997) standard and other studies (Cademartori *et al.* 2014, 2015; Missio *et al.* 2016), the size of the samples were: 2.0 x 2.0 x 35.0 cm^3 (radial, tangential and longitudinal direction) for static bending; 2.5 x 2.5 x 10 cm^3 for compression parallel to grain and physical tests; 3.0 x 3.0 x 10 cm^3 for Janka hardness; and 2.0 x 2.0 x 30 cm^3 for impact resistance. All the samples were prepared from the first log (3 m in length from the base of the trees), thus avoiding defects and the presence sapwood in the samples.

Methods

Two-stage freeze-heat treatments

The sample treatments were carried out on the basis of previously published research methods (Missio *et al.* 2015). The wood samples subjected to the freezing stage were first immersed in water to attain saturated moisture conditions, while the samples not subjected to the freezing stage were kept in a climatic chamber (20 °C and 65% of relative humidity). Then, five combined treatments were performed, as well as a control treatment (Table 1).

Table 1. Thermal Treatments Performed on the Rose Gum and the Gympie Messmate Woods

	Freezing stage*			Heating stage**		
Treatment	Natural condition	Temperature (°C)	Time (h)	Natural condition	Temperature (°C)	Time (h)
Control	-	-	-	-	-	-
WF	Wet	-22 ± 2	72	-	-	-
WFT180	Wet	-22 ± 2	72	Climatic chamber (20 °C and 65% de UR)	180 ± 1	3.5
WT180	-	-	-	Climatic chamber (20 °C and 65% de UR)	180 ± 1	3.5
WFT200	Wet	-22 ± 2	72	Climatic chamber (20 °C and 65% de UR)	200 ± 1	3.5
WT200	-	-	-	Climatic chamber (20 °C and 65% de UR)	200 ± 1	3.5

W: wood; F: freeze-treated; T: heat-treated * freezing rate = 0.04 °C/min; defrosting rate until 0°C = 0.6°C/min; ** heating rate = 0.09 °C/min.

The wood freezing (WF) was carried out in a conventional freezer without air circulation. After this stage, all the samples were dried at 40 °C in an oven with forced air circulation, and the moisture content of the samples was monitored using the methodology described by Severo (2000). The heat treatment (WT) was performed in the same oven with forced air circulation. After this step, the temperature was reduced to 100 °C and all the samples were kept in a climatic chamber (20 °C and 65% of relative humidity). The Two-stage freezing-heat treatment (WFT) was then performed with the same parameters which are described above with the freezing and heat treatments taken into consideration.

Physical and mechanical analysis

Thirty replicates were used for all tests. The mass percent loss (WL) after the sample treatment was obtained by measuring the acclimated masses of the samples before and after the treatments. For this procedure, the sample's mass was normalized after taking into account the moisture content. The equilibrium moisture content (EMC) and the basic density (ρ_b) were obtained following the procedures from ASTM D143-94 (2000).

The mechanical tests of static bending, compression parallel to grain, and Janka hardness were determined according to the ASTM D143-94 standard (2000). The impact resistance test was carried out using a Charpy pendulum as described by NBR 7190 (ABNT 1997). Thus, the modulus of elasticity (MOE), modulus of rupture (MOR), resistance to compression parallel to grain (σ_{max}), Janka hardness in the tangential (H_T), radial (H_R), and longitudinal (H_L) directions, and impact resistance (F_{max}) were measured.

Data analysis

The collected data were analyzed with descriptive statistics and analysis of variance (ANOVA) using Statgraphics Centurion XVI. The assumption of normality of data ($p > 0.05$) and homogeneity of variance ($p > 0.05$) were checked by the White and Shapiro-Wilk test. The results obtained in these assumptions tests allowed the further performing of the ANOVA and LSD Fisher parametric statistical tests.

The statistical analysis took into consideration a factorial arrangement of 2 x 3 with two levels of freezing (with and without) and three levels of temperature (20 °C – control, 180 °C, and 200 °C), in which the average values were compared by means of the F-test at 1% and 5% significance. If the null hypothesis was rejected ($p < 0.05$), the average values for each treatment were compared by means of the LSD (Least Significant Difference) Fisher test at 1% and 5% significance.

RESULTS AND DISCUSSION

Physical Properties

The factorial ANOVA (Table 2) was not applied for WL, as it was not determined for the control treatment. The two-stage freeze-heat treatments affected the EMC, which demonstrated its combined effect in this property. However, this influence was not observed for ρ_b. The variation of ρ_b was not significant because of a similar loss of mass and volume (Hill 2006), as previously stated in the literature (Cademartori *et al.* 2014, 2015; Delucis *et al.* 2014). When the variations of both mass and volume of wood were similar, the basic density did not significantly change. Nevertheless, other studies with heating treatments in juvenile/adult wood (Bal and Bektas 2012) and in heartwood/sapwood (Todorovic *et al.* 2012) observed that the effect of high temperatures

in the basic density were different for each type of wood. Thus, the effect of heating treatments on wood properties should not be based on the basic density changes, since the tendency of this wood property were not well-defined for these conditions.

Table 2. Summary of ANOVA for the Physical Properties of Thermally Treated Rose Gum and Gympie Messmate Woods

Properties	Rose gum			Gympie messmate	
	Factor	df	F-value	df	F-value
ρ_b (g/cm^3)	Freezing (A)	1	0,03^{ns}	1	0,08^{ns}
	Temperature(B)	2	0,96^{ns}	2	2,81^{ns}
	A x B	2	0,20^{ns}	2	2,23^{ns}
	Residue	135	-	124	-
EMC (%)	Freezing (A)	1	20,43**	1	64,11**
	Temperature (B)	2	1484,15**	2	2604,59**
	A x B	2	12,75**	2	17,01**
	Residue	166		167	

ρ_b: Density (g/cm^3); EMC: Equilibrium moisture content (%); df: Degree of freedom; ns: Not significant; **Significant at 1% significance.

The freezing treatment by itself was not sufficient to cause any significant changes to the mass of the wood samples; however, when combined with the heating treatment stage (both at 180 and 200 °C), the WL values decreased (Table 3). The attenuation of WL may be due to the improved permeability of the wood after freezing, enabling the removal of water, and thus avoiding the drastic vapor pressures during the heating treatment. The temperature in the heating stage was positively related to the WL. An increase of WL at high temperatures was related to the thermal stability of hemicelluloses (Esteves *et al.* 2007), which probably affects the mechanical properties of the wood.

Furthermore, the WL can indicate the severity of heating treatments (Almeida *et al.* 2009), especially in closed process, wet conditions, with oxygen atmosphere and for hardwoods (Hill 2006). The WL increases with increasing the temperature of treatment (Almeida *et al.* 2009; Gunduz *et al.* 2010; Bal and Bektas 2012; Todorovic *et al.* 2012; Cademartori *et al.* 2014; Conte *et al.* 2014; Pertuzzatti *et al.* 2015).

The EMC decreased with increasing temperatures, which can be clearly observed at 200 °C. These results occurred because of the *in situ* dehydration of sugars into hydroxymethyl furfural (HMF) and furfural, which are less polar compounds than sugars (Rowell *et al.* 2009). On the other hand, the freezing stage significantly increased the EMC of the rose gum (both the control and sample heated at 180 °C) and the Gympie messmate. The improved permeability of wood after freezing (Glossop 1994; Ilic 1995) could lead to an increase of the fractional volume of wood vessels and, consequently, to an increase of longitudinal permeability and EMC (Siau 1984). This phenomenon was also highlighted in other studies (Bal 2014; Cademartori *et al.* 2014; Esteves *et al.* 2014; Missio *et al.* 2016).

Table 3. The Physical Properties of the Control Wood Samples and the Two-Stage Freeze-Heat Treated Wood Samples

			Control	180 °C	200 °C
WL (%)	Rose gum	NF	-	3.11 aA (1.19)	6.81 bB (0.95)
		F	-0.10 a (0.04)	2.74 bA (0.98)	4.81 cA (0.84)
	Gympie messmate	NF	-	4.67 aB (1.03)	7.44 bA (1.15)
		F	-0.93 a (0.51)	4.06 bA (0.89)	6.92 cA (0.61)
ρ_b (g/cm³)	Rose gum	NF	0.43 (0.03)	0.42 (0.03)	0.44 (0.04)
		F	0.43 (0.04)	0.43 (0.04)	0.44 (0.05)
	Gympie messmate	NF	0.69 (0.04)	0.68 (0.03)	0.69 (0.04)
		F	0.67 (0.04)	0.68 (0.04)	0.70 (0.04)
EMC (%)	Rose gum	NF	11.18 cA (0.71)	7.80 bA (0.51)	5.85 aA (0.54)
		F	11.95 cB (0.40)	8.45 bB (0.73)	5.63 aA (0.5)
	Gympie messmate	NF	11.42 cA (0.36)	7.58 bA (0.49)	5.29 aA (0.51)
		F	12.62 cB (0.57)	7.72 bA (0.35)	5.76 aB (0.62)

Average (standard deviation); WL: Mass loss (%); ρ_b: Density (g/cm³); EMC: Equilibrium moisture content; NF: Non-freeze; Average values followed by the same lowercase letters in the line and uppercase letter in the column are not statistically different according to LSD Fisher test at 5% significance. The absence of a letter in the column and/or line means that there was no significant difference.

Mechanical Properties

The interaction between the two stages of the treatment was significant for F_{max}, σ_{max} (in the rose gum), and H_T (in the Gympie messmate), which indicated the combined action of the freezing and heating treatments in wood properties. The MOR, σ_{max}, and H_L for the rose gum, and H_R for the Gympie messmate were significantly affected by the freezing stage. The heating temperature factor was not significant for H_R of the rose gum and for MOE of the Gympie messmate wood, illustrating that at least one temperature affected the other properties (Table 4).

The MOE of the rose gum wood increased by approximately 8.5% after the treatment at 200 °C (Table 5). On the other hand, MOE of the Gympie messmate wood did not vary significantly. According to Esteves and Pereira (2009), the MOE increases at mild-temperature heating treatments and decreases at high-temperature heating treatments. This mechanism was driven by the thermal decomposition of wood. In fact, mass loss of up to 4% suggests an increase of MOE, whereas a higher increase of mass loss results in the reduction of MOE. The mass losses of the rose gum wood were 2.9% and 5.8% after treatments at 180 and 200 °C, respectively. The mass losses of the Gympie messmate wood were 4.4% and 7.2% at the same temperatures. Even with mass losses higher than 4% after some treatments, the MOE did not decrease. This suggests a relation between MOE and a significant reduction of EMC at high temperatures. This reduction was 7.8% at 180 °C and

5.5% at 200 °C. The lower equilibrium moisture content can increase some of the mechanical properties of wood (Bodig and Jayne 1982).

Table 4. A Summary of ANOVA for the Mechanical Properties of the Thermally Treated the Rose Gum and the Gympie Messmate Woods

Properties	Rose gum			Gympie messmate	
	Factor	df	F-value	df	F-value
MOE (GPa)	Freezing (A)	1	0.10^{ns}	1	0.53^{ns}
	Temperature (B)	2	8.19^{**}	2	2.22^{ns}
	A x B	2	0.06^{ns}	2	0.05^{ns}
	Residue	164	-	164	-
MOR (MPa)	Freezing (A)	1	13.36^{**}	1	0.83^{ns}
	Temperature (B)	2	10.51^{**}	2	29.32^{**}
	A x B	2	0.63^{ns}	2	0.64^{ns}
	Residue	168		165	
F_{max} (kJ/cm^2)	Freezing (A)	1	1.22^{ns}	1	0.02^{ns}
	Temperature (B)	2	62.86^{**}	2	131.24^{**}
	A x B	2	4.65^{*}	2	0.34^{ns}
	Residue	135	-	132	-
σ_{max} (MPa)	Freezing (A)	1	12.42^{**}	1	0.06^{ns}
	Temperature (B)	2	48.57^{**}	2	64.71^{**}
	A x B	2	6.53^{**}	2	1.33^{ns}
	Residue	102	-	94	-
H_T (kgf/cm^2)	Freezing (A)	1	0.24^{ns}	1	0.10^{ns}
	Temperature (B)	2	4.12^{*}	2	21.1^{**}
	A x B	2	2.75^{ns}	2	4.13^{*}
	Residue	99	-	95	-
H_R (kgf/cm^2)	Freezing (A)	1	0.08^{ns}	1	4.30^{*}
	Temperature (B)	2	2.62^{ns}	2	11.37^{**}
	A x B	2	1.61^{ns}	2	2.14^{ns}
	Residue	102	-	94	-
H_L (kgf/cm^2)	Freezing (A)	1	5.53^{*}	1	3.21^{ns}
	Temperature (B)	2	7.47^{**}	2	11.13^{**}
	A x B	2	1.91^{ns}	2	0.31^{ns}
	Residue	106	-	97	-

df: Degree of freedom; ^{ns}: Not significant; *: Significant at 5% significance; **: Significant at 1% significance.

The variation of MOE of heat-treated wood in oxygen atmosphere was related by other researchers. Calonego *et al.* (2012) did not observe changes in MOE after heating treatments of *E. grandis* at 180 and 200 °C. On the one hand, Pfriem *et al.* (2010) found an increase of 36% in *Picea abies* wood heat-treated at 180 °C. Todorovic *et al.* (2012) observed a significant increase of MOE of *Fagus sylvatica* wood heat-treated at 170 and 190 °C. On the other hand, Bal and Bektas (2013) verified a reduction of ~21% in MOE of

E. grandis wood treated at 180 °C, and Kačíková *et al.* (2013) observed reduction of 9.45% in MOE of *Picea abies* wood treated at 187 °C.

The wood freezing caused decreases in some mechanical properties of the rose gum. The MOR and σ_{max} values changed by -15% when compared with the control samples, while F_{max} changed by -21.8%. According to Szmutku *et al.* (2013), the pressure developed by the expansion of the ice during the freezing process in the lumen may be able to break the H-bonds in the water/wood interface, which leads to the formation of microcracks in the cell wall structure and reduces the mechanical strength. The absence of strength loss for the Gympie messmate wood can be related to its lower moisture content (~70% dry basis) during freezing, in comparison with the Rose gum wood (~140% dry basis). The mechanical changes of wood promoted by the freezing treatment were associated with compression forces against the cell wall of wood attributed to the water expansion (Ilic 1995). This phenomenon is aggravated by the faster freezing rate (Szmutku *et al.* 2013).

The MOR and F_{max} presented an inverse relation to the heat temperature (Table 5). The MOR of the Rose gum wood decreased by 11.9% at 180 °C, and the MOR of the Gympie messmate wood decreased by 23.2% at 200 °C. The F_{max} of Rose gum wood decreased by 55.6% (NF) and 32.1% (F), respectively at 180 °C, and 64.5% (NF) and 56.9% (F) at 200 °C. Regarding the Gympie messmate wood, the heating temperature did not influence the F_{max}. However, the F_{max} decreased by 51% in relation to the control samples. In contrast to what was observed by Korkut and Budakçi (2009), there was no relation between the heating temperature and F_{max} loss. This change occurred because of the attenuation effect promoted by the freezing stage which avoided excessive thermal decomposition of wood (Missio *et al.* 2015).

The decrease of the MOR found in this study was lower than those observed by Calonego *et al.* (2012) for *E. grandis* wood heat-treated at 180 and 200°C. The authors found reductions of MOR of 24 and 33% for 180 and 200°C, respectively. Likewise, Bal and Bektas (2013) observed for the same wood specie the reduction of 27.20% at 180°C in the bending strength.

If no freezing stage was applied, the heating treatments led to a cleavage of secondary bonds that connect the hemicelluloses to cellulose, which have a high correlation with F_{max}. Treatments at high temperatures can promote the cleavage of covalent bonding (depolymerization) inside the microfibrils/fibrils of cellulose, which generates a proportional increase of the crystalline cellulose and, consequently, a potentially negative effect in F_{max} (Boonstra *et al.* 2007; Kačíková *et al.* 2013) was noticed.

Of all the wood properties, F_{max} is often affected by the heating treatments, in which its decrease is directly proportional to the increase of both time and temperature of treatment (Korkut and Budakçi 2009). The same authors found reductions of 16.33% and 32.25% in F_{max} of *Sorbus aucuparia* wood after 6 h of treatment at 150 and 180 °C, respectively. Likewise, for similar conditions (4 h at 180 °C), Bal and Bektaş (2013) observed a decrease of 58.72% and 42.18% of impact resistance of both juvenile and adult wood, respectively, of *E. grandis*. Boonstra *et al.* (2007), under hydrothermolysis conditions, verified a high loss of impact resistance of *Pinus sylvestris* (56%), *Picea abies* (79%), and *Pinus radiate* (80%). The σ_{max} increased after the heating treatments. Regarding *E. grandis* wood, the σ_{max} increased as temperature increased. The σ_{max} of *E. cloeziana* wood significantly increased after heating. However, the level of modification was statistically equal between 180 and 200 °C. The same positive effect in the σ_{max} was observed by Boonstra *et al.* (2007) treating *Pinus sylvestris* wood.

Table 5. Average Values of the Mechanical Properties of the Untreated and Thermally Treated Rose Gum and Gympie Messmate Woods

			Control	180 °C	200 °C
MOE (GPa)	Rose gum	NF	10.53 a (1.32)	11.10 ab (1.37)	11.35 b (1.40)
		F	10.42 a (0.92)	11.01 ab (0.90)	11.38 b (1.14)
	Gympie messmate*	NF	14.00 (1.49)	14.17 (1.60)	14.59 (1.78)
		F	13.88 (1.34)	13.88 (1.45)	14.46 (1.86)
MOR (MPa)	Rose gum	NF	78.19 bB (11.12)	68.92 aB (15.33)	61.47 aA (19.51)
		F	66.19 bA (10.24)	60.01 abA (17.04)	56.07 aA (19.42)
	Gympie messmate	NF	119.07 b (12.50)	112.08 a (19.65)	97.25 a (17.42)
		F	120.90 c (12.20)	107.24 b (20.30)	92.90 a (21.49)
F_{max} (kJ/cm^2)	Rose gum	NF	46.44 bB (17.85)	20.64 aA (9.50)	15.08 aA (7.20)
		F	36.31 cA (14.58)	24.66 bA (9.67)	15.64 aA (6.66)
	Gympie messmate	NF	74.55 b (19.57)	37.80 a (9.26)	34.03 a (8.49)
		F	73.90 b (9.56)	36.48 a (6.35)	36.90 a (9.59)
σ_{max} (MPa)	Rose gum	NF	46.25 aB (3.99)	53.09 bB (6.37)	54.52 bA (5.76)
		F	39.04 aA (3.12)	47.49 bA (4.53)	56.04 cA (7.14)
	Gympie messmate	NF	60.88 a (6.21)	72.21 b (6.99)	75.96 b (7.06)
		F	59.50 a (5.35)	75.39 b (5.07)	75.10 b (6.09)
H_T (MPa)	Rose gum	NF	36.01 aA (12.38)	34.74 aA (4.14)	33.55 aB (5.65)
		F	33.51 abA (8.26)	39.21 bA (8.43)	29.25 aA (6.81)
	Gympie messmate	NF	81.24 bA (11.98)	73.32 aA (5.45)	72.31 aB (11.17)
		F	88.10 bA (6.59)	72.38 aA (12.51)	64.36 aA (11.80)
H_R (MPa)	Rose gum	NF	32.22 a (10.95)	29.80 a (8.76)	27.92 a (7.90)
		F	28.78 ab (7.20)	33.45 b (8.69)	26.32 a (8.15)
	Gympie messmate	NF	76.64 bA (8.55)	62.45 aA (19.95)	66.01 aA (9.51)
		F	80.25 bA (8.10)	73.48 bB (6.44)	65.66 aA (9.78)
H_L (MPa)	Rose gum	NF	47.19 a (10.94)	51.12 a (8.27)	49.05 a (7.65)
		F	39.74 a (6.62)	51.54 b (8.81)	44.22 a (9.14)
	Gympie messmate	NF	84.20 b (4.74)	80.42 ab (16.24)	71.10 a (17.86)
		F	80.86 b (5.61)	76.58 b (16.10)	62.84 a (19.40)

Average (standard deviation); Average values followed by the same lowercase letters in the line and uppercase letter in the column are not statistically different according to the LSD Fisher test at 5% significance. * Average values without significant difference. No letter in the column and/or line represent no significant difference.

The freezing stage did not influence the Janka hardness; however, the heating treatment at 200 °C decreased the hardness by 11.9%. On the other hand, the freezing stage contributed to a 17.7% increase of H_R in the Gympie messmate wood. As previously stated for other mechanical properties, the freezing stage promotes damage to the wood cell wall, which may decrease or increase the mechanical properties as a function of the damage level (Szmutku *et al.* 2013). In summary, the Janka hardness decreased as temperature increased. Regarding the Gympie messmate wood, H_L decreased by 22.3% (NF) and H_T decreased by 26.9% (F) at 200 °C. Similar results have been observed in previous studies (Unsal *et al.* 2003; Korkut *et al.* 2008; Calonego *et al.* 2012; Bakar *et al.* 2013; Priadi and Hiziroglu 2013). The reduction of hardness was attributed to the decomposition of the hemicelluloses during the heating treatments. On the other hand, H_T and H_L of the rose gum wood increased by 17% and 29.7%, respectively, at 180 °C (F). The heating treatment at 200 °C resulted in no significant variation of hardness.

CONCLUSIONS

1. The freezing treatment decreased the level of mass loss and increased the moisture content of wood.
2. The modulus of rupture, compression parallel to grain, and impact maximum strength of the rose gum wood decreased after the freezing treatment.
3. The heating treatments decreased the moisture content and increased the mass loss for both species. The modulus of elasticity and compression strength increased only after the heating stage, while decrements were found for modulus of rupture, impact maximum strength, and Janka hardness.
4. The two-stage treatments did not prevent the decrease in mechanical properties. The two-stage freezing and heating treatments in the rose gum wood caused the highest decrements of the mechanical properties, especially at 180 °C for modulus of rupture, compression strength parallel to the grain, and tangential hardness. Regarding the Gympie messmate wood, the two-stage freezing and heating treatments only significantly influenced radial hardness at 180 °C. Therefore, regarding Gympie messmate wood, two-stage freezing and heating treatments can be used in places where wood is exposed to flexure tensions and high hardness, such as for flooring applications.

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