Physical and Colorimetric Changes in *Eucalyptus grandis* Wood after Heat Treatment

Antônio José Vinha Zanuncio,[a,*] Javan Pereira Motta,[b] Teodorico Alves da Silveira,[b] Elias De Sá Farias,[b] and Paulo Fernando Trugilho [b]

Planted forests can meet the world's demand for wood. In Brazil, eucalypt species are cultivated on a large scale, but their dimensional instability and color limit their use, which makes heat treatment necessary. The aim of this study was to evaluate physical and colorimetric properties of *Eucalyptus grandis* after heat treatment at 140, 170, 200, and 230 °C for 3 h. Mass loss, shrinkage, equilibrium moisture content, volumetric swelling, fiber saturation point (FSP), and colorimetric parameters were determined; photos were also taken with a scanning electron microscope for all treatments. Heat treatment reduced the wood mass by 0.33 to 10.64% and caused shrinkage by 0.23 to 5.16%. Treatment at 230 °C reduced oven dry density. Equilibrium moisture content was 9.40, 9.34, 8.55, 6.55, and 5.05% for control and test samples treated at 140, 170, 200, and 230 °C, respectively. Heat treatment reduced thickness swelling and FSP by 59.65% and 56.31%, respectively. Heat treatment also reduced the *L** (lightness), *a** (green–red coordinate), and *b** (blue–yellow coordinate) values of the wood samples. Heat treatment improved physical properties and darkened the wood; however, the damage observed in scanning electron microscope images could reduce the mechanical properties of wood.

Keywords: Equilibrium moisture; Eucalyptus grandis; Heat treatment; Lightness; Volumetric swelling

*Contact information: a: Department of Forestry Engineering at Federal University of Viçosa, MG 36570-000, Brazil; b: Department of Forest Science, Federal University of Lavras, P.O. Box 3037,37200-000, Lavras/MG, Brazil; *Corresponding author: ajvzanuncio@gmail.com*

INTRODUCTION

Wood from rainforests have excellent physical, mechanical, and colorimetric properties. In the past, unsustainable exploitation has devastated forests, decreasing the supply and increasing the price of native timber, with an imbalance between supply and demand.

Fast-growth cultivated forests can meet the wood demand. Brazil has about 6.5 million hectares of planted forests, with 74.8% of this forest comprising various eucalypt species (ABRAF 2012), especially *E. camaldulensis*, *E. globulus*, *E. grandis*, *E. robusta*, *E. saligna*, *E. tereticornis*, and *E. urophylla* (ABRAF 2012). However, the anisotropy coefficient of these species, ranging between 1.30 and 2.22, and its color (Caixeta *et al*. 2003; Oliveira *et al*. 2003; Pelozzi *et al*. 2012) are undesirable characteristics for solid wood.

The heat treatment process consists of heating the wood with the process variables of time, pH, wood moisture content, and pressure (Esteves and Pereira 2009). The process degrades hemicelluloses and breaks down monomers such as arabinose, galactose, mannose, and xylose (Brito *et al*. 2008; Severo *et al*. 2012; Brosse *et al*. 2010). Heat treatment releases extractives, such as acetic acid, furfural, and mono-, sesqui-, and di-terpenes (Esteves *et al*. 2008a; Esteves *et al*. 2011). Cellulose and lignin are less influenced by heat treatment (Yildiz *et al*. 2006; Tumen *et al*. 2010).

The mass loss resulting from heat treatment depends on wood characteristics, being higher for hardwood treated by high temperatures and long retention time (Esteves *et al*. 2007; Korkut, 2012; Severo *et al*. 2011; Kasemsiri *et al*. 2012).

The destruction of hemicelluloses reduces the hygroscopic sites of wood (Korkut 2012). Heat treatment at 220 °C for 2.5 h reduced the equilibrium moisture content of *Eucalyptus grandis* wood by 49.3% (Calonego *et al*. 2012).

Reduction of the equilibrium moisture improves wood's dimensional stability. The swelling of *Hevea brasiliensis* wood decreased from 7.3% to 3.1% with heat treatment at 180 °C for 10 h (Ratnasingam and Ioras 2012).

The oxidation of hemicelluloses reduces timber lightness (Bourgois *et al*. 1991), especially for hardwoods (Esteves *et al*. 2008b; Moura and Brito 2011). The concentration and volatilization of extractives may influence the *a** (green–red coordinate) and *b** (blue–yellow coordinate) values of wood (Aydemir *et al*. 2012).

For the mechanical properties, heat treatment at 230 °C for 4 h reduced the dynamic elasticity modulus of *E. grandis* by 13% (Garcia *et al*. 2012). Heat treatment at 180 to 210 °C for 3 h reduced the rupture modulus of *Pinus nigra* wood from 61.4 to 43.0 to 39.3 N/mm^2 and the elasticity modulus from 5606.8 to 4783.5 to 4537.5 N/mm^2 at the temperatures of 180 °C and 210 °C for 3 h (Dundar *et al*. 2012).

Most studies dealing with heat treatment in *Eucalyptus* wood have separated the juvenile and mature wood and have used oriented samples (Esteves and Pereira 2009; Bal and Bektaş 2012). Such experimentation does not match typical industrial practices, where the wood from mature and juvenile wood are used together and the lumbers do not have perfect orientation in radical and tangential direction. Such differences can make it difficult to interpret the results of these studies in terms of the real situations. Random sampling may make the evaluation of thermally treated wood faster and easier to obtain samples and better corresponds with industrial situations. The aim of this study was to evaluate the physical and colorimetric properties of *E. grandis* wood after being subjected to different heat treatment temperatures using a random sampling.

EXPERIMENTAL

Three 15-year-old *E. grandis* trees were used. Logs were cut from the trees at 1.3 to 2.3 m height. This species was selected because it is widely planted in Brazil and the age is the recommended one for solid wood. These logs were cut to lumber and dried naturally until reaching equilibrium moisture content. Afterwards, 30 samples per treatment (2 × 2 × 3 cm each) were randomly collected along the radial and axial direction. These samples were dried in an oven until they reached 0% moisture content, and the mass and volume of each sample were obtained. Heat treatment was done at 140 °C, 170 °C, 200 °C, and 230 °C with a heating rate of 5 °C/min and residence time of 3 h in the same oven at atmospheric pressure and in the presence of air. Samples were withdrawn after cooling down to 25 °C, and the mass and volume were measured again.

Mass losses of samples were calculated with the formula,

$$ML = [(M_i - M_f)/M_i)*100] \quad (1)$$

where ML is the mass loss (%), M_i is the mass before heat treatment, and M_f is the mass after heat treatment. The volumetric loss by heat treatment was calculated by mercury displacement method using the equation,

$$CV = [(V_i - V_f)/V_i)*100] \quad (2)$$

where CV is the volumetric loss by heat treatment (%), V_i is the volume before heat treatment, and V_f is the volume after heat treatment. Density was calculated by,

$$D = M/V \quad (3)$$

where D is the oven dry density (g/cm^3), M is the mass at 0% moisture (g), and V is the volume at 0% moisture (cm^3).

The samples were conditioned at 23 °C and 65% relative humidity; samples were weighed daily until mass stabilization occurred. A curve of moisture absorption was then calculated. Sample moisture was calculated as,

$$Mo\ (\%) = [(M_w - M_d)/M_d)*100] \quad (4)$$

where Mo (%) is the moisture of the sample, M_w is the mass of the wet sample, and M_d is the mass of the dry sample. Then, the samples were placed in a humidity chamber and subjected to temperatures of 25 to 35 °C and relative humidities of 40, 60, and 80%. The moisture of the samples was determined after mass stabilization.

Samples were saturated to constant mass to obtain the saturated volume. The swelling volume was calculated by mercury displacement method using the equation,

$$VS = [(V_f - V_i)/V_i)*100] \quad (5)$$

where VS is volumetric swelling (%), V_i is the anhydrous volume of the sample, and V_f is the saturated volume of the sample.

The fiber saturation point (FSP) was calculated according Bal and Bektaş (2012), with the equation FSP (%) = VS/D, where VS is the volumetric swelling (%) and D is the oven-dry density (g/cm^3).

The L (lightness), a^* (green–red coordinate), and b^* (blue–yellow coordinate) values were measured with a Konica Minolta Spectrophotometer CM-2500D.

The anatomical structures of heat-treated wood were investigated using a scanning electron microscope (SEM). Untreated and heat-treated wood (4 m × 4 mm × 3 mm) were scraped with a blade. The sample was put under a vacuum and coated with a thin film of gold using an ion sputtering device. The micrographs of the surfaces were taken with an SEM model LEOEVO40 PVX.

The physical and colorimetric parameters of heat treated wood were tested using the Tukey test at the 0.05 probability level.

RESULTS AND DISCUSSION

Mass losses of *E. grandis* after heat treatment ranged from 0.33 to 10.64%, whereas the shrinkage increased from 0.23 to 5.16% and the oven dry density decreased from 0.627 to 0.570 g/cm^3 (Table 1). Increasing the temperature decreased the variation coefficient, which indicated that the treatment homogenized the samples. Mass losses of *E. grandis* wood showed a trend similar to the shrinkage, being less than 1% in the treatments at 140 °C and 170 °C due to the volatilization of polar extractives that occurs between 130 °C and 250 °C (Mészáros *et al*. 2007). Degradation of hemicelluloses at 200 °C and 230 °C caused higher mass losses

(Musinguzi *et al*. 2011; Barnetoa *et al*. 2011; Poletto *et al*. 2010). *Eucalyptus grandis* wood has shown mass losses from 0.57% to 1.65% at treatment temperatures of 150 °C and 180 °C, respectively, at 4 h (Bal and Bektaş 2012). This species has shown mass losses of 5.19 to 9.68% at 180 and 200 °C (Brito *et al*. 2006) and 17% at 250 °C for 5 h (Almeida *et al*. 2009).

Table 1. Mass Losses, Volumetric Loss by Heat Treatment, and Oven Dry Density of *Eucalyptus grandis* Samples Subjected to Heat Treatment for Three Hours

Treatment	Mass losses (%)	VLHT* (%)	Oven dry density (g/cm^3)
Control	-	-	0.617 (8.18) a
140°C	0.33 (9.15)^A a	0.23 (15.67) a	0.623 (9.54) a
170°C	0.63 (6.97) a	0.40 (14.54) a	0.627 (10.13) a
200°C	2.73 (3.69) b	1.36 (11.35) b	0.622 (9.11) a
230°C	10.64 (3.24) c	5.16 (9.65) c	0.570 (6.12) b

* Volumetric loss by heat treatment ^{A}Variation coefficient; Means followed by the same lower case letter per column do not differ between them by Tukey test at the 0.05 probability level

The oven-dry density of *E. grandis* wood was lower at 230 °C due to the degradation of hemicelluloses and the evaporation of volatile extractives (Brito *et al*. 2008; Boonstra and Tjeerdsma 2006). This phenomenon also occurred at lower temperatures (Musinguzi *et al*. 2012; Mészáros, 2007), but the period for which the samples were subjected to heat treatment was insufficient to change its density. The temperature at which significant density losses occurs varies with species and exposition period (Korkut 2012).

The equilibrium moisture content of *E. grandis* wood ranged from 5.5% to 9.41% with a reduction of 9.04, 30.31, and 46.27% at temperatures of 170, 200, and 230 °C, respectively, without differences in the period in which the sample reached the equilibrium moisture content (Fig. 1). Low values of equilibrium moisture in control samples were due to the hysteresis effect. Heat induces hemicellulose degradation and increases the area of crystalline cellulose (Kocaefe *et al*. 2008; Brito *et al*. 2008; Bhuiyan and Hirai 2005; Tjeerdsma and Boonstra 2006).

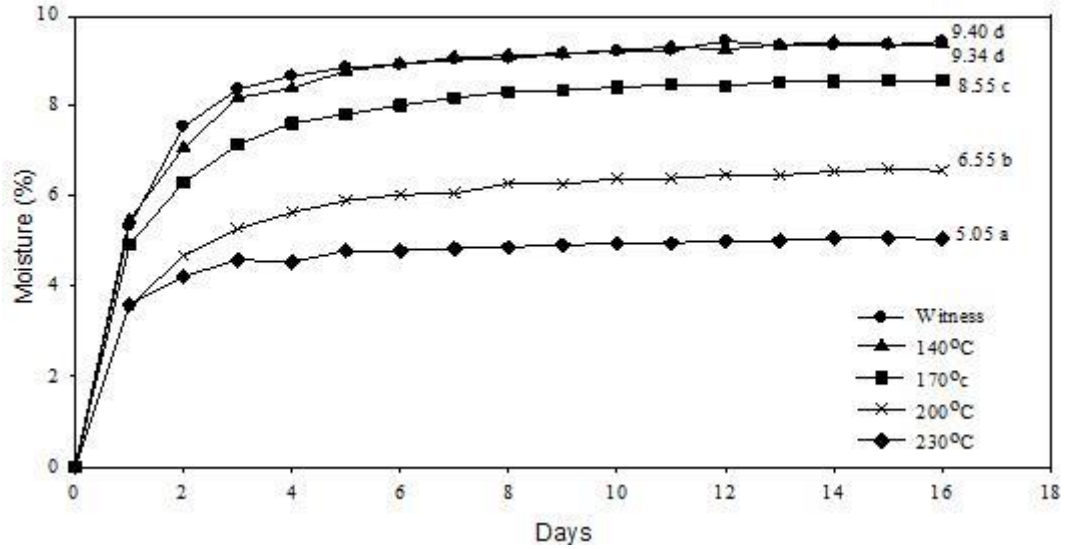

Fig. 1. Water adsorption of *Eucalyptus grandis* wood heat treated in a climatic chamber at 20 ± 2° C and 65 ± 3% RH

The equilibrium moisture content of *E. grandis* wood increased with relative humidity at 25 °C and 35 °C, with higher values at 25 °C and 80% relative humidity and lower at 35 °C and 40% relative humidity (Table 2). Variations in wood moisture between 25 °C and 80% relative humidity and 35 °C with 40% relative humidity were 5.56, 5.85, 5.32, 4.8, and 3.16% for the control and for the samples at temperatures of 140, 170, 200, and 230 °C, respectively. Heat treatment reduced the equilibrium moisture content of the wood and its variation when subjected to different environmental conditions.

Table 2. Wood Moisture (%) at 25 and 35 °C and Relative Humidity of 40, 60, and 80%

Treatment	25 °C			35 °C			Variation* (%)
	40%	60%	80%	40%	60%	80%	
Control	7.86	9.27	12.72	7.16	8.72	12.40	5.56
140°C	7.88	9.03	12.63	6.78	8.87	11.92	5.85
170°C	7.28	8.27	11.68	6.36	7.77	10.91	5.32
200°C	5.51	6.42	9.70	4.90	6.16	8.70	4.8
230°C	4.09	4.81	6.77	3.61	4.60	6.25	3.16

*Difference between the highest and lowest moisture in each treatment

Increasing the temperature reduced the water absorption when the wood was saturated in water (Table 3). This can be explained by the release of organic acids due to depolymerization of the hemicelluloses, which decreased the linkages with hydroxyl groups (Kasemsiri *et al*. 2012) and consequently reduced water absorption (Kocaefe *et al*. 2010).

Volumetric swelling of *E. grandis* wood was lower when treated at 170 °C (Table 3), with a reduction of 7.16, 30.86, and 59.65% at 170, 200, and 230 °C, respectively. Values from 6.8% to 55.21% have been found at 180 °C and 240 °C for *E. grandis* wood treated for 4 to 8 h (de Cademartori *et al*. 2012; Calonego *et al*. 2012). The reduction of hydroxyl groups reduces wood swelling (Severo *et al*. 2012).

The fiber saturation point (FSP) for *E. grandis* wood decreased when treated at 170 °C (Table 3), with a reduction of 9.22, 32.23, and 56.31% for samples subjected to 170, 200, and 230 °C, respectively, for 3 h. Hemicelluloses degrade faster at higher temperatures (Musinguzi *et al*. 2012; Barnetoa *et al*. 2011; Poletto *et al*. 2010), which reduces the content of hydroxyl groups and therefore reduces FSP. Juvenile and adult *E. grandis* wood have shown a reduction in FSP of 15.98% and 14.17% after treatment at 180 °C for 8 h (Bal and Bektaş 2012).

Table 3. Water Absorption, Volumetric Swelling, and Fiber Saturation Point (FSP) of *Eucalyptus grandis* Subjected to Temperatures between 140 °C and 230 °C

Treatment	Water absorption (%)	Volumetric swelling (%)	FSP (%)
Control	119.49 (8.11)[A] a	17.30 (7.55) a	28.39 (8.31) a
140°C	117.19 (8.22) a	17.43 (7.18) a	28.18 (7.12) a
170°C	103.83 (5.55) b	16.06 (5.12) b	25.68 (5.34) b
200°C	89.32 (5.13) c	11.96 (4.66) c	19.17 (5.54) c
230°C	69.64 (4.12) d	6.98 (3.14) d	12.36 (4.13) d

[A] Variation coefficient; Means followed by the same lower case letter per column are not significantly different by Tukey test at the 0.05 probability level.

The SEM photographs (Fig. 2) of wood treated at 140 °C showed no damage to the samples, but cracks were found near the vessel elements in those treated at 170 °C. This damage was intensified in samples subjected to treatment at 200 °C and 230 °C, with more widespread fiber degradation. Anatomical damage has been found to vary with wood features and process variables (Boonstra *et al*. 2006; Awoyemi and Jones 2011; Kasemsiri *et al*. 2012).

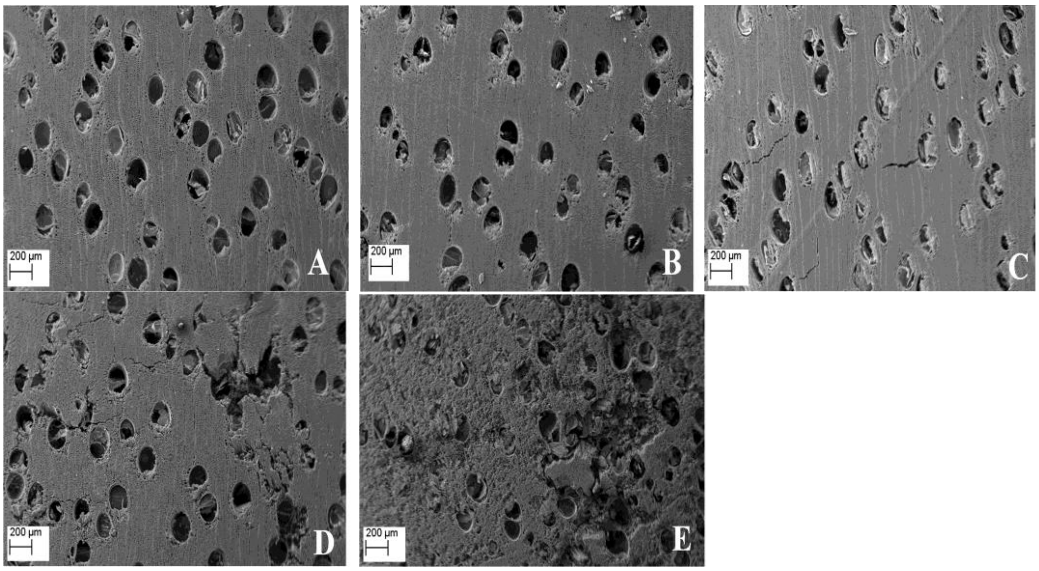

Fig. 2. SEM images of *Eucalyptus grandis* samples subjected to different temperatures. A- control; B- 140 °C; C- 170 °C; D- 200 °C; and E- 230 °C

The lightness value of *E*. *grandis* wood ranged from 29.69 to 56.16; the *a** value ranged from 2.5 to 12.9; and the *b** value ranged from 2.18 to 13.60. The reduction in lightness value of *E*. *grandis* was highest at 140 to 200 °C; it was reduced by 16.18% at 140 to 170 °C and 14.76% at 170 to 200 °C compared to the control. Increasing the treatment temperature gradually turned the wood into charcoal, which, irrespective of the species, is always black. The *a** value showed greater reduction at 170 to 200°C (33.41%), with a similar trend for the *b** value at the same temperature range (25.95%).

Table 4. Lightness, a* Value (Green-Red Coordinate), and b* Value (Blue-Yellow Coordinate) of *Eucalyptus grandis* Samples at Temperatures from 140 to 230 °C

Treatment	Lightness (L)	Green-red coordinate (a*)	Blue-yellow coordinate (b*)
Control	56.15 (6.26) [A] a	12.90 (8.82) a	13.60 (7.44) a
140°C	50.20 (5.84) b	12.03 (4.13) b	10.85 (7.86) b
170°C	41.11 (4.44) c	9.34 (5.75) c	8.36 (6.22) c
200°C	32.82 (4.22) d	5.03 (4.23) d	4.83 (9.67) d
230°C	29.69 (2.23) e	2.50 (6.56) e	2.18 (10.05) e

[A] Variation coefficient; Means followed by the same lower case letter per column do not differ between them by Tukey test at the 0.05 probability level.

The reduction of green-red coordinate (*a**) is associated with volatilizing of some chemical compounds such as phenolic extractives, which confer red color to wood, while the reduction of blue-yellow coordinate (*b**) is associated with the

chromophores in the lignin and extractives, as well as organometallic compounds in extractives that are degraded by increasing of temperature (Pincelli *et al.* 2012).

Pincelli *et al.* (2012) reported changes in lightness (*L**) from 65.6 to 45.1, in green-red coordinate (*a**) from 12.8 to 10.5, and the blue-yellow coordinate (*b**) from 18.6 to 17.6 for wood of *Eucalyptus saligna* treated at 180 °C. On the other hand, Cademartori *et al.* (2013) reported reductions of 53.95%, 24.9% and 40.81% for lightness (*L**), green-red coordinate (*a**), and blue-yellow coordinate in *E. grandis* wood treated at 240 °C for 8 h.

Color change is not desired during drying, but heat treatment can add value to wood. In addition, methods of coloring wood emit toluene and xylene, which are dangerous to human health and the environment (Korkut *et al*. 2012).

The highest coefficient of variation for the physical and colorimetric parameters for timber without heat treatment was 15.67% and 8.82%, respectively. Except for the coordinated blue-yellow, these values decreased as the heat treatment was intensified and were similar in relation to studies using oriented specimens (Canolego *et al.* 2012; Cademartori *et al.* 2012; Korkut *et al.* 2012). This indicates that the random sampling may be used for this type of study, primarily for heat treatment at higher temperatures.

CONCLUSIONS

1. The heat treatment promoted mass loss between 0.33 and 10.64%, as well as volumetric loss by heat treatment between 0.23 and 5.16%. The treatment also reduced the oven dry density from 0.617 to 0.570 (g/cm^3).
2. The heat treatment reduced the equilibrium moisture content in 46.27% but without differences in the period in which the sample reached the equilibrium moisture content when conditioned at 23 °C and 65% relative humidity.
3. The heat treatment reduced the variation of equilibrium moisture content in wood under different conditions of humidity and temperature.
4. The heat treatment decreased the water absorption from 119.49 to 69.64%, the volumetric swelling from 17.3 to 6.98%, and *FSP* from 28.39 to 12.36%.
5. The heat treatment reduced all colorimetric parameters. The lightness (*L**) from 56.15 to 26.69, the green-red coordinate (*a**) from 12.9 to 2.5, and blue-yellow coordinate (*b**) from 13.6 to 2.18.
6. A random sampling can be used for evaluation of heat treated wood, since all parameters evaluated had low variation coefficient. This sampling allows saving time, ease of obtaining samples, and a better adaptation to the industrial situation.

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