Prediction of the physical, mechanical and colorimetric properties of *Eucalyptus grandis* heat-treated wood using artificial neural networks

Predição das propriedades físicas, mecânicas e colorimétricas da madeira termorretificada de *Eucalyptus grandis* com redes neurais artificiais

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Resumo

A termorretificação é importante para alterar a cor da madeira e aumentar sua estabilidade dimensional, entretanto existem poucos estudos sobre controle da qualidade e modelagem durante o processo. O objetivo deste trabalho foi avaliar a eficiência das redes neurais artificiais em função da temperatura para predizer as propriedades da madeira de *Eucalyptus grandis* termorretificada. As amostras foram termorretificadas a 140, 170, 200 e 230°C por três horas sob pressão atmosférica e presença de ar, sendo avaliadas as propriedades físicas, mecânicas e colorimétricas da madeira. Uma rede neural artificial com base na temperatura de termorretificação foi criada para predição da umidade de equilíbrio, inchamento volumétrico, módulo de ruptura, claridade (L) e coordenadas vermelho-verde (a*) e azul-amarelo (b*). A umidade de equilíbrio, inchamento volumétrico e módulo de ruptura (MOR) foram afetados a partir de 170°C, o módulo de elasticidade a partir de 200°C, densidade anidra a partir de 230°C e parâmetros colorimétricos a partir de 140°C. As redes neurais artificiais utilizadas estimaram os resultados com precisão e sem tendências, evidenciando seu potencial para o controle da qualidade no processo de termorretificação.

Palavras-chave: Umidade de equilíbrio; Inchamento volumétrico; Propriedades mecânicas

Abstract

Heat treatment is important to change wood color and increase its dimensional stability, though the quality control and modeling during this process need to be further studied. The aim of this study was to evaluate the efficiency of artificial neural network based in temperature to predict the *Eucalyptus grandis* wood properties after heat treatment. The physical, mechanical and colorimetric properties of the wood samples after heat treatment at 140, 170, 200 and 230°C for three hours under atmospheric pressure and the presence of air were evaluated. An artificial neural network based on the heat treatment temperature was established to predict the equilibrium moisture content, total volumetric swelling, modulus of rupture, modulus of elasticity, lightness (L*), green-red coordinate (a*) and blue-yellow coordinate (b*) of the wood. The equilibrium moisture content, total volumetric swelling and modulus of rupture (MOR) were affected from 170°C, the modulus of elasticity from 200°C, the oven-dry density from 230°C and colorimetric parameters from 140°C onward. The artificial neural network estimated results with precision, showing its potential for quality control in the wood heat treatment process.

Keywords: Equilibrium moisture content; Mechanical properties; Volumetric swelling

INTRODUCTION

Eucalyptus wood has potential to produce solid wood (ANANIAS et al., 2014; SEVERO et al., 2010), but its coloring (CADEMARTORI et al., 2013a; CARVALHO et al., 2014) and high dimensional instability (SRINIVAS;PANDEY, 2012) limits its use for this purpose. Heat treatment can reduce these problems (ESTEVES; PEREIRA, 2009). The literature reports reduction from 6.8% to 55.21% for total volumetric swelling at 180°C and 240°C for *E. grandis* wood treated during 4 and 8 h (CALONEGO et al. 2012).

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The heat treatment involves the application of temperature between 160°C and 260°C (ESTEVES; PEREIRA, 2009), combined with other variables, such as time (KESIK et al., 2014; YALCIN; SAHIN, 2015), pH (WANG et al., 2012), presence of atmosphere (CANDELIER et al., 2013; KUZMAN et al., 2015) and relative humidity (OUMAROU et al., 2015). This process degrades the hemicellulose and cellulose of the wood (BRITO et al., 2008), limiting moisture adsorption capacity and increasing the wood dimensional stability (AYTIN et al., 2015; BASTANI et al., 2015; ESTEVES et al., 2008). This technique has high importance because no chemicals are used and it is of easy application (ESTEVES; PEREIRA, 2009; KORKUT, 2012).

The gains from the heat treatment may be higher with rigorous process control. Artificial neural networks, used in different areas of science (CATALOGNA et al., 2012; WERE et al., 2015; ATA, 2015) including wood technology (FINI et al., 2015; IGLESIAS et al., 2015; TIRYAKI et al., 2014a), can accurately predict results. Artificial neural networks are biologically-inspired computation models, consisting of simple processing elements that implement a particular mathematical function data when activated, generating the results desired (SCHUMAN; BIRDWEl, 2013).

Heat treatment has a proven potential to increase the applicability of the wood, but modeling and quality control of this process need to be further evaluated. The aim of this study was to evaluate neural networks to predict *Eucalyptus grandis* wood properties after heat treatment.

MATERIAL AND METHODS

Biological material

Three 15-years-old *Eucalyptus grandis* trees were selected. The trees were planted in Lavras-MG, Brazil (21° 14′ 43″ S and 44° 59′ 59″ W), the site planting had 1000 trees/ha. The average DBH was 30,1cm and mean height was 31 m. This species was chosen because it is one of the most used in Brazil and because of its potential as solid wood.

A central plank (plain-sawn) was removed from the base of each harvested tree and dried at open air until the wood equilibrium moisture content (EMC). Wood samples from 20 mm x 20 mm x 300 mm (Radial × tangential × longitudinal) were prepared for mechanical tests (static bending) , and specimens from 20 mm × 20mm × 30 mm (Radial × tangential × longitudinal) were prepared to evaluate the physical and colorimetric properties. The specimens were taken along the radial direction and samples coming from the same local within the boards were proportionally distributed in each heat treatment in order to avoid sampling effect, with 30 samples per treatment.

Heat treatment and mass loss

All wood samples were dried in an oven at 103°C for 24 hours until dry. Then, they were separated per treatment, their mass was recorded and the samples were heat treated at 140°C, 170°C, 200°C and 230°C at a heating rate of 5°C/min and time at maximum temperature of three hours at atmospheric pressure and under presence of air. The mass loss was calculated according to the equation 1:

$$MI = \frac{(M1 - M2) * 100}{M1} \qquad \textit{(Equation 1)}$$

Where:
Ml= mass loss (%),
M1= mass before heat treatment,
M2= mass after heat treatment.

Oven-dry density

The oven-dry density was calculated according to the equation 2:

$$D=M/V \qquad \textit{(Equation 2)}$$

Where:
D= oven-dry density (g/cm^3),
M= sample mass at dry condition (g),
V= volume of the sample at dry condition (cm^3).

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Equilibrium moisture content

The heat treated specimens were conditioned for 15 days in a climatic chamber at 23°C and 50% relative humidity to stabilize its mass. The EMC of the samples was calculated according to the equation 3:

$$EMC\,(\%) = \frac{(Mw - Md) * 100}{Md} \qquad \textit{(Equation 3)}$$

Where:
EMC (%) = equilibrium moisture content (%);
Mw= mass of samples after conditioning;
Md= mass of oven-dry sample.

Total volumetric swelling

Wood samples were then saturated with a vacuum pump connected to the desiccators so as to obtain the saturated volume. Total volumetric swelling was calculated with the mercury displacement method using the equation 4:

$$VS = \frac{(V2 - V1) \times 100}{V1} \qquad \textit{(Equation 4)}$$

Where:
VS= Total volumetric swelling (%),
V1= oven-dry volume of sample,
V2 = the saturated volume of the sample.

Mechanical characterization

The wood samples (2 × 2 × 30 cm) were submitted to mechanical tests and their modulus of elasticity (MOE) and rupture (MOR) determined according to ASTM (1994).

Colorimetric analysis

Wood colorimetric analysis was obtained with a spectrophotometer Konica Minolta CM-2500D, in its longitudinal surface, with four measurements per sample. Colorimetric parameters measured were lightness (L*), red-green coordinate (a*) and blue-yellow coordinate. The chroma (C*) was calculated by equation 5 and hue angle (H) by equation 6:

$$C= [(a^*)^2+ (b^*)^2]^{0.5} \qquad \textit{(Equation 5)}$$

$$H= arctang\,(b^*/a^*) \qquad \textit{(Equation 6)}$$

where:
C = chroma,
H = hue angle,
a* = green-red coordinate,
b* = blue-yellow coordinate.

The parameters obtained were based on the color system CIELAB 1976.

Statistical analysis

Variance homogeneity (Bartlett›s test at 5% significance) and normality (Shapiro-Wilk test at 5% significance) tests were performed prior to the analysis of variance. The contrast between the treatments means was determined by Tukey test at 5% significance level.

Artificial neural network

An artificial neural network with the Scaled Conjugate Gradient algorithm was established with 70% of the data for its training and 30% for its validation. The EMC, total volumetric swelling, modulus of rupture (MOR), modulus of elasticity (MOE), lightness (L*), green-red coordinate (a*) and blue-yellow coordinate (b*) were estimated with the same artificial neural network with the temperature as an input variable.

RESULTS AND DISCUSSION

The effect of heat treatment varied with the parameter evaluated, with an exponential increase of the mass loss as the temperature increased. The EMC, total volumetric swelling and MOR were affected from 170°C on, the modulus of elasticity from 200°C and the oven-dry density from 230°C onward (Table 1).

Tabela 1. Mass loss, equilibrium moisture content (Equi. moist.), total volumetric swelling (Vol. Swel.), oven dry density (Oven dry), modulus of rupture (MOR) and modulus of elasticity (MOE) of heat treated *Eucalyptus grandis* wood.
Table 1. Perda de massa, umidade de equilíbrio (Equi. moist) e inchamento volumétrico (Vol. Swel.) e densidade anidra (Oven dry) da madeira de *Eucalyptus grandis* termorretificada sob diferentes temperaturas.

Temperatures	Mass loss (%)	Equi. moist. (%)	Vol. Swel. (%)
Control	-	$9.41^{2.09}$ d	$17.25^{6.17}$ d
140 °C	$0.35^{3.32}$ a	$9.33^{2.14}$ d	$17.15^{3.73}$ d
170 °C	$0.66^{3.77}$ a	$8.55^{2.77}$ c	$16.06^{3.76}$ c
200 °C	$2.72^{2.88}$ b	$6.61^{3.11}$ b	$12.10^{3.78}$ b
230 °C	$10.53^{3.15}$ c	$5.02^{4.74}$ a	$7.12^{5.19}$ a
Temperatures	**Oven dry (g/cm³)**	**MOE (MPa)**	**MOR (MPa)**
Control	$0.611^{5.95}$ b	$6482^{5.52}$ a	$95.84^{4.70}$ d
140 °C	$0.611^{6.26}$ b	$6581^{8.83}$ a	$95.40^{6.89}$ d
170 °C	$0.614^{5.92}$ b	$6679^{7.68}$ a	$86.22^{6.54}$ c
200 °C	$0.617^{5.25}$ b	$7173^{5.42}$ b	$66.90^{7.66}$ b
230 °C	$0.566^{5.50}$ a	$7189^{4.33}$ b	$52.33^{6.27}$ a

Means followed by the same letter does not differ significantly by the Tukey test at 5%.
Values in superscript represent the coefficient of variation, in percentage.
Médias seguidas de mesma letra não diferem significativamente pelo teste de Tukey a 5% de probabilidade.
Valores em sobrescrito representam o coeficiente de variação, em porcentagem.

The EMC of heat treated wood at 170°C was 11.69% lower than control samples. This reduction was more severe at higher temperatures, being 46.65% at 230°C, a temperature at which degradation of carbohydrates is higher in hardwoods (CADEMARTORI et al., 2013b) and softwoods (SEVERO et al., 2012) and thus, resulting in higher EMC reduction. The total volumetric swelling, which is related to the holocellulose content and water adsorption capacity (CADEMARTORI et al., 2015; DUBEY et al., 2012), followed a similar trend of that of EMC, dropping significantly from 170°C onward and with an intense decrease at higher temperatures.

The wood oven-dry density was reduced only at 230°C, possibly explained by the evaporation of extractives and degradation of carbohydrates (BRITO et al., 2008; MÉSZÁROS et al., 2007). The reduction in wood density by heat treatment can occur at lower temperatures (YALCIN; SAHIN, 2015; KASEMSIRI et al., 2012), but the period for which the wood samples were subjected to heat up to 200°C was not enough to degrade their constituents and to reduce their oven-dry density. In addition, temperature that causes reduction of wood density varies according to species (KORKUT, 2012).

The reduction of the MOR followed the degradation of wood carbohydrates, reducing its values from 170°C onward, similar to that of the EMC and total volumetric swelling. The hemicelluloses are degraded at lower temperatures; this component has an important structural function, connecting the structure of cellulose and lignin. Its degradation changes the cell wall arrangement, reducing the wood mechanical strength (KACIKOVA et al., 2013).

Increasing the temperature from 200°C onward increased the MOE of the wood. This may be due to degradation of hemicellulose, reducing the wood capacity to resist to elastic deformations when subjected to mechanical stress and thus, reducing its deformation before rupture (SANTOS, 2000). A similar trend was reported for *Eucalyptus globulus* (SANTOS, 2000); however, in other works, the modulus of elasticity has not altered (CADEMARTORI et al., 2015) or its value was reduced after heat treatment (ZHANG et al., 2015). This shows that the modulus of elasticity behavior can vary with heat treatment.

The EMC and total volumetric swelling reduction of the heat treated wood render its use easier in environments with high variation of relative humidity, but the reduction of its mechanical strength limits its use for structural purposes (ESTEVES; PEREIRA, 2009). This indicates that it is necessary to

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find the optimum temperature which improves the physical properties and reduces the mechanical strength of treated wood.

The artificial neural network had a high accuracy for predicting the physical and mechanical properties of wood, with a correlation coefficient above 94% and a mean squared error lower than 7% (Table 2). This indicates that artificial neural network use can explain at least 95% of the data. The validation of the wood physical and mechanical parameters showed a correlation coefficient lower than that of the training except for the modulus of elasticity, which remained constant.

Tabela 2. Characteristics of selected artificial neural network to estimate the equilibrium moisture content (Equi. moist.), volumetric swelling (Vol. Swel.), modulus of elasticity (MOE) and modulus of rupture (MOR).
Table 2. Características da rede neural artificial selecionada para estimar a umidade de equilíbrio (Equi. moist.), Inchamento volumétrico (Vol. Swel.), módulo de elasticidade (MOE) e o módulo de ruptura (MOR).

ANN	Type	Coef.	Mean squared error (%)
Equi. Moist. (%)	Training	99.26	2.77
	Validation	96.01	2.73
Vol. Swel. (%)	Training	98.48	5.07
	Validation	97.62	3.92
MOE (MPa)	Training	95.06	6.78
	Validation	95.08	5.34
MOR (MPa)	Training	96.16	6.51
	Validation	94.56	6.90

ANN= Parameter estimated by artificial neural network; Coef.= correlation coefficient.

ANN= Parâmetro estimado pela rede neural artificial; Coef.= Coeficiente de correlação.

The correlation coefficient of artificial neural network found in this work was higher than others reported for wood properties using this technique in literature, as fracture toughness in solid wood, 0.62 (SAMARASINGHE et al., 2007); modulus of elasticity and rupture in structural panels, 0.73 and 0.66 respectively (FERNANDEZ et al., 2012) and similar to those of heat treated wood properties of *Fagus orientalis* and *Picea orientalis*, above 0.99 (TIRYAKI et al., 2014b).

Due to the lower coefficient of variation between samples in the same treatment, the error of values estimated by artificial neural network was lower for equilibrium moisture and volumetric swelling (Figure 1). However, all parameters showed errors homogeneously distributed and without homoscedasticity, showing the quality of the developed artificial neural network.

Heat treatment reduced the wood colorimetric parameter values. The highest reduction for the lightness (L*) was found at 170°C and 200°C, the green-red coordinate (a*), blue-yellow coordinate (b*) and chroma (C*) at 200°C, and finally, the hue angle from 140°C (Table 3).

Tabela 3. Lightness (L*), green-red coordinate (a*), blue-yellow coordinate (b*), chroma (C*) and hue angle (H) of *Eucalyptus grandis* heat treated wood.
Table 3. Claridade (L*), matriz vermelho-verde (a*) e azul-amarelo (b*) da madeira termorretificada de *Eucalyptus grandis*.

Treatment	L*	b*	b*	C*	H
Control	$56.21^{6.99}$ e	$12.85^{7.26}$ e	$13.59^{2.67}$ e	$18.7^{4.56}$ e	$46.73^{4.67}$ b
140 °C	$50.44^{6.46}$ d	$12.03^{4.10}$ d	$10.82^{6.60}$ d	$16.18^{5.67}$ d	$42.95^{5.15}$ a
170 °C	$41.06^{4.42}$ c	$9.36^{6.1}$ c	$8.41^{6.86}$ c	$12.59^{6.45}$c	$41.87^{6.33}$ a
200 °C	$32.43^{3.57}$ b	$4.84^{3.43}$ b	$4.63^{5.32}$ b	$6.7^{4.24}$b	$41.76^{4.54}$ a
230 °C	$29.81^{4.8}$ a	$2.58^{5.68}$ a	$2.29^{6.44}$ a	$3.45^{6.11}$a	$41.41^{6.23}$ a

Means followed by the same letter does not differ by the Tukey test at 5%. Values in superscript represent the coefficient of variation, in percentage.
Médias seguidas de mesma letra na vertical por clone não diferem pelo teste de Tukey a 5% de probabilidade. Valores em sobrescrito representam o coeficiente de variação, em porcentagem.

The reduction in lightness (L*) values of *E. grandis* wood heat treated was higher from 140 to 200°C, highlighting its darkening. This darkening at lower temperatures is due to the degradation of polar extractives and at 200°C due that of hemicelluloses (BRITO et al., 2008; MÉSZÁROS et al., 2007). At 230°C, the lightness (L*) reduction was less intense, because most of the compounds that darken the wood had been degraded at lower temperatures (BRITO et al., 2008; ESTEVES; PEREIRA, 2009). The wood darkening with increasing temperature was also reported for *E. grandis* (ZANUNCIO et al., 2014), *Eucalyptus saligna* (CADEMARTORI et al., 2015) and *Pinus pinaster* heat treated (ESTEVES et al., 2014), besides pre-hydrolysated *Eucalyptus grandis* and *Pinus elliottii* (CARVALHO et al., 2014).

a* and b* values were close for the control samples. The a* values were higher than b* values for heat treated wood at 140°C and 170°C. At 200°C, the a * and b * values were also similar again and this trend was repeated at 230°C. The a* value reduction in heat treated wood occurs due to degradation of some chemical compounds such as phenolic extractives (ESTEVES; PEREIRA, 2009). The b* values are associated with chromophores in the lignin and extractives (PINCELLI et al., 2012), which are degraded by heat. Values of C followed a trend similar to those of a* and b*, with decreased as temperature increased.

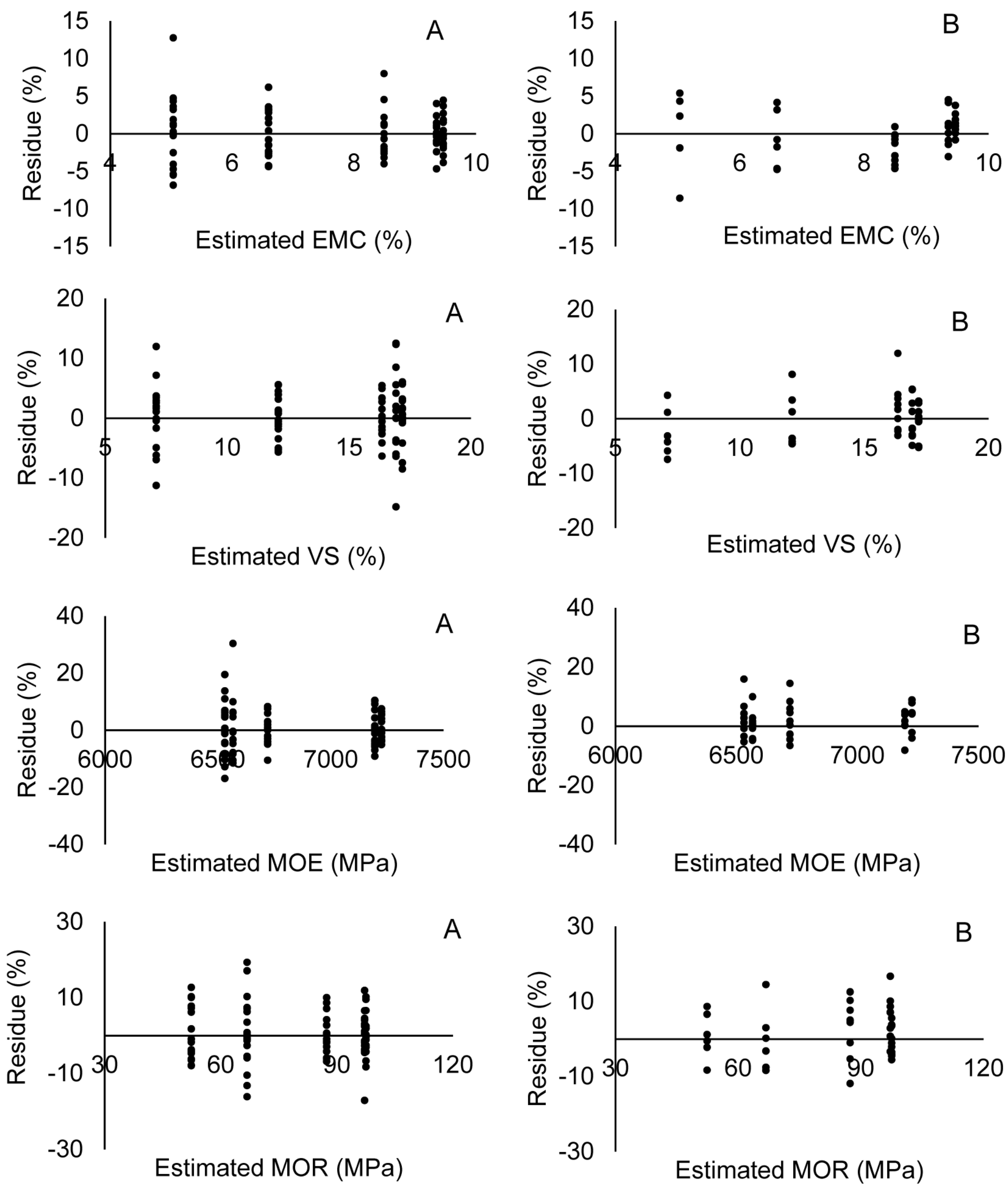

Figure 1. Distribution of residues of the physical and mechanical wood properties estimated by artificial neural networks. EMC= Equilibrium moisture content; VS= volumetric swelling. Graphs with the letter "A" represents the training and those with "B" the validation of artificial neural network.

Figura 1. Distribuição dos resíduos das propriedades físicas e mecânicas estimadas por redes neurais artificiais. EMC= umidade de equilíbrio higroscópico; VS= inchamento volumétrico. Gráficos com a letra "A" representam o treino e aqueles com a "B" a validação das redes neurais artificiais.

a* and b* values decreased in 6.38% and 20.38% at 140°C, respectively, which also reduced the hue angle. At higher temperatures, the a* and b* values reduction were similar and, therefore, the hue angle remained constant.

The artificial neural network also showed high precision to estimate the colorimetric parameters, with a correlation coefficient above 96% and a mean squared error below 9%. The correlation

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coefficient for the training was lower to that of the validation for colorimetric parameters, a trend contrary to those of physical and mechanical parameters (Table 4).

Tabela 4. Characteristics of selected artificial neural network to estimate the Lightness (L*), green-red coordinate (a*), blue-yellow coordinate (b*).
Table 4. Características da rede neural artificial selecionada para estimar a claridade (L*), matriz vermelho-verde (a*) e azul-amarelo (b*).

Est. par. by ANN	Type	Coef.	Mean squared error (%)
L	Training	96.19	6.93
	Validation	99.15	7.29
a*	Training	97.81	7.5
	Validation	98.50	7.39
b*	Training	96.70	8.87
	Validation	98.28	8.52

Est. par. by ANN= Estimated parameter by artificial neural network; Coef.= correlation coefficient.
Est. par. by ANN= Parâmetro estimado pela rede neural artificial; Coef.= Coeficiente de correlação.

The errors showed homogeneous distribution for all parameters. However, the homoscedasticity for lightness (L*) was due to a greater variability of the control data with high values of this parameter and increasing the mean standard error for the wood (Figure 2).

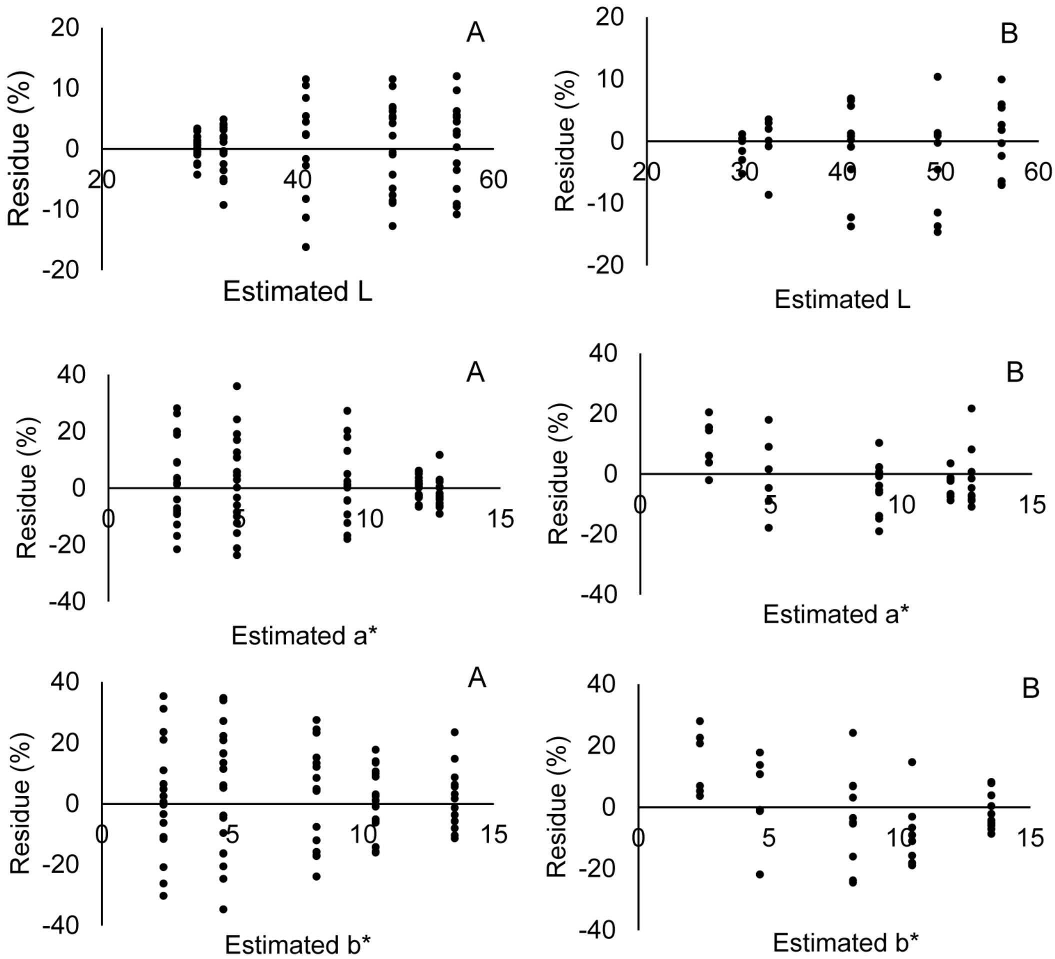

Figure 2. Distribution of residues for the colorimetric properties of wood estimated by artificial neural networks. Graphs with the letter "A" represents the training and those with "B" the validation of artificial neural network.
Figura 2. Distribuição dos resíduos para as propriedades colorimétricas estimadas por redes neurais artificiais. Gráficos com a letra "A" representam o treino e aqueles com a letra "B" a validação das redes neurais artificiais.

The artificial neural network was effective to predict the physical, mechanical and colorimetric parameter of *E. grandis* heat treated wood. This increased the potential of this method of easy application and low environmental impact.

CONCLUSIONS

The wood color started changing at 140°C. From 170°C on, the heat treatment improved the physical properties and decreased mechanical strength of the wood, with more intense effect at higher temperatures. The heat treatment improved the physical properties and promoted color changes, these facts assign new uses for the wood with a technique with low environmental impact. Artificial neural networks were effective for predicting the main quality parameters of heat treated wood, with a correlation coefficient above 95% and normal distribution of errors for all parameters, demonstrating its potential for controlling the quality of this process.

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