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COLOUR RESPONSES OF *Eucalyptus grandis* WOOD TO THE BRAZILIAN PROCESS OF THERMAL MODIFICATION

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ABSTRACT

A study of colour assessment, using the CIEL*a*b* system, was conducted with samples of Eucalyptus grandis wood thermally treated by the Brazilian industrial process of thermal modification, VAP HolzSysteme®, at three different temperatures, i.e. 140, 160 and 180 °C. Previous to the treatment, the samples were classified into three groups according to their distance to the wood pith, on the radial direction. All thermally modified samples presented a noticeable colour change, confirmed by high values of ΔE*. As the intensity of the treatment increased, the eucalypt samples presented an increase of red colour tone (a*) (up to 160 ºC) and a decrease of colour lightness (L*). Significant colour differences were found among the classified groups, for both untreated and thermally modified samples. The eucalypt samples groups showed different colour responses when thermally treated at 140 and 160 ºC. At 180 ºC the groups didn't show a significant colour response variation.

Keywords: Colorimetry, eucalypt wood, heat treatment, VAP HolzSysteme®, wood grading.

INTRODUCTION

The existing variability of wood colour found at the timber market is mainly due to a large number of commercial wood species. Nevertheless, despite that wood colour differs widely among species, the influence of growth conditions, genetics, and the wood anatomy can produce high variation in colour within a single tree (Dianiskova *et al.* 2008, Moya and Berrocal 2010). This variation can also be related to earlywood and latewood, tangential and radial cuts, or heartwood and sapwood (Brischke *et al.* 2007). The thermal modification process can change the wood colour and homogenise some properties, including the colour (Esteves and Pereira 2008). Understanding the wood colour variation combined with the application of thermal modification treatments may be led to the uniformity of the wood surface colour and enhancement of the final product.

The colour is an important wood property for the final consumer which often prioritizes the decorative point of view (Esteves *et al.* 2008). The preference for dark coloured or pale coloured wood depends on the timber markets, application areas, wood species, individual choices of the final client, trends or fashion and may be very diverse. However, a strong heartwood-sapwood colour variation can be a negative property for appearance products. Therefore, uniformity is a desirable trait since the variability in colour might reduce the aesthetic wood value for some nobles uses, like furniture or parquet floors (Bush 2008, Montes *et al.* 2008).

Equipment for exact colour reproduction and measurement have become increasingly important to respond to the present demand for timber markets for higher quality products. Among the different ways of determining the colour, the colorimetry, a non-destructive technique, arose from the need for

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an objective and accurate description of this feature. The colour determination can provide a useful means to classify, match and join pieces of wood in parquet floors, joinery, and furniture manufacturing (Janin *et al.* 2001).

Eucalyptus trees have been widely planted in Brazil, covering in total approximately 5,7 million hectares, which represents 72 % of the total area of planted trees in this country, and the *Eucalyptus grandis* is one of the main planted species (IBA 2017). This species is mainly used in the pulp and paper industry and used as energy fuel, whereas there is a limited interest on this wood for the sawn timber production. Usually, fast-growing trees from forest plantations present wood of inferior quality in such attributes as colour, density and mechanical properties, which makes that it fetches a lower price in the timber market (Thulasidas *et al.* 2006, Moya and Berrocal 2010). The surface colour and dimensional instability limit the use of the *Eucalyptus grandis* wood (Zanuncio *et al.* 2014). During the thermal modification process, the wood becomes more dimensionally stable and the wood colour is changed and acquires a dark tonality, what could be advantageous for species with unappealing colour like eucalypt (Esteves *et al.* 2008). Bekhta and Niemz (2003) studied the strong influence of thermal treatment on changing the wood colour of Norway spuce (Picea abies). As the temperature of treatment was increased all the colorimetric parameters were changed and the thermal treatment induced extensive darkening and reddening of spruce wood.

Extensively studies have shown the potential of thermal modification processes to enhance some of the less favourable wood properties, such as aesthetic characteristics, dimensional stability, and durability (Hill 2006, Cademartori *et al.* 2013, Zanuncio *et al.* 2014, Batista *et al.* 2016). From earlier research studies, it is known that after thermal modification at high temperatures several wood species like beech, poplar, pine, spruce, birch, and the eucalypt become more dimensionally stable (Militz 2002, Hill 2006, Esteves *et al.* 2007, Esteves and Pereira 2008). The lightness as well as the equilibrium moisture content, shrinkage-swelling, mechanical strength and hardness decrease progressively as the intensity of thermal treatment increases, while colour variation, anti-swelling efficiency, durability and resistance to weathering, increase (Vital *et al.* 1983, Brischke *et al.* 2007, Esteves *et al.* 2008, Esteves and Pereira 2008, Militz and Altgen 2011). From a chemical viewpoint, high temperatures of thermal modification usually cause the degradation of hemicelluloses and lignin. During the thermal modification of spruce and oak wood, it was found that the content of cellulose, lignin, and extractives increased, while the hemicellulose content dropped considerably for both species (Sikora *et al.* 2018).

Due to environmental concerns, only recently the development of industrial scale processes and the commercialization of thermally modified products were encouraged around the world, however, in some European countries, there are already several commercial applications for these products (Esteves *et al.* 2008, Sandberg and Kutnar 2016). The thermal modification of wood, without the use of chemicals, is an environment-friendly alternative method for improving several properties of wood (Sikora *et al.* 2018). The thermal treatment using saturated water steam is an alternative to reach different shades and change the wood colour (Dzurenda 2018). The thermo-mechanical densification can also modify the wood colour and promising results related to colour changes control during the industrial process of densification have been already achieved (Bekhta *et al.* 2014). Nowadays, the thermally modified wood could be found in several application areas, for outdoor use in the cladding, decking, flooring, garden furniture, window frames and indoors for parquet, kitchen furniture, and decoration (Willems *et al.* 2015).

In this paper, we analysed the colour responses of untreated and thermally modified *Eucalyptus grandis* samples at three different temperatures of thermal modification by the Brazilian industrial process VAP HolzSysteme®. Before the thermal modification we classified the eucalypt samples into three groups according to their distances to the wood pith, on the radial direction. First, we assessed the colour variation of all samples, without consider the classification and then we compared the results with the colour responses of the classified samples groups. This research paper will show the colour responses of three classified groups of eucalypt wood to different intensities of thermal modification and the variation of colour on the radial direction of the untreated wood.

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MATERIAL AND METHODS

Eucalypt boards

Five *Eucalyptus grandis* trees of 18 years-old were used for the sampling preparation. The trees are from a commercial plantation located at the state of Paraná, Brazil. Fifteen logs of 3300 mm length each were machined, and three boards were produced by log. The position of the boards at the eucalypt log is presented in transverse section at Figure 1. The boards were classified in three groups, i.e. *S*, *I* and *H*, named according to their distance to the wood pith, on the radial direction of the wood. We refer in this paper the external boards near the bark as *S* (sapwood), the central boards as *I* (internal) and the boards near the wood pith as *H* (hardwood). Each board has 30 mm of thickness.

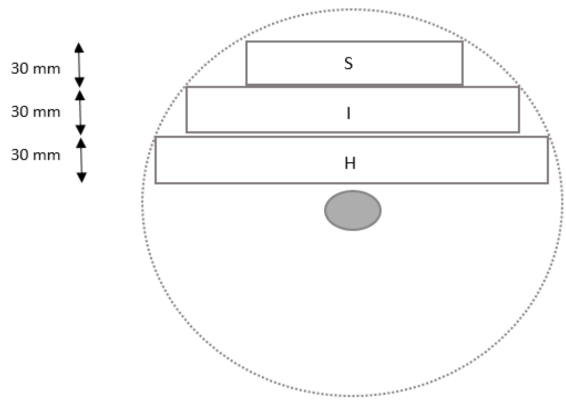

Figure 1: Position of the boards in the eucalypt log.

Thermal modification

The eucalypt boards were thermally modified according to the Brazilian industrial process VAP HolzSysteme®, at the TW Brazil Company. The boards were divided in four batches corresponding to the control samples and each of the final cycle temperature of thermal modification, i.e. 140, 160, 180 º C. The exact schedule of the thermal treatment is proprietary information, but a general description of the process can be found in Batista *et al.* (2015).

Sample preparation

The research was designed in 12 groups, resulted from the combination of the three groups of boards (*S, I* and *H*) and the four treatments (control, 140, 160 and 180 °C). After the thermal modification of the eucalypt boards, six samples of 70 x 25 x 5 mm^3 with clear transverse, radial and tangential faces were prepared from each of the 12 groups of boards thermally treated, which makes a total of 72 samples. The samples were all hand-sanded with a 150-grit sandpaper for a uniform surface.

To prevent colour changes and/or photo-oxidation of surfaces the samples were placed in a conditioned room at 20 ºC and 65 % relative humidity until the start of the experiments.

Colour measurements

The colour of the eucalypt samples was measured with a Konica Minolta CM-5 spectrophotometer. Ten points per sample were measured on the radial surfaces to have an objective characterisation of the samples colour, before and after thermal modifications. The wood colour was determined according to the CIE 1976 $L^*a^*b^*$ Colour space (ISO 11664-4-2008) using CIE standard illuminates D65 and a 10º standard observer. In the three-dimensional CIE $L^*a^*b^*$ Colour space, each colour is expressed as a point in the Euclidean space defined by three main parameters: the vertical coordinate for lightness (L^*) representing the position on the black-white axis, and the chromatic coordinates a^* (green/red) and b^* (blue/yellow) representing the position on the horizontal plane (González-Peña and Hale 2009). The values of colour saturation chroma (C^*) and hue angle (h) were calculated using the following equation 1 and 2:

$$C^* = \sqrt{(a^*)^2 + (b^*)^2} \qquad (1)$$

$$h = arctan\left(\frac{b^*}{a^*}\right) \qquad (2)$$

The values of the parameters *L**, *a** and *b** were used to calculate the colour change (*ΔE**) according to the equation 3 bellow:

$$\Delta E^* = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \qquad (3)$$

Where *ΔL**, *Δa** and *Δb** are the colorimetric differences between two different states of the samples.

Statistical analysis

The analysis of variance (ANOVA) was performed to identify differences between the samples. The experimental design was completely randomized, with a 99 % confidence level for all tests. If a statistically significant difference was identified between averages, the Tukey's multiple range test was performed to determine which averages were different.

RESULTS AND DISCUSSION

Quantification of colour of untreated and thermally modified wood samples – Unclassified

Figure 2 shows the effects of three thermal modification intensities on the surface colour of the eucalypt wood samples, regardless the classification by groups. All thermally modified samples have presented a noticeable process of aesthetic modification, specially at 180 ºC. By visual inspection it is possible to observe significantly different colours between the untreated and thermally modified samples. As the temperature of thermal modification increased the wood surface colour turned darker. While the untreated samples of *Eucalyptus grandis* wood present light-yellow to pale pink colour tone, the thermally modified samples at the three different final temperatures have presented the following perceived colours: at 140 ºC a pink to dark-pink surface colour, at 160 ºC a light-brown colour and at 180 ºC a dark-brown colour surface.

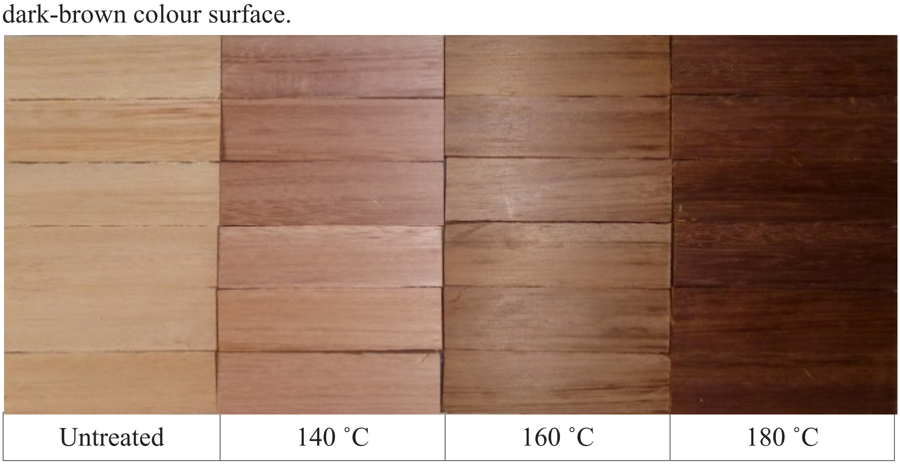

Figure 2: Colour variation of the samples according to the temperature of thermal modification.

Table 1 shows the results of the quantitative analysis obtained with CIE *L*a*b** Colour space of untreated and thermally modified *E. grandis* samples, and the colour change (*ΔE**).

The untreated samples have presented high mean value for lightness, i.e. *L** = 70, therefore this species can be classified as a light colour wood, according to Camargos 1999. Despite the important role of the coordinate *a** (red colour tone) on the wood colour composition, the coordinate *b** (yellow colour tone) is the main responsible component for the perceived aspect of this wood.

The values of the parameter *L** (lightness) decreased with the increase of the thermal treatment temperatures. The reduction of *L** was of 14,5 % at 140 °C; 37,6 % at 160 °C and almost 50 % at 180 °C. The reduction of the lightness index (*L**) is directly related to the darkening of the wood colour. The changes on the wood exposed to heating involves reduction in the polymerization degree of cellulose by bond breaking, elimination of water, formation of free radicals and finally, formation of carbonaceous char that darkens the wood surface (Mitsui *et al.* 2004, Sundqvist 2004, Hill 2006, Esteves *et al.* 2008, Moura and Brito 2011).

At 140 °C the values of *a** (red colour tone) increased 18,5 %, resulting in wood samples with pinkish colour tone surfaces, noticeable by visual inspection. As thermal modification intensity increased, at 160 and 180 °C, the eucalypt samples tended to present a less reddish colour. This trend has also been observed in studies with *Eucalyptus grandis* by Zanuncio *et al.* (2014), Cademartori *et al.* (2013) and Moura and Brito (2011) for different thermal treatment process. The reduction of red colour tone in the thermally modified wood is associated with the volatilization of phenolic extractives that confer a red colour to the eucalypt wood (Pincelli *et al.* 2012).

The effects of thermal modification on the values of the coordinate *b** (yellow colour tone) have been observed in three stages: a) reduction of the mean value at 140 °C; b) light recovering at 160 °C and c) second more intensive reduction at 180 °C. The decrease of yellow colour tone (*b**) at 140 °C and increase of this coordinate at temperatures above 140 °C up to 180 °C has been reported for the *Eucalyptus saligna* wood by Pincelli *et al.* (2012). On the other hand, a progressively reduction of yellow colour tone (*b**) for treatment temperatures above 140 °C up to 200 °C were reported by Moura and Brito (2011) for the thermally modified *Eucalyptus grandis* wood. The yellowness of the wood is primarily related to the photochemistry of the major wood components, especially lignin (Gierlinger *et al.* 2004).

The reduction of the values of coordinates *a** and *b** contributed to the colour darkening of the wood. At 180 °C, the coordinate *b** presented the lowest mean value. At this temperature range the darkening of the wood surface became more pronounced and this effect is due to the photo degradation of phenolic constituents of wood extracts (Ishiguri *et al.* 2003).

Based on the observed values of the chroma parameter (*C**) this species presents a medium saturation colour. The thermal treatment at 140 ºC, did not significantly influence the chroma parameter (*C**) and the wood maintained the same level of colour saturation than as when untreated. The required treatment intensity to significantly decrease chroma was for temperatures of 160 and 180 ºC, when the colour changes perceived were more noticeable.

The hue angle (*h*), which is calculated as a function of the coordinates *a** and *b**, assists in defining the colour. The higher the value of *h* (up to 90 º) the closer the colour approaches to the axis of coordinate *b**, indicating higher yellow colour tone. The reduction of *h* due to the thermal treatment represents a substitution of yellow tones to red tones. The reduction of the hue angle was not statistically significant for samples treated at 160 and 180 ºC and at 140 ºC the hue angle was closer to the red colour tone.

Table 1: Mean colour data of unclassified untreated and thermally modified *Eucalyptus grandis* wood samples.

Treatments	*L**	*a**	*b**	*C**	*h*	*ΔE**
Untreated	70,63 a (3,92)	10,81 c (14,32)	18,23 a (6,19)	21,22 b (7,39)	59,44 a (5,06)	
140 °C	60,40 b (6,23)	12,81 a (9,93)	16,94 b (8,28)	21,29 b (5,82)	52,87 c (7,34)	**11,32 (35,71)**
160 °C	44,08 c (7,55)	12,21 b (4,87)	18,15 a (15,03)	21,92 a (11,03)	55,71 b (6,58)	**26,77 (16,50)**
180 °C	36,86 d (10,91)	9,60 d (14,67)	14,07 c (20,30)	17,04 c (18,37)	55,36 b (3,80)	**34,28 (15,97)**
F-Ratio	689,48*	469,52*	288,56*	367,66*	252,26*	**465,30***

*Significant at p<0,01 level. Means followed by the same lower-case letter per column do not differ between them by the Tukey test. Coefficient of variation in % between brackets.

At Table 1 the colour change (*ΔE**) was used to assess the colorimetric variation between the untreated and the thermally modified samples. As expected, the *ΔE** increased as the temperature of thermal modification has raised. This trend was previously identified by visual inspection of the wood samples, which were darkened by the thermal modification. The highest colour change, between untreated and thermally treated wood, were observed at 180 ºC. The high values of colour change confirm the significant effects caused by the thermal modification.

Quantification of colour of untreated and thermally modified wood samples – Classified by groups

Table 2 shows the results of the quantitative analysis of colour obtained with CIE *L*a*b** Colour space of untreated and thermally modified *E. grandis* samples, classified by groups. Table 2 also shows the colour change (*ΔE**), calculated to assess how each group of classified samples differed from the mean colour data of the unclassified samples, for each corresponding treatment presented at Table 1.

Table 2: Mean colour data of untreated and thermally modified *Eucalyptus grandis* wood by groups: sapwood (S), internal (I) and heartwood (H).

Treatments	Groups	*L**	*a**	*b**	*C**	*h*	*ΔE**
Untreated	S	70,65 ab (2,69)	10,91 c (7,26)	17,35 c (4,23)	20,51 d (4,05)	57,86 c (3,32)	0,89
	I	69,29 b (4,69)	11,89 b (14,21)	19,29 b (5,39)	22,69 b (7,35)	58,51 c (4,77)	**2,02**
	H	71,96 a (3,22)	9,64 d (11,25)	18,04 c (2,93)	20,47 d (4,09)	61,95 a (3,95)	1,78
140 °C	S	58,23 d (3,64)	13,11 a (3,95)	15,20 d (3,19)	20,08 d (1,72)	49,23 h (3,59)	**2,80**
	I	64,23 c (5,53)	12,00 b (15,08)	17,98 c (4,87)	21,66 c (6,64)	56,43 d (6,34)	**4,05**
	H	58,76 d (3,16)	13,34 a (4,18)	17,66 c (3,05)	22,14 bc (1,52)	52,93 g (3,42)	1,87
160 °C	S	41,60 g (3,81)	12,08 b (5,45)	15,49 d (7,13)	19,65 d (6,29)	52,01 g (2,02)	**3,64**
	I	43,75 f (6,97)	12,38 b (4,94)	18,03 c (11,24)	21,89 bc (8,52)	55,33 e (4,82)	0,39
	H	46,90 e (5,76)	12,15 b (3,74)	20,92 a (7,18)	24,20 a (6,09)	59,79 b (2,16)	**3,95**
180 °C	S	36,28 h (7,03)	9,65 d (14,41)	14,10 e (19,97)	17,10 e (18,08)	55,26 ef (3,83)	0,58
	I	36,61 h (16,52)	9,32 d (19,05)	13,13 f (25,14)	16,11 f (23,05)	54,25 f (3,42)	1,01
	H	37,53 h (7,56)	9,83 d (8,89)	14,97 d (13,49)	17,92 e (11,95)	56,57 d (2,92)	1,15
F-Ratio		2566,28*	190,73*	221,55*	197,20*	308,90*	

*Significant at p<0,01 level. Means followed by the same lower-case letter per column do not differ between them by the Tukey test. Coefficient of variation in % between brackets.

Untreated *Eucalyptus grandis* samples have shown statistically significant differences in colour among the three classified groups.

The untreated samples from the group S have shown colorimetric mean values similar to the unclassified untreated samples, but with a significant reduction of the intra-group colour variation.

The untreated samples from the internal group (I) have presented the lowest lightness (L^* = 69,29) while showed the highest red colour tone (a^* = 11,89), yellow colour tone (b^* = 19,29) and chroma (C^* = 22,69). This group presented higher percentage of coefficient of variation than the others, for all the colour parameters, revealing a notable intra-group colour variation, similar to the one presented by the untreated unclassified samples.

The untreated samples from the group H presented the highest mean value for lightness (L^* = 71,96) and hue angle (h = 61,95), while they presented the lowest mean value for red colour tone (a^* = 9,64) and chroma (C^* = 20,47). This group presented lower percentage of coefficient of variation than the untreated unclassified samples from Table 1, what represent a reduction of the intra-group colour variation.

The pronounced colour lightness at the heartwood group (H), can be explained by the presence of younger cells at this region, therefore shorter, with thinner walls and less lignified. This effect was previously confirmed by similar data reported by Gonçalez and Macedo (2003). In great measure, the colour of this group contrasts to others mainly due to the statistically significant differences in hue angle, which is the parameter where deviations are most perceived by human vision (Malacara 2002).

Among the three groups of untreated samples, the coordinate a^* (red colour tone) presented an increasing trend from the wood pith to bark, similar to the results reported by Gonçalez and Macedo (2003) for the same wood species.

When thermally treated at 140 ºC the three groups of samples presented a decrease of lightness and yellow colour tone and an increase of red colour tone. The increase of red colour tone, regarding the untreated unclassified wood, was of 20 % for the group S, 38 % for group H and a small increase of 0,95 % for the group I. At this temperature, the pinkish hue, caused by the increase of the red parameter, was more evident and uniform on samples from groups S and H. In addition, thermally treated samples from groups S and H presented mean values not statistically different of lightness (L^*) and red colour tone (a^*). The internal group (I) was the group that presented the highest difference of colour in respect to unclassified samples, at two cases: for untreated samples (ΔE^* = 2,02), and for samples treated at 140 ºC (ΔE^* = 4,05).

The thermally treated samples at 160 °C showed an increase of colour lightness on the radial direction, from bark to the wood pith. The samples also presented a small increase of red colour tone (a^*), and the mean values of this parameter were not statistically different among the three groups. However, at this temperature, the groups S and I showed a reduction of yellow colour tone (b^*) in respect to untreated unclassified samples of 11 % and 6,5 %, respectively, while the group H showed an increase of 16 % for the same parameter. Samples from group H thermally treated at 160 ºC presented higher mean values of colour lightness, yellow colour tone, chroma and hue angle than the other two groups. Opposing to the two treatments commented previously, at 160 ºC the internal group (I) showed the lower colour difference (ΔE^* = 0,39), which means that the classification didn't have a significant impact on the colour of this group at this temperature.

At 180 °C, all colorimetric parameters presented a very noticeable reduction, however, a significant increase of the percentage of coefficient of variation was observed. At this temperature, the mean values of colour lightness (L^*) and red colour tone (a^*) were not statistically different among the three groups. And the internal group (I) presented the higher intra-group colour variation.

The samples thermally modified at 180 ºC from the three groups, when assessed by visual inspection presented a largely uniform colour, despite of the high percentages of coefficient of variation. The thermal modification at this temperature produced a pronounced darkening of the wood colour that have contributed to dissimulate the intra-group colour variation. Therefore, the classification of samples in groups didn't produced a significant impact on the colour variation of the thermally treated samples at 180 ºC.

CONCLUSIONS

All thermally modified samples, by the three temperatures, showed a significant colour change, confirmed by high values of ΔE*. At 140 ºC the eucalypt samples presented a significant increase of red colour tone (a*), at 160 ºC the levels of yellow colour tone (b*) were similar to untreated wood and at 180 ºC a reduction of 50 % of colour lightness (L*) was observed.

Untreated Eucalyptus grandis samples have shown statistically significant colour differences among the three classified groups. An increasing trend for the red colour tone (a*) was identified on the radial direction of the wood. And the classified samples at the H group presented higher colour lightness and low intra-group colour variation.

The classification of eucalypt samples has allowed the identification of different colour responses of the groups to thermal treatments at 140 and 160 ºC. However, at 180 ºC, the samples didn't show a significant variation in the colour responses among the groups.

These results have shown that the light yellow colour of the untreated Eucalyptus grandis wood can be darkened and homogenised by this industrial process of thermal modification. It has also shown that classifying the eucalypt samples in groups, according to their distance to the wood pith, allows to reduce intra-group colour variation and, therefore, to achieve a better wood quality for aesthetics applications that require a low tolerance of colour variation.

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