### Bruno Esteves, Umit Ayata, Levent Gurleyen

# EFFECT OF HEAT TREATMENT ON THE COLOUR AND GLOSSINESS OF BLACK LOCUST, WILD PEAR, LINDEN, ALDER AND WILLOW WOOD

In this study, samples of wood from black locust (Robinia pseudoacacia Lipsky), wild pear (Pyrus communis L.), linden (Tilia platyphyllos), alder (Alnus glutinosa L. Gaertn.) and willow (Salix alba) were treated at 212°C for 1 hour and 2 hours according to the ThermoWood heat treatment procedure at the Novawood Factory in Gerede, Bolu (Turkey). Colour and glossiness were tested and compared with untreated samples. The results showed that overall, lightness decreased, while the behaviour of a\* and b\* values depended on the species and on the initial colour. Differences in lightness and a\* between species were equalized by heat treatment. Gloss at 20° and 60° angles decreased with heat treatment for both perpendicular and parallel directions, while at 85° an equilibrium was reached between the decrease in gloss due to the heat treatment and the increase due to the lower roughness of the heat-treated surface.

Keywords: ThermoWood, heat treatment, glossiness, colour

# Introduction

Wood modification was introduced many decades ago with studies by Stamm et al. [1946], but in the last decade there has been a substantial increase in studies on modified wood. Heat treatment has been the most studied wood modification procedure, with several processes attaining commercial status, including ThermoWood<sup>®</sup>, Platowood<sup>®</sup> and Perdure<sup>®</sup>. The main objective of heat treatment has been to increase dimensional stability [Tjeerdsma et al. 1998; Bekhta and Niemz 2003; Srinivas and Pandey 2012; Esteves et al. 2014] and durability against decay [Kim et al. 1998; Tjeerdsma et al. 2002; Boonstra et al. 2007; Metsä-Kortelainen and Viitanen 2009; Dubey 2010], although other

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properties such as reduced thermal conductivity [Kol and Sefil 2011] or colour homogeneity [Johansson and Morén 2006] throughout the wood surface are advantageous for some types of products. Usually the treatment is performed on low-value species to increase their commercial value and allow their utilization in products that require better dimensional stability and durability. In the last few years some studies have considered changes in the colour of the surface of higher-value species, with the aim of using them as indoor flooring materials, avoiding the use of stains, varnishes or other chemical finishing.

Colour is one of the most important properties for consumers, and the possibility of changing the natural colour of wood without chemicals is important for some markets. Colour measurements are usually made by the CIE L\*a\*b\* method, which uses a three-dimensional space with coordinates L\*, called lightness, and a\* and b\* representing green-red and blue-yellow tones respectively. The colour changes due to various heat treatment processes have been extensively studied for several species. All of the tests have shown that lightness decreases with heat treatment and that the decrease is greater for harsher treatments [Bekhta and Niemz 2003; Aksoy et al. 2011; Srinivas and Pandev 2012: Barcík et al. 2015]. Moreover, the use of a shielding gas has been shown to reduce the decrease in lightness. For instance, Esteves et al. [2007] treated a softwood (Pinus pinaster Ait) and a hardwood (Eucalyptus globulus Labill) by two different heat treatment methods, with and without steam as shielding gas, and concluded that with the heat treatment the wood became darker. L\* decreased more under treatment without shielding gas, and under the same treatment conditions (with or without steam) the decrease was greater for eucalyptus wood. Changes in the chroma coordinates a\* and b\* depended on the initial colour of the samples and on the species undergoing heat treatment. Results have shown that heat treatment intended to produce colour changes has to be tailored to the particular species [Ayata et al. 2017b; Gurleyen et al. 2017].

The reasons for the colour changes have been studied by several authors. Amongst others, degradation products from wood polymers and extractive compounds have been identified as responsible for colour formation during hydrothermal treatment [Sundqvist and Morén 2002]. Similarly, González-Peña and Hale [2009] report that colour changes are due to chemical changes in the main wood polymers, more in lignin than in polysaccharides, due to the darkening of lignin itself. This is associated with the generation of chromophoric groups, mostly attributed to the appearance of quinone species.

Colour has also been associated with the possibility of predicting properties of heat-treated wood [Brischke et al. 2007]. Strong correlations between the total colour change and modulus of elasticity and bending strength were reported by Bekhta and Niemz [2003] and also by Todorovic et al. [2012] with R<sup>2</sup> reaching 0.76 for MOR. However, Johansson and Morén [2006] stated that colour is not a useful parameter for prediction of strength. The worst results were achieved for impacted bending, where the correlation was too small to give useful results.

Glossiness is important for some products used indoors, since it affects the dispersion of light in the room. Heat treatment reduces gloss for all species, although the decrease depends on the species and on the initial gloss of the samples, as reported previously [Aksoy et al. 2011; Karamanoglu and Akyildiz 2013; Gurleyen et al. 2017].

This paper reports the changes in colour (L\*, a\*, b\*,  $\Delta E^*$ ) and glossiness (parallel and perpendicular to the grain at 20°, 60° and 85°) for ThermoWood--treated black locust, wild pear, linden, alder and willow, with a view to their use for interior applications. The treatment will enable the production of different colours according to consumers' preferences, without the application of any varnishes or stains on the wood surface.

## Materials and methods

### Wood materials

Wood from the species black locust (*Robinia pseudoacacia* Lipsky), wild pear (*Pyrus communis* L.), linden (*Tilia platyphyllos*), alder (*Alnus glutinosa* L. Gaertn.) and willow (*Salix alba*) were obtained from suppliers of forest products in Duzce, Turkey. Ten samples measuring 100 mm by 100 mm by 10 mm were used for colour and glossiness measurements. All samples were conditioned at 20 ( $\pm$ 2)°C and 65% ( $\pm$ 5%) RH (relative humidity) before testing (ISO 554:1976).

### Heat treatment

The ThermoWood<sup>®</sup> process was applied according to Viitaniemi et al. [2001]. The treatment was performed at 212°C for 1 and 2 hours at the commercial Novawood factory in Gerede, Bolu (Turkey). After treatment all samples were conditioned at 20 ( $\pm$ 2)°C and relative humidity 65% ( $\pm$ 5%) before colour and glossiness measurements.

### Colour

The colour of the tangential surface of heat-treated and control samples was obtained by an X-Rite Ci62 Series Portable Spectrophotometer made in Switzerland (wavelength resolution 10 nm, measurement geometry D/8°) with a D65 standard illuminant. The CIELAB system, characterized by the three axes L\*, a\*, and b\*, was used. The L\* axis represents lightness, varying from 100 (white) to zero (black); a\* is the red(+)-green(-) tone; and b\* is the yellow(+)--blue(-) tone. The corresponding changes  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  on heat treatment were calculated using heat-treated and control samples from the equations:

 $\Delta L^* = L^*$ heat-treated – L\*reference,

 $\Delta b^* = b^*$ heat-treated – b\*reference,

 $\Delta a^* = a^*heat$ -treated  $-a^*reference$ .

The total colour difference ( $\Delta E^*$ ) was calculated by:

 $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2] 1/2.$ 

#### Glossiness

Measurement of the glossiness of the tangential surface of heat-treated and control test specimens was performed in a Gloss Meter Poly Gloss GL0030 TQC (Netherlands) in parallel and perpendicular directions at 20°, 60° and 85° according to the ISO 2813:1994 standard.

#### Statistical analysis

SPSS 17 software (Sun Microsystems, Inc.; 4150 Network Circle, Santa Clara, California 95054, U.S.A.) was used for ANOVA and Duncan values. Glossiness and colour parameters were measured using ten replicates of each sample, and an average value was reported.

### **Results and discussion**

Colour parameters determined for untreated and heat-treated wood at 212°C for 1 h and 2 h are presented in table 1. Initial lightness is highest for willow (81.4), followed by linden (81.1), black locust (61.3), alder (61.1) and wild pear (59.9). After heat treatment all samples exhibited lower lightness than the untreated wood. The greatest differences are seen between untreated and heat-treated wood, although overall a longer treatment (2 h) leads to lower lightness than shorter treatments (1 h) except in the case of linden. The lightness differences between species are much greater for untreated wood (59.9-81.4) than for heat--treated wood (36.0-41.7), showing that heat treatment equalizes lightness between species. Similar results have been reported previously, for instance: for Scots pine [Aksoy et al. 2011], for Pinus pinaster and Eucalyptus globulus [Esteves et al. 2007] and for the exotic wood species afrormosia, doussie, frake and iroko [Ayata et al. 2017a]. Changes in a\* were different for each species, increasing for black locust, linden and willow, but decreasing for wild pear and alder. This different behaviour is possibly due to the initial value of a\*, since for all of the samples after heat treatment (2 h) a\* is in the range 9.5-11.3. The largest changes are therefore found in the linden samples, which had the lowest a\* before the treatment (3.8). Similar results were reported by Ayata et al. [2017a] for afrormosia, doussie, frake and iroko, and by Torniainen et al. [2015] for pine (Pinus sylvestris L.) and spruce (Picea abies L.), where following Thermo D treatment both species took a\* values in the range 9.8-10.2.

The value of b\* (yellow-blue tone) for the control samples is highest for black locust, followed by linden and alder, wild pear and willow. The variation of b\* with heat treatment depends on the species and on the initial value. A decrease after 1 h of treatment followed by an increase after 2 h of treatment is observed for black locust and linden, while for wild pear and alder there is a decrease in the value with the intensity of the treatment. In the case of willow, there is an increase in b\* followed by a decrease.

Wood species Duration		L* mean	a* mean	b* mean
Black locust	Control	61.25 (0.55)	9.14 (0.47)	27.16 (0.77)
(Robinia pseudoacacia	212°C – 1 hour	41.44 (1.27)	10.98 (0.76)	19.20 (1.54)
Lipsky)	$212^{\circ}C - 2$ hours	40.07 (0.58)	11.28 (0.23)	19.85 (0.50)
***** 1	Control	59.88 (0.67)	13.55 (0.22)	21.90 (0.22)
Wild pear (Purus communis I)	212°C – 1 hour	37.83 (0.69)	10.70 (0.22)	15.58 (0.74)
(1 yrus communis L.)	$212^{\circ}C - 2$ hours	36.00 (0.50)	9.50 (0.15)	12.99 (0.43)
** 4	Control	81.05 (1.40)	3.82 (1.03)	23.25 (1.55)
Linden (Tilia platyphyllos)	212°C – 1 hour	39.47 (0.86)	9.65 (0.21)	18.41 (0.70)
(1111a platyphytios)	$212^{\circ}C - 2$ hours	41.81 (0.88)	10.34 (0.20)	20.63 (0.62)
Alder	Control	61.08 (0.92)	11.54 (0.91)	22.90 (1.78)
(Alnus glutinosa L.	212°C – 1 hour	44.85 (1.11)	11.35 (0.29)	21.92 (1.21)
Gaertn.)	$212^{\circ}C - 2$ hours	40.99 (0.83)	10.69 (0.36)	18.88 (0.72)
	Control	81.39 (0.46)	5.48 (0.27)	15.66 (0.70)
Willow	212°C – 1 hour	44.12 (0.81)	10.59 (0.22)	20.20 (0.50)
(Salix alba)	$212^{\circ}C - 2$ hours	41.73 (0.43)	10.01 (0.18)	17.94 (0.38)

Table 1. L\*, a\* and b\* for untreated wood and wood subjected to heat treatment at 212°C for 1 h and 2 h

Values in parentheses are standard deviations. \*Highest value.

Figure 1 shows a graphic representation of the CIE L\*a\*b\* space for untreated and heat-treated woods, where a\* is shown on the x-axis, b\* is shown on the y-axis, and the size of the balls represents lightness (L\*). This representation clearly shows that there is a higher dispersion in the colour parameters of the control samples while there is a higher concentration of values in heat-treated wood, again confirming that heat treatment equalizes the initial colour differences between species. Furthermore there is also greater homogeneity in each sample, due to the darker colour that masks many blemishes and discolorations, and throughout the thickness of the board, which allows further machining of the wood without compromising the colour of the surface, as has been stated previously [González-Peña and Hale 2009].

Two-way ANOVA (table 2) was performed to determine whether there is a significant difference between species and heat treatments. The results show that L\*, a\* and b\* values are significantly different for wood species (A), heat treatment (B) and interaction (AB), at the selected level of significance (p < 0.05). A significant interaction effect means that the effect of one factor depends on the other factor. In this case the effect of heat treatment on colour varies with species. This confirms that, in order to impart a darker colour to wood, the heat treatment should be tailored to each species, as has been reported previously [Ayata et al. 2017a].



● Control ● 1 h treatment ● 2 h treatment

Fig. 1. CIE L\*a\*b\* space, where L\* is represented by the size of the balls

Test	Source	Type sum of squares	df	Mean square	F	Sig.
	Wood species (A)	2583.420	4	645.855	901.937	0.000*
	Heat treatment (B)	26372.009	2	13186.005	18414.266	0.000*
	Interaction (AB)	3051.415	8	381.427	532.663	0.000*
$L^*$	Error	96.670	135	0.716		
	Total	410050.282	150			
	Corrected total	32103.515	149			
	Wood species (A)	273.979	4	68.495	310.395	0.000*
	Heat treatment (B)	110.529	2	55.265	250.440	0.000*
<b>*</b>	Interaction (AB)	420.262	8	52.533	238.061	0.000*
u.	Error	29.790	135	0.221		
	Total	15555.670	150			
	Corrected total	834.561	149			
	Wood species (A)	614.123	4	153.531	172.889	0.000*
	Heat treatment (B)	460.625	2	230.313	259.352	0.000*
1 *	Interaction (AB)	657.974	8	82.247	92.617	0.000*
<i>D</i> +	Error	119.884	135	0.888		
	Total	60447.333	150			
	Corrected total	1852.607	149			

Table 2. Two-way	<b>ANOVA</b> for	L*,	a*	and b*
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Table 3 shows the Duncan homogeneous groups for  $L^*$ ,  $a^*$  and  $b^*$  values. The results show that both wood species and treatment duration have a significant effect on lightness, and the same is true for the effect of treatment duration on  $a^*$  and  $b^*$ . Although, generally, wood species has a significant effect on  $a^*$  and  $b^*$ , wild pear and alder have statistically similar  $a^*$ , and alder and linden have statistically similar  $b^*$ .

W/ 1	N	L*		a'	*	b*	
wood species		mean	HG	mean	HG	mean	HG
Wild pear (Pyrus communis L.)	30	44.57	Е	11.25	A*	16.82	D
Black locust <i>(Robinia pseudoacacia</i> Lipsky)	30	47.58	D	10.47	В	22.07	A*
Alder (Alnus glutinosa L. Gaertn.)		48.97	С	11.19	А	21.23	В
Linden (Tilia platyphyllos)		54.11	В	7.94	D	20.76	В
Willow (Salix alba)	30	55.75	A*	8.69	С	17.93	С
Duration	N	L	*	a'	*	b	*
Duration		mean	HG	mean	HG	mean	HG
Control	50	68.93	A*	8.70	С	22.17	A*
212°C – 1 hour	50	41.54	В	10.65	A*	19.06	В
212°C – 2 hours	50	40.12	С	10.36	В	18.06	С

Table 3. Duncan homogeneity groups for L\*, a\* and b\* values

Number of measurements, HG: Homogeneous group, \*Highest value.

Table 4 gives the variations between untreated and heat-treated wood and the total colour change for the tested species. The total colour ( $\Delta E^*$ ) is in the range 16.3-42.26. The highest  $\Delta E^*$  was found for linden and the lowest for alder, and the same was true for  $\Delta L^*$ . This shows that  $\Delta E^*$  is highly dependent on changes in L\*, since these are much greater than the changes in a\* and b\*.

Along with colour, gloss is one of the most important aesthetic properties, particularly in terms of consumer preferences. Table 5 presents the results of perpendicular and parallel gloss measurements in untreated wood and wood that has undergone heat treatment for 1 h and 2 h, at the angles 20°, 60° and 85°. The initial gloss of the samples at the respective angles lay in the ranges 1.41-2.55, 2.62-3.95 and 1.07-3.06 for perpendicular gloss and 1.35-2.37, 3.00-4.65 and 0.99-5.58 for parallel gloss. Although according to ISO 2813:1994 the correct angle for measuring gloss is 85° (since at 60° the quantity of gloss units is less than 10), all three angles were tested. Gloss at 20° decreased with heat treatment for both perpendicular and parallel directions. The largest differences were found

Wood species	Duration	$\Delta L^*$	∆a*	$\Delta b^*$	$\Delta E^*$
Black locust <i>(Robinia pseudoacacia</i> Lipsky)	212°C – 1 hour	-19.81	1.84	-7.96	21.43
	212°C – 2 hours	-21.18	2.14	-7.31	22.51
Wild pear (Pyrus communis L.)	$212^{\circ}C - 1$ hour	-22.05	-2.85	-6.32	23.11
	$212^{\circ}C - 2$ hours	-23.88	-4.05	-8.91	25.81
Linden (Tilia platyphyllos)	212°C – 1 hour	-41.58	5.83	-4.84	42.26
	212°C – 2 hours	-39.24	6.52	-2.62	39.86
Alder (Alnus glutinosa L. Gaertn.)	212°C – 1 hour	-16.23	-0.19	-0.98	16.26
	212°C – 2 hours	-20.09	-0.85	-4.02	20.51
Willow (Salix alba)	212°C – 1 hour	-37.27	5.11	4.54	37.89
	212°C – 2 hours	-39.66	4.53	2.28	39.98

Table 4. Colour changes due to heat treatment for 1 h and 2 h

between untreated and heat-treated wood; the differences between 1 h and 2 h of treatment were small or non-existent. For instance, there is a small decrease for black locust and alder in the perpendicular and parallel directions, an increase for willow ( $\perp$ ) and for linden (||), but in most cases, as with wild pear and linden ( $\perp$ ) and wild pear and willow (||), the samples have similar gloss. A decrease in glossiness has previously been reported for several species, including Scots pine [Aksoy et al. 2011; Gurleyen et al. 2017], wild cherry [Korkut et al. 2013], Anatolian black pine, Calabrian pine, sessile oak and chestnut [Karamanoglu and Akyildiz 2013], and afrormosia, doussie, frake and iroko [Ayata et al. 2017a].

Glossiness at a  $60^{\circ}$  angle is similar to that at  $20^{\circ}$ , although the decrease on heat treatment is smaller. Wild pear even exhibited an increase in glossiness in the perpendicular and parallel directions. This angle is usually the reference angle, since it is less influenced by the direction of measurement and the natural features of the wood substrate [Scrinzi et al. 2011].

At the highest angle  $(85^{\circ})$  there is no consistent variation for all of the species. In some cases there is a decrease (black locust  $\pm 85^{\circ}$ ), in others an increase (wild pear and willow  $\pm$  and ||), and in the remainder a decrease followed by an increase. This is probably due to differences in surface roughness between treated and untreated wood. Several authors have stated that heat treatment leads to a large decrease in surface roughness, and that this decrease is greater for longer treatments [Kamdem et al. 2002; Korkut et al. 2008; Korkut and Guller 2008; Unsal and Ayrilmis 2005]. At the 85° angle the surface grains are highlighted, with consequent lower gloss values, as reported previously [Scrinzi et al. 2011]. Therefore, there is an equilibrium between the decrease in gloss due to the heat treatment and the increase due to the lower roughness of the heat-treated surface.

Wood species Duration		⊥20° mean	⊥ 60° mean	$\pm 85^{\circ}$ mean
Black locust <i>(Robinia pseudoacacia</i> Lipsky)	Control 212°C – 1 hour 212°C – 2 hours	1.41 (0.03) 0.82 (0.06) 0.70 (0.00)	2.95 (0.23) 2.11 (0.43) 1.88 (0.14)	3.06 (0.54) 2.08 (0.42) 1.40 (0.16)
Wild pear (Pyrus communis L.)	Control 212°C – 1 hour 212°C – 2 hours	1.50 (0.05) 0.83 (0.12) 0.84 (0.21)	2.62 (0.09) 2.77 (0.37) 3.31 (0.99)	1.93 (0.08) 2.69 (0.47) 3.66 (0.86)
Linden (Tilia platyphyllos)	Control 212°C – 1 hour 212°C – 2 hours	2.55 (0.10) 0.82 (0.09) 0.85 (0.05)	3.38 (0.25) 1.75 (0.21) 1.73 (0.18)	1.07 (0.09) 1.01 (0.17) 1.03 (0.11)
Alder (Alnus glutinosa L. Gaertn.)	Control 212°C – 1 hour 212°C – 2 hours	1.63 (0.05) 1.14 (0.05) 0.94 (0.05)	3.95 (0.19) 2.80 (0.32) 2.53 (0.24)	2.25 (0.21) 1.76 (0.33) 2.34 (0.33)
Willow (Salix alba)	Control 212°C – 1 hour 212°C – 2 hours	2.39 (0.11) 1.08 (0.08) 1.30 (0.08)	3.30 (0.17) 3.09 (0.53) 3.58 (0.19)	1.32 (0.09) 1.74 (0.49) 2.21 (0.10)
Wood species	Duration	20° mean	60° mean	85° mean
Wood species Black locust (Robinia pseudoacacia Lipsky)	Duration Control 212°C – 1 hour 212°C – 2 hours	20° mean 1.35 (0.07) 0.76 (0.05) 0.65 (0.05)	60° mean 3.38 (0.30) 3.11 (0.19) 2.71 (0.38)	85° mean 4.04 (0.94) 5.88 (0.56) 4.01 (1.06)
Wood species Black locust (Robinia pseudoacacia Lipsky) Wild pear (Pyrus communis L.)	Duration Control 212°C – 1 hour 212°C – 2 hours Control 212°C – 1 hour 212°C – 2 hours	20° mean 1.35 (0.07) 0.76 (0.05) 0.65 (0.05) 1.43 (0.05) 0.71 (0.13) 0.67 (0.13)	60° mean 3.38 (0.30) 3.11 (0.19) 2.71 (0.38) 3.00 (0.18) 3.63 (0.36) 3.10 (0.81)	85° mean 4.04 (0.94) 5.88 (0.56) 4.01 (1.06) 4.68 (1.15) 8.74 (1.47) 8.48 (3.78)
Wood species Black locust (Robinia pseudoacacia Lipsky) Wild pear (Pyrus communis L.) Linden (Tilia platyphyllos)	Duration Control $212^{\circ}C - 1$ hour $212^{\circ}C - 2$ hours Control $212^{\circ}C - 1$ hour $212^{\circ}C - 2$ hours Control $212^{\circ}C - 1$ hour $212^{\circ}C - 1$ hour $212^{\circ}C - 1$ hour $212^{\circ}C - 1$ hour $212^{\circ}C - 1$ hour	20° mean 1.35 (0.07) 0.76 (0.05) 0.65 (0.05) 1.43 (0.05) 0.71 (0.13) 0.67 (0.13) 2.37 (0.07) 0.65 (0.05) 0.81 (0.09)	60° mean 3.38 (0.30) 3.11 (0.19) 2.71 (0.38) 3.00 (0.18) 3.63 (0.36) 3.10 (0.81) 3.81 (0.17) 2.20 (0.21) 2.29 (0.26)	85° mean 4.04 (0.94) 5.88 (0.56) 4.01 (1.06) 4.68 (1.15) 8.74 (1.47) 8.48 (3.78) 0.99 (0.07) 1.64 (0.41) 1.41 (0.30)
Wood species Black locust (Robinia pseudoacacia Lipsky) Wild pear (Pyrus communis L.) Linden (Tilia platyphyllos) Alder (Alnus glutinosa L. Gaertn.)	Duration Control $212^{\circ}C - 1$ hour $212^{\circ}C - 2$ hours Control $212^{\circ}C - 1$ hour $212^{\circ}C - 2$ hours Control $212^{\circ}C - 1$ hour $212^{\circ}C - 2$ hours Control $212^{\circ}C - 1$ hour $212^{\circ}C - 1$ hour	20° mean 1.35 (0.07) 0.76 (0.05) 0.65 (0.05) 1.43 (0.05) 0.71 (0.13) 0.67 (0.13) 2.37 (0.07) 0.65 (0.05) 0.81 (0.09) 1.49 (0.09) 0.90 (0.05) 0.78 (0.10)	60° mean 3.38 (0.30) 3.11 (0.19) 2.71 (0.38) 3.00 (0.18) 3.63 (0.36) 3.10 (0.81) 3.81 (0.17) 2.20 (0.21) 2.29 (0.26) 4.65 (0.28) 3.04 (0.44) 3.15 (0.48)	85° mean 4.04 (0.94) 5.88 (0.56) 4.01 (1.06) 4.68 (1.15) 8.74 (1.47) 8.48 (3.78) 0.99 (0.07) 1.64 (0.41) 1.41 (0.30) 5.58 (0.30) 3.83 (1.32) 6.08 (1.72)

Table 5.	Perpendicular	and parallel	l gloss at	: 20°, 60°	and 8	85° for	untreated	wood
and woo	d subjected to l	heat treatmen	nt for 1 h	and 2 h				

Table 6 presents the results of two-way ANOVA for perpendicular and parallel gloss at 20°, 60° and 85°. The results show that values of perpendicular and parallel gloss at 20°, 60° and 85° are significantly different for wood species (A), heat treatment (B) and interaction (AB), at the selected level of significance (p < 0.05). The fact that the interaction is significant means that the effect of heat treatment on parallel and perpendicular gloss differs with species.

Test	Source	Type sum of squares	Df	Mean square	F	Sig.
	Wood species (A)	7.571	4	1.893	238.794	0.000*
	Heat treatment (B)	30.980	2	15.490	1954.354	0.000*
	Interaction (AB)	6.833	8	0.854	107.756	0.000*
$\pm 20^{\circ}$	Error	1.070	135	0.008		
	Total	282.080	150			
	Corrected total	46.453	149			
	Wood species (A)	26.067	4	6.517	47.377	0.000*
	Heat treatment (B)	15.901	2	7.950	57.801	0.000*
	Interaction (AB)	23.592	8	2.949	21.440	0.000*
⊥60°	Error	18.569	135	0.138		
	Total	1246.170	150			
	Corrected total	84.128	149			
	Wood species (A)	48.190	4	12.048	88.647	0.000*
	Heat treatment (B)	1.995	2	0.997	7.339	0.001*
	Interaction (AB)	32.903	8	4.113	30.263	0.000*
⊥85°	Error	18.347	135	0.136		
	Total	683.570	150			
	Corrected total	101.435	149			
	Wood species (A)	5.141	4	1.285	189.010	0.000*
	Heat treatment (B)	34.831	2	17.415	2561.069	0.000*
	Interaction (AB)	6.338	8	0.792	116.510	0.000*
20°	Error	0.918	135	0.007		
	Total	233.600	150			
	Corrected total	47.228	149			
	Wood species (A)	23.960	4	5.990	42.937	0.000*
	Heat treatment (B)	16.162	2	8.081	57.925	0.000*
	Interaction (AB)	23.247	8	2.906	20.830	0.000*
60°	Error	18.833	135	0.140		
	Total	1733.570	150			
	Corrected total	82.201	149			
	Wood species (A)	563.807	4	140.952	77.132	0.000*
	Heat treatment (B)	81.652	2	40.826	22.341	0.000*
110.50	Interaction (AB)	181.479	8	22.685	12.414	0.000*
83°	Error	246.702	135	1.827		
	Total	4030.680	150			
	Corrected total	1073.640	149			

Table 6. Multiple variance analysis for perpendicular and parallel gloss at 20°, 60° and 85°

Table 7 shows Duncan homogeneity groups for perpendicular glossiness values. In relation to wood species, only for glossiness at 20° does each species belong to a different homogeneous group, meaning that there are significant differences between all species. Although, generally, there are significant differences between wood species, this does not apply to black locust and linden

 $(\pm 60^{\circ})$  or black locust and alder  $(\pm 85^{\circ})$ . No significant differences were found between treatments, although there was a significant difference between untreated (control) and treated wood for perpendicular glossiness at 20° and 60°.

Wood species		⊥20°		⊥60°		⊥85°
wood species	IN	mean	HG	mean	HG	Mean
Wild pear (Pyrus communis L.)	30	1.06	D	2.90	С	2.76
Black locust (Robinia pseudoacacia Lipsky)	30	0.98	Е	2.31	D	2.18
Alder (Alnus glutinosa L. Gaertn.)	30	1.24	С	3.09	В	2.12
Linden (Tilia platyphyllos)	30	1.41	В	2.29	D	1.04
Willow (Salix alba)	30	1.59	<b>A*</b>	3.32	<b>A*</b>	1.76
Duration	N	⊥2	0°	⊥60	)°	⊥85°
Duration	1	mean	HG	mean	HG	mean
Control	50	1.90	A*	3.24	A*	1.93
212°C – 1 hour	50	0.94	В	2.50	В	1.86
$212^{\circ}\text{C} - 2$ hours		0.93	В	2.61	В	2.13

Table 7. Duncan homogeneity groups for  $\perp 20^\circ, \perp 60^\circ$  and  $\perp 85^\circ$  glossiness values

Number of measurements, HG: Homogeneous group, \*Highest value.

Table 8.	Duncan	homogeneity	groups for	r   20°,   (	60° and   8	85° glossiness	values

Washmasia		20°		∥60°		85°
Wood species	Ν	mean	HG	mean	HG	mean
Wild pear (Pyrus communis L.)	30	0.94	D	3.24	С	7.30
Black locust (Robinia pseudoacacia Lipsky)	30	0.92	D	3.07	С	4.64
Alder (Alnus glutinosa L. Gaertn.)	30	1.06	С	3.61	В	5.16
Linden (Tilia platyphyllos)	30	1.28	В	2.77	D	1.35
Willow (Salix alba)	30	1.38	<b>A*</b>	3.90	A*	3.75
		20	)°	60	)°	85°
Duration	Ν	mean	HG	mean	HG	mean
Control	50	1.80	A*	3.78	A*	3.48
212°C – 1 hour	50	0.79	В	3.10	В	4.57
$212^{\circ}C - 2$ hours	50	0.76	В	3.07	В	5.27

Number of measurements, HG: Homogeneous group, \*Highest value.

Table 8 shows Duncan homogeneity groups for parallel glossiness values. There were no significant differences between all species at any measurement angle, the same holding for the treatments. However, similarly to the case of perpendicular glossiness, there was a significant difference between untreated and treated wood for  $20^{\circ}$  and  $60^{\circ}$ .

### Conclusions

- 1. Lightness decreases with heat treatment, with the greatest differences seen between untreated and heat-treated wood. The lightness differences between species are much greater for untreated wood, showing that heat treatment equalizes lightness between species.
- 2. Changes in a\* were different for each species, increasing for black locust, linden and willow, and decreasing for wild pear and alder.
- 3. The change in a\* and b\* on heat treatment depends on the species and on the initial value.
- 4. L\*, a\* and b\* values are significantly different for wood species (A), heat treatment (B) and interaction (AB) at the selected level of significance (p < 0.05).
- 5. Gloss at a 20° angle decreased with heat treatment for both the perpendicular and parallel directions. Glossiness at a 60° angle is similar to that at 20°, although the decrease on heat treatment is smaller. With the 85° angle there is an equilibrium between the decrease in gloss due to the heat treatment and the increase due to the lower roughness of the heat-treated surface.
- 6. Perpendicular and parallel glossiness at the angles  $20^{\circ}$ ,  $60^{\circ}$  and  $85^{\circ}$  are significantly different for wood species (A), heat treatment (B) and interaction (AB) at the selected level of significance (p < 0.05).

### References

- Aksoy A., Deveci M., Baysal E., Toker H. [2011]: Colour and gloss changes of Scots pine after heat modification. Wood Research 56 [3]: 329-336
- Ayata U., Gurleyen L., Esteves B. [2017a]: Effect of heat treatment on the surface of some exotic wood species. Drewno 60 [199]. DOI: 10.12841/wood.1644-3985.198.08
- Ayata U., Gurleyen L., Esteves B., Gurleyen T., Cakıcıer N. [2017b]: Effect of heat treatment [ThermoWood] on some surface properties of parquet Beech [*Fagus orientalis Lipsky*] with different layers of UV system applied. BioResources 12 [2]: 3876-3889. DOI: 10.15376/biores.12.2.3876-3889
- **Barcík Š., Gašparík M., Razumov E.Y.** [2015]: Effect of temperature on the color changes of wood during thermal modification. Cellulose Chemistry and Technology 49 [9-10]: 789-798
- Bekhta P., Niemz P. [2003]: Effect of high temperature on the change in color dimensional stability and mechanical properties of spruce wood. Holzforschung 57 [5]: 539-546
- Boonstra M., Van Acker J., Kegel E., Stevens M. [2007]: Optimisation of a two-stage heat treatment process: durability aspects. Wood Science and Technology 41: 31-57
- **Brischke C., Welzbacher C.R., Brandt K., Rapp A.O.** [2007]: Quality control of thermally modified timber: Interrelationship between heat treatment intensities and CIE L\* a\* b\* color data on homogenized wood samples. Holzforschung 61 [1]: 19-22

- **Dubey M.K.** [2010]: Improvements in stability durability and mechanical properties of radiata pine wood after heat-treatment in a vegetable oil. PhD Thesis in Forestry at the University of Canterbury
- **Esteves B., Nunes L., Domingos I., Pereira H.** [2014]: Improvement of termite resistance dimensional stability and mechanical properties of pine wood by paraffin impregnation. European Journal of Wood and Wood Products 72 [5]: 609-615
- Esteves B., Velez Marques A., Domingos I., Pereira H. [2007]: Heat-induced colour changes of pine [*Pinus pinaster*] and eucalypt [*Eucalyptus globulus*] wood. Wood Science and Technology 42 [5]: 369-384. DOI: 10.1007/s00226-007-0157-2
- González-Peña M.M., Hale M.D. [2009]: Colour in thermally modified wood of beech Norway spruce and Scots pine. Part 1: Colour evolution and colour changes. Holzforschung 63 [4]: 385-393
- Gurleyen L., Ayata U., Esteves B., Cakicier N. [2017]: Effects of heat treatment on the adhesion strength pendulum hardness surface roughness color and glossiness of Scots pine laminated parquet with two different types of UV varnish application. Maderas. Ciencia y tecnología 19 [2]: 213-224. DOI: 10.4067/S0718-221X2017005000019
- Johansson D., Morén T. [2006]: The potential of colour measurement for strength prediction of thermally treated wood. Holz als Roh- und Werkstoff 64 [2] 104-110. DOI: 10.1007/s00107-005-0082-8
- Kamdem D.P., Pizzi A., Jermannaud A. [2002]: Durability of heat-treated wood. Holz als Roh-und Werkstoff 60 [1]: 1-6
- Karamanoglu M., Akyildiz M.H. [2013]: Colour gloss and hardness properties of heat treated wood exposed to accelerated weathering. Pro Ligno 9 [4]: 729-738
- Kim G.-H., Yun K.-E., Kim J.-J. [1998]: Effect of heat treatment on the decay resistance and the bending properties of radiata pine sapwood. Material und Organismen 32 [2] 101-108
- Kol H.Ş., Sefil Y. [2011]: The thermal conductivity of fir and beech wood heat treated at 170 180 190 200 and 212°C. Journal of Applied Polymer Science 121 [4]: 2473-2480. DOI: 10.1002/app.33885
- Korkut D.S., Guller B. [2008]: The effects of heat treatment on physical properties and surface roughness of red-bud maple [*Acer trautvetteri* Medw.] wood. Bioresource Technology 99 [8]: 2846-2851. DOI: 10.1016/j.biortech.2007.06.043
- Korkut D.S., Hiziroglu S., Aytin A. [2013]: Effect of heat treatment on surface characteristics of wild cherry wood. BioResources 8 [2]: 1582-1590
- Korkut D.S., Korkut S., Bekar I., Budakçı M., Dilik T., Çakıcıer N. [2008]: The effects of heat treatment on the physical properties and surface roughness of Turkish hazel [*Corylus colurna* L.] wood. International Journal of Molecular Sciences 9 [9]: 1772--1783. DOI: 10.3390/ijms9091772
- Metsä-Kortelainen S., Viitanen H. [2009]: Decay resistance of sapwood and heartwood of untreated and thermally modified Scots pine and Norway spruce compared with some other wood species. Wood Material Science and Engineering 4 [3-4]: 105-114
- Scrinzi E., Rossi S., Deflorian F., Zanella C. [2011]: Evaluation of aesthetic durability of waterborne polyurethane coatings applied on wood for interior applications. Progress in Organic Coatings 72 [1]: 81-87. DOI: 10.1016/j.porgcoat.2011.03.013
- Srinivas K., Pandey K.K. [2012]: Effect of heat treatment on color changes, dimensional stability and mechanical properties of wood. Journal of Wood Chemistry and Technology 32 [4]: 304-316
- Stamm A.J., Burr H.K., Kline A.A. [1946]: Staybwood heat-stabilized wood. Industrial Engineering Chemistry 38: 630-634

- Sundqvist B., Morén T. [2002]: The influence of wood polymers and extractives on wood colour induced by hydrothermal treatment. European Journal of Wood and Wood Products 60 [5]: 375-376
- Tjeerdsma B.F., Boonstra M., Pizzi A., Tekely P., Militz H. [1998]: Characterisation of thermally modified wood: molecular reasons for wood performance improvement. Holz als Roh-und Werkstoff 56 [3]: 149-153
- **Tjeerdsma B.F., Stevens M., Militz H., Van Acker J.** [2002]: Effect of process conditions on moisture content and decay resistance of hydro-thermally treated wood. Holzforschung und Holzverwertung 54 [5]: 94-99
- Todorovic N.V., Popović Z., Milić G., Popadić R. [2012]: Estimation of heat-treated beechwood properties by color change. BioResources 7 [1]: 0799-0815
- **Torniainen P., Elustondo D., Dagbro O.** [2015]: Industrial validation of the relationship between color parameters in thermally modified spruce and pine. BioResources 11 1369-1381
- Unsal O., Ayrilmis N. [2005]: Variations in compression strength and surface roughness of heat-treated Turkish river red gum [*Eucalyptus camaldulensis*] wood. Journal of Wood Science 51 [4]: 405-409
- Viitaniemi P., Jämsä S., Ek P. and Viitanen H. [2001]: Pat. EP 0695408 (BE,DE,FR, ES,IT,AT,GR,PT,NL,IE,GB,CH), 2001. Method for improving biodegradation resistance and dimensional stability of cellulosic products. VTT. Appl. 94915166.6, 13.5.1994. Publ. 10.1.2001

#### List of standards

- **ISO 2813:1994** Paints and varnishes Determination of specular gloss of non-metallic paint films at 20 degrees, 60 degrees and 85 degrees, International Organization for Standardization.
- ISO 554:1976 Standard atmospheres for conditioning and/or testing Specifications.

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