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THE EFFECTS OF HEAT TREATMENT ON COLOUR AND GLOSSINESS OF SOME COMMERCIAL WOODS IN TURKEY

In this study, hornbeam (Carpinus betulus L.), poplar (Populus deltoides), chestnut (Castanea sativa Mill.) and Uludağ fir (Abies bornmülleriana Mattf.) were heat treated at 212°C for 1 hour and 2 hours by the ThermoWood® method. Colour and glossiness were determined and compared with untreated samples. Results showed that lightness (L) decreased for all tested samples and that the decrease was higher with more intense treatments. Colour parameter a* (red/green) initially increased with heat treatment, then decreasing afterwards. The colour parameter b* (yellow/blue) on the other hand, decreased for Hornbeam and Chestnut and increased for Poplar and Uludag fir. Generally, the glossiness decreased with heat treatment for both measurements, along and across the grain. All factors and interactions were found to be significant (according to α = 0.05) with the exception of interaction (AB) for parallel (//) glossiness in 60°. The highest total colour change was achieved for Hornbeam (ΔE* = 41.58).*

Keywords: ThermoWood, heat treatment, wood, glossiness, colour

Introduction

Wood modification methods are used to improve wood properties without the introduction of harmful chemicals into the wood. These methods are usually divided into heat treatment, which includes treatments like Thermowood®, Plato®, OHT® or Perdure®, chemical modifications like acetylation (Accoya®) and impregnation modifications like furfurylation (Kebony®) [Esteves and Pereira 2009]. The most widespread wood modification procedure is heat

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treatment, probably because it is simple with no added chemicals and can be applied to all kinds of wood. On the other hand, processes like furfurylation and acetylation that use high quantities of chemicals are not suitable for some species. The most important properties improved by these treatments are dimensional stability [Viitaniemi et al. 1997; Tjeerdsma et al. 1998a; Bekhta and Niemz 2003; Esteves et al. 2007] and durability [Tjeerdsma et al. 1998a; Kamdem et al. 2002; Hakkou et al. 2006; Boonstra et al. 2007; Dubey 2010]. On the other hand, heat treatments deteriorate mechanical properties [Kim et al. 1998; Kubojima et al. 2000; Bekhta and Niemz 2003; Esteves et al. 2007; Kocaefe et al. 2008; Dubey 2010].

The CIELAB method created by the *Commission International de l' Éclairage* is used in most wood colour studies, probably because it is a simple method that can easily describe colour changes. This method quantifies colour by a three axes system: lightness (L^*) from 0% (black) and 100% (white), a^* from green (-*a*) to red (+*a*), and b^* from blue (-*b*) to yellow (+*b*), these last two are called the chroma coordinates [Esteves et al. 2008]. From the aesthetic point of view, colour changes are also important for the consumers. Colour changes are also often linked to other wood properties and can be used to determine the treatment intensity to achieve the desired properties. According to Bourgois et al. [1991], there is a close relationship between chemical modifications and some colour parameters. Bekhta and Niemz [2003] found strong correlations between total colour difference and both modulus of elasticity and bending strength for heat treated spruce wood while Brischke et al. [2007] reported good linear correlation between a cumulated measure of L^* and b^* and the heat treatment intensity for spruce (*Picea abies* Karst.), pine (*Pinus sylvestris* L.), and beech (*Fagus sylvatica* L.). Esteves et al. [2008] reported that lightness decrease was related to chemical changes, with good correlations with glucose (R^2 = 0.96), hemicelluloses (R^2 = 0.92) and lignin (R^2 = 0.86). Todorovic et al. [2012] used colour changes due to heat treatment to predict the mass loss, density loss, and modulus of rupture (MOR) in both sapwood and heartwood and concluded that the PLS-R showed the best results and presented the highest coefficients of determination.

The darker colour obtained from heat treatment has been attributed to several factors like the formation of coloured degradation products from hemicelluloses [Sundqvist 2002; Sehlstedt-Persson 2003] or to extractives [Sundqvist and Morén 2002]. Other authors attribute the heat-treated colour, to oxidation products such as quinones that are formed during treatment [Tjeerdsma et al. 1998b; Mitsui et al. 2001; Bekhta and Niemz 2003; Sehlstedt-Persson 2003] and also suggested that the change in colour resulting from hemicellulose degradation might be due to a hydrolysis reaction similar to a Maillard reaction, which is a well-known process in the food industry. The change in glossiness has also been reported in the last few years. In accordance with Aksoy et al. [2011], heat treatment caused a decrease in gloss values of Scots pine. Similar results were presented by Korkut et al. [2013] for wild cherry wood and Karamanoglu and Akyildiz [2013] for Anatolian black pine, Calabrian pine, Sessile oak and Chestnut woods. Different results were attained by Bekhta et al. [2014] with thermally densified wood; however, the obtained increase in glossiness was attributed to densification, rather than to heat treatment. These authors also reported that gloss measurements for all investigated wood species, when measured along the grain were higher than those measured across the grain.

The objective of this work was to determine the effects of heat treatment on the colour and glossiness of some commercial woods in Turkey. The colour parameters (L^*, a^*, b^*, ΔE^*, ΔL^*, Δa^*, and Δb^*) and glossiness (parallel and perpendicular to the grain at 20°, 60°, and 85°) of heat treated and untreated hornbeam (*Carpinus betulus* L.), poplar (*Populus deltoides*), chestnut (*Castanea sativa* Mill.) and Uludag fir (*Abies borülleriana* Mattf.) were determined. Although glossiness measurement is usually done in just one angle in accordance to the surface brightness, measurements were made at 20°, 60° and 85º to determine if the heat treatment has any special effect in one of these angles.

Materials and methods

Materials

All wood species (hornbeam (*Carpinus betulus* L.), poplar (*Populus deltoides*), chestnut (*Castanea sativa* Mill.) and Uludag fir (*Abies bornmülleriana* Mattf.) were obtained from organized industrial zones of Duzce forest products. Approximately the same amount of all wood specimens measuring 100 mm by 100 mm by 10 mm were used for colour and glossiness measurements. Three samples were prepared for each experiment and they were derived from the sapwood of the logs since the heat treatment is usually applied to wood from young trees. All wood species samples were conditioned to 12% MC (moisture content) in a special room at 20°C (±2) and 65% (±5) RH (relative humidity) [ISO 554:1976].

Heat treatment process

The ThermoWood® process was applied to air-dried wood in a Novawood factory in Gerede, Bolu, Turkey, following a schedule commonly used for hardwoods. The boards were introduced inside the kiln where there was a fast increase in temperature with heat and steam and less than 3 to 5% oxygen until 100°C was attained, followed by a slower increase up to 130°C. At this stage, the wood nearly reaches zero moisture content . Afterwards, the heat treatment was carried out at 212°C for 1 or 2 hours. The temperature of the wooden materials was then decreased to about 60°C by water spraying. Usually, the moisture level reaches about 6%. The duration of the cooling and conditioning steps takes between 24 to 30 hours in accordance to the width and thickness of the wooden material [ThermoWood Handbook 2003].

Methods

The colour change of all wood species heat treated, and untreated control was analysed by a spectrophotometer (Datacolour 110, Datacolour Technology Suzhou Co., Ltd., China) (wavelength resolution 10 nm, measurement geometry D/8°) with a D65 standard illuminant. Colour parameters were measured in the centre of each sample using ten replicates of each heat treated and untreated control sample and an average value was reported. The CIELAB system is characterized by three parameters, L^*, a^*, and b^*. The L^* axis represents lightness, $+a^*$ is the red, minus a^* for green, $+b^*$ for yellow, minus b^* for blue, and L^* varies from 100 (white) to zero (black) [Zhang et al. 2009]. The corresponding variations ΔL^*, Δa^* and Δb^* with heat treatments were calculated using the untreated control as a reference (equations 2, 3, 4). The total colour difference (ΔE^*) between heat treated and untreated control was calculated using equation 1.

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

$$\Delta L^* = L^*_{\text{heat treated}} - L^*_{\text{reference}}, \quad (2)$$

$$\Delta b^* = b^*_{\text{heat treated}} - b^*_{\text{reference}}, \quad (3)$$

$$\Delta a^* = a^*_{\text{heat treated}} - a^*_{\text{reference}} \quad (4)$$

The glossiness change of all wood species heat treated, and untreated control was analysed with a gloss meter (Novo-Gloss Trio, Rhopoint Instruments Ltd., UK) (20°, 60° and 85°) [ISO 2813:1994].

Statistical Analysis

Statistical evaluations were calculated by a SPSS 17.0 Software Package program (IBM, USA). In total, 450 colour measurements were done (3 treatment durations × 4 wood types × 10 measurement × 3 colour parameter (L^*, a^* and b^*) = 360 measurement). In relation to glossiness 720 measurements were done (3 heat treatment × 4 wood types × 10 measurement × 2 glossiness parameter (perpendicular (⊥) and parallel (||)) × 3 gloss degree (20°, 60° and 85°) = 720 measurement). In total, 1080 measurements were done in this research.

Results and discussion

Changes in colour and glossiness

The results of the variance analysis of L^*, a^*, b^*, perpendicular (⊥) and parallel (||) glossiness at 20°, 60° and 85° for heat treated and untreated wood from hornbeam (*Carpinus betulus* L.), poplar (*Populus deltoides*), chestnut (*Castanea sativa* Mill.), and Uludag fir (*Abies bornmülleriana* Mattf.) are given in table 1.

Table 1. Results of the variance analysis of glossiness and colour values

Test	Factors	Sum of Squares	Degree of Freedom	Mean Square	F Number	P < 0.05
*L**	Wood types (A)	2721.891	3	907.297	1646.536	0.000*
	Treatment duration(B)	36855.450	2	18427.725	33442.095	0.000*
	Interaction (AB)	181.119	6	30.186	54.782	0.000*
	Error	59.512	108	0.551		
*a**	Wood types (A)	3.792	3	1.264	18.439	0.000*
	Treatment duration(B)	1011.111	2	505.556	7375.548	0.000*
	Interaction (AB)	184.078	6	30.680	447.587	0.000*
	Error	7.403	108	0.069		
*b**	Wood types (A)	838.721	3	279.574	577.891	0.000*
	Treatment duration(B)	19.736	2	9.868	20.398	0.000*
	Interaction (AB)	561.603	6	93.600	193.476	0.000*
	Error	52.249	108	0.484		
⊥20°	Wood types (A)	7.216	3	2.405	201.074	0.000*
	Treatment duration(B)	45.875	2	22.938	1917.383	0.000*
	Interaction (AB)	0.456	6	0.076	6.355	0.000*
	Error	1.292	108	0.012		
⊥60°	Wood types (A)	60.882	3	20.294	123.730	0.000*
	Treatment duration(B)	38.711	2	19.356	118.009	0.000*
	Interaction (AB)	15.948	6	2.658	16.205	0.000*
	Error	17.714	108	0.164		
⊥85°	Wood types (A)	183.714	3	61.238	282.975	0.000*
	Treatment duration(B)	2.928	2	1.464	6.765	0.002*
	Interaction (AB)	20.706	6	3.451	15.947	0.000*
	Error	23.372	108	0.216		
‖20°	Wood types (A)	3.601	3	1.200	184.926	0.000*
	Treatment duration(B)	54.121	2	27.061	4169.106	0.000*
	Interaction (AB)	0.477	6	0.079	12.244	0.000*
	Error	0.701	108	0.006		
‖60°	Wood types (A)	113.269	3	37.756	147.123	0.000*
	Treatment duration(B)	39.333	2	19.667	76.634	0.000*
	Interaction (AB)	2.069	6	0.345	1.344	0.244**
	Error	27.716	108	0.257		
‖85°	Wood types (A)	5221.626	3	1740.542	673.312	0.000*
	Treatment duration(B)	75.561	2	37.781	14.615	0.000*
	Interaction (AB)	86.161	6	14.360	5.555	0.000*
	Error	279.185	108	2.585		

*Significant at 95% confidence level.
**Insignificant.

Only the interaction (AB) for parallel (||) glossiness in 60° was found to be insignificant, all other factors and interactions were found to be significant (according to $\alpha = 0.05$). This shows that wood types and treatment duration play an important role in colour and glossiness proving that the treatment has to be tailored to each species in order to achieve the desired final properties. The highest error was observed for glossiness at an 85° angle due to the high dispersion of results at this angle as stated before [Ayata et al. 2017].

The colour and glossiness of untreated and heat-treated woods are presented in table 2. As expected, lightness (*L**) decreased with heat treatment for all the tested species corresponding to the darkening of the wood surface. The highest decrease was observed for Hornbeam with a decrease from 78.51 to 37.57 which corresponded to a 52% decrease in relation to initial lightness. The smallest decrease (42%) was attained for Uludag fir (84.32 to 48.90). These results are in accordance with the changes presented by several authors like Bekhta and Niemz [2003] for Spruce, Esteves et al. [2008] with *Pinus pinaster* and *Eucalyptus globulus*, Aksoy et al. [2011] for Scots pine, Karamanoglu and Akyildiz [2013] for Anatolian black pine, Calabrian pine, Sessile oak and Chestnut woods.

Overall *a** increases in the first hour of the treatment decreasing afterwards. The changes in *b** differ a lot for all of the treated samples. Generally, *b** decreases for Hornbeam and Chestnut and increases for Poplar and Uludag fir. Similar changes in chroma parameters have been reported before by several authors [Esteves et al. 2008; Aksoy et al. 2011; Chen et al. 2012].

In accordance with the results, there is a significant difference in *L** between wood treated for 1 hour or 2 hours for Hornbeam and Poplar as can be seen in table 2. Nevertheless, for chestnut and Uludag fir, this does not happen, probably because the main changes in lightness happened in the first hour of treatment at 212°C. In relation to chroma parameters, only for the Uludag fir are the differences between 1 h and 2 h treatment not significant.

The changes in colour due to heat treatment can be attributed to the fact that the duration and high temperatures of treatment applied to the wood cause a change in its chemical structure. The cellulose, hemicellulose and lignin structures become different. Some studies indicate that there is a close relation between lightness decrease and chemical composition. For instance, Esteves et al. [2008] found good correlations between chemical changes in glucose ($R^2 = 0.96$) and hemicelluloses ($R^2 = 0.92$). They also found a weaker relation with lignin ($R^2 = 0.86$). These results show that the changes in cellulose and hemicelluloses play an important role in the colour changes due to heat treatment. This is in accordance with several authors who attribute colour changes with heat treatment to the formation of coloured degradation products from hemicelluloses [Sundqvist and Morén 2002; Sehlstedt-Persson 2003].

Nevertheless, the changes in colour are most likely due to the sum of several factors which include the formation of degradation products and the migration of extractives to the surface.

The specular gloss is the amount of light that is reflected by a given surface when it focuses a light with a given angle of incidence. The light that is reflected from the surface depends on the uniformity, the smooth or rough surface as well as the refractive index of the finishing. Higher incidence angles correspond to higher glossiness. That is why gloss is usually measured at a 20° angle for more

Table 2. Colour parameters and glossiness results

Woodtype	Duration	L^*			a^*			b^*	
		Mean^{HG}	Min	Max	Mean^{HG}	Min	Max	Mean^{HG}	Min
WT$_1$	TW$_0$	78.51^{C} (0.59)	77.57	79.21	3.72^{I} (0.16)	3.37	3.91	19.08^{D}(0.48)	18.30
	TW$_1$	38.38^{H} (1.27)	36.37	40.04	10.02^{D} (0.27)	9.53	10.39	15.55^{F} (0.87)	14.00
	TW$_2$	37.57^{I} (0.82)	36.51	39.18	9.46^{E} (0.27)	9.01	9.71	14.62^{G} (0.64)	13.78
WT$_2$	TW$_0$	83.52^{B} (0.95)	81.66	84.81	1.57^{K} (0.61)	0.73	2.69	17.87^{E} (1.44)	15.35
	TW$_1$	48.51^{F} (0.25)	47.96	48.89	11.24^{A*} (0.18)	10.86	11.50	22.70^{B} (0.42)	21.71
	TW$_2$	43.93^{G} (0.56)	43.03	44.85	10.66^{B} (0.14)	10.40	10.85	20.70^{C} (0.44)	20.19
WT$_3$	TW$_0$	73.27^{D} (0.69)	72.07	74.22	6.72^{H} (0.28)	6.17	7.16	19.30^{D} (0.30)	18.91
	TW$_1$	37.72^{HI} (0.59)	36.72	38.31	9.12^{F} (0.22)	8.88	9.57	15.29^{F} (0.60)	14.49
	TW$_2$	37.52^{I} (0.34)	36.75	38.06	8.55^{G} (0.10)	8.37	8.65	13.72^{H} (0.22)	13.27
WT$_4$	TW$_0$	84.32^{A*} (0.78)	83.44	86.13	3.41^{J} (0.25)	2.94	3.68	18.79^{D} (0.49)	17.95
	TW$_1$	49.50^{E} (0.58)	48.51	50.36	10.58^{BC} (0.08)	10.49	10.78	23.79^{A} (0.62)	22.85
	TW$_2$	48.90^{EF} (0.90)	47.52	50.03	10.38^{C} (0.12)	10.14	10.53	24.35^{A*} (0.92)	22.76

Woodtype	Duration	⊥20°			⊥60°			⊥85°	
		Mean^{HG}	Min	Max	Mean^{HG}	Min	Max	Mean^{HG}	Min
WT$_1$	TW$_0$	2.51^{B} (0.12)	2.30	2.70	5.98^{A*} (0.70)	5.00	7.00	5.22^{A*} (0.94)	3.60
	TW$_1$	1.24^{E} (0.05)	1.20	1.30	5.06^{B} (0.35)	4.50	5.60	5.18^{A} (0.46)	4.60
	TW$_2$	1.06^{F} (0.12)	0.90	1.30	3.34^{EF} (0.66)	2.50	4.20	3.41^{B} (0.43)	2.50
WT$_2$	TW$_0$	2.72^{A*} (0.09)	2.60	2.90	4.03^{C} (0.17)	3.80	4.20	1.53^{DE} (0.13)	1.40
	TW$_1$	1.46^{D} (0.12)	1.20	1.60	3.25^{EFG} (0.31)	2.80	3.60	1.75^{DE} (0.23)	1.40
	TW$_2$	1.34^{E} (0.14)	1.20	1.60	3.11^{FG} (0.32)	2.60	3.50	1.91^{CD} (0.16)	1.70
WT$_3$	TW$_0$	1.96^{C} (0.13)	1.80	2.20	3.55^{DE} (0.43)	3.10	4.40	1.92^{CD} (0.32)	1.50
	TW$_1$	0.85^{G} (0.11)	0.60	1.00	2.73^{H} (0.55)	1.80	3.40	2.29^{C} (1.01)	1.40
	TW$_2$	0.80^{G} (0.00)	0.80	0.80	2.88^{GH} (0.11)	2.70	3.10	2.25^{C} (0.14)	2.10
WT$_4$	TW$_0$	2.71^{A} (0.10)	2.60	2.90	3.88^{CD} (0.27)	3.60	4.30	1.30^{E} (0.13)	1.20
	TW$_1$	1.26^{E} (0.07)	1.20	1.40	2.63^{H} (0.12)	2.40	2.80	1.55^{DE} (0.12)	1.40
	TW$_2$	1.31^{E} (0.17)	1.00	1.60	2.68^{H} (0.32)	2.00	2.90	1.67^{DE} (0.24)	1.30

Woodtype	Duration	‖20°			‖60°			‖85°	
		Mean^{HG}	Min	Max	Mean^{HG}	Min	Max	Mean^{HG}	Min
WT$_1$	TW$_0$	2.35^{B} (0.11)	2.20	2.50	7.23^{A*} (0.89)	6.10	8.40	17.48^{B} (2.75)	13.30
	TW$_1$	0.84^{F} (0.07)	0.80	1.00	5.84^{B} (0.54)	5.00	6.80	21.50^{A*} (2.55)	16.30
	TW$_2$	0.78^{F} (0.06)	0.70	0.90	5.50^{B} (0.75)	4.30	6.30	20.43^{A} (2.66)	15.80
WT$_2$	TW$_0$	2.56^{A*} (0.05)	2.50	2.60	4.74^{C} (0.15)	4.60	5.00	3.16^{E} (0.68)	2.30
	TW$_1$	1.06^{D} (0.05)	1.00	1.10	3.84^{D} (0.33)	3.20	4.30	3.83^{E} (0.44)	2.90
	TW$_2$	0.96^{E} (0.13)	0.90	1.30	3.80^{D} (0.35)	3.10	4.20	6.50^{C} (0.95)	5.20
WT$_3$	TW$_0$	1.90^{C} (0.07)	1.80	2.00	4.55^{C} (0.67)	3.70	5.60	5.72^{C} (1.63)	3.80
	TW$_1$	0.69^{G} (0.07)	0.60	0.80	3.67^{D} (0.61)	2.80	4.60	6.43^{C} (1.75)	4.00
	TW$_2$	0.65^{G} (0.07)	0.60	0.80	3.41^{D} (0.24)	3.10	3.90	5.41^{CD} (0.83)	4.10
WT$_4$	TW$_0$	2.39^{B} (0.06)	2.30	2.50	4.76^{C} (0.30)	4.30	5.20	3.01^{E} (0.69)	2.00
	TW$_1$	1.03^{DE} (0.07)	1.00	1.20	3.46^{D} (0.18)	3.20	3.70	3.86^{E} (0.84)	2.60
	TW$_2$	1.00^{DE} (0.12)	0.90	1.30	3.40^{D} (0.44)	2.40	3.80	4.16^{DE} (0.85)	2.30

Values in parentheses are standard deviations, *HG*: Homogeneous group, WT$_1$: Hornbeam, WT$_2$: Poplar, WT$_3$: Chestnut, WT$_4$: Uludag fir, TW$_0$: Control, TW$_1$: 212°C for 1 hour, TW$_2$: 212°C for 2 hours, *: Maximum value, Mean: Average

bright surfaces, 60° for medium brightness and 85° for low brightness. At 20° and 60° glossiness generally decreases with heat treatment for both measurements, along and across the grain. This shows that glossiness decreases with heat treatment. Similar results were presented before for different wood species [Aksoy et al. 2011; Korkut et al. 2013; Karamanoglu and Akyildiz 2013; Bekhta et al. 2014]. In relation to 85°, there is no clear trend probably due to the high dispersion of results seen in table 1. The main differences between parallel and perpendicular glossiness were that generally glossiness was higher along the grain for 20° but smaller for 60° and 85°.

Total colour differences (Δ*E**) were calculated and presented in table 3. The highest increase in colour was observed for Hornbeam (Δ*E** = 41.58), followed by poplar (Δ*E** = 40.72), Uludag fir (Δ*E** = 36.52) and Chestnut (Δ*E** = 36.23).

Table 3. Colour changes (Δ*E) of heat treated wood at 212°C for 1 hour and 2 hours**

Wood types	Duration	Δ*E**
Hornbeam (*Carpinus betulus* L.)	212°C – 1 hour	40.77
	212°C – 2 hours	41.58
Poplar (*Populus deltoides*)	212°C – 1 hour	36.64
	212°C – 2 hours	40.72
Chestnut (*Castanea sativa* Mill.)	212°C – 1 hour	35.86
	212°C – 2 hours	36.23
Uludag fir (*Abies bornmülleriana* Mattf.)	212°C – 1 hour	35.90
	212°C – 2 hours	36.52

Conclusions

1. Colour changes depend on the species and the intensity of the treatment. Colour is suitable for quality control if a study is made for each species individually.
2. Lightness (*L**) decreases while *a** increases initially decreasing afterwards with heat treatment. The *b** decreases for Hornbeam and Chestnut and increases for Poplar and Uludag fir.
3. The highest total colour change was achieved for Hornbeam.
4. Generally, glossiness decreases with heat treatment for both measurements, along and across the grain.

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