

Effect of Heat Treatment (ThermoWood) on Some Surface Properties of Parquet Beech (*Fagus orientalis* Lipsky.) with Different Layers of UV System Applied

Umit Ayata,^{a,*} Levent Gurleyen,^b Bruno Esteves,^c Tugba Gurleyen,^d and Nevzat Cakicier^e

Heat treatment is known to improve the stability and durability of various woods. However, in the process some surface properties are changed. This paper reports the changes in surface properties such as color, glossiness, pendulum hardness, and surface adhesion on coated heat-treated and untreated beech wood. The wood was coated with an epoxy acrylic resin sealer followed by a polyacrylic-based resin varnish that is normally applied on parquet with single and double layers according to the manufacturer's recommendations. The results showed that in relation to color parameters, the lightness decreased while a^* increased in response to heat treatment. This was followed by a decrease in the redness (a^*) and a decrease in yellow (b^*) with the intensity of the treatment. There was an initial increase in the glossiness but it decreased later on with the severity of the treatment. The hardness and adhesion also decreased with the severity of the treatment for beech coated with single and double layers. The wood surface properties depended on the treating time and the temperature of the treatment.

Keywords: Parquet Beech; Heat treatment; Glossiness; Color, Adhesion

Contact information: a: Forestry and Forest Products, Oltu Vocation School, Ataturk University, Oltu/Erzurum, Turkey; b: Yigilca Multi-Program Anatolian High School, Yigilca/Duzce, Turkey; c: Superior School of Technology Polytechnic Institute of Viseu, Viseu, Portugal; d: Department of Furniture and Decoration, Vocational High School, Duzce University, Duzce, Turkey; e: Department of Forest Industry Engineering, Duzce University, Duzce, Turkey; * Corresponding author: umitayata@atauni.edu.tr

INTRODUCTION

Heat treatment has been one of the most studied wood modification procedures in the last few years. The absence of chemicals in the process has been mentioned as the main reason for the green status of this treatment. Several commercial processes have arisen, in Finland (ThermoWood), Holland (Plato), Germany (OHT), and in France (Bois Perdure and Rectification). Currently, the main producers are ThermoWood in Europe and Perdure in Canada because the process now belongs to PCI industries (Quebec). The ThermoWood process, patented by Viitaniemi *et al.* (1997), is made with vapour, with less than 3% to 5% of oxygen, without any pressure.

The process starts by a fast increase of oven temperature until 100 °C, followed by a steady increase up to 130 °C until the moisture content reaches almost zero. Subsequently, the temperature is increased to the desired treatment temperature (over 180 °C), with the heat and vapour staying at this temperature for 2 to 3 h, before cooling to a temperature around 80 to 90 °C (Esteves and Pereira 2009).

Heat treatment is known to improve the dimensional stability (Tjeerdsma *et al.* 1998; Bekhta and Niemz 2003; Esteves *et al.* 2006) and durability of wood against fungal biodegradation, depending on the intensity of the treatment (Dirol and Guyonnet 1993; Kim *et al.* 1998; Tjeerdsma *et al.* 2002; Boonstra *et al.* 2007a). At the same time several other wood properties are changed with the treatment. Mechanical properties including bending strength or impact bending decrease in response to more intense treatments (Kim *et al.* 1998; Kubojima *et al.* 2000). Some other properties exhibit a slight decrease or increase, such as the hardness or bending stiffness, depending on the intensity of the treatment and on the wood species.

From the consumer's point of view, surface properties are some of the most important wood properties, because they influence the aesthetical look that is frequently a factor for the selection of a specific wood. The main surface properties changed by a heat treatment are colour, glossiness, surface hardness, and wettability. These parameters considerably affect wood's adhesion properties.

Recently, the surface property of colour has been extensively studied. Treated wood becomes darker, although the final colour depends on the initial colour and on the intensity of the treatment, as reported before by several authors (Mitsui *et al.* 2001; Bekhta and Niemz 2003; Esteves *et al.* 2007). The darker colour of heat-treated wood is frequently attributed to the formation of coloured degradation products from the hemicelluloses and from some extractives (Sundqvist 2002; Sundqvist and Morén 2002; Sehlstedt-Persson 2003). Hemicellulose hydrolysis has been reported to occur by a reaction similar to a Maillard reaction, a well-known process in the food industry (Sehlstedt-Persson 2003).

The main colour studies are done by the CIE $L^*a^*b^*$ method, which represents colour with a three axes system representing L^* (lightness), a^* (green-red) and b^* (blue-yellow). With heat treatment, the lightness decreases while effects on a^* and b^* differ amongst wood species. Reports show that the glossiness of a heat-treated surface decreases (Aksoy *et al.* 2011; Korkut *et al.* 2013). The wettability of heat-treated wood is known to decrease for heat treatments above 130 °C until about 190 °C. Several reasons have been suggested to account for this decrease, such as the formation of degradation compounds (Pecina and Paprzycki 1988) or the increase of cellulose crystallinity (Pétrissans *et al.* 2003).

The influence of a heat treatment on the surface hardness has been found to depend on the wood species, test directions, and time and temperature of the treatment (Shi *et al.* 2007). These authors stated that hardness generally increased for pine (*Pinus* spp.) and birch (*Betula* spp.), while it decreased for aspen (*Populus* spp.). Scots pine (*Pinus sylvestris* L.) hardness increased for wood that was treated by the Plato process (Boonstra *et al.* 2007a) and decreased with an oven treatment (Korkut *et al.* 2008). According to Sundqvist *et al.* (2006), the initial increase in the surface hardness might be linked to condensation reactions in the lignin and hemicellulose. According to Kvietková *et al.* (2015b), heat treatment has no effect on surface roughness of beech after plane milling but slightly reduces surface roughness of birch (Kvietková *et al.* 2015a).

This paper reports the changes in the surface properties of glossiness, color, pendulum hardness and surface adhesion on coated heat-treated (ThermoWood) and untreated beech (*Fagus orientalis* Lipsky.) wood by a UV system applied on KPS company.

EXPERIMENTAL

Materials

The beech (*Fagus orientalis* Lipsky.) wood samples were taken from Arın Forest Products Industry in Duzce, Turkey. Wood samples measuring 550 mm length × 100 mm width × 20 mm thickness was used in this study.

Methods

Heat treatment process

In the ThermoWood method, the application of a heat-treatment on freshly cut or air-dried wooden materials is recognized to involve three stages (Aytin 2013). The first stage is the increasing of the kiln temperature and the stage of drying at high temperatures. The temperature of the kiln was first immediately increased to 100 °C fast *via* making use of the heat and vapour and then it was slowly increased to 130 °C to fulfil the drying process at a higher temperature. The moisture content of the wooden material was decreased to almost 0% during this stage, which lasted between 14 to 30 h (Aytin 2013). The second stage is the stage of the heat treatment where the temperature in the kiln is increased to the desired heat treatment temperature which is usually between 185 and 215 °C for a period lasting to 6 to 8 h. In this study the samples were treated at 190 °C for 2 h and 212 °C for 1 and 2 h. The vapour was transferred into the kiln to prevent the wooden materials from becoming damaged (Aytin 2013). The final stage is the stage of cooling and conditioning. The temperature of the wooden materials was decreased to 50 and 60 °C by using a water spray system. The process was performed until the moisture content of the wooden materials reached 4% to 6%. The cooling and conditioning lasts for 24 and 30 h, according to the width and thickness of the wooden material. The total processing duration for ThermoWood lasts approximately 50 to 80 h (Aytin 2013).

The ThermoWood process was done in a private commercial Novawood factory in Gerece, Bolu, Turkey. The samples were heat treated according to the ThermoWood method and all of the untreated samples were conditioned to 12% moisture contents in a special room at 20 °C ± 2 °C and a relative humidity of 65% (± 5%) (ISO 554 1976).

Parquet Flooring Material

In this study, different layers of the UV system were produced and applied by the KPS Company. The production method of parquet flooring is shown in Table 1. Specimens were cut measuring 100 mm × 100 mm × 10 mm. In this study, the parquet samples have no paint application. All of the specimens were conditioned to 12% moisture contents (MC) in a special room at 20 °C ± 2 °C and 65% (± 5%) relative humidity (RH) (ISO 554 1976).

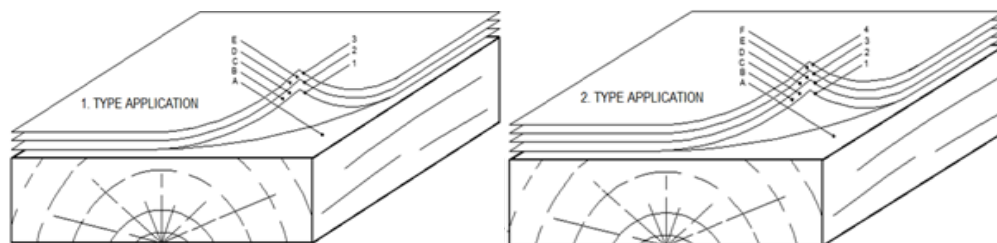


Fig. 1. 1. and 2. Type of Varnish Application Process

Table 1. Two Different Types of UV Varnish Application Process

1. Type of Varnish Application Process	A. Sanding (3 cylinders) & Calibrating Machines	80-120-220 grit sandpaper
	1. A43-0646-UV Sanding Sealer	50 g/m ²
	B. UV lamp drying (mercury)	2 x 80 W
	C. Sanding 2 cylinders	280-320 grit sandpaper
	2. N93-0910 Nanolacke UV Matt Varnish	7.5 g/m ²
	D. UV lamp drying	2 x 80 W
2. Type of Varnish Application Process	3. N93-0910 Nanolacke UV Matt Varnish	7.5 g/m ²
	E. UV lamp drying	400 W
	A. Sanding (3 cylinders) & Calibrating Machines	80-120-220 grit sandpaper
	1. A43-0646-UV Sanding Sealer	35 g/m ²
	B. UV lamp drying (mercury)	2 x 80 W
	2. A43-0646-UV Sanding Sealer	35 g/m ²
	C. UV lamp drying	400 W
	D. Sanding (2 cylinders)	280-320 grit sandpaper
	3. N93-0910 Nanolacke UV Matt Varnish	7.5 g/m ²
	E. UV lamp drying	2 x 80 W
4. N93-0910 Nanolacke UV Matt Varnish	7.5 g/m ²	
F. UV lamp drying	400 W	

Table 2. Some Properties of the Chemicals Used in the Production of Flooring UV System

	A43-0646 - UV Sanding Sealer	N93-0910 Nanolacke UV Matt Varnish
Description	Epoxy acrylic resin, ultraviolet ray curing sealers.	Polyacrylic-based resin, nano-containing minerals, nanocomposites Ultra Violet curing (UV) varnish.
Colour	Transparent	
Solids (wt.%)	95 to 97	95 to 100
Density (20 °C, g/cm ³)	1:15 to 1:20	1:09 to 1:15
Application	Solid hardwood, chipboard, medium density fibreboard; for application to the surface.	

Color Measurement

The color change of the heat-treated samples according to the ThermoWood method and the untreated beech (*Fagus orientalis* Lipsky.) of the one and two layers UV system that was applied on the parquet flooring materials was analyzed by a spectrometer (Datacolor 110, Suzhou Industrial Park, Suzhou, China) (Wavelength Resolution 10 nm, Measurement Geometry D/8°) with a D65 standard illuminant. The CIELAB system is characterized by three parameters, L^* , a^* , and b^* . The L^* axis represents the lightness, $+a^*$ is the red, $-a^*$ for green, $+b^*$ for yellow, $-b^*$ for blue, and L^* varies from 100 (white) to zero (black) (Zhang *et al.* 2009). The total color difference (ΔE^*) was calculated by Eq. 1:

$$\begin{aligned}
 \Delta L^* &= L^*_{\text{heat-treated and UV Varnishing System Applied}} - L^*_{\text{control and UV Varnishing System Applied}}, \\
 \Delta b^* &= b^*_{\text{heat-treated and UV Varnishing System Applied}} - b^*_{\text{control and UV Varnishing System Applied}}, \\
 \Delta a^* &= a^*_{\text{heat-treated and UV Varnishing System Applied}} - a^*_{\text{control and UV Varnishing System Applied}}, \\
 \Delta E &= [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (1)
 \end{aligned}$$

Glossiness Measurement

Surface brightness measurements were made of the different layers of the UV system that was applied to the heat-treated (ThermoWood) and untreated beech (*Fagus orientalis* Lipsky.) according to ISO 2813 (1994) with a Gloss Meter (PICOGLOSS 562 MC Erichsen, Measuring geometry: 20°/60°, Hemer, Germany) device. These measurements were carried out both perpendicular and parallel at an angle of 60°.

Adhesion Test

The adhesion resistance strength of the one and two layers of the UV system that was applied to the heat-treated and untreated beech (*Fagus orientalis* Lipsky.) was determined according to ASTM D-4541 (1995) with ALSA Laboratory Equipment Inc. brand 1 ton (10 kN) in an electromechanical universal testing machine (Umraniye / Istanbul, Turkey). The 404 Chemistry Inc. brand (Cekmekoy - Istanbul, Turkey) adhesive plastic steel was used. The adhesion resistance was calculated according to Eq. 2,

$$X = 4F / \pi \cdot d^2 \quad (2)$$

where F is the rupture force (Newton) and d is the diameter of the experiment cylinder (mm) (ASTM D-45411995).

Pendulum Hardness

The pendulum hardness of the heat-treated and untreated beech (*Fagus orientalis* Lipsky.) with both single and double layers of coating was determined with the König device (Pendulum Damping Tester, Model 299/300 Erichsen, Hemer, Germany) and in accordance to ASTM D 4366-95 (1984).

Statistical Analysis

The pendulum hardness, glossiness, color lightness, the total color, red color tone, and yellow color tone were measured using thirty replicates of each sample and an average value was reported. The adhesion resistance test (MPa) values were obtained with ten replicates of each sample. The Statistical Package for the Social Sciences (SPSS) 17 Software (Sun Microsystems, Inc., 4150 Network Circle, Santa Clara, California 95054, U.S.A.) programme was used for determining the statistical analysis.

RESULTS AND DISCUSSION

Color

Table 3 presents the statistical analysis of the variation of the color parameters due to the heat treatment. The lightness decreased with the increase of both the temperature and the duration of the treatment. For example, in the wood treated for 2 h, L^* decreased from about 70 to 39 at 190 °C and to 22 at 212 °C, while the wood treated at 212 °C for 1 h presented an L^* value of 44. With the exception of the wood treated at 190 °C for 2 h, all of the remaining samples with two layers presented a lower lightness. Similar results regarding the decrease in lightness have been reported for several other heat-treated woods (Sundqvist 2002; Bekhta and Niemz 2003; Sehlstedt-Persson 2003; Esteves *et al.* 2007; Barcık *et al.* 2014, 2015)

The variations in a^* chroma parameter were not consistent. Generally there was an increase followed by a decrease in redness (a^*). In relation to b^* , there was a decrease in the yellow (b^*) with the intensity of the treatment. Similar behaviour has been observed before. For instance, Esteves *et al.* (2007) heat-treated eucalypt (*Eucalyptus globulus*) and observed a slight increase followed by a decrease for coordinate a^* reaching -46%, -29% and -60% in relation to the initial wood. For coordinate b^* there was a decrease with the treatment time and temperature, with a maximum decrease of -71%, -66% and -86% for transverse, radial and tangential sections, respectively. Previous results have shown that the color chroma parameters behave differently according to the wood species. Both a^* and b^* increased for the heat-treated black locust (*Robinia pseudoacacia*) (Chen *et al.* 2012), while for Scots pine (*Pinus sylvestris*) a^* increased and b^* increased, decreasing afterwards (Aksoy *et al.* 2011). For rubberwood (*Hevea brasiliensis*) and silver oak (*Grevillea robusta*), heat treated at temperatures between 210 and 240 °C, a^* and b^* increased initially, decreasing afterwards (Srinivas and Pandey 2012). The effect of the application of two layers instead of one single layer in lightness, a^* and b^* was considered to be significantly different at the 95% level, which is shown in Table 3.

Table 3. Color Parameters for Finished Heat-Treated and Untreated Wood

Test	Heat treatment	Layer thickness	N	Mean	HG	Std. Deviation	Minimum	Maximum	
L*	Control	1 layer	30	70.03	A*	0.38	69.33	70.65	
		2 layers	30	63.19	B	0.36	62.41	63.64	
	190 °C - 2 h	1 layer	30	39.01	C	0.23	38.49	39.45	
		2 layers	30	39.23	C	0.48	38.42	39.88	
	212 °C - 1 h	1 layer	30	32.26	D	0.76	31.12	33.77	
		2 layers	30	30.33	E	0.43	29.51	30.95	
	212 °C - 2 h	1 layer	30	21.96	F	0.67	20.96	23.30	
		2 layers	30	20.34	G	0.37	19.54	20.76	
	a*	Control	1 layer	30	7.03	H	0.12	6.85	7.24
			2 layers	30	8.99	F	0.14	8.79	9.32
190 °C - 2 h		1 layer	30	14.09	C	0.07	13.98	14.30	
		2 layers	30	13.94	D	0.22	13.64	14.33	
212 °C - 1 h		1 layer	30	15.94	A*	0.13	15.61	16.13	
		2 layers	30	15.07	B	0.19	14.62	15.37	
212 °C - 2 h		1 layer	30	9.55	E	0.39	8.89	10.52	
		2 layers	30	7.92	G	0.20	7.32	8.20	
b*		Control	1 layer	30	27.48	B	0.18	27.14	27.91
			2 layers	30	29.39	A*	0.13	28.99	29.60
	190 °C - 2 h	1 layer	30	29.15	A	0.28	28.59	29.81	
		2 layers	30	27.64	B	0.38	27.18	28.92	
	212 °C - 1 h	1 layer	30	24.85	C	0.82	23.67	26.13	
		2 layers	30	21.19	D	0.62	20.04	22.07	
	212 °C - 2 h	1 layer	30	8.67	E	0.61	7.63	10.33	
		2 layers	30	7.30	F	0.27	6.47	7.69	

HG: Homogeneous Group, N: Number of measurements, Mean: Average, *: Maximum value

The ΔE^* parameter, which represents the total colour change (Table 4), increased with both treatment time and temperature, ranging from 31.8 and 51.7 for single coat and from 24.5 to 48.2 for double coated wood. Overall the total colour change is smaller for wood coated with two layers.

Table 4. The Color Changes (ΔL^* , Δa^* , Δb^* and ΔE^*) of Coated Heat-Treated Wood

Duration	Layer thickness	ΔL^*	Δa^*	Δb^*	ΔE^*
190 °C - 2 hours	1 layer	-31.02	7.06	1.67	31.86
	2 layers	-23.96	4.95	-1.75	24.53
212 °C - 1 hour	1 layer	-37.77	8.91	-2.63	38.90
	2 layers	-32.86	6.08	-8.20	34.41
212 °C - 2 hours	1 layer	-48.07	2.52	-18.81	51.68
	2 layers	-42.85	-1.07	-22.09	48.22

With heat treatment, both extractives and structural compounds are affected. In the first stages of the treatment most of the original wood extractives migrate to the surface, disappearing or being degraded afterwards but at the same time new ones are created from the degradation of the structural polymers (Esteves *et al.* 2008). Hemicelluloses are the first structural compounds affected by the heat treatment. Their deacetylation leads to the release of acetic acid that acts as a depolymerisation catalyst that further degrades wood

components (Tjeerdsma *et al.* 1998). Dehydration reactions occur simultaneously, inducing the formation of furfural and hydroxymethylfurfural (Tjeerdsma *et al.* 1998). Amorphous cellulose is the first to be affected, leading to the increase of cellulose crystallinity, while in lignin the ether linkages are cleaved and new free phenolic hydroxyl groups and α - and β -carbonyl groups are created, inducing crosslinking via formation of methylene bridges (Tjeerdsma *et al.* 1998). Several studies have supported the idea that these chemical changes are responsible for the changes in colour. For instance Sundqvist *et al.* (2006) found good correlations between lightness decrease and the formation of acids. Sehistedt-Persson (2003) attributed these changes to the formation of coloured degradation products from hemicelluloses and suggested that this colour change might be due to hydrolysis by a reaction similar to a Maillard reaction which is a well-known process in the food industry. Sundqvist and Morén (2002) reported that extractives seem to participate in the colour formation of hydrothermally treated wood. There has also been mentioned the formation of oxidation products such as quinones as the reason for colour change (Tjeerdsma *et al.* 1998; Mitsui *et al.* 2001; Bekhta and Niemz 2003).

Glossiness

Glossiness is an important property of finished wood surfaces to which finishing layers have been applied. Table 5 presents the glossiness for untreated and heat-treated wood with one or two layers of finishing. The results showed that although there was an initial increase in the glossiness for wood treated at 190 °C, it decreased later with the severity of the treatment. The final decrease is higher for glossiness that is parallel to the grain. Korkut *et al.* (2013) presented similar results that reported the change in glossiness due to a heat treatment on wild cherry wood and concluded that the glossiness decreased with time and temperature of the treatment. Aksoy *et al.* (2011) reported similar results with heat-treated scots pine. The initial increase in glossiness might result from the migration of the resins to the wood surface, as mentioned earlier. With the increase of the intensity of the treatment the resins are degraded or leave the wood resulting, in a decrease in glossiness. Rautkari *et al.* (2008) observed the same phenomenon in densified wood.

Table 5. Glossiness of Finished Heat-Treated and Untreated Wood

Test	Heat treatment	Layer thickness	N	Mean	HG	Std. deviation	Minimum	Maximum
Glossiness 60°T	Control	1 layer	30	17.23	CD	0.69	15.40	19.00
		2 layers	30	16.89	D	0.82	15.50	18.30
	190 °C - 2 h	1 layer	30	18.76	A*	0.66	17.20	19.80
		2 layers	30	17.96	B	0.96	15.50	19.40
	212 °C - 1 h	1 layer	30	17.72	B	0.57	16.60	18.90
		2 layers	30	17.55	CB	0.72	15.70	18.70
	212 °C - 2 h	1 layer	30	16.19	E	0.92	14.40	18.20
		2 layers	30	16.08	E	1.41	13.20	17.70
Glossiness 60°//	Control	1 layer	30	23.75	CD	1.89	19.20	26.20
		2 layers	30	23.23	DE	1.00	20.70	25.50
	190 °C - 2 h	1 layer	30	26.17	A*	1.30	22.70	27.80
		2 layers	30	25.61	A	1.44	23.10	28.00
	212 °C - 1 h	1 layer	30	24.90	B	0.92	22.80	26.20
		2 layers	30	24.24	BC	1.32	21.10	26.80
	212 °C - 2 h	1 layer	30	20.57	F	1.50	17.60	22.70
		2 layers	30	22.92	E	1.15	20.00	24.20

HG: Homogeneous Group, N: Number of measurements, Mean: Average, *: Maximum value

Hardness

Table 6 presents the pendulum hardness for untreated and heat-treated wood with both a single and a double layer. The hardness decreased with the heat treatment for beech coated with both single and double layers. Similar results were presented before for uncoated heat-treated aspen (Shi *et al.* 2007) and Scots pine (Korkut *et al.* 2008). Contrary results were reported for Scots pine treated by the Plato process, where the hardness increased (Boonstra *et al.* 2007b). Other authors have not found any change in the hardness for heat-treated beech and soft maple at 180 °C (Nejad *et al.* 2013). Sundqvist *et al.* (2006) justified these different results by stating that the initial increase in surface hardness might be linked to condensation reactions in the lignin and hemicellulose. Hardness of the samples and their surface quality are function of heat treatment. Salca and Hiziroglu (2014) stated that although hardness is affected by the heat treatment an important impact on hardness is induced by the wood species itself.

The finishing increased the hardness of the surface because double finished wood has a higher hardness. These results showed that the application of a double layer is favourable for applications where a good hardness is necessary such as in flooring.

Table 6. Pendulum Hardness for Finished Heat-Treated and Untreated Wood

Test	Heat treatment	Layer thickness	N	Mean	HG	Std. deviation	Minimum	Maximum
Pendulum Hardness	Control	1 layer	30	70.07	C	5.62	62.00	84.00
		2 layers	30	79.53	A*	4.59	70.00	90.00
	190 °C - 2 h	1 layer	30	69.37	CD	4.85	60.00	82.00
		2 layers	30	79.37	A	5.64	65.00	92.00
	212 °C - 1 h	1 layer	30	67.00	D	4.79	59.00	77.00
		2 layers	30	76.03	B	5.33	66.00	86.00
	212 °C - 2 h	1 layer	30	69.67	CD	5.40	60.00	86.00
		2 layers	30	74.90	B	5.14	66.00	89.00

HG: Homogeneous Group, N: Number of measurements, Mean: Average, *: Maximum value

Adhesion

With heat treatment the wood surface becomes more hydrophobic and less suitable for some adhesives, mainly aqueous adhesives. Table 7 presents the adhesion test for finished heat-treated and untreated wood with an epoxy acrylic resin sealer followed by a polyacrylic-based resin varnish. The results showed that the adhesion strength of this resin decreased for the heat-treated surfaces and that the decrease was higher for more severe treatments. Also the highest decrease was observed for double finished wood. Similar results were presented before for heat-treated Scots pine (*Pinus sylvestris*), aspen (*Populus tremuloides*), yellow poplar (*Liriodendron tulipifera*), and jack pine (*Pinus banksiana*) that was bonded with phenol resorcinol formaldehyde (PRF) and polyurethane (PUR) adhesives. All of the heat-treated wood presented a lower bonding strength than the untreated with aspen showing the worse results (Poncsák *et al.* 2007).

Table 7. Adhesion Test for Finished Heat-Treated and Untreated Wood

Test	Heat treatment	Layer thickness	N	Mean	HG	Std. deviation	Minimum	Maximum
Adhesion (MPa)	Control	1 layer	10	2.150	B	0.72	1.213	3.271
		2 layers	10	4.412	A*	0.31	3.892	4.831
	190 °C - 2 h	1 layer	10	2.142	B	0.58	1.505	3.228
		2 layers	10	2.196	B	0.50	1.631	3.137
	212 °C - 1 h	1 layer	10	1.744	BC	0.41	1.061	2.592
		2 layers	10	1.734	BC	0.27	1.248	2.229
	212 °C - 2 h	1 layer	10	1.564	C	0.46	1.134	2.258
		2 layers	10	1.972	BC	0.36	1.475	2.446
HG: Homogeneous Group, N: Number of measurements, Mean: Average, *: Maximum value								

The adhesion strength of water-based wood varnishes was found to decrease with the increase in temperature and time of the heat treatment (Kesik and Akyildiz 2015). This decrease has been attributed to the lower wettability of heat-treated wood which in accordance with Pecina and Paprzycki (1988) is owed to the formation of degradation compounds, and according to Pétrissans *et al.* (2003), to the increase of cellulose crystallinity. On the other hand, Hakkou *et al.* (2005) proposed that the wettability decrease could be owed to a modification of the conformational arrangement of wood biopolymers due to loss of residual water or, possibly, to the plasticization of lignin.

The adhesion on oil heat-treated wood surfaces was even more challenging because it was highly dependent on the amount of oil on the surface. For example, the adhesion on soft maple oil heat-treated wood was just one level lower than on untreated wood while in beech wood samples that had an oilier surface there was a remarkable adhesion loss for all of the coatings (Nejad *et al.* 2013).

The results showed that the wood surface properties depended on the treating time and temperature of the treatment but also on the wood species. Therefore an optimization procedure is needed for each species to attain the best combination between wood durability and stability and surface properties.

CONCLUSIONS

1. The results showed that there was a decrease in the lightness and in the yellow tone (b^*), while there was an increase followed by a decrease in the redness tone (a^*) with the intensity of the heat treatment.
2. There was an initial increase in glossiness, but it decreased later on with the severity of the treatment.
3. The hardness decreased with the severity of the treatment for beech coated with both single and double layers.
4. The adhesion decreased with the increase in temperature and duration of treatment.

ACKNOWLEDGEMENTS

The authors would like to thank ASD Laminat Factory in Duzce, Turkey for all the color (L^* , a^* and b^*) measurements, the Novawood factory in Gerece, Bolu, Turkey for the heat treatment and KPS company in Duzce, Turkey.

REFERENCES CITED

- Aksoy, A., Deveci, M., Baysal, E., and Toker, H. (2011). "Colour and gloss changes of Scots pine after heat modification," *Wood Research* 56(3), 329-336.
- ASTM D 4366-95 (1984). "Standard test methods for hardness of organic coatings by pendulum test," ASTM, Philadelphia, PA.
- ASTM D-4541 (1995). "Standard test method for pull-off strength of coatings using portable adhesion testers," ASTM International, West Conshohocken, USA.
- Aytin, A. (2013). *Effect of High Temperature Treatment on Physical, Mechanic, and Technologic Properties of Wild Cherry (Cerasus avium (L.) Monench)*, Doctoral Thesis, University of Duzce, Duzce, Turkey.
- Barcák, Š., Gašparík, M., and Horejš, P. (2014). "Influence of thermal modification on nail withdrawal strength of spruce wood," *BioResources* 9(4), 5963-5975.
- Barcák, Š., Gašparík, M., and Razumov, E. Y. (2015). "Effect of thermal modification on the colour changes of oak wood," *Wood Res* 60(3), 385-396.
- Bekhta, P., and Niemz, P., (2003). "Effect of high temperature on the change in color, dimensional stability and mechanical properties of spruce wood," *Holzforchung* 57(5), 539-546. DOI: 10.1515/HF.2003.080
- Boonstra, M., Van Acker, J., Kegel, E., and Stevens, M. (2007a). "Optimisation of a two-stage heat treatment process: Durability aspects," *Wood Sci. Technol.* 41(1), 31-57. DOI: 10.1007/s00226-006-0087-4
- Boonstra, M. J., Van Acker, J., Tjeerdsma, B. F., and Kegel, E. V. (2007b). "Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents," *Ann. For. Sci.* 64(7), 679-690. DOI: 10.1051/forest:2007048

- Chen, Y., Fan, Y., Gao, J., and Stark, N. M. (2012). "The effect of heat treatment on the chemical and color change of black locust (*Robinia pseudoacacia*) wood flour," *BioResources* 7(1), 1157-1170. DOI: 10.15376/biores.7.1.1157-1170
- Dirol, D., and Guyonnet, R. (1993). *Durability by Rectification Process* (IRGWP 93-40015), International Research Group on Wood Protection, Stockholm, Sweden.
- Esteves, B., Graça, J., and Pereira, H. (2008). "Extractive composition and summative chemical analysis of thermally treated eucalypt wood," *Holzforschung* 62, 344-351. DOI: 10.1515/HF.2008.057
- Esteves, B. M., and Pereira, H. (2009). "Wood modification by heat treatment: A review," *BioResources* 4(1), 340-404. DOI: 10.15376/biores.4.1.340-404
- Esteves, B., Marques, A. V., Domingos, I., and Pereira, H. (2006). "Influence of steam heating on the properties of pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood," *Wood Sci. Technol.* 41, 193-207. DOI: 10.1007/s00226-006-0099-0
- Esteves, B., Velez Marques, A., Domingos, I., and Pereira, H. (2007). "Heat-induced colour changes of pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood," *Wood Sci. Technol.* 42, 369-384. DOI: 10.1007/s00226-007-0157-2
- Hakkou, M., Pétrissans, M., El Bakali, I., Gérardin, P., and Zoulalian, A. (2005). "Wettability changes and mass loss during heat treatment of wood," *Holzforschung* 59(1), 35-37. DOI: 10.1515/HF.2005.006
- 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, Geneva, Switzerland
- ISO 554 (1976). "Standard atmospheres for conditioning and/or testing – Specifications," International Organization for Standardization, Geneva, Switzerland.
- Kesik, H. I., and Akyildiz, M. H. (2015). "Effect of the heat treatment on the adhesion strength of water based wood varnishes," *Wood Res.* 60(6), 987-994.
- Kim, G. -H., Yun, K. E., and Kim, J. J. (1998). "Effect of heat treatment on the decay resistance and the bending properties of radiata pine sapwood," *Mater. Organismen* 32(2), 101-108.
- Korkut, D. S., Hiziroglu, S., and Aytin, A. (2013). "Effect of heat treatment on surface characteristics of wild cherry wood," *BioResources* 8(2), 1582-1590. DOI: 10.15376/biores.8.2.1582-1590
- Korkut, D. S., Korkut, S., Bekar, I., Budakçi, M., Dilik, T., and Cakicier, N. (2008). "The effects of heat treatment on the physical properties and surface roughness of Turkish Hazel (*Corylus colurna* L.) wood," *Int. J. Mol. Sci.* 9(9), 1772-1783. DOI: 10.3390/ijms9091772
- Kubojima, Y., Okano, T., and Ohta, M. (2000). "Bending strength and toughness of heat-treated wood," *J. Wood Sci.* 46(1), 8-15. DOI: 10.1007/BF00779547
- Kvietková, M., Gaff, M., Gašparík, M., Kaplan, L., and Barčík, S. (2015a). "Surface quality of milled birch wood after thermal treatment at various temperatures," *BioResources* 10(4), 6512-6521. DOI: 10.15376/biores.10.4.6512-6521
- Kvietková, M., Gašparík, M., and Gaff, M. (2015b). "Effect of thermal treatment on surface quality of beech wood after plane milling," *BioResources* 10(3), 4226-4238. DOI: 10.15376/biores.10.3.4226-4238

- Mitsui, K., Takada, H., Sugiyama, M., and Hasegawa, R. (2001). "Changes in the properties of light-irradiated wood with heat treatment. Part 1: Effect of treatment conditions on the change in color," *Holzforschung* 55(6), 601-605. DOI: 10.1515/HF.2001.098
- Nejad, M., Shafaghi, R., Ali, H., and Cooper, P. (2013). "Coating performance on oil-heat-treated wood for flooring," *BioResources* 8(2), 1881-1892. DOI: 10.15376/biores.8.2.1881-1892
- Pecina, H., and Paprzycki, O. (1988). "Interactions between the temperature treatment of the wood and its wettability," *Holzforsch. Holzverw.* 40(1), 5-8 (in German).
- Pétrissans, M., Gérardin, P., and Serraj, M. (2003). "Wettability of heat-treated wood," *Holzforschung* 57(3), 301-307. DOI: 10.1515/HF.2003.045
- Poncsák, S., Shi, S. Q., Kocafe, D., and Miller, G. (2007). "Effect of thermal treatment of wood lumbers on their adhesive bond strength and durability," *J. Adhes. Sci. Technol.* 21(8), 745-754. DOI: 10.1163/156856107781362653
- Rautkari, L., Properzi, M., Pichelin, F., and Hughes, M. (2008). "An innovative thermo densification method for wooden surfaces," in: Proceedings of the 10th World Conference on Timber Engineering.
- Salca, E. A., and Hizirolu, S. (2014). "Evaluation of hardness and surface quality of different wood species as function of heat treatment," *Mater. Des.* 62, 416-423. DOI: 10.1016/j.matdes.2014.05.029
- Sehlstedt-Persson, M. (2003). "Colour responses to heat treatment of extractives and sap from pine and spruce," in: 8th International IUFRO Wood Drying Conference, Brasov, Romania, pp. 459-464.
- Shi, J. L., Kocafe, D., and Zhang, J. (2007). "Mechanical behaviour of Quebec wood species heat-treated using ThermoWood process," *Holz. Roh. Werkst.* 65(4), 255-259. DOI: 10.1007/s00107-007-0173-9
- Srinivas, K., and Pandey, K. K., (2012). "Effect of heat treatment on color changes, dimensional stability, and mechanical properties of wood," *J. Wood Chem. Technol.* 32(4), 304-316. DOI: 10.1080/02773813.2012.674170
- Sundqvist, B. (2002). "Color response of Scots pine (*Pinus sylvestris*), Norway spruce (*Picea abies*), and birch (*Betula pubescens*) subjected to heat treatment in capillary phase," *Holz. Roh. Werkst.* 60(2), 106-114. DOI: 10.1007/s00107-001-0273-x
- Sundqvist, B., and Morén, T. (2002). "The influence of wood polymers and extractives on wood colour induced by hydrothermal treatment," *Holz. Roh. Werkst.* 60(5), 375-376. DOI: 10.1007/s00107-002-0320-2
- Sundqvist, B., Karlsson, O., and Westermarck, U. (2006). "Determination of formic-acid and acetic acid concentrations formed during hydrothermal treatment of birch wood and its relation to colour, strength and hardness," *Wood Sci. Technol.* 40, 549-561. DOI: 10.1007/s00226-006-0071-z
- Tjeerdsma, B. F., Boonstra, M., Pizzi, A., Tekely, P., and Militz, H. (1998). "Characterisation of thermally modified wood: Molecular reasons for wood performance improvement," *Holz. Roh. Werkst.* 56, 149-153. DOI: 10.1007/s001070050287
- Tjeerdsma, B. F., Stevens, M., Militz, H., and Van Acker, J. (2002). "Effect of process conditions on moisture content and decay resistance of hydro-thermally treated wood," *Holzforsch. Holzverwer.* 54(5), 94-99.

Viitaniemi, P., Jämsä, S., and Viitanen, H. (1997). “Method for improving biodegradation resistance and dimensional stability of cellulosic products,” U. S. Patent No. 5678324.

Zhang, J., Kamdem, D. P., and Temiz, A., (2009). “Weathering of copper-amine treated wood,” *Appl. Surf. Sci.* 256(3), 842-846. DOI: 10.1016/j.apsusc.2009.08.071

Article submitted: October 24, 2016; Peer review completed: January 11, 2017; Revised version received: January 13, 2017; Accepted: January 21, 2017; Published: April 12, 2017.

DOI: 10.15376/biores.12.2.3876-3889

Copyright of BioResources is the property of North Carolina State University c/o Forest Biomaterials and its content may not be copied or emailed to multiple sites or posted to a listserv without the copyright holder's express written permission. However, users may print, download, or email articles for individual use.