SURFACE CHARACTERISTICS OF ORIENTAL BEECH AND SCOTS PINE WOODS HEAT⁻TREATED ABOVE 200°C

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ABSTRACT

Heat-treated of Oriental beech (*Fagus orientalis* L.) and Scots pine (*Pinus sylvestris* L.) woods were carried out by hot air in an oven for 1, 3, and 5 hours at 205, 220, and 235°C. After heat treatments, some surface properties such as color and gloss changes of both wood specimens were evaluated.

Our results showed that heat treatment caused decrease in gloss values of Oriental beech and Scots pine wood specimens. Higher treatment temperature and duration resulted in higher gloss loss of wood specimens after heat treatments. Heat-treated wood became darker tonality, especially; it became more darkening after 3 and 5 hours heat treatments. Generally according to our results, Δa^* and Δb^* decreased after heat treatments. Oriental beech wood tended to become less reddish than Scots pine after heat treatments. Δa^* , Δb^* and ΔE^* of heat-treated Oriental beech and Scots pine decreased with increasing treatment temperature and duration.

INTRODUCTION

Wood is sustainable and environmentally friendly natural material widely used for both structural and non-structural applications (Tomak et al. 2012, Trisna and Hiziroglu 2013). However, being biological material, it is susceptible to environmental degradation. It is necessary to treat wood to provide long service life and improve specific surface properties on the intended application (Srinivas and Pandey 2012a). Heat treatment, as a modification method, is an effective method to improve biological durability and dimensional stability of wood (Poncsak et al. 2006, Shi et al. 2007, Kocaefe et al. 2008a, Korkut et al. 2008, Yildiz et al. 2011). In addition, it is alternative to the preservation methods using chemical agents (Kocaefe et al. 2008a, 2013). Heat treated wood products can be used for an extensive range of applications such as wood pallets, decking, window frames, garden fences, sauna furnishing and exterior joinery (Yildiz et al. 2011). Thus, heat treatment has been widespread in the productions of value added forest products.

A typical heat treatment is applied at temperature levels and exposure times ranging from 120 to 250°C and from 15 min to 24 h, respectively, depending on the process, species, sample size, moisture content and the desired target utilization (Sevim Korkut and Guller 2008, Kasemsiri et al. 2012, Salca and Hiziroglu 2014). It is stated that heat treatment of wood at relatively high temperatures (in the range of 150-250°C) is more effective to improve biological durability of wood (Kocaefe et al. 2008b). The high temperature heat treatment technology for wood has recently attracted a lot of interest in various countries (Kocaefe et al. 2007). In this process, wood is heated to temperatures above 200°C (Bekhta and Niemz 2003, Kocaefe et al. 2007, 2013). However, physical, mechanical and chemical properties of wood under heat treatment change at temperature near 150°C and it continues with increasing temperature (Yildiz et al. 2006, Korkut and Guller 2008, Salca and Hiziroglu 2014). Wood treated at high temperature has less hygroscopicity than natural wood (Gunduz et al. 2008). When wood becomes less hydroscopic after heat treatment, chemical modification and degradation of wood components take place through dehydration, hydrolysis, oxidation, decarboxylation, and transglycosylation (Kocaefe et al. 2008b). During thermal treatment at high temperatures, the color of wood tends to darken due to the considerable changes in the chemical composition of wood, such as the degradation of the amorphous carbohydrates (Kamperidou et al. 2012). Hemicelluloses start to decompose first among the wood polymers, due to the low molecular weight that makes them more reactive (Hakkou et al. 2005, Kamperidou et al. 2012). Additionally, lignin softens; cellulose and hydrophilic groups modify (Bekhta and Niemz 2003, Kocaefe et al. 2007). As a result of this process, heat treated wood loses its reabsorbing water capacity, acquiring a more hydrophobic behavior compared to untreated wood (Kocaefe et al. 2007, Kamperidou et al. 2012). It has been observed in the studies of wood modification at high temperatures that the mechanical properties of wood decrease (Bekhta and Niemz 2003, Yildiz et al. 2006, Kocaefe et al. 2008b, Mburu et al. 2008, Srinivas and Pandey 2012b). On the other hand, heat treatment at high temperatures provides new physical properties to wood such as reduced hygroscopy, improved dimensional stability, better resistance to degradation by insects and micro-organisms, and most importantly, attractive darker color (Huang et al. 2012). From earlier investigations, it is known that wood exposed to high temperature become darker than untreated wood (Bekhta and Niemz 2003, Aksoy et al. 2011, Srinivas and Pandey 2012b). Heat treatment gives wood an attractive darker color for aesthetic uses (Huang et al. 2012, Todorovic et al. 2012, Cademartori et al. 2014) and uniform tone (Srinivas and Pandey 2012b). Wood color and gloss are important for wood applications in terms of aesthetic considerations, and sometimes may determine its value in the market (Pandey 2005, Tolvaj et al. 2011, Huang et al. 2012). Thus, in this study, it was aimed to investigate some surface characteristics such as color and gloss changes of Oriental beech and Scots pine woods heat-treated above 200°C.

MATERIALS AND METHODS

Preparation of test specimens

Specimens 10 x 100 x 150 mm (radial by tangential by longitudinal) were machined from the air-dried sapwood of Oriental beech (*Fagus orientalis* L.) and Scots pine (*Pinus sylvestris* L.) lumber. All specimens were conditioned at 20°C and 65 % relative humidity for two weeks before tests.

Heat treatment

Heat treatment was performed using a temperature-controlled laboratory oven. Three different temperatures (205, 220, and 235°C) and three treatment durations (1, 3, and 5 hours) were applied to Oriental beech and Scots pine wood specimens under atmospheric pressure and in the presence of air.

Color test

The colors of the specimens were measured by a colorimeter (X-Rite SP Series Spectrophotometer) before and after the heat treatments. The spectrophotometer measures color space that measures sample lightness (L) and color coordinates (a and b). This system is called CIELAB and works according to the CIE standard. The color space is given Fig. 1.

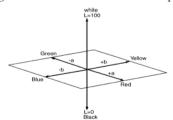


Fig. 1: The CIE L*a*b* color space.

The *L* values mean the samples are lightening or fading (i.e., a positive ΔL for lightning and negative ΔL for darkening). Coordinate a represents the red-green coordinate while *b* coordinate represents the yellow-blue direction. In this way, *a* positive Δa value represents a color shift towards red while *a* negative Δa value represents a shift towards green. Similarly, *b* positive Δb values represent a color shift towards yellow while *b* negative Δb value represents a shift towards blue. The total color change ΔE is calculated according to ASTM D 2244 (2015) (Xue et al. 2012, Turkoglu et al. 2015).

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

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where: ΔL^* , Δa^* and Δb^* - the differences between the initial and final values (before and after heat treatment) of L^* , a^* and b^* , respectively. Values were measured before and after the heat treatment at three different locations for each set of samples (n = 10) and mean value calculated (Srinivas and Pandey 2012b). The average color values, standard deviations and 95 % confidence intervals (5 % significance level), based on distribution, were calculated assuming normal distribution (Korkut et al. 2012, Akgul et al. 2013). Color measurements were made in the direction parallel to the fiber.

Gloss test

The gloss of wood specimens was determined using a gloss meter (BYK Gardner, MicroTRI-Gloss) according to ASTM D523-08 (2008). The chosen geometry was an incidence angle of 60°. Results were based on a specular gloss value of 100, which relates to the perfect condition under identical illuminating and viewing conditions of a highly polished, plane, black glass surface. Gloss measurements were made in the direction parallel to the fiber.

Evaluations of test results

The results of total color and gloss changes were evaluated by a computerized statistical program composed of analysis of variance and following Duncan tests at the 95 % confidence level. Statistical evaluations were made on homogeneity groups of which different letters reflected statistical significance in the tables.

RESULTS AND DISCUSSIONS

Gloss changes

Gloss values of the Oriental beech and Scots pine at a 60° incidence angle measured before and after heat treatments are given in Tabs. 1 and 2, respectively. Glossiness, the property of reflecting light in a mirror is very important for the aesthetic and decorative appearance of surfaces (Cakicier et al. 2011). Our results showed that heat treatments decreased gloss values of the Oriental beech and Scots pine wood specimens to some extent.

Heat	Time	Before heat	treatment	After heat	Difference		
treatment (°C)	(hour)	Mean	SD	Mean	SD	(%)	
	1	4.80	1.74	3.94	1.10	-17.92ª	
205	3	5.70	2.15	4.05	1.54	-28.94 ^{abc}	
	5	4.04	1.60	2.50	0.83	-38.11 ^{cd}	
	1	4.72	1.85	3.76	1.05	-20.34 ^{ab}	
220	3	6.80	2.38	3.72	1.10	-45.29 ^{de}	
	5	5.04	1.39	2.40	0.61	-52.38e	
235	1	4.38	1.54	3.04	0.92	-30.59 ^{bc}	
	3	3.90	1.21	2.04	0.73	-47.69 ^{de}	
	5	5.62	2.20	1.96	0.65	-65.12 ^f	

Tab. 1: The gloss values of Oriental beech before and after heat treatment.

Note: Small letters given as superscript over difference of gloss values represent homogeneity groups obtained by statistical analysis program. Different letters reflect statistical significance at the 95 % confidence level. SD: Standard deviation. Ten replicates were made for each treatment group.

According to our results, gloss value was the lowest degree when Oriental beech and Scots pine were heat-treated for 5 h at 235°C. From this result, the maximum decrease in gloss was obtained for Oriental beech and Scots pine wood specimens at 235°C for 5 h as 65.12 and 69.38 %, respectively. The gloss loss of Oriental beech and Scots pine changed 17.92 to 65.12 % and 15.50 to 69.38 %, respectively after heat treatments. In literature, Baysal et al. (2014) found that 39.69 % gloss loss of heat-treated Oriental beech at 200°C for 8 h. Sevim Korkut et al. (2013) reported that gloss loss of wild cherry wood was 36.6 % after heat treatment at 212°C for 2.5 h.

Heat treatment	Time	Before heat treatment		After h	eat treatment	Difference(%)	
(°C)	(hour)	Mean	SD	Mean	SD	Difference(%)	
	1	5.16	1.66	4.36	1.34	-15.50a	
205	3	3.36	0.90	1.85	0.49	-44.94bc	
	5	5.46	1.53	2.82	0.95	-48.35c	
	1	4.18	1.68	3.44	1.07	-17.70a	
220	3	6.30	2.45	3.27	1.35	-48.09c	
	5	4.74	1.53	2.18	0.70	-54.01c	
	1	4.06	1.93	2.69	0.95	-33.74b	
235	3	5.73	2.27	2.51	0.80	-56.19c	
	5	5.88	1.85	1.80	0.65	-69.38d	

Tab. 2: The gloss values of Scots pine before and after heat treatment.

Note: Small letters given as superscript over difference of gloss values represent homogeneity groups obtained by statistical analysis program. Different letters reflect statistical significance at the 95 % confidence level. SD: Standard deviation. Ten replicates were made for each treatment group.

Aksoy et al. (2011) found that gloss loss values for Scots pine wood after heat treatment at 200°C for 4 h was 36.6 %. Therefore, gloss loss of wood after heat treatment depends on heat treatment temperature and treatment time. The results of this study on the effect of heat treatment on Oriental beech and Scots pine are generally compatible with the findings in literature on the effect of heat treatment on loss of gloss. However, in our study gloss loss of heat-treated Oriental beech and Scots pine wood specimens were higher than aforementioned studies. It can be resulted from higher heat treatment temperature was applied compared to the mentioned above in literature. Our results showed that gloss values of heat-treated Oriental beech and Scots pine wood specimens decreased with increasing treatment temperature and duration. Similar results were recorded by Korkut et al. 2012, Korkut 2012, Baysal et al. 2014 who studied the effects of heat treatment on different wood species of which gloss values decreased with increasing heat treatment time and temperature. Moreover, gloss loss of heat-treated Oriental beech and Scots pine wood specimens for 1 h was rather lower than heat-treated both wood specimens for 3 and 5 h. For example, gloss loss of Oriental beech and Scots pine wood specimens were 30.59 and 33.74 %, respectively for 1 h at 235°C, gloss loss of Oriental beech and Scots pine wood specimens were 65.12 and 69.38 %, respectively for 5 h at 235°C. Our results showed that there was a statistical difference for Oriental beech and Scots pine in gloss loss values between heat treated at 235°C for 5 h and other treatment groups.

Color changes

 L^* , a^* , b^* values and the changes of ΔL^* , Δa^* , Δb^* , and ΔE^* of non-heated and heat- treated Oriental beech and Scots pine before and after heat treatment are given in Tabs. 3 and 4, respectively. The decrease in L^* values after heat treatment indicates that the specimens become

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darker. Darkening was increased with increasing heat treatment temperature and duration and this finding is compatible with earlier studies (Aksoy et al. 2011, Militz 2002, Yildiz et al. 2011, Akgul and Korkut 2012, Guller 2012, Cademartori et al. 2013a, b Baysal et al. 2014). For instance, L^* of Scots pine after heat treatment at 205, 220, and 235°C for 1 h , decreased 7.11, 11.63, and 44.76 % , respectively and the decline of L^* values within 3 h of treatment was observed 43.91, 57.58, and 63.28 %, respectively. Our results clearly showed that except for at 235°C for 1 h treatment of Oriental beech and Scots pine, ΔL^* values of heat-treated Oriental beech and Scots pine wood specimens for 1 h at 205 and 220°C were remarkably lower compared to for 3 and 5 h at same temperatures. Esteves et al. (2008) reported a 52.9 % decrease in lightness of pine wood for a treatment at 200°C for 24 h. Gunduz and Aydemir (2009) found a 64.23 % decrease in lightness of Hornbeam wood for a treatment at 200°C for 12 h.

Oriental beech											
Heat	Time (hour)	Before heat treatment			After heat treatment						
treatment (°C)		L_i^*	a_i^*	b_i^*	L_{f}^{*}	a_f^*	b_{f}^{*}	ΔL^*	Δa^*	Δb^*	ΔE^*
205	1	67.64	11.28	19.58	62.00	10.95	18.88	-5.64	0.33	-0.70	5.69a
	3	66.60	11.92	20.04	39.15	8.37	13.13	-27.45	-3.55	-6.91	28.53b
	5	68.94	11.03	19.15	30.30	4.92	6.49	-38.63	-6.11	-12.65	41.11cd
220	1	67.04	11.41	19.55	57.48	10.83	18.38	-9.57	-0.58	-1.17	9.66a
	3	67.36	10.90	19.64	28.86	2.79	3.19	-38.50	-8.11	-16.45	42.64cd
	5	66.18	11.84	20.06	27.23	1.08	1.08	-38.95	-10.75	-18.98	44.64d
235	1	66.70	11.83	20.50	34.22	6.89	10.54	-32.48	-4.94	-9.96	34.33bc
	3	66.09	11.91	20.01	28.02	1.73	1.83	-38.08	-10.18	-18.17	43.41cd
	5	68.25	10.98	19.62	25.99	0.57	0.46	-42.26	-10.40	-19.15	47.55d

Tab. 3: The color changes of Oriental beech before and after heat treatment.

Note: Small letters given as superscript over difference of total color change represent homogeneity groups obtained by statistical analysis program. Different letters reflect statistical significance at the 95 % confidence level. Ten replicates were made for each treatment group.

Scots pine											
Heat treatment (°C)	Time (hour)	Before heat treatment			After heat treatment						
		L_i^*	a_i^*	<i>b</i> _{<i>i</i>} *	L_{f}^{*}	a_f^*	b_{f}^{*}	ΔL^*	Δa^*	Δb^*	ΔE^*
205	1	76.1	7.43	28.72	70.69	8.96	31.48	-5.41	1.53	2.76	6.26a
	3	72.72	8.76	30.4	40.79	10.91	17.88	-31.93	2.14	-12.52	34.37b
	5	74.40	7.75	29.47	31.48	7.87	9.73	-42.93	0.12	-19.74	47.25c
220	1	77.89	6.64	28.38	68.83	9.15	28.83	-9.06	2.51	0.45	9.41a
	3	75.69	7.72	30.60	32.12	7.80	10.47	-43.58	0.07	-20.13	48.00c
	5	75.46	7.38	29.81	28.75	1.09	0.25	-46.71	-6.29	-29.55	55.63c
235	1	75.73	7.75	30.72	41.83	11.77	21.2	-33.90	4.01	-9.53	35.44b
	3	73.72	8.23	30.66	27.07	3.15	2.69	-46.65	-5.08	-27.97	54.63c
	5	77.94	6.34	29.34	27.62	-0.13	-0.57	-50.33	-6.47	-29.91	58.90c

Tab. 4: The color changes of Scots pine before and after heat treatment.

Note: Small letters given as superscript over difference of total color change represent homogeneity groups obtained by statistical analysis program. Different letters reflect statistical significance at the 95 % confidence level. Ten replicates were made for each treatment group.

In our study, the maximum lightness reduction of heat-treated Oriental beech and Scots pine were 61.92 and 64.58 %, respectively at 235°C after 5 h of treatment. The darkening of heattreated Oriental beech and Scots pine pine might be due to degradation of lignin and other noncellulosic polysaccharides (Hon and Chang 1985, Grelier et al. 2000, Petric et al. 2004). Bourgios et al. (1991) reported that wood darkening after heating might be related to a reduction in holocellulose content as the temperature increases. As wood is heated, acetic acid is formed from acetylated hemicelluloses by hydrolysis (Forsman 2008). The released acid serves as a catalyst in the hydrolysis of hemicelluloses to the soluble sugars (Finnish Thermowood Association 2003). The heat caramelizes the sugar to a brown color which affects the color of wood. Mitsui (2004) reported that decline in lightness in Cryptomeria japonica wood was attributed to the extractives and low molecular weight compounds produced by hemicelluloses and lignin. While the positive values of Δa^* indicate a tendency of wood surface to become reddish, the negative values of Δa^* indicate a tendency of wood surface to become greenish. Except for 1 h at 205°C for Oriental beech, heat-treated Oriental beech wood surface become greenish after heat treatment. The Δa^* of heat-treated Oriental beech decreased with increasing treatment temperature and duration, it became more greenish. Our results showed that Oriental beech wood tended to become less reddish than Scots pine wood after heat treatments. For example, heat-treated Oriental beech wood showed a tendency to reddish only for 1 h at 205°C. Pincelli et al. (2012) reported that Eucalyptus saligna wood tended to become less reddish as the maximum temperature in thermal rectification increased. Moura and Brito (2011) found that decreasing in red color of Eucalyptus grandis after heat treatments is related with volatilizing of some chemical compounds that give red color to *Eucalyptus grandis*. Positive values of Δb^* indicate a tendency of wood surface to yellowish while negative values mean a tendency to bluish. Negative values of Δb^* for Oriental beech indicated that wood surface became bluish after all heat treatment conditions. The Δb^* of heat-treated Oriental beech decreased with increasing treatment temperature and duration, it became more bluish. For Scots pine wood, positive values of Δb^* was seen only heat-treated for 1 h at 205 and 220°C. Other all heat-treated Scots pine wood surface became bluish after heat treatments. Gunduz and Aydemir (2009) found that while the b* value of heat-treated Hornbeam wood increased at 170°C, and then the b^* value decreased when heated at 190 and 210°C. Johansson and Morén (2006) studied color characteristics of heat-treated birch wood. They found that the yellow b^* coordinate was fairly constant for the different times at 175°C. When treated at 200°C, the b^* decreased over time. Bekhta and Niemz (2003) reported that the b^* value of spruce wood specimens increased with increase in treatment temperature up to 150°C (reaches a maximum value at this temperature), and afterwards began to decrease. Akgul and Korkut (2012) studied color changes of heat-treated Uludag fir wood. They found that heat-treated wood specimens, with increasing yellowness initially and decreased yellowness later for more severe treatments. Baysal et al (2014) reported that b^* of Oriental-beech wood initially increased at 140 and then decreased at 170 and 200°C. Moura and Brito (2011) reported that a severe decrease of yellow color in Pinus caribaea var. hondurensis heat treated above 160°C. Schnabel et al. (2007) investigated color characteristics of thermally modified European ash and European beech. They found that the b* value increased slightly due to low heat treatment. However, even when high temperatures were applied, the b^* value decreased. Generally aforementioned studies, b^* values of wood was decreased when applied more severe treatment conditions. Therefore, wood surface became more bluish after severe heat treatment conditions. The results of this study are consistent with these finding. The highest color difference (ΔE^*) value was 47.55 for Oriental beech and 58.90 for Scots pine after heat treatment at 235°C for 5 h. The total color difference of wood appeared to be well correlated with treatment temperature and time for Oriental beech and Scots

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pine. The total color change (ΔE^*) of heat-treated Oriental beech and Scots pine were changed from 5.69 to 47.55 and 6.26 to 58.90, respectively. It was found that the total colour changes of heat-treated Oriental beech and Scots pine wood increased with increasing treatment temperature and duration. Also, it was determined that ΔE^* values of both wood specimens heat-treated for 3 and 5 h were highly higher than heat-treated of both wood specimens for 1 h. Our results showed generally total color changes of Scots pine were higher than Oriental beech wood. According to the statistical test results, in general there was a statistical difference in ΔE^* values between heattreated for 3 and 5 hours at 220 and 235°C and other treatment groups.

CONCLUSIONS

Gloss and color changes of Oriental beech and Scots pine wood specimens heat- treated at 205, 220, and 235°C for 1, 3, 5 hours were studied. Heat treatments caused loss gloss of wood specimens. Moreover, it more pronounced at higher treatment temperatures and durations. Heat-treated wood surface became darken and it increased with increasing treatment temperatures and durations. Generally, Δa^* and Δb^* values of both wood specimens showed negative values after heat treatments. The Δa^* and Δb^* values decreased with increasing treatment temperature and durations. Higher temperature and duration resulted in higher total color changes of wood. In general, total colors changes of Scots pine wood were higher than that of Oriental beech wood. Gloss and color changes of both wood specimens were highly higher for 3 and 5 hours heat-treated wood then that of 1 hour heat-treated wood.

In conclusion, heat treatments caused gloss loss, darkening, bluish, and greenish of wood. Higher treatment temperature and longer times resulted in higher gloss loss, darkening, bluish, and greenish wood. Color is a very important wood property for the final consumer and, in some cases it is determining factor for the selection of a specific wood since the visual decorative point of is often prevailing (Esteves et al. 2008). Especially, heat-treated wood acquired a darker color similar to most tropical woods, which is an aesthetical advantage for some applications (Tuong and Li 2010). Moreover, it has a darkened color which is highly preferred in furniture industry. Also, because of its high dimensional stability, reduced hygroscopicity, good weathering and decay resistance etc., heat-treated wood may be alternative structural material for outdoor applications. It is also suited for end uses where it is an advantage that resin has flown out of wood and heat insulation has increased like interiors in bathrooms and saunas (Jämsä and Viitaniemi 2001). However, heat treatment causes strength loss of wood. Thus, heat-treated wood should not be used where strength is a dominant factor.

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