

ESTIMATION OF HEAT-TREATED BEECHWOOD PROPERTIES BY COLOR CHANGE

Nebojša Todorović,* Zdravko Popović, Goran Milić, and Ranko Popadić

Changes in color (CIE $L^*a^*b^*$) and properties (density, mass loss, density loss, and bending properties) of heat-treated beechwood were researched, as well as the possibilities of predicting these properties based on color. Considering the different market values of sapwood and red heartwood, the aim of this study was to establish whether these parts of beechwood differ after a heat treatment. Samples were exposed to temperatures of 170°C, 190°C, and 210°C, respectively, for 4 hours. In order to predict the properties, a linear regression with color change (ΔE) and ΔL predictors was used, as well as the partial least squares (PLS) regression with 12 color variables. It has been shown that heat treatment reduces the properties of sapwood and red heartwood in the same manner, and equalizes the colors. The PLS-R showed the best results of prediction and presented the very high coefficients of determination for the mass loss, density loss, and modulus of rupture (MOR) in both sapwood and red heartwood. The equalized colors of heat-treated red heartwood and sapwood can significantly increase the use of products made out of red heartwood. Color can be an important indicator of the quality of such beechwood.

Keywords: Beechwood; Red heartwood; Heat treatment; Color; Multivariate estimation

Contact information: University of Belgrade, Faculty of Forestry, Department of Wood Processing, Kneza Visislava 1, 11030 Belgrade, Serbia; * Corresponding author: nebojsa.todorovic@sfb.bg.ac.rs

INTRODUCTION

Heat treatment can reduce wood hygroscopicity and improve its dimensional stability and durability, but it may decrease some mechanical properties, mainly bending (Kubojima et al. 2000; Yildiz 2002; Bekhta and Niemz 2003; Johansson and Morén 2006; Esteves et al. 2007; Shi et al. 2007; Kocafe et al. 2008). Along with the improvement of hygroscopicity and durability, one of the important reasons for heat-treating is the change in wood color (Mitsui et al. 2001; Bekhta and Neimz 2003; Johansson and Morén 2006; Esteves et al. 2008b; González-Peña and Hale 2009; Toung and Li 2010). Heat treatment gives wood a darker color that has become very interesting on the market, which enables a broader use and an increased market value of less-valued species. The changes in color seem to originate from complex changes and degradation of hemicelluloses, lignin, and certain extractive compounds (Tjeerdma et al. 1998; Sundqvist and Moren 2002; Bekhta and Neimz 2003; Sehistedt-Persson 2003; Sundqvist 2004; Windeisen et al. 2009; Kocafe et al. 2008; Niemz et al. 2010; Aydemir et al. 2011). Because the process of heat treatment changes both chemical and some of the physical and mechanical properties, color could be used to predict these changes, and it

could, therefore, become an important indicator of the quality of heat-treated wood. Along with color, NIR spectra and ESR spectra are used for assessing the properties and classifying the heat-treated wood (Hinterstoisser et al. 2003; Schwanninger et al. 2004; Esteves and Pereira 2008c; Ahajji et al. 2009; Bächle et al. 2010).

Patzelt et al. (2003) suggested that color could be used as a classification method of treated wood, because it has a significant correlation with mass loss. Brischke et al. (2007) found good correlation between heat treatment intensity and the color of milled wood, with less variation compared to wood surface measurements. Schnabel et al. (2007) used cluster analysis to prove that color can be used to classify bending strengths of heat-treated beechwood and ash wood. Studies of correlation between color and strength of heat-treated wood have shown contradictory results. Bekhta and Niemz (2003) reported a strong correlation ($R^2 = 0.99$) between changes of color and bending strengths of spruce wood, whereas Johanson and Morén (2006) have used partial least squares (PLS) analysis and concluded that there is no strong correlation between changes of color and bending strengths at heat-treated birch wood. These authors have used – as independent variables – color, density, moisture content (MC), modulus of elasticity in bending strength (MOE), thickness, and width of the sample, treatment time, treatment temperature, and position in a board. González-Peña and Hale (2009b) used color to predict the physical and mechanical properties of wood of Scots pine, Norway spruce, and beech, by using both PLS and linear regression. Their results indicate that color could be used to estimate most of wood properties.

Previous studies indicate that color assessment can be used as an inexpensive and non-destructive method to determine the quality of heat-treated wood. Published results need to be verified in the future research of wood species that have inhomogeneous color and whose value can be significantly increased by heat treatment. Beech is one of the most important wood species in Europe. However, a common problem is red heartwood, out of which products of lower value are made, due to its naturally darker color and a potential presence of fungi. The process of heat treatment modifies the color, and there is a need to establish whether the wood of red heartwood differs from sapwood by its color and properties. Therefore, the aim of this research was to determine changes in color and in properties (density, density loss, mass loss, and bending properties) of heat-treated beech sapwood and red heartwood, as well as to explore the possibilities of predicting the properties by the color itself.

EXPERIMENTAL

Materials

Eleven beech trees that were randomly chosen were obtained from the forest area of Goč Mountain (southwestern Serbia). The average breast height diameter was 45 cm. All the trees had a similar amount of red heartwood (around 50%) with the absence of visible decay and were cut into 2 m long logs. Three logs were cut from each tree (33 logs in total): above breast height, at the middle, and at the height of first green branches. Each log was cut into eight radial boards 30 mm thick (four from sapwood and four from

red heartwood – Fig. 1). From total of 264 kiln-dried boards, 72 sapwood and 72 red heartwood boards (with no visible defects and deformations) were selected. Four samples from the central part of selected boards were cut (1 untreated plus 3 for heat treatment). The samples (marked 1, 2, 3, and 4) had clearly defined anatomic directions and had no visible defects. They were cut into specimens that were used to determine the physical and mechanical properties (Fig. 1). Specimens that were used for determining the moisture content (MC), the density (oven dried-ODD, air dried-ADD), the mass loss (MI), and the density loss (DI) had dimensions: 20x20x20 mm, and specimens for determining the bending properties – modulus of rupture (MOR) and modulus of elasticity (MOE) were 20x20x320 mm (radial, tangential and longitudinal). There were a total of 576 specimens: 288 out of sapwood (72 + 3x72) and 288 out of red heartwood (72 + 3x72).

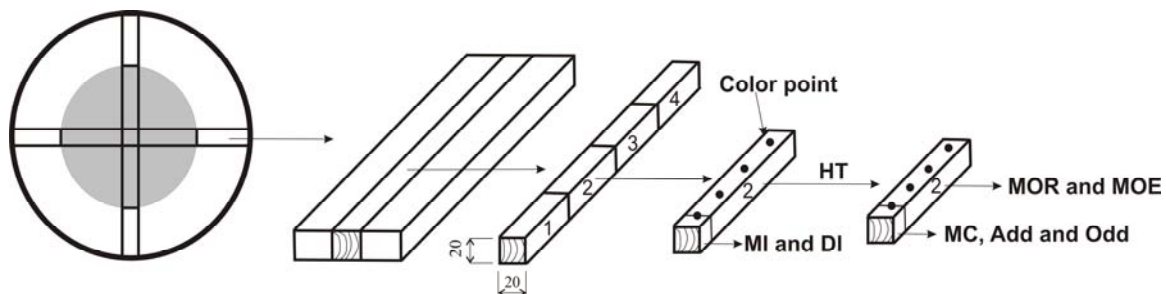


Fig. 1. Sawing pattern and test samples. (1-untreated, 2-170°C, 3-190°C, 4 -210°C, HT – heat treatment, MOR – modulus of rupture, MOE – modulus of elasticity, MC –moisture content, ODD – oven dry density, ADD – air dry density, MI – mass loss, DI – density loss).

Heat Treatment

Heat treatment was carried out in a laboratory chamber (1 m³, ± 1°C sensitivity) for heat treatment filled with water vapor, where samples were exposed to temperatures of 170°C, 190°C, or 210°C. It took approximately twenty-four hours to heat the samples from room temperature to the treating temperature, after which the temperature was kept constant for four hours. The chosen schedules are often used in industrial heat-treating of beech timber.

Determination of the Physical and Mechanical Properties

Before heat treatments, all specimens (including specimens for MI and DI determination that were previously oven-dried) were conditioned at 23°C and relative humidity of 50% during eight weeks. The same procedure of conditioning was carried out after the heat treatment. Then, the mechanical properties (bending properties) and physical properties (ADD, ODD, and MC) were determined. MOR and MOE were determined by a three-point bending test on specimens measuring 20x20x320 mm. Distance from supports was 280 mm. ADD, ODD, and MC (20x20x20) were determined gravimetrically subsequent to the bending tests.

Masses and volumes of oven-dried specimens (20x20x20 mm) that were to be heat-treated were measured. Then they were conditioned with other specimens and, after

heat treatment, they were oven-dried and had their masses and volumes measured again. According to these data, MI and DI of heat-treated wood were calculated.

Color Assessment

Color assessments were performed on radial surfaces of untreated and heat-treated samples by BYK (BYK Gardner GmbH) colorimeter. Color was assessed in four locations, and average values were used for further calculations. The sensor head was 11 mm in diameter. Measurements were made using a D65 illuminant and a 10° standard observer. The coordinates L^* (black-white), a^* (green-red), and b^* (blue-yellow), measured before and after the treatment, were used to determine: ΔL , Δa , Δb (exemplified for $\Delta L = L^*_{\text{treated}} - L^*_{\text{untreated}}$), ΔE – total color difference, h_{ht} – hue after the heat treatment, C_1 – saturation before the heat treatment, C_2 – saturation after the heat treatment, ΔC – change in saturation, and H_{ab} – change in hue.

One-way ANOVA and Tukey's multiple range test (SPSS 13.0 software) were used for comparing and determining any significant differences in color and wood properties between the groups.

Prediction of Properties According to Color

In order to determine correlation between color and physical and mechanical properties of heat-treated beechwood, simple linear, multiple linear, and PLS regressions were used. Average values of measured properties were taken as dependent variables in all three treatments. As predictors in simple linear regression, ΔE and ΔL were used and 12 color variables (L^* , a^* , b^* , ΔL , Δa , Δb , ΔE , h , C_1 , C_2 , ΔC , H_{ab}) were used in multiple linear regressions and in PLS. Simple and multiple linear regressions were calculated by SPSS 13.0 software. PLS analysis was done by the Unscrambler (CAMO AS, Norway) software version 9.7. PLS calibration was done with two subsets (calibration set and validation set) with a maximum 10 latent variables (LV). Samples for calibration and validation sets were taken manually. First, the samples were ranked in ascending order of their dependent variables and every third or fourth sample was taken into the validation set. This produced a calibration set (150 samples) and a validation set (66 samples) with roughly the same standard deviation. Secondly, 12 color variables of 150 samples were regressed against the tested properties by cross-validation of five randomly chosen groups. The number of factors used was determined by The Unscrambler software. Model efficiency was then tested by the validation set. The following calibration and validation parameters were used: determination coefficient of calibration set (R_c^2), standard error of calibration (SEC), standard error of cross-validation (SECV), determination coefficient of validation set (R_p^2) (value calculated to show the ability of the calibration to account for the variation in the validation set), standard error of prediction (SEP) (the measure of the calibrations ability to predict wood properties in samples not used in the calibration set), and ratio of performance to deviation (RPD), which evaluates the predictive ability of the calibration and is the ratio of the standard deviation of the measured data to the SEP (Williams and Sobering 1993). According to Workman (2008), values of SEC, SECV, and SEP should be as low as possible, while R^2 and RPD should be as high as possible. For use in the forestry science, Schimleck et al.

(2001) claim that a RPD greater than 1.5 is considered satisfactory for screenings and preliminary predictions, while Schimleck and Evans (2004) consider a RPD of 2.5 sufficient for tree-selection in breeding programs.

RESULTS AND DISCUSSION

Color Changes

Mean values of color parameters for untreated and treated beechwood are shown in Table 1, and average values of color differences in all three treatments are shown in Fig. 2. Measured coordinates had positive values both before and after treatment. Colors of untreated sapwood and red heartwood differ significantly. The L^* values are higher in sapwood than in red heartwood, whereas a^* and b^* coordinates are lower (Table 1). The applied heat treatments have had the greatest influence on the change of L^* coordinate, as it made the wood darker, which was also shown in earlier studies of heat-treated wood (Militz 2002; Bekhta and Neimz 2003; Mitsui et al. 2001, 2003; Johanson and Morén 2006; González-Pena and Hale 2009 a,b). The L^* coordinate decreased significantly (Fig. 2), and its reduction was greater in sapwood (-36.8) than in red heartwood (-26.1). In sapwood, coordinates a^* and b^* increased at 170°C, but decreased at 190°C and 210°C. However, these changes were smaller than the changes of L^* coordinate. Red heartwood showed a decrease of a^* and b^* with the increase of temperature, and the average values of all heat treatments were negative ($\Delta a = -2.0$, $\Delta b = -5.8$). Sapwood showed average Δa of 0.8, and Δb of (-4.2).

Table 1. Mean Values for Three Color Coordinates and Color Difference in Heat-Treated Beech

Part of beechwood	Treatment	L^*	a^*	b^*	ΔE
Sapwood	Untreated (N=72)	79.1 (*) (2.4)	6.3 (*) (1.0)	18.9 (*) (1.1)	-
	170°C (N=72)	55.9 (4.9)	8.4 (*) (0.5)	19.8 (1.1)	23.3 (*) (5.8)
	190°C (N=72)	39.8 (3.3)	7.7 (*) (0.9)	15.2 (2.0)	39.5 (*) (4.0)
	210°C (N=72)	30.9 (1.2)	5.1 (0.5)	8.9 (0.9)	49.2 (*) (3.4)
Red heartwood	Before (N=72)	68.3 (3.4)	9.5 (1.1)	20.5 (1.1)	-
	170°C (N=72)	55.4 (3.9)	9.2 (0.8)	20.0 (1.4)	12.9 (4.3)
	190°C (N=72)	40.4 (4.5)	8.3 (1.0)	15.2 (2.3)	28.4 (5.7)
	210°C (N=72)	30.8 (1.7)	5.1 (0.6)	8.8 (1.3)	39.5 (3.4)
N – number of samples. The standard deviations are in parentheses.					
(*) statistically significant difference between sapwood and red heartwood ($p < 0.05$).					

A significant difference in values of L^* , a^* , and b^* between sapwood and red heartwood (Table 1) was noticed in L^* (untreated), a^* (untreated, 170°C and 190°C), and b^* (untreated). There was no difference between the values of a^* and b^* in red heartwood treated at 170°C in comparison with untreated wood. In all other cases, both sapwood and red heartwood showed significant differences in L^* , a^* , and b^* as compared to untreated wood.

As was expected, a higher mean value of total color difference (Fig. 2) was obtained for sapwood ($\Delta E=37.3$) than for red heartwood ($\Delta E=27.0$). According to a table frequently used for color classification (e.g. Allegretti et al. 2008), the color difference between sapwood and red heartwood before treatment was high ($\Delta E= 11.4 (>6)$), whereas after heat treatment it was small at 170°C ($\Delta E=0.96<2$) and 190°C ($\Delta E=0.85<2$), and not visible at 210°C ($\Delta E=0.14<0.2$).

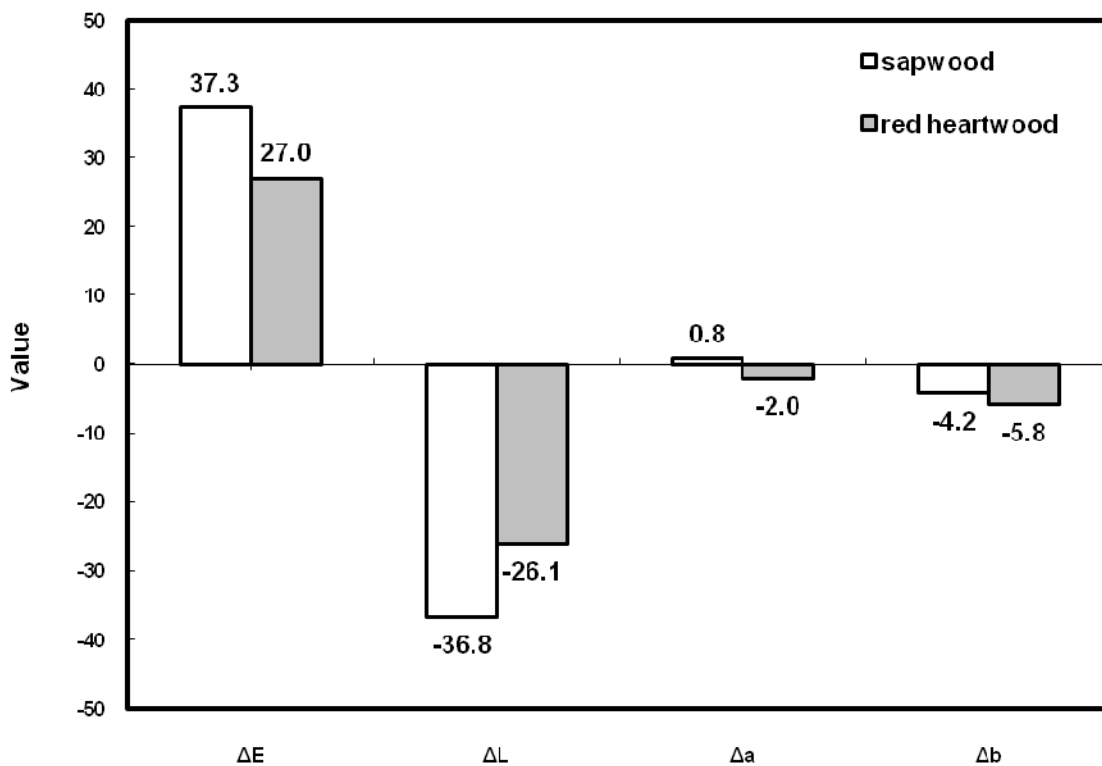


Fig. 2. Color difference between untreated and heat treated wood (average of all heat treatments)

Properties of Heat-Treated Beechwood

Hygroscopicity of wood decreased significantly as a consequence of heat treatment. Untreated wood (after 8 weeks of conditioning) had an average MC of 9.0% in sapwood and 9.6% in red heartwood. Mean values of MC for treated samples were 4.7% (3.0 to 6.6%) in sapwood and 4.2% (2.3 to 6.2%) in red heartwood. Values of other physical and mechanical properties are shown in Table 2. Properties of untreated and heat-treated sapwood and red heartwood mostly did not differ much and were within the

values from previous research studies. ODD of untreated beechwood was 0.676 g/cm³ (ADD – 0.703 g/cm³) in sapwood and 0.677 g/cm³ (ADD – 0.700 g/cm³) in red heartwood. Densities of untreated sapwood and red heartwood were no different and the values were similar to those in previous research studies (Pöhler et al. 2006; Popadić and Todorović 2008; Popović et al. 2010). As expected, heat treatment was associated with decrease in density (Yildiz 2002; Boonstra et al. 2007; Korkut and Guller 2008). There were no difference in wood density at 170°C compared to untreated samples, but strong reduction was found at 190°C and 210°C.

Table 2. Physical and Mechanical Properties of Untreated and Treated Samples

Part of beechwood	Treatment	ODD (g/cm ³)	ADD (g/cm ³)	MI (%)	DI (%)	MOR (N/mm ²)	MOE (N/mm ²)
Sapwood	Untreated (N=72)	0.676 (0.048)	0.703 (0.048)			126.9 (16.0)	11739 (1629)
	170°C (N=72)	0.667 (0.031)	0.695 (0.033)	4.7 (0.9)	0.8 (0.2)	132.1 (15.9)	12500* (1864)
	190°C (N=72)	0.644* (0.030)	0.659* (0.030)	9.2 (1.1)	5.2 (*) (1.4)	95.7* (*) (16.8)	12540* (1910)
	210°C (N=72)	0.619* (0.027)	0.633* (0.029)	16.9 (1.1)	9.7 (2.5)	69.6* (14.1)	11721 (1705)
Red heartwood	Untreated (N=72)	0.677 (0.051)	0.700 (0.049)			124.1 (17.9)	12095 (1513)
	170°C (N=72)	0.674 (0.038)	0.696 (0.040)	5.0 (0.7)	0.7 (0.2)	127.9 (21.6)	12663* (1840)
	190°C (N=72)	0.650* (0.033)	0.667* (0.035)	9.3 (0.8)	4.0 (1.3)	88.7* (24.7)	12624* (2076)
	210°C (N=72)	0.621* (0.033)	0.635* (0.030)	17.0 (1.1)	10.0 (2.9)	65.7* (17.4)	11638 (1982)
N – number of samples. The standard deviations are in parentheses.							
* statistically significant difference as compared to untreated wood (p<0.05).							
(*) statistically significant difference between sapwood and red heartwood (p<0.05).							

This research confirmed that mass loss increases with the rise of temperature (Bourgois and Guyonnet 1988; Zaman et al. 2000; Alén et al. 2002; Esteves et al. 2008a) both in sapwood and in red heartwood. Average mass loss (for all three treatments) was 10.3% in sapwood and 10.4% in red heartwood. Average density loss was lower than mass loss and it was 5.2% in sapwood and 4.9% in red heartwood. There was no difference between sapwood and red heartwood in density loss, except for treatment at 190°C. Compared to untreated samples, MOR decreased significantly at higher temperatures (190 and 210°C), which is in agreement with results in previous studies (Schnabel et al. 2007; Esteves et al. 2007; Kocaefe et al. 2008; Windeisen et al. 2009;). There was no difference between sapwood and red heartwood in MOR both for untreated and samples treated at 170°C and 210°C. Higher value in sapwood than in heartwood at 190°C was found, mainly as a consequence of similar trend in density loss.

The heat treatment at 170°C and 190°C slightly improved MOE, while at 210°C it was not different compared to untreated wood. No difference between sapwood and red heartwood in MOE was obtained both in untreated and treated samples.

Prediction of Properties by Linear Regression

Table 3. Linear Regression Analysis of Prediction of Physical and Mechanical Properties of Heat-Treated Beechwood using ΔL or ΔE as Predictors

Part of beechwood	Predictor	N	Stand. error	R^2	F_{sig}	b_0	b_1	b_2
ODD (g/cm ³)								
Sapwood	ΔL	216	0.029	0.35	117.0	0.71	0.002	
	ΔE	216	0.029	0.41	150.3	0.72	-0.002	
Red heartwood	ΔL	216	0.034	0.31	97.3	0.71	0.002	
	ΔE	216	0.032	0.26	74.6	0.69	-0.002	
Total	ΔL	432	0.032	0.30	184.1	0.70	0.002	
	ΔE	432	0.031	0.31	192.7	0.70	-0.002	
ADD (g/cm ³)								
Sapwood	ΔL	216	0.029	0.41	150.3	0.72	-0.002	
	ΔE	216	0.030	0.45	174.0	0.75	0.002	
Red heartwood	ΔL	216	0.033	0.47	188.8	0.75	0.003	
	ΔE	216	0.030	0.42	156.6	0.74	-0.002	
Total	ΔL	432	0.033	0.40	291.4	0.74	0.002	
	ΔE	432	0.031	0.43	322.9	0.74	-0.002	
MI (%)								
Sapwood	ΔL	216	2.32	0.86	667.2	11.8	0.72	0.018
	ΔE	216	1.32	0.96	2501	13.4	-0.80	0.019
Red heartwood	ΔL	216	2.16	0.87	731.8	5.86	0.26	0.016
	ΔE	216	1.40	0.95	1896	6.97	-0.30	0.015
Total	ΔL	432	3.68	0.64	381.8	1.42	-0.21	0.004
	ΔE	432	2.81	0.80	862.4	4.81	-0.09	0.009
DI (%)								
Sapwood	ΔL	216	1.82	0.74	298.3	0.48	0.11	0.006
	ΔE	216	1.62	0.77	357.3	-0.14	-0.06	0.005
Red heartwood	ΔL	216	2.28	0.57	142.1	-1.66	-0.17	0.002
	ΔE	216	1.88	0.67	211.1	-1.72	0.16	0.002
Total	ΔL	432	2.36	0.55	262.3	-1.72	0.16	0.001
	ΔE	432	2.04	0.62	347.8	-1.72	0.15	0.001
MOR (N/mm ²)								
Sapwood	ΔL	216	14.8	0.76	667.1	184.7	2.38	
	ΔE	216	15.4	0.76	662.2	186.7	-2.44	
Red heartwood	ΔL	216	21.6	0.58	299.1	160.6	2.53	
	ΔE	216	19.9	0.65	392.8	159.6	-2.46	
Total	ΔL	432	22.7	0.49	417.2	156.8	1.93	
	ΔE	432	21.9	0.54	512.1	159.2	-2.01	
MOE (N/mm ²)								
Sapwood	ΔL	216	1152	0.32	104.4	15140	72.0	
	ΔE	216	1236	0.36	122.6	15398	-82.6	
Red heartwood	ΔL	216	1649	0.27	80.0	15019	98.6	
	ΔE	216	1519	0.31	98.9	14990	-94.8	
Total	ΔL	432	1485	0.22	124.3	14537	65.5	
	ΔE	432	1431	0.29	174.4	14785	-75.1	
Models of the form: $y=b_0+b_1x+b_2x^2$. All coefficients are significant at $p<0.001$.								

Table 3 shows the results of linear regression assessment of basic physical and mechanical properties of beech wood, based on ΔL and ΔE as predictors. Most of properties had a linear correlation with ΔL and ΔE .

According to coefficient of determination (R^2), ΔL and ΔE had the best correlation with mass loss (Fig. 3), density loss, and MOR. R^2 for mass loss were roughly the same for both parts of wood and were 0.86 (ΔL) and 0.96 (ΔE) in sapwood, and 0.87 (ΔL) and 0.95 (ΔE) in red heartwood. Density loss and MOR had roughly the same prediction efficiency by ΔL and ΔE , but lower than mass loss. R^2 in density loss varied between 0.57 (ΔL in red heartwood) and 0.77 (ΔE in sapwood). Similar results were found in MOR – the lowest R^2 was in red heartwood – 0.58 (ΔL) and the highest was in sapwood – both ΔL and ΔE had R^2 values of 0.76.

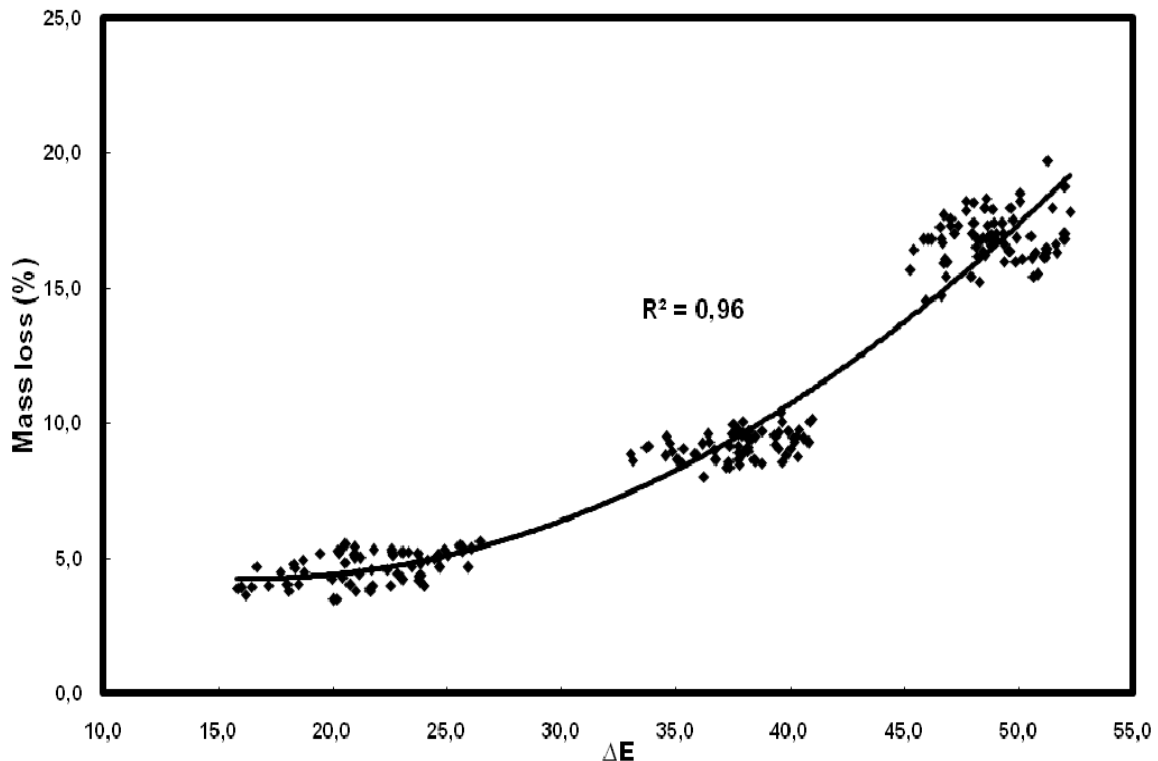


Fig. 3. Correlation between mass loss and ΔE in sapwood of heat-treated beechwood. Grouped data originate from different heat treatments

A parabolic correlation was present between ΔL and/or ΔE and mass loss, which coincides with the results of González-Peña and Hale (2009b), but there was a linear correlation between these predictors and bending properties (Fig. 4), which differs from the results reported by same authors. Standard errors of mass loss, MOR, and MOE were significantly higher in red heartwood than in sapwood. Compared to results reported by González-Peña and Hale (2009b), standard errors in assessment of MOR and MOE in sapwood were smaller, and those of density loss were similar. The reason for this is

probably a narrower range of applied temperatures and therefore a narrower range of color change ($\Delta E=15-56$).

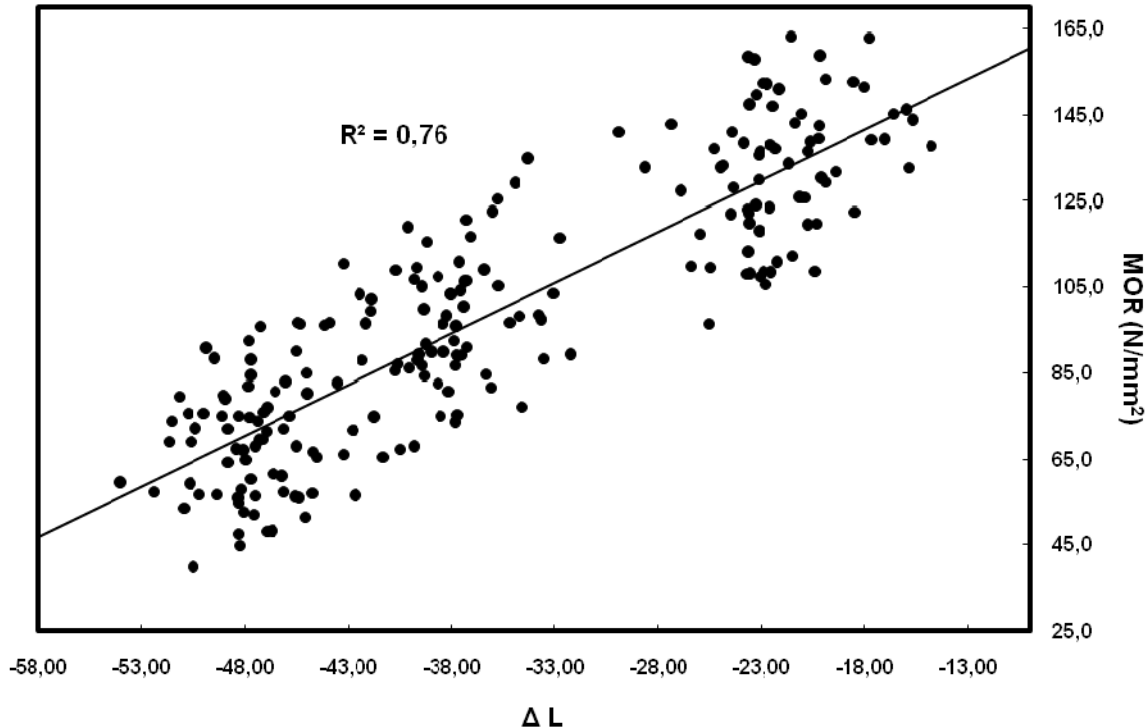


Fig. 4. Correlation between MOR and ΔL in sapwood of heat-treated beechwood

ΔE was a better predictor than ΔL for most of the properties, except for red heartwood density. Sapwood had higher R^2 than heartwood for all investigated properties. This was probably caused by sapwood more homogeneous color that provided a better correlation between color parameters and the tested properties.

Multiple linear regression based on twelve independent color variables showed a higher R^2 than simple linear regression for density loss and MOE in sapwood ($R^2 = 0.89$ and $R^2 = 0.42$). In all other cases, simple linear regression was equally or more efficient in predicting.

Prediction of Properties by PLS Regression

PLS regression improved ability of properties prediction (with density loss this goes up to 70%) compared to simple and multiple linear regressions (Table 4). Red heartwood had higher values of R^2 than linear regression in mass loss ($R^2=0.97$), density loss ($R^2=0.89$), and MOE ($R^2=0.38$). PLS regression in sapwood showed higher values of RPD with all tested properties. Highest values of RPD in sapwood were with mass loss (RPD=6.42), density loss (RPD=3.92), and MOR (RPD=2.69). ODD had the lowest values of RPD. Compared to linear predictors ΔL and ΔE , PLS was better with ODD in red heartwood ($R^2=0.34$) and with ADD in sapwood ($R^2=0.50$).

Table 4. Summary of Statistics for Calibration and Predictions of Physical and Mechanical Properties of Heat-treated Beechwood

Part of beechwood	Calibration					Validation			
	N_c	Factors	SEC	SECV	R_c^2	N_b	SEP	R_p^2	RPD
ODD (g/cm ³)									
Sapwood	150	2	0.037	0.038	0.36	66	0.038	0.38	1.29
Red heartwood	150	4	0.034	0.036	0.31	66	0.036	0.34	1.22
Total	300	1	0.037	0.038	0.34	132	0.030	0.36	1.23
ADD (g/cm ³)									
Sapwood	150	3	0.026	0.027	0.47	66	0.025	0.50	1.44
Red heartwood	150	3	0.030	0.031	0.38	66	0.030	0.38	1.33
Total	300	1	0.033	0.033	0.40	132	0.027	0.43	1.33
MI (%)									
Sapwood	150	2	1.05	1.06	0.97	66	1.05	0.97	6.42
Red heartwood	150	2	1.16	1.19	0.97	66	1.10	0.97	5.74
Total	300	3	1.05	1.07	0.97	132	0.97	0.98	6.14
DI (%)									
Sapwood	150	2	0.95	0.99	0.93	66	0.92	0.94	3.92
Red heartwood	150	2	1.16	1.26	0.87	66	1.08	0.89	2.98
Total	300	2	1.12	1.14	0.90	132	0.93	0.93	3.81
MOR (N/mm ²)									
Sapwood	150	4	11.5	12.05	0.83	66	11.6	0.86	2.69
Red heartwood	150	2	19.7	20.4	0.53	66	19.2	0.60	1.57
Total	300	5	15.3	15.7	0.71	132	15.3	0.74	2.00
MOE (N/mm ²)									
Sapwood	150	3	992	1040	0.57	66	823	0.56	1.52
Red heartwood	150	2	1460	1506	0.38	66	1350	0.38	1.27
Total	300	5	1206	1242	0.46	132	1061	0.45	1.45

RPD values were higher in sapwood than in red heartwood. Considering the limit value of 1.5 (Schimleck et al. 2001), the color of heat-treated beechwood is a good predictor of mass loss, density loss (Fig. 5), and MOR. In sapwood, RPD was on the limit for MOE, but it was a weak predictor for red heartwood. Color was a poor indicator of density in both sapwood and red heartwood.

R^2 had the lowest values in predicting ADD, ODD, and MOE in both linear and PLS regression. The explanation for this is that the color change in heat-treated wood is an indicator of the change in chemical composition and mass loss; therefore color was a better indicator of properties that change more with the change in chemical composition and mass loss. This means that color of heat-treated beechwood can be used more accurately in predicting the strength than density and elasticity in bending. A small change in density was caused by the simultaneous mass loss and volume loss; still volume loss was significantly lower than mass loss (Popadić and Todorović 2008).

The research confirms that the increase in temperature makes sapwood and red heartwood darker while bending strength decreases, which could be directly associated with thermal degradation of wood components (Ponscak et al. 2006; Esteves et al. 2008b). With heat treatment, the color of wood is modified, acquiring a darker tonality,

which is often justified by the formation of colored degradation products from hemicelluloses (Sehistedt-Persson 2003; Sundqvist 2004).

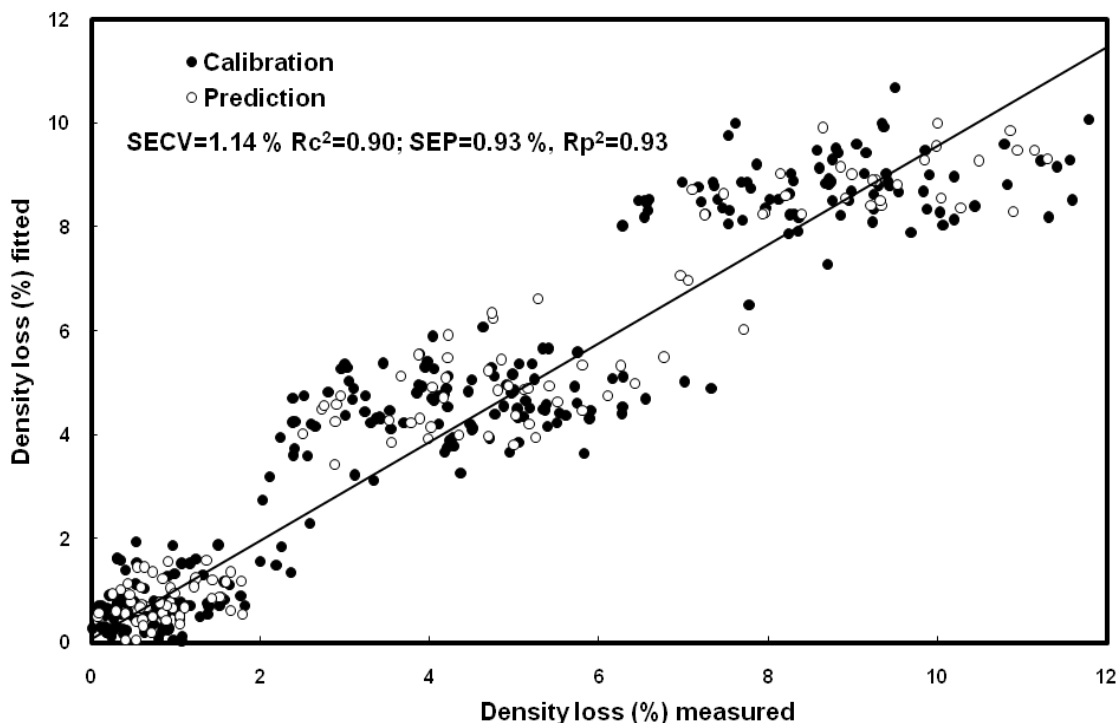


Fig. 5. Calibration plot for DI of heat-treated beechwood using PLS 12 color variables (all 432 samples)

Fengel and Wegener (1989) explained that the reason for the color change is the production of chromophores as a result of the hydrolytic reactions that occur during heat treatment. The decrease of hemicelluloses content has a smaller effect on change in elasticity than on bending strength, for which a strong correlation was reported by several authors (Winandy and Lebow 2001; Esteves et al. 2008a; Windeisen et al. 2009; Niemz et al. 2010). This research didn't examine the content of chemical compounds; however there was a distinctive difference between NIR spectra recorded on beechwood radial surface before and after treatment (Todorović et al. 2010). NIR spectra (resolution 8 cm^{-1} and 100 scans) were recorded on the same samples that were used in this research. The heat treatment was observed in the second derivative mode of the NIR spectra. The hemicelluloses are more sensitive to high temperature than are lignin and cellulose. There is a decrease of the band at 5800 cm^{-1} , the first overtone of CH stretching vibrations, which is caused by the degradation hemicelluloses and to a decrease of the absorption band assigned to the amorphous region cellulose at 7000 cm^{-1} (Windeisen et al. 2009; Bächle et al. 2010; Schwanninger et al. 2003; Mitsui et al. 2008). The increase of the bands 5950 cm^{-1} and 5981 cm^{-1} , the first overtone of aromatic skeletal CH stretching vibrations, may be due to a relative increase of lignin. Also, there is an increase of the

absorption band around 6900 cm^{-1} , which can be assigned to the phenolic hydroxyl groups originating from lignin (Mitsui et al. 2008).

CONCLUSIONS

This research assessed colors and properties of heat-treated beech sapwood and red heartwood and the possibilities of estimation their properties by color were examined. The following conclusions can be drawn:

1. Coordinates of sapwood and red heartwood color have positive values both before and after treatment. Untreated sapwood had L^* values higher than red heartwood, while a^* and b^* coordinates were lower. Changes in color after heat treatment were caused by the decrease in L^* , whereas the influence of changes in a^* and b^* was small. Differences in color (ΔE) between sapwood and red heartwood before treatment were high, but after treatment they were small at 170°C and 190°C , and not visible at 210°C .
2. Most of properties of sapwood and red heartwood showed no significant difference both before and after treatment. Higher temperatures caused a rise in mass loss and density loss, and a reduction of bending strength, whereas the effect on MOE was small.
3. In general, color could be used for predicting the properties of sapwood and red heartwood. In linear regression, the majority of examined properties had a linear correlation with ΔL and ΔE , while ΔE was a better predictor for all properties, except for density of red heartwood. PLS regression based on twelve independent color variables (L^* , a^* , b^* , ΔL , Δa , Δb , ΔE , h , C_1 , C_2 , ΔC , and H_{ab}) considerably enhanced the estimation of most of the examined properties. Both linear and PLS regressions made the best estimations on sapwood mass loss, density loss, and MOR.
4. Heat treated red heartwood and sapwood had similar color and properties, which can increase the usage of beech red heartwood for products made out of heat-treated wood.

ACKNOWLEDGMENTS

Authors would like to thank the company "Tarkett" d.o.o. Bačka Palanka, Serbia for technical support in carrying out this research. This paper was funded from the Ministry of Educational and Science of the Republic of Serbia (TR 37008).

REFERENCES CITED

- Ahajji, A., Diouf, P. N., Aloui, F., Elbakali, I., Perrin, D., Merlin, A., and George, B. (2009). "Influence of heat treatment on antioxidant properties and colour stability of beech and spruce wood and their extractives," *Wood Sci. Technol.* 43, 69-83.

- Alén, R., Kotilainen, R., and Zaman, A. (2002). "Thermochemical behavior of Norway spruce (*Picea abies*) at 180-225°C," *Wood Sci. Technol.* 36, 163-171.
- Allegretti, O., Travan, L., and Cividini, R. (2008). "Drying techniques to obtain white beech," *ProLigno* 4 (1), 11-19.
- Arnold, M. (2010). "Effect of moisture on the bending properties of thermally modified beech and spruce," *J. Mater. Sci.* 45, 669-680
- Aydemir, D., Gunduz, G., Altuntas, E., Ertas, M., Turgut Sahin, H., and Hakki Alma, M. (2011). "Investigating changes in the chemical constituents and dimensional stability of heat-treated hornbeam and uludag fir wood," *BioResources* 6(2), 1308-1321.
- Bächle, H., Zimmer, B., Windeisen, E., and Wegener, G. (2010). "Evaluation of thermally modified beech and spruce wood and their properties by FT-NIR spectroscopy," *Wood Sci. Technol.* 44, 421-433.
- Bekhta, P., and Niemz, P. (2003). "Effect of high temperature on the change of color, dimensional stability and mechanical properties of spruce wood," *Holzforschung.* 57, 539-546.
- Boonstra, M., Van Acker, J., Tjeerdsma, B., and Kegel, E. (2007). "Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents," *Ann. Forest. Sci.* 64, 679-690.
- Borrega, M., and Kärenlampi P. (2008). "Mechanical behavior of heat-treated spruce (*Picea abies*) wood at constant moisture content and ambient humidity," *Holz Roh Werkst.* 66, 63-69.
- Bourgeois, J., and Guyonnet, R. (1988). "Characterization and analysis of torrefied wood," *Wood Sci. Technol.* 22, 143-155.
- Brischke, C., Welbacher, C.R., Brandt, K., and 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.
- Esteves, B., Velez Marques, A., Domingos, I., and Pereira H. (2007). "Influence of steam heating on the properties of pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood," *Wood Sci. Technol.* 41, 193-207.
- Esteves, B.M., Domingos, I. J., and Pereira, H.M. (2008a). "Pine wood modification by heat treatment in air," *BioResources* 3(1), 142-154.
- Esteves, B., Velez Marques, A., Domingos, I., and Pereira, H. (2008b). "Heat induced color changes of pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood," *Wood Sci. Technol.* 42(5), 369-384.
- Esteves, B., and Pereira, H. (2008c). "Quality assessment of heat-treated," *Holz Roh-Werkst.* 66, 323-332.
- Fengel, D., and Wegener, G. (1989). *Wood Chemistry Ultrastructure Reactions*, Walter de Gruyter.
- González-Peña, M., and Hale, M. (2009a). "Colour in thermally modified wood of beech, Norway spruce and Scots pine, Part 1: Colour evolution and colour changes," *Holzforschung* 63, 385-393.

- González-Peña, M., and Hale, M. (2009b). "Colour in thermally modified wood of beech, Norway spruce and Scots pine, Part 2: Property predictions from colour changes," *Holzforschung* 63, 394-401.
- Hinterstoisser, B., Schwanninger, M., Stefke, B., Stingl, R., and Patzelt, M. (2003). "Surface analysis of chemically and thermally modified wood by FT-NIR," *The First European Conference on Wood Modification ECWMI*, Ghent, Belgium, 65-70.
- Johansson, D., and Morén, T. (2006). "The potential of colour measurement for strength prediction of thermally treated wood," *Holz Roh-Werkst.* 64, 104-110.
- Kocaeffe, D., Poncsak, S., and Bulok Y. (2008). "Effect of thermal treatment on the chemical composition and mechanical properties of birch and aspen," *BioResources* (3)2, 517-537.
- Korkut, D., and Guller, B. (2008). "The effects of heat treatment on physical properties and surface roughness of Red-bud maple (*Acer trautvetteri* Medw.) wood," *Bioresour. Technol.* 99, 1861-1868.
- Kubojima, Y., Okano, T., and Ohta, M. (2000). "Bending strength of heat-treated wood," *J. Wood Sci.* 46, 8-15.
- Militz, H. (2002). "Heat treatment of wood. European processes and their background," In: *International Research Group Wood Pre.*, Section 4-Processes N°IRG/WP 02-40242.
- 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, 601-605.
- Mitsui, K., Murata, A., Kohara M., and Tsuchikawa, S. (2003). "Colour modification of wood by light-irradiation and heat treatment," In: *Abstracts of the First European Conference on Wood Modification*, Ghent, Belgium.
- Mitsui, K., Inagaki, T., and Tsuchikawa, S. (2008). "Monitoring of hydroxyl groups in wood during heat treatment using NIR spectroscopy," *Biomacromolecules* 9, 286-288.
- Niemz, P., Hofmann, T., and Rétfalvi, T. (2010). "Investigation of chemical changes in the structure of thermally modified wood," *Maderas y Science Tecnologia.* 12 (2), 69-78.
- Patzelt, M., Emsenhuber, G., and Stingl, R. (2003). "Color measurement as means of quality control of thermally treated wood," In: *Abstract of the First European Conference on Wood Modification*, Ghent, Belgium.
- Pöhler, E., Klingner, R., and Künniger T. (2006). "Beech (*Fagus sylvatica* L.) – Technological properties, adhesion behavior and colour stability with and without coatings of the red heartwood," *Ann. For. Sci.* 63, 129-137.
- Poncsak, S., Kocaeffe, D., Bouzara, M., and Pichette, A. (2006). "Effect of high temperature treatment on the mechanical properties of birch (*Betula Pendula*)," *Wood Sci. Technol.* 40 (8), 647-663.
- Popadić, R., and Todorović, N. (2008). "Uticaj visokotemperaturnog tretmana na neka fizička svojstva bukovog drveta," *Prerada drveta* (23), 5-10.

- Popović, Z., Todorović, N., Popadić, R., and Nešovanović B. (2010). "Uticaj visokotemperaturnog tretmana na neka svojstva bukovog drveta iz predela iz beljike i lažne srčevine," *Prerada drveta* (29), 5-13.
- Schimleck, R., Evans, R., and Ilic, J. (2001). "Estimation of eucalyptus delegatensis wood properties by near infrared spectroscopy," *Canadian Journal of Forest Research* 31(10), 1671-1675.
- Schimleck, R., and Evans, R. (2004). "Estimation of *Pinus radiata* D. Don tracheid morphological characteristics by near infrared spectroscopy," *Holzforschung* 58, 66-73.
- Schnabel, T., Zimmer, B., Petutschnigg, A., and Schönberger, S. (2007). "An approach to classify thermally modified hardwoods by color," *For. Prod. J.* 57(9), 105-110.
- Schwanninger, M., Gierlinger, N., Hanger, J., Hansmann, C., Hinterstoiser, B., and Wimmer, R. (2003). "Characterization of thermally treated beech wood by UV-microspectrophotometry, FT-MIR and FTIR spectroscopy," *In: Proceedings of 12th ISWPC*, Madison, 55-58.
- Schwanninger, M., Hinterstoiser, B., Gierlinger, N., Wimmer, R., and Hanger, J. (2004). "Application of Fourier transform near infrared spectroscopy (FT-NIR) to thermally modified wood," *Holz Roh-Werkst* 62, 483-485.
- Sehistedt-Persson, M. (2003). "Colour responses to heat treatment of extractives and sap from pine and spruce," *8th International IUFRO Wood Drying Conference*, Brasov, Romania, 459-464.
- Shi, J., Kocaefe, D., and Zhang, J. (2007). "Mechanical behavior of Quebecwood species heat-treated using ThermoWood process," *Holz Roh-Werkst* 65, 255-259.
- Sundqvist, B. (2004). "Colour changes and acid formation in wood during heating," *Doctoral thesis*, Lulea University of Technology.
- Sundqvist, B., and Morén, T. (2002). "The influence of wood polymers and extractives on wood colour induced by hydrothermal treatment," *Holz Roh-Werkst.* 60, 375-376.
- Tjeerdsma, B., Bonstra, M., Pizzi, A., Tekely, P., and Militz, H. (1998a). "Characterization of thermally modified wood: Molecular reasons for wood performance improvement," *Holz Roh-Werkst* 56, 149-153.
- Tjeerdsma, B., Boonstra, M., and Militz, H. (1998b). "Thermal modification of non-durable wood species. Part 2. Improved wood properties of thermally treated wood," *In: International Research Group Wood Pre.*, Document no. N°IRG/WP 98- 40124.
- Todorović, N., Schwanninger, M., and Popović, Z. (2010). "Prediction of mechanical properties of thermally modified beech wood by use of near infrared spectroscopy," *The Fifth European Conference on Wood Modification ECWM5*, Riga, Latvia, 187-191.
- Toung, M. V., and Li, J. (2010). "Effect of heat treatment on the change in color and dimensional stability of acacia hybrid wood," *BioResources* 5(2), 1257-1267.
- Williams, P. C., and Sobering, D. C. (1993). "Comparison of commercial near infrared transmittance and reflectance instruments for analysis of whole grains and seeds," *J. Near Infrared Spec.* 1, 25-33.

- Winandy, J., and Lebow, P. (2001). "Modeling strength loss in wood by chemical composition. Part I. An individual component model for southern pine," *Wood Fiber Sci.* 33(2), 239-254.
- Windeisen, E., Bächle, H., Zimmer, B., and Wegener, G. (2009). "Relations between chemical changes and mechanical properties of thermally treated wood," *Holzforschung* 63, 773-778.
- Workman, J. J. (2008). "NIR spectroscopy calibration basics," *In: Handbook of Near-Infrared Analysis*, 3rd Edn., CRC Press, Boca Raton, 123-150.
- Yildiz, S. (2002). "Physical, mechanical, technological and chemical properties of beech and spruce wood treated by heating," PhD dissertation *Karadeniz Tech. Univ., Trabzon, Turkey*.
- Zaman, A., Alen, R., and Kotilainen, R. (2000). "Heat behavior of *Pinus sylvestris* and *Betula pendula* at 200-230 °C," *Wood Fiber Sci.* 32(2), 138-143.

Article submitted: November 15, 2011; Peer review completed: December 17, 2011;
Revised version received and accepted: January 3, 2012; Published: January 6, 2012.