EFFECT OF TEMPERATURE ON THE COLOR CHANGES OF WOOD DURING THERMAL MODIFICATION

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This paper deals with the determination of color change for oak, birch and pine wood. Color changes were measured on native and thermally modified wood. The thermal modification was carried out at four different temperatures: 160 °C, 180 °C, 210 °C and 240 °C. Color changes were evaluated according to CIE *L*a*b** method by parameters *L**, *a**, *b** and *ΔE**. After the heat treatment, the lightness *L** of wood was reduced for all wood species. The *a** values had a similar character for oak and birch wood, while the values of pine showed an opposite character. Increasing the temperature resulted in a decrease in the values of the parameter *b** for oak and birch wood. However, pine wood achieved the highest *b** values during thermal modification at a temperature of 160 °C. Further increases in temperature led to a gradual lowering of these values. The overall color change *ΔE** gradually grew with the increase in temperature for oak and birch, while pine had the highest value at a temperature of 160 °C.

Keywords: color, overall color change, thermally modified wood, temperature, oak, birch, pine wood

INTRODUCTION

Color is one of the quality attributes that influence the customers' perceptions of wood products. In wood processing processes, wood is mechanically converted by sawing, planning and milling, as well as by drying or thermal modification.

Thermal modification is a process whereby wood is modified by high temperature treatment. Depending on the type of thermal modification, nowadays in Europe a variety of temperature ranges are used. Most often, these temperatures are in the range of 180-260 ºC.[1-2] The most widely used types of thermal modification are the ThermoWood® process (Finland), Plato-Process (Holland), OHT process (Germany), and Rectification process and Perdure Bois (France).[3]

Thermal modification reduces the mechanical properties of wood, but increases the dimensional stability and resistance to decay.[3-7] Thermal modification has important effects on the color and the chemical composition of wood. In general, in terms of color, the thermal modification affects most basic components of wood – lignin, cellulose and hemicellulose. Significant and also profound changes in the structure of wood appear at 180-250. °C. Carbonization of wood begins above 250 °C, its products are carbon monoxide and other combustible substances.[8]

Color is a very important wood property and according to Abrahão[9] color homogeneity is important because it determines the appearance of the final product. Most researches evaluate the color of the wood using the CIE *L*a*b** method with a three axis system (Fig. 1).[10-13] This system is used to accurately determine the color, color changes being usually measured also by colorimeters and spectrophotometers. The principle of the CIE Lab system is based on the rules and conditions of CIE (Commission Internationale de L'Eclairage) and can measure lightness (*L**), chromaticity coordinates (*a** and *b**), chroma (*C**) and hue angle (*h**).

Bekhta and Niemz[12] reported that when the wood is thermally modified at 200 °C, the color change is most intense and a stronger color is

reached within the first few hours of modification. On the other hand, Patzelt *et al.*[14] observed that the color change of thermally treated wood is similar whether the high temperature and short time treatment or the lower temperature and longer time treatment is applied. The darker color of thermally modified wood is often attributed to the formation of degradation products from hemicelluloses.[15-16]

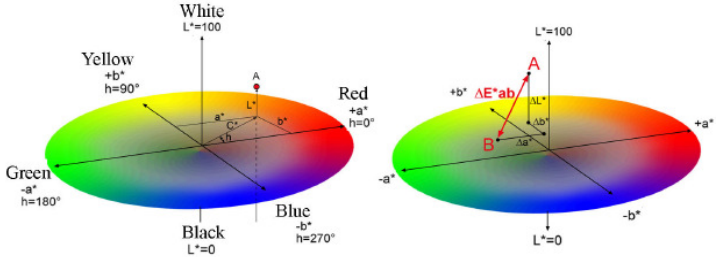

Figure 1: Graphical representation of the CIE L*a*b* color space

Table 1
Conditions for thermal modification

Input technical parameters		
Wood	Moisture content of wood	10.5 to 12%
Thermal chamber	Chamber power	5.3 kW
	Heating	up to 260 °C
	Cooling	down to 20 °C
	Maximum reached temperatures	160 °C, 180 °C, 210 °C and 240 °C

Taking these facts into account, the aim of this research was to evaluate the effect of thermal modification on the color change of wood from selected species. Color measurement was evaluated according to the CIE *L*a*b** method by parameters *L**, *a**, *b** and *ΔE**.

EXPERIMENTAL

Materials

The experimental Pedunculate oak trees (*Quercus robur* L.) (75 years old), Scotch pine trees (*Pinus sylvestris* L.) (65 years old) and Silver birch trees (*Betula verrucosa* Ehr.) (60 years old) grew in the central region of Mari El Republic. The zones suitable for sampling were cut from the trunk at a height of 2 m from the stump. The areas situated at the middle distance between the pith and bark were chosen for sample preparation. These parts were cut into 200 cm long sections, which contained 3 mm wide (oak), 2 mm wide (pine) and 2.5 mm wide (birch) annual rings. Samples with gross dimensions of 40 × 100 × 500 mm were used. All samples were air-conditioned in the conditioning room (ϕ = 65 ± 3% and t = 20 ± 2 ºC) for more than six months to achieve an equilibrium moisture content (EMC) of 12%. The actual EMC of each sample was measured by a weighing method after conditioning.

A number of the air-conditioned samples from all wood species were left as native samples and the rest were used for thermal modification. Each tree species was represented by 10 samples, *i.e.* 2 samples per treatment type. The whole investigation contained 30 samples. Native (unmodified) wood samples served as reference.

A special group of samples was prepared for determining and verifying the physical properties (moisture content, density) of native and thermally modified wood. The dimensions of these samples were in compliance with relevant standards and these samples were only used for determination of these properties.

Procedure

The whole process of sample preparation and thermal modification was carried out in collaboration with Volga State University of Technology in Yoskhar-Ola, Mari El Republic, which provided apparatus and testing facilities.

The thermal treatment was carried out according to the ThermoWood® process developed by VTT, Finland. The modification process was realized in a thermal chamber XVC 304 with cooling system (produced by UNOX Company) adapted for laboratory use. Thermal modification parameters are listed in Table 1.

The wood samples intended for thermal modification were placed on a metal grate in the thermal chamber and modified in three phases. The first phase consisted of drying the wood and heating the chamber to the required temperature, from 160 to 240 °C using steam as protective vapor. In the second stage, the desired temperature was kept for the specified time (5 h) (Table 2). In the third phase, the

chamber and wood were gradually cooled. During this phase, the wood was re-moisturized in order to achieve the end-use moisture (5-7%).

All samples were then machined to final thickness (25 mm) using a thickness planer. Subsequently both faces of each sample intended for color measurement were gently smoothed with a belt sander (grain size 200). Thermally modified samples were conditioned (ϕ = 65 ± 3% and t = 20 ± 2 ºC) for three weeks. Thus, native and thermally modified samples (dimensions 25 × 100 × 500 mm) (Fig. 2) were prepared for color measuring.

Table 2
Phases of thermal treatment

Final thermal temperature (°C)	Thermal treatment			
	I phase (h)	II phase (h)	III phase (h)	Total time (h)
160	4	5	2	11
180	5	5	2.5	12.5
210	6	5	3	14
240	7	5	3.5	15.5

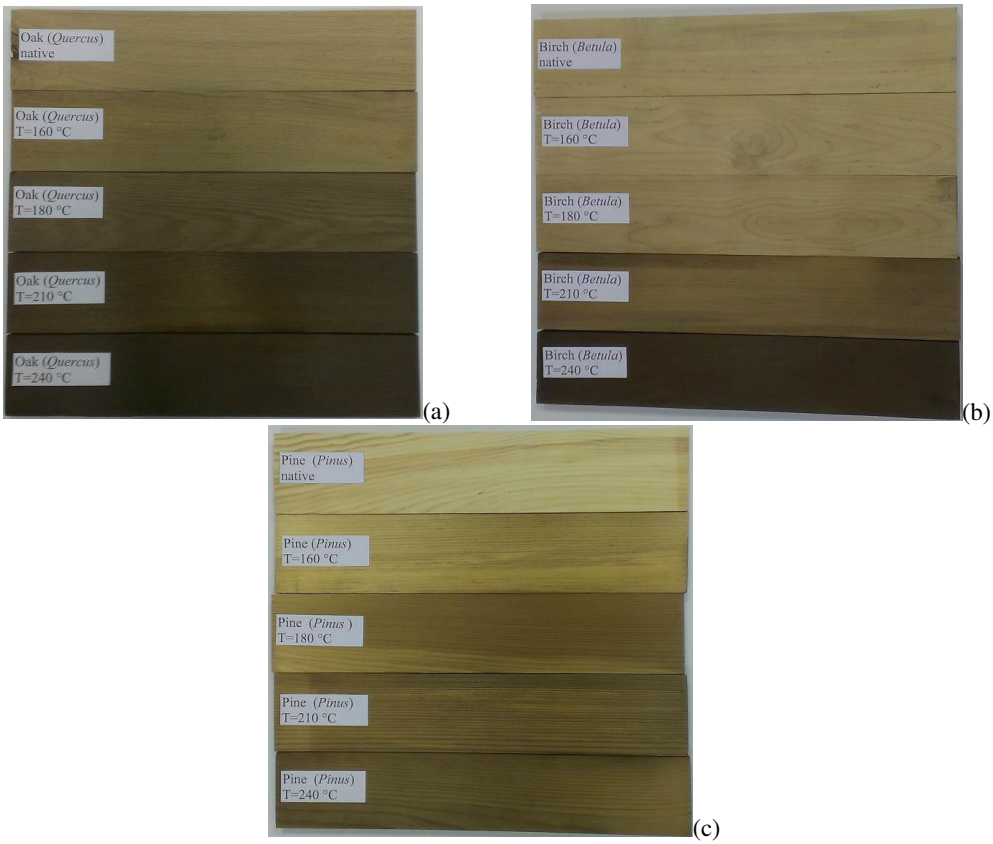

Figure 2: Native and thermally modified samples – oak (a), birch (b) and pine (c)

Color measurements

Color measurements were performed on native and thermally modified samples with a portable spectrophotometer Konica Minolta CM-600d (10° standard observer, D65 standard illuminate, color difference format ΔE^*ab). Measurements were taken on 10 locations on each sample (5 per face), and the arithmetic mean of these measurements was calculated for each wood species. The coordinates L^* (lightness or black-white relation), a^* (coordinate red-green), b^* (coordinate yellow-blue), measured on native and thermally modified wood, were used to determine: ΔL^*, Δa^*, Δb^* (exemplified for $\Delta L^* = L^*_{modified} - L^*_{native}$), overall color change ΔE^* by using the CIE

L*a*b* color measuring system according to ISO 11664-2[17] and ISO 11664-4.[18] The overall color change *ΔE** was determined by the rules of color change distribution, according to Cividini *et al.*[19] (Table 3).

Table 3
Evaluation criteria of overall color change *ΔE**

$0.2 < \Delta E^*$	Invisible difference
$0.2 < \Delta E^* < 2$	Small difference
$2 < \Delta E^* < 3$	Color change visible with high-quality filter
$3 < \Delta E^* < 6$	Color change visible with medium-quality filter
$6 < \Delta E^* < 12$	High color changes
$\Delta E^* > 12$	Different color

Evaluation and calculation

The color differences were evaluated by the overall color change, which was calculated according to Eq. 1 from ISO 11664-4[17] and ISO 11664-6:[20]

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

where *ΔE** is the overall color change, *ΔL**, *Δa**, and *Δb** are the differences between the initial and the final values (before and after thermal modification, *e.g.* between native and thermally modified wood) of *L**, *a**, and *b**, respectively.

The density was determined as an auxiliary indicator. Density was calculated according to Eq. 2 from ISO 13061-2:[21]

$$\rho_w = \frac{m_w}{a_w * b_w * l_w} = \frac{m_w}{V_w} \quad (2)$$

where ρ_w is the density of the sample at certain moisture content w [kg/m^3], m_w is the weight of the sample at certain moisture w [kg], a_w, b_w, and l_w are dimensions of the sample at certain moisture w [m], and V_w is the volume of the sample at a certain moisture w [m^3].

The density of the wood after treatment was calculated according to Eq. 3 from ISO 13061-2:[21]

$$\rho_{tw} = \frac{m_{tw}}{a_{tw} * b_{tw} * l_{tw}} = \frac{m_{tw}}{V_{tw}} \quad (3)$$

where ρ_{pl} is the density of the sample after the treatment [kg/m^3], m_{pl} is the weight of the sample after treatment [kg], a_{pl}, b_{pl}, and l_{pl} are dimensions of the sample after treatment [m], and V_{pl} is the volume of the sample after treatment [m^3].

The moisture content of the samples was determined before and after the thermal treatment. These calculations were carried out according to ISO 13061-1[22] and Eq. 4:

$$w = \frac{m_w - m_0}{m_0} * 100 \quad (4)$$

where w is the moisture content of the samples [%], m_w is the weight of the sample at a certain moisture w [kg], and m_0 is the weight of the oven-dry sample [kg].

Drying to an oven-dry state was also carried out according to ISO 13061-1,[22] using the following procedure. The samples were placed in the drying oven at a temperature of 103 ± 2 °C until a constant mass was reached. It was considered that a constant mass was reached if the loss between two successive weighing measurements, carried out at an interval of 6 h, was equal to or less than 0.5% of the mass of sample. After cooling the samples to approximately room temperature in a desiccator, they were weighed rapidly enough to avoid an increase in moisture content of more than 0.1%. The accuracy at weighing should be at least 0.5% of the mass of the sample.

RESULTS AND DISCUSSION

Physical properties

Table 4 contains the average density of native and thermally modified samples for all three wood species.

Some heat treatments, such as steaming, can slightly increase the wood density, while thermal modification conversely reduces the density of wood. Our density values correspond to the values mentioned by Knapic *et al.* for oak,[23] Ahmed *et al.*[24] for birch and Kamperidou *et al.*[25] for pine. In general, wood density gradually decreased with increasing temperature during thermal modification. The lowest density was found for thermally modified wood at 240 °C. Oak wood presented the smallest decline in density, namely 9.1%, birch wood had a bigger decline – of 14.3%, and the highest decline, of 16.9%, was found for pine wood. For comparison, Shchupakivskyy *et al.*[26] found a 12% decrease in density of oak after heat treatment at 220 °C, while Boonstra *et al.*[27] reported a 10% decrease in density for heat treated Scots pine.

Density reduction was caused by the loss of moisture and hence the reduction of volume, as well as by the degradation of wood structure. Boonstra *et al.*[27] is convinced that the degradation of hemicelluloses into volatile products and the

evaporation of the extracts are the main reasons for density reduction.

Color changes

As stated previously, the color change was evaluated by the parameters *L**, *a**, *b** and *ΔE**. The values of these parameters for all the wood species are listed in Table 5.

As expected, the lightness *L** was changed the most. The lightness *L** gradually decreased with an increase in temperature. The thermally modified samples of all three wood species achieved significant decreases in lightness in comparison with the unmodified wood. For all wood species, these values decreased by more than half at 240 °C. The lightness of pine wood was the highest before and after the modification, while the oak had the lowest lightness. These results were confirmed by Čermák and Dejmal,[13] who found a reduction in the lightness of oak at the temperature of 230 °C by an average of 36 units, whereas the lightness of the native wood was in the range of 69-75 and after modification in the range of 29-35. Esteves *et al.*[28] presented a lightness value of 67.3 for native pine wood, and reported a 60.4% decrease in lightness for wood heated at 200 °C, while in our research, the lightness of native pine wood was 81.7 and the maximum decrease after modification was of 42.7%.

Table 4
Average density of native and thermally modified wood

Wood species			Density (kg/m^3)		
			Minimum value	Maximum value	Average value
Oak	Native		695.1	802.5	739.1
	TMW	160 °C	693.8	796.6	722.4
		180 °C	675.3	774.7	718.8
		210 °C	675.2	704.8	687.8
		240 °C	649.9	696.4	671.5
Birch	Native		585.3	658.3	613.7
	TMW	160 °C	557.8	625.3	596.6
		180 °C	529.1	590.0	568.4
		210 °C	488.5	565.8	541.9
		240 °C	475.4	537.7	525.8
Pine	Native		461.2	574.6	502.1
	TMW	160 °C	453.3	523.3	484.1
		180 °C	429.5	481.0	456.1
		210 °C	392.4	433.5	421.7
		240 °C	384.0	421.5	416.9

Table 5
Average values of three color coordinates and overall color change for native and thermally modified wood

Wood species	Treatment	Color coordinates			Overall color change
		*L**	*a**	*b**	*ΔE**
Oak	Native wood (untreated)	64.46	6.77	20.17	-
	160 °C	57.91	6.77	18.84	1.01
	180 °C	46.22	6.35	14.47	9.96
	210 °C	42.90	7.73	15.42	11.21
	240 °C	30.82	4.78	7.40	26.05
Birch	Native wood (untreated)	73.06	7.05	19.72	-
	160 °C	72.10	7.24	20.37	1.16
	180 °C	64.64	7.58	20.83	4.52
	210 °C	52.38	9.09	19.58	2.56
	240 °C	32.25	6.70	10.87	17.77

	Native wood (untreated)	81.72	4.98	22.81	-
	160 °C	64.98	9.34	27.14	16.45
Pine	180 °C	48.57	10.51	22.92	8.77
	210 °C	47.83	9.83	20.81	6.81
	240 °C	46.82	8.23	21.24	5.96

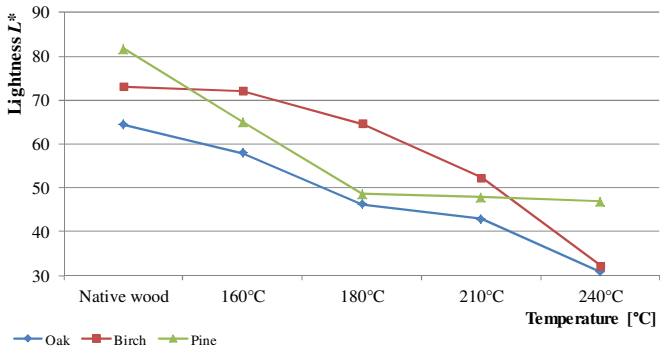

Figure 3: Lightness *L** of native and thermally modified wood for all wood species

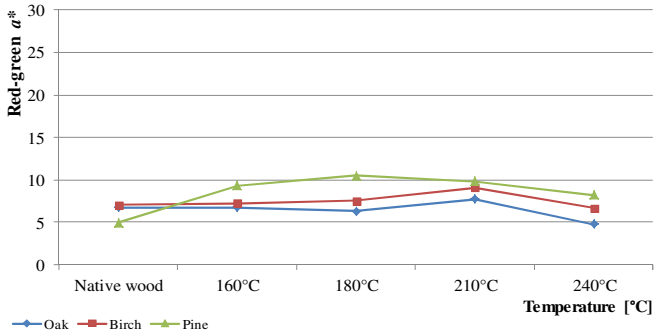

Figure 4: Color coordinate *a** of native and thermally modified wood for all wood species

Kamperidou *et al.*,[25] who heat-treated pine wood for 4, 6 and 8 hours, also found a decrease in lightness by half after an 8 hour heat treatment. The decrease in the lightness of birch wood (approx. 56%), which has been found in our research (Fig. 3), is not unusual. Other authors found similar values. For example, Johansson and Morén,[29] who thermally treated birch wood at the temperature of 175 °C and 200 °C for 0, 1, 3, and 10 hours, found a 50% decrease in lightness after a 10 hour heat treatment at 200 °C.

The a* values of modified oak and birch wood had a very similar character. First, they gradually increased until 210 °C. Then, these values dropped below the values for native wood. The differences, between the *a** values for oak and birch wood were not great. The *a** values of pine showed a different character (Fig. 4). The values gradually increased up to the temperature of 180 ° C, then they began to decrease. The values at 240 °C were higher than those of native wood. Interestingly, however, the *a** values of pine at 180 °C were more than twice higher than those for native wood. Čermák and Dejmal[13] found a small increase of *a** for thermally modified oak, the maximum was up to 2 units. Kamperidou *et al.*[25] also found an increase in *a**values after 4 hour heating of pine wood, but further heating did not bring more significant growth. Esteves *et al.*[28] found that the *a** increased for less intense treatments, reaching a 72% increase for wood treated at 170 °C for 24 hour treatment, but decreasing with the increase in treatment severity.[28] On the other hand, Johansson and Morén[29] found an increase of the values of the parameter *a** in the range of 4-6 units in comparison with native wood, even if the curve had a similar behavior (shape) as that for pine, *i.e.* first a slight growth was recorded, reaching the maximum, but with gradual increase of the heating time it decreased again.

In general, parameter *b** values changed more than those of parameter *a**. The biggest change was found for oak, where *b** values gradually decreased (Fig. 5) with increasing temperature and were almost by two thirds (12.8 units) lower than those for native wood. Conversely, *b** values for birch slightly rose up to 180 °C and then fell below the values of native wood (45% decrease at 240 °C).

Completely different values of the parameter *b** were found for pine. In this case, the values sharply increased up to the temperature of 160 °C and then suddenly began to decrease slightly below values of native wood. Similar values of parameter *b** for oak were reported by Čermak and Dejmal,[13] who found a decrease of 10.4 units, as well as by Weigl *et al.*,[30] who found a value of 8 for oak. Kamperidou *et al.*,[25] who measured color changes of treated and untreated pine wood, found a similar behavior of parameter *b** after treatment, *i.e.* first a slight increase, reaching the maximum and then a slight decline close to the values of untreated wood. Esteves *et al.*[28] found that the *b** for pine wood decreased with treatment time for all temperatures, with a maximum decrease of 86%. Johansson and Morén[29] found that the *b** coordinate was after the thermal treatment of birch wood higher than for untreated wood, but was fairly constant for the different treatment times at 175 °C. When treated at 200 °C, *b** decreased over time, and was back to the level of the untreated wood after a 10 hour treatment.

The overall color change ΔE^* is an important indicator that assesses the color change by parameters *L**, *a** and *b** (Fig. 6). Gonzales-Peña and Hale[31] proposed that ΔE^*_{ab} should be more suitable for predicting than ΔL^* for most properties of wood. The assessment criteria presented in Table 3 were used in a substantial part of the research on wood color. The overall color change ΔE^* of oak and birch had a very similar character, but the values of oak were substantially higher. In the temperature range 160-180 °C, the increase of the overall color change was sharp, and then the growth was mitigated at temperatures of 180-210 °C, but began again to rise rapidly at temperatures of 210-240 °C. However, the overall color change of pine had an opposite character. The biggest change occurred at 160 °C, and then the values gradually decreased with an increase in temperature. The value of the overall color change at the temperature of 160 °C was three times higher than that at the temperature of 240 °C (Table 6).

On the other hand, Čermak and Dejmal[13] found an overall color change of oak at 180 °C (2 hour and 4 hour treatment) in the range of 17-22 units and for the temperature of 230 °C (2 hour and 4 hour treatment) in the range of 37-41 units. Their values are higher than the values determined in our research. Kamperidou *et al.*[25] found average values of overall color change in the range of 19.6-34.4 for pine wood. Johansson and Morén[29] found that the average values of ΔE^* were 4.1, 4.8, 3 and 4.9 at 170 °C and 3.7, 1.8, 3.3 and 3.6 at 200 °C during 0, 1, 3, and 10 hour treatments. These values are consistent with our results of ΔE^*. The different behavior of the overall color change between birch and pine was also reported by Sundqvist,[32] who found that pine had darker and more saturated color.

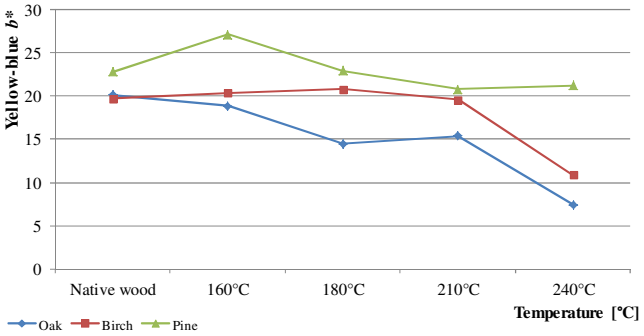

Figure 5: Color coordinate *b** of native and thermally modified wood for all wood species

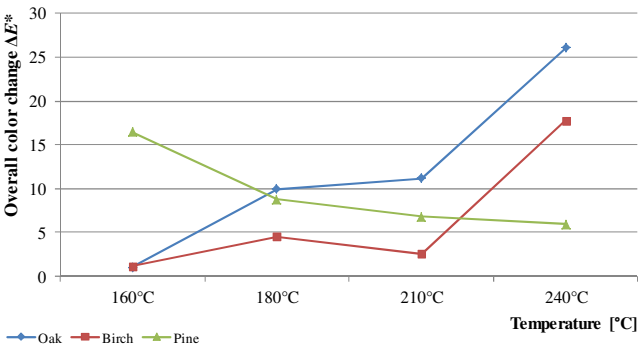

Figure 6: Overall color change ΔE^* of native and thermally modified wood for all wood species

Table 6
Evaluation of overall color change for native and thermally modified wood

Wood species	Treatment temperature	Overall color change ΔE^*	Evaluation	
			Criteria	Description
Oak	160 °C	1.01	$0.2 < \Delta E^* < 2$	Invisible difference
	180 °C	9.96	$6 < \Delta E^* < 12$	High color changes
	210 °C	11.21	$6 < \Delta E^* < 12$	High color changes
	240 °C	26.05	$\Delta E^* > 12$	Different color
Birch	160 °C	1.16	$0.2 < \Delta E^* < 2$	Invisible difference
	180 °C	4.52	$3 < \Delta E^* < 6$	Color change visible with medium-quality filter
	210 °C	2.56	$2 < \Delta E^* < 3$	Color change visible with high-quality filter
	240 °C	17.77	$\Delta E^* > 12$	Different color
Pine	160 °C	16.45	$\Delta E^* > 12$	Different color
	180 °C	8.77	$6 < \Delta E^* < 12$	High color changes
	210 °C	6.81	$6 < \Delta E^* < 12$	High color changes
	240 °C	5.96	$3 < \Delta E^* < 6$	Color change visible with medium-quality filter

The overall color change is characterized by darkening of the wood surface. This decrease of luminance of the wood should be caused by the formation of hemicelluloses and extractives, thermal degradation products or lignin polymerization reactions during treatment.[25] Dirckx *et al.*[33] contends that lignin is a major component responsible for coloring wood by the influence of thermal modification. For example, oak wood contains a relatively large amount of extractives, which come to the surface and spread out by the effects of thermal modification, resulting in discoloration of the wood surface. The color of oak wood thus presents a decreasing yellowness as a function of the increasing treatment temperature. Tolvaj and Faix[34] reported that yellow color was primarily caused by oligomeric chromophores, which probably originated from leuco chromophores. On the other hand, the main cause of the difference in the overall color change for pine is based on its anatomical structure. Pine wood presents significant differences between spring and summer wood, and therefore a sharp color change at lower temperatures occurs. Further temperature increase does not substantially change the color of the wood. This fact was confirmed by McDonald *et al.*,[35] who found that phenolic extractives, such as the stilbenes, can cause color changes during thermal modification. Besides phenolic extractives, degradation products from hemicelluloses and lignin resulting from thermal treatment, can be a reason for the coloring processes.[36] The influence of hemicellulose degradation in thermally treated pine wood is due to the released acetic acid, which further catalyzes the lignin.[37] Moisture content, which is a important characteristic of wood, has an impact on the degradation of various components during heating or exposure to heat.[38-41]

CONCLUSION

1. After heat treatment, the lightness of wood was reduced for all wood species. Birch wood

achieved the largest decrease in lightness – of 56%, while oak wood had a decrease of lightness of 52.2% and pine wood achieved the lowest decline of 42.7%.

2. In general, no great differences in the values of *a** were found between native and thermally modified wood. The *a** values for oak and birch wood had a similar character, while the values of pine showed an opposite character. The highest differences of *a** values were found for pine wood.
3. The increase in temperature resulted in a decrease of the parameter *b** for oak and birch wood. The differences in the values between these two wood species were less pronounced. The highest *b** value was found for pine thermally modified at the temperature of 160 °C (16% increase compared to the native wood), but it gradually decreased with further increase in temperature.
4. The overall color change *ΔE** gradually increased with increasing temperature for oak and birch, while pine achieved the highest value at the temperature of 160 °C. Based on the evaluation of the overall color change, it can be concluded that the highest differences were found for pine wood, and the lowest overall color change was visible with medium-quality filter.

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