

EFFECT OF THERMAL MODIFICATION ON THE
COLOUR CHANGES OF OAK WOOD

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ABSTRACT

This paper deals with the examination of colour and colour changes of oak wood. The colour parameters (L^* , a^* , b^* , and ΔE^*) were determined by the CIE- $L^*a^*b^*$ colour space on native and thermally modified wood oak wood as well as on sub-fossil oak wood. The thermal modification has been carried out at four different temperatures: 160, 180, 210 and 240°C for oak wood. The results show that heat treatment resulted in the darkening of wood tissues and the lightness (L^*) of oak wood decreased. The values of a^* and b^* also decreased, although the decline of parameter a^* was only slight, while the parameter b^* dropped more significantly. The overall colour change (ΔE^*) increased with raising the temperature of modification with a maximum at 240°C. All the investigated characteristics have achieved the biggest changes at the highest temperature of modification. The equalized colours of thermally modified wood can significantly increase the use of products. Colour can be an important indicator of the quality of wood surface.

KEYWORDS: Colour; overall colour change; thermal modification; temperature; oak wood; sub-fossil oak.

INTRODUCTION

Colour is one of the quality attributes that influences perceptions by customers of wood products. Colour of the wood is a factor that affects initial contact of man with the product (Abrahão 2005). It is a characteristic that is closely linked with the selection of a given product according to the visual, decorative and functional point of view.

The most widespread method of evaluating colour or colour changes of the wood is CIE $L^*a^*b^*$ colour space with a three axis system (Fig. 1) (Brischke et al. 2007; Baar and Gryc 2012; Bekhta and Niemz 2003; Čermák and Dejmál 2013; Dzurenda 2014). This method is used for accurate determination of colour, discolouration or colour difference most often in combination with colorimeters and spectrophotometers. The method is based on the conditions set by CIE (Commission Internationale de L'Eclairage), which make it ideal for measuring various parameters of colours: lightness (L^*), chromaticity coordinates (a^* and b^*), chroma (C^*) and hue (h^*) (Konica Minolta 2007).

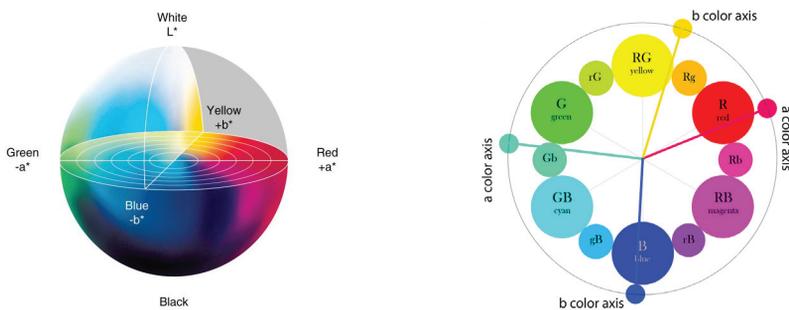


Fig. 1: Graphical representation of the CIE $L^*a^*b^*$ color space.

Although the colour is the result of the structure as well as composition of the wood itself, during the production is greatly influenced by the different processes. One of the processes that substantially alter the colour of the wood is the thermal modification (ThermoWood). During this process the wood is heated to a given temperature for certain, predetermined time. Therefore the discolouration of wood depends on the type of wood species, temperature and heating time (Hill 2006; Sahin et al. 2011). Most often, these temperatures are in the range of 150 - 260°C (Barčík and Gašparík 2014).

In general, thermal modification changes the colour of the wood, because it directly affects the constituents in the wood – lignin, cellulose and hemicellulose. Although these constituents of wood are relatively resistant, but a certain temperature starts their degradation as well as break of their chemical bonds. Significant and irreversible changes in wood start in the range of temperatures 180 – 250°C. Bekhta and Niemz (2003) report that thermally modified wood at 200°C has the most significant change of colour and the biggest change is achieved in the first hours of modification. On the other hand, Patzelt et al. (2003) claims that there is no difference in the colour change of thermally modified wood between high temperature and short time compared with the lower temperature and longer time. Darker colour of thermally modified wood is caused by degradation of hemicelluloses and subsequently arising products formed from it (Sehlstedt-Persson 2003; Sundqvist 2004). Carbonization of wood begins at 250°C, where appear emerging carbon oxides as well as other combustible substances (Kačíková and Kačík 2011).

The aim of this research was to evaluate the effect of different temperatures during thermal modification on the colour change of oak wood. Colour measurement was evaluated according to CIE $L^*a^*b^*$ colour space by parameters L^* , a^* , b^* and ΔE^* .

MATERIAL AND METHODS

Material

The experiment used two types of oak, Pedunculate oak (*Quercus robur* L.) and sub-fossil oak. The experimental Pedunculate oak trees (*Quercus robur* L.) (75 years old) was harvested in the central region of Mari El Republic. The zones, which were in the middle distance between the pith and bark, were chosen for sample preparation. From these parts were cut 200-cm long sections which contained 3-mm-wide annual rings. For the experiments, samples with gross dimensions of 40 × 100 × 500 mm were used. These samples were air-conditioned in the conditioning room ($\phi = 65 \pm 3\%$ and $t = 20 \pm 2^\circ\text{C}$) for more than six months to achieve an equilibrium moisture content (EMC) of 12%. All of the air-conditioned samples were divided into two basic groups— samples of native wood and samples for thermal modification. Each group was represented by 25 samples, e.g. 5 samples per treatment type. Native (untreated) wood samples served as reference standard.

Sub-fossil oak was found in the alluvium of the river Sura in Mari El Republic at a depth of 5-7 meters from the water level. These samples were divided into three groups A, B and C, according to the age of the sub-fossil wood (Tab. 1). Each group was represented by 5 samples. Samples of sub-fossil oak were intended only for the comparison of colour with native and thermally-treated oak. Samples were also air-conditioned in the conditioning room ($\phi = 65 \pm 3\%$ and $t = 20 \pm 2^\circ\text{C}$) 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.

Tab. 1: Categorization of sub-fossil oak.

Wood Species	Group	Radiocarbon dating method	
		Radiocarbon age, years ago	Calibrated age interval, beginning - end (with accuracy 95.4%)
Sub-fossil oak	A	2320 ± 50 years	550 - 200 BC
	B	950 ± 30 years	1020 - 1170 AD
	C	770 ± 50 years	1160 - 1310 AD

A special group of samples have been prepared for identification of physical properties of native, thermally modified and sub-fossil wood. The dimensions of these samples were in compliance with relevant standards and these samples were only used for identification and verification 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 Yoshkar-Ola, Mari El Republic, which provided apparatus and testing laboratory.

Wood samples intended for thermal modification were put on a metal grate and subsequently placed into thermal furnace XVC 304 with cooling system (basic parameters are listed in Tab. 2),

produced by UNOX company (subsequently adapted for laboratory use) and modified in three phases. During first phase (heating) the wood was dried and heated to required final temperature of 180 – 240°C. During second phase (thermal modification) the desired temperature was kept for the whole duration – 5 hours. Last third phase (cooling) was characterized by a gradual decrease in temperature.

Tab. 2: Conditions for thermal treatment.

Input technical parameters		
Thermal Furnace	Furnace Power	5.3 kW
	Heating	up to 260°C
	Cooling	down to 20°C
	Maximum reached temperatures	160, 180, 210 and 240°C

At the temperature 100°C the conditioning of wood was carried out (facilitated by using dispersed water) in the transverse direction. Refer to Tab. 3 for times of all thermal treatment phases. All samples were then machined to final thickness (25 mm) using a thickness planer. Subsequently both faces of each sample were intended for measuring of colour and therefore were gently grinded with a belt sander (grain size 200).

Then, the native and thermally modified oak samples as well as sub-fossil samples were conditioned ($\phi = 65 \pm 3 \%$ and $t = 20 \pm 2^\circ\text{C}$) for three weeks. Thus, native and thermal modified samples (final clear dimensions 25 × 100 × 500 mm) were prepared for colour measuring.

Tab. 3: Phases of thermal treatment.

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

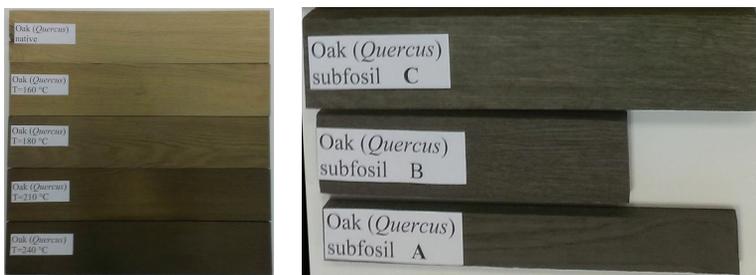


Fig. 2: Native, thermally modified and sub-fossil samples - oak (left), and sub-fossil oak (right).

Colour measurements

Colour measurements were performed on native and thermally modified samples with a portable spectrophotometer Konica Minolta CM-600d (10° standard observer, D65 standard illuminate, colour difference format ΔE^*_{ab}). Measurements were taken on the 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) were used to determine: ΔL^* , Δa^* , Δb^* (exemplified for $\Delta L^* = L^*_{\text{modified}} - L^*_{\text{native}}$), overall colour change ΔE^* by using the CIE $L^*a^*b^*$ colour measuring system according to ISO 11664-2 (2007), ISO 11664-4 (2008), and ISO 11664-6 (2013). The overall colour change ΔE^* was classified under the rules distribution of colour changes according to Cividini et al. (2007) (Tab. 4).

Tab. 4: 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



Fig. 3: Laboratory thermal furnace with samples. Fig. 4: Spectrophotometer Konica Minolta (left) and color measurement (right).

Evaluation and Calculation

The colour differences were evaluated by overall colour change which was calculated according to Eq. 1 from ISO 11664-4 (2008) and ISO 11664-6 (2013),

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

where: ΔE^* - overall colour change,
 ΔL^* , Δa^* and Δb^* - the differences between the initial and the final values (before and after thermal modification, e.g. between native and thermally modified wood or sub-fossil wood) of L^* , a^* , and b^* , respectively.

The density was determined as an auxiliary indicator. Density was calculated according to Eq. 2 from ISO 3131 (1975),

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

where: ρ_w - the density of the test sample at certain moisture content w ($\text{kg}\cdot\text{m}^{-3}$),
 m_w - the mass (weight) of the test sample at certain moisture w (kg),
 a_w , b_w and l_w - dimensions of the test sample at certain moisture w (m),
 V_w - the volume of the test sample at a certain moisture w (m^3).

The density of wood after thermally treatment was calculated according to Eq. 3 from ISO 3131 (1975),

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

where: ρ_{tt} - the density of the test sample after thermally treatment ($\text{kg}\cdot\text{m}^{-3}$),
 m_{tt} - the mass (weight) of the test sample after thermally treatment (kg),
 a_{tt} , b_{tt} and l_{tt} - dimensions of the test sample after thermally treatment (m),
 V_{tt} - the volume of the test sample after thermally treatment (m^3).

Moisture content of samples was determined and verified before and after thermal treatment. These calculations were carried out according to ISO 3130 (1975) and Eq. 4,

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

where: w - the moisture content of the samples (%),
 m_w - the mass (weight) of the test sample at a certain moisture w (kg),
 m_0 - the mass (weight) of the oven-dry test sample (kg).

Drying to oven-dry state was also carried out according to ISO 3130 (1975), using the following procedure: The samples were placed in the drying oven at a temperature of $103 \pm 2^\circ\text{C}$ until a constant mass had been reached. Constant mass is considered to be reached if the loss between two successive weighing carried out at an interval of 6 h is equal to or less than 0.5 % of the mass of the test sample. After cooling the test samples to approximately room temperature in a desiccator, the sample was weighed rapidly enough to avoid an increase in moisture content by more than 0.1 %. The accuracy at weighing should be at least 0.5 % of the mass of the test sample.

RESULTS AND DISCUSSION

Physical and mechanical properties

Density values of the native, thermally modified as well as sub-fossil oak wood are shown in Tab. 5. These values have been measured after conditioning.

Our density values of native oak wood ($639 \text{ kg}\cdot\text{m}^{-3}$) correspond to the values mentioned by Knapić et al. (2008) for *Quercus robur* L., i.e. $500\text{--}660 \text{ kg}\cdot\text{m}^{-3}$. Wood density gradually decreased with increasing temperature during thermal modification. The highest decrease of wood density (10.6 %) was found at 240°C . Shchupakivskyy et al. (2014) found 12 % decrease in density of oak heat-treated at 220°C , which corresponds with our results. Reduced density was caused not only moisture loss during thermal modification and thereby reducing the volume of wood, but also by degradation of its structure. Boonstra et al. (2007) argues that the degradation of hemicellulose into volatile products and evaporation of extracts are the main causes of reduced density.

Tab. 5: Density of native, thermally modified and sub-fossil wood.

Wood species Density (kgm ⁻³)	Pedunculate oak					Sub-fossil oak		
	Native	TMW				Native		
		160°C	180°C	210°C	240°C	A	B	C
Minimum value	595	594	575	575	546	656	630	597
Maximum value	696	673	657	605	596	825	761	726
Average value	639	622	619	588	571	765	696	687

When comparing of densities of current oak and sub-fossil oak in our research there has been found that the oldest sub-fossil wood had a 19 % higher density. On the other hand, Govorčin and Sinković (1995) argue that the sub-fossil oak wood has a similar or slightly increased density compared to the current wood. However, it is necessary to take into account the variability of density within tree species, growth locations, location in trunk etc.

Colour changes

As stated earlier, the colour change was evaluated by means of the parameters L^* , a^* , b^* and ΔE^* . Tab. 6 shows the L^* , a^* , b^* and ΔE^* values of native, thermally modified and sub-fossil oak wood.

Lightness L^* has changed the most and was gradually decreased with increasing temperature as we expected, i.e. the lowest lightness of thermally modified wood was at temperature 240°C (Fig. 5). All thermally modified samples had a significant decrease of lightness compared with the native wood. At 240°C, the Lightness of wood decreased by more than half. Similar results also indicate Čermák and Dejmál (2013), who found that thermally-modified oak at temperature of 230°C had lower lightness by an average of 36 units. Lightness L^* has changed the most and was gradually decreased with increasing temperature as we expected, i.e. the lowest lightness of thermally modified wood was at temperature 240°C (Fig. 5).

Tab. 6: Average values of three color coordinates and overall color change.

Wood Species	Treatment/ Group	Color coordinates			Overall color change
		L^*	a^*	b^*	ΔE^*
Pedunculate oak	Native wood	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
Sub-fossil oak	A	33.35	0.62	3.26	27.83
	B	34.68	0.85	4.70	25.88
	C	38.38	1.56	7.19	21.52

All thermally modified samples had a significant decrease of lightness compared with the native wood. At 240°C, the lightness of wood decreased by more than half. Similar results also

indicate Čermák and Dejmál (2013), who found that thermally modified oak at temperature of 230°C had lower lightness by an average of 36 units. In their research, the lightness of native wood was 69-75 and after modification was decreased to 29-35. Same lightness of native oak wood were also found by Babiak et al. (2004), who studied 19 domestic wood species. Change of lightness has been caused by that the wood has reached a dark shade which was similar to the shade of sub-fossil wood (near-black shade). However, the shade of sub-fossil wood is caused by ferric component dissolved in the water, which reacted with the tannins contained in oak. The degree of darkening is directly dependent on the time at which the wood was deposited under these conditions, as well as the nature of the sediments (Kolář et al. 2010).

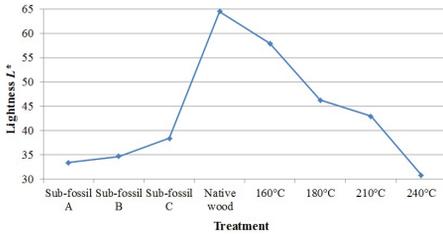


Fig. 5: Lightness L^* of native, thermally modified and sub-fossil oak.

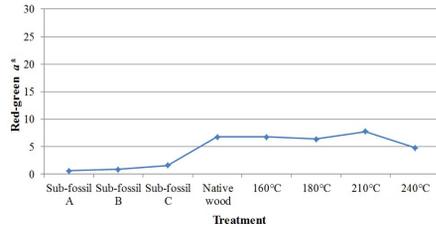


Fig. 6: Color coordinate a^* of native, thermally modified and sub-fossil oak.

The a^* values of modified oak first slightly decreased and then increased with peak at 210°C, then values dropped again down below values of native wood (Fig. 6). Čermák and Dejmál (2013) found a relatively small increase in a^* values not exceeding 2 units. Although parameter a^* gradually decreased with increasing temperature of modification, the lowest achieved value had not significant differences compared to native wood. Similarly, small differences were found between the different age groups of sub-fossil oak. The a^* values of sub-fossil wood were quite a bit lower compared to native and thermally modified wood.

In general, the b^* values had greater changes than at parameters a^* . The increasing temperature of modification caused gradual decrease values of the parameter b^* (Fig. 7), and as well as in the previous case, the biggest change occurred at 240°C. Although values gradually decreased with increasing temperature, at temperature 210°C occurred slightly increasing, and then values dropped again. The biggest change were almost by two thirds (12.8 units) lower than for native wood. The b^* values achieved at 240°C were nearly identical to the values of the sub-fossil oak group C. Similar changes of parameter b^* were obtained by other authors in their research. Čermák and Dejmál (2013) found decrease in the values of 10.4 units while Weigl et al. (2012), found the value of 8 units for parameter b^* .

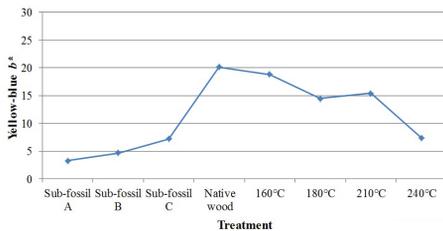


Fig. 7: Color coordinate b^* of native, thermally modified and sub-fossil oak.

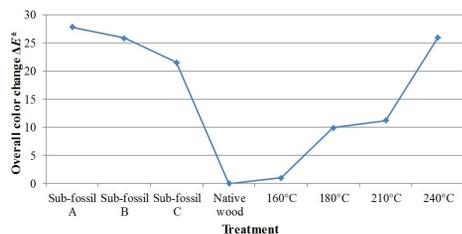


Fig. 8: Overall color change ΔE^* of native, thermally modified and sub-fossil oak.

The overall colour change ΔE is an important indicator that assesses the colour change based on changes in all parameters L^* , a^* and b^* . In part of Materials and Methods was presented table with the assessment criteria, which was used for evaluation of wood colour change. In the temperature range 160 – 180°C, the increase of overall colour change was sharp, and then at temperatures of 180 – 210°C growth was mitigated, but again began to rise rapidly at temperatures of 210 – 240°C (Tab. 7) (Fig. 8).

Other authors have achieved higher values of overall colour change. Čermak and Dejmál (2013), found that the overall colour change of native oak wood heated at 180°C (2-hour and 4-hour heating) was in range 17 – 22 units and for temperature 230°C (2-hour and 4-hour heating) was in range 37 – 41 units.

The overall colour change is always caused by darkening the wood surface. This darkening of the wood surface can be explained by thermal degradation of hemicelluloses and extractives or potential polymerization reactions of lignin (Kamperidou et al. 2013). Oak wood is known for its high content of extractives, which are extruded to the surface during thermal of modification, causing undesirable colour change.

Tab. 7: Evaluation of overall color change for thermally modified and sub-fossil oak.

Wood species	Treatment/Group	Overall color change ΔE^*	Evaluation	
			Criteria	Description
Pedunculate 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
Sub-fossil oak	A	27.83	$\Delta E^* > 12$	Different color
	B	25.88	$\Delta E^* > 12$	Different color
	C	21.52	$\Delta E^* > 12$	Different color

CONCLUSIONS

The lightness of wood decreased with increasing temperature during thermal modification. The largest decline was found at 240°C namely 52.2 %. Thermal modification was relatively little effect on the change of the parameter a^* . However, the temperature rise gradually reduced the a^* values. Also in this case, the largest decrease was found at 240°C, namely 16.9 %. Increasing temperature resulted in a decrease in the values of the parameter b^* of oak wood. In this case, the course of the decrease was not unequivocal. Parameter b^* was first decreased to a temperature of 180°C, then slightly increased at 210°C and then dropped again. The overall colour change ΔE^* increases with increasing temperature. Growth of the overall colour change was characterized by mild onset, then rapid growth, followed by a slight rise again and ultimately a sharp increase. As with the previous parameters, also overall colour change reached a maximum at the highest temperature.

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