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EFFECT OF HEAT TREATMENT ON COLOUR, DENSITY AND DIMENSIONAL STABILITY OF SUBFOSSIL OAK WOOD

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ABSTRACT

The subject of this paper was analysis of the influence of heat treatment on colour change and dimensional stability of subfossil oak wood. The subfossil oak logs used in this experiment originated from the Morava River in Central Serbia. Heat treatment was conducted on oak samples in a laboratory vacuum chamber at temperature of 180˚C for 4 hours. After the heat treatment, subfossil oak wood retained its natural colour, but its density and dimensional stability were changed. Density decreased from 0.637 g/cm³ to 0.620 g/cm³ and shrinkage was reduced by 25% in radial and 35% in tangential direction.

Key words: subfossil oak, heat treatment, wood colour, dimensional stability

1. INTRODUCTION

Subfossil wood is unfossilized wood which has been deposited in rivers, swamps or moraine sediments for hundreds or thousands of years. This material is interesting to wood marketers and wood industry because of the high market value that it can reach as a result of its age, specific colour and texture. In anaerobic conditions wood is protected from the main factors that affect its degradation - xylophagous insects and lignikol fungi, and it can be preserved for an extremely long period of time.
Although protected against the influence of fungi and insects, many processes take place in the wood over time. This material changes its properties over a long period of deposition in water or soil. The change in colour is the most obvious difference between subfossil and recent oak wood. The colour of wood turns gold-brown, dark gray or black on the outer layer of the trunk. It is caused by reaction of the ferric components from the water and the tannins present in oak (Bürck et al. 2012). The intensity of the shade is primarily determined by the time period during which the wood had been deposited in water and soil (Kolář and Rybníček 2010). The mechanical properties of subfossil oak wood are lower by 10-40% compared to recent oak, but the biggest drawback is a dimensional change which is twofold compared to recent oak (Veizović et al. 2017). Structural alterations in the wood cell wall components, together with a decrease in crystallinity of cellulose, contributed to increase in the number of water bonding sites, clarifying the increment in EMC (Juan Guo et al. 2017). Increased EMC leads to increased dimensional changes.

It is known that thermal treatment leads to an increase in dimensional stability. Changes that occur in wood and lead to rise in dimensional stability are: (1) loss of hygroscopic hemicellulose polymers (Esteves and Pereira 2009), (2) cross-linking or bridging of cellulose chains (Stamm and Tarkow 1947, as cited in Kocaefe et al. 2015), (3) cross-linking of aromatic rings in lignin (Tjeerdsma et al. 1998).

The aim of this study was to analyze the possibility of improving dimensional stability of subfossil oak wood by using heat treatment. An additional goal was to analyze colour, as one of the main advantages of subfossil wood in comparison with the recent one.

2. MATERIAL AND METHODS

The research involved trunks of subfossil Oak (*Quercus spp.*) which had been deposited under the surface of alluvial plains of the Great Morava river in Central Serbia. Unedged timber was obtained after sawing. The nominal thickness of the boards was 25 mm, the width was 15-40 cm and the length 2m. After sawing, timber was air-dried to about 20% moisture content (MC), followed by kiln-drying to 9.5% MC. Drying quality was evaluated both after air drying and kiln-drying processes. It was found that the drying process has no significant effect on change in colour of the subfossil wood (Veizović et al. 2018a).

From the three boards a total of 54 samples (20 mm x 20 mm x 300 mm) were prepared (Fig. 1). The samples labeled with T were used for the thermal treatment. Dimensions, mass and density of specimens were determined. Other samples were used for SEM imaging and FTIR spectroscopy (Veizović et al. 2018b)

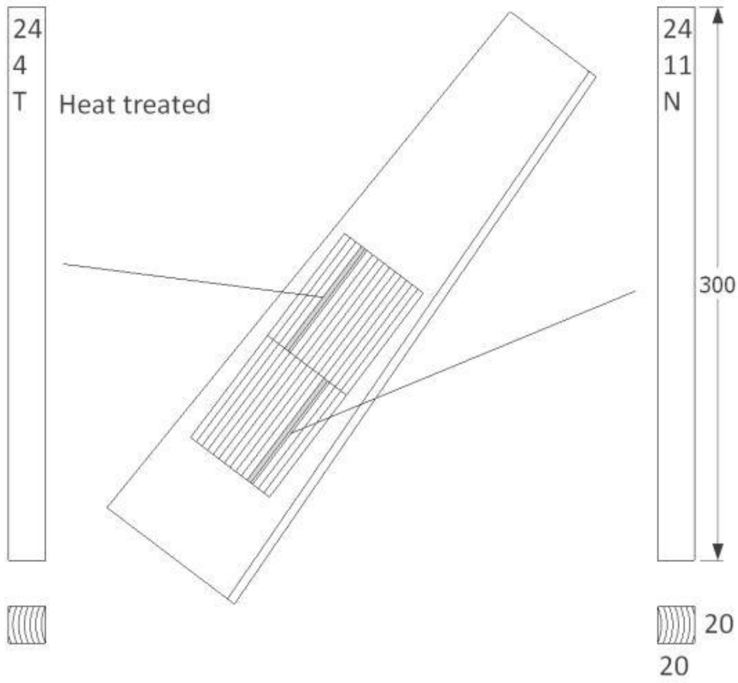

Figure 1. *Boards cutting plan*

Heat treatment was conducted in a laboratory vacuum oven (Fig. 2), at a temperature of 180° C and a pressure of 100 mbar, lasting for 4 hours.(Tab. 1). The colour of subfossil oak wood was measured before and after heat treatment, using Easyco (Erichsen). The diameter of the lenses was 10 mm, the device was set at the observing angle of 10˚, and illumination D65 for daylight. Measured colour was presented in CIEL*a*b* colour system. Based on the obtained coordinate values, the colour difference was determined applying the following formula:

$$\Delta E = \sqrt{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2}$$

The classification was made according to the criteria shown by Allegretti et al. (2008).

The samples of 20 mm x 20 mm x 20 mm were used for investigation of density and dimensional stability. Firstly the samples were drenched in water with temperature 20 ± 2˚C until their dimensions were stable. Their weight was measured with an accuracy of 0.01 g and dimensions with an accuracy of 0.1 mm. Afterwards samples were dried to 0% moisture content, followed by measurements of mass and dimensions in oven dry state.

Table 1. *Heat treatment schedule*

Phase		Duration [h]	T [°C]	P [mbar]
1	Heating up	2	50	1000
2	Predrying	13	60	100
3	HT	1	75	100
4	HT	1	90	100
5	HT	1	105	100
6	HT	1	120	100
7	HT	1	135	100
8	HT	1	150	100
9	HT	1	165	100
10	HT	4	180	100
11	Cooling	18	20	1000

Figure 2. *Laboratory vacuum chamber*

3. RESULTS AND DISCUSSION

The process was stable and the overall time of the heat treatment was 45h (Fig. 3). Data obtained in this work showed that heat treatment improved dimensional stability of subfossil oak wood. Non treated subfossil oak wood shrinkage was about 9.2% (R) and about 16.9% (T). Shrinkage of heat treated subfossil oak wood was about 7.1% (R) and 10.9% (T) (Fig. 4). The reduction of shrinkage was about 25% in radial and about 35% in tangential direction. These results confirm that thermal treatment has a positive effect on dimensional stability of wood, and shows that this also applies to subfossil wood.

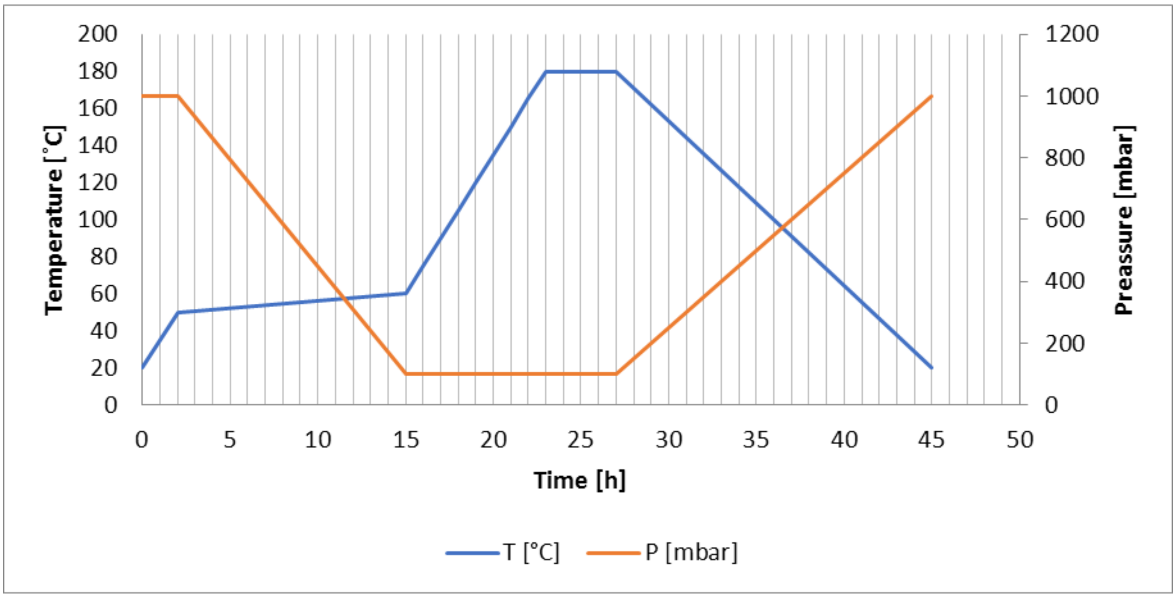

Figure 3. *Heat treatment process of subfossil oak wood in a vacuum chamber*

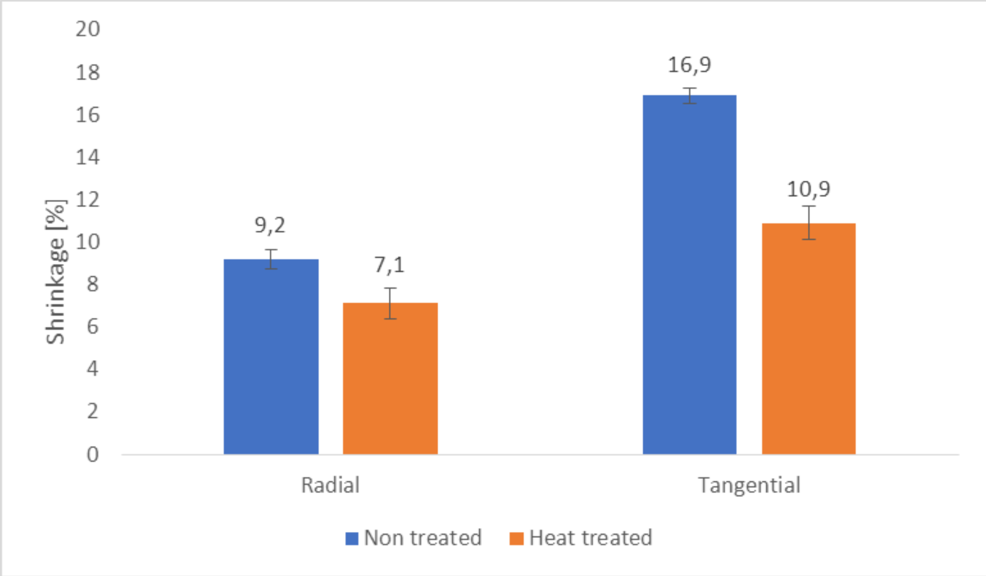

Figure 4. *Shrinkage of non treated and heat treated subfossil oak wood*

After the heat treatment, the mean value of density of the subfossil oak wood was 0.620 g/cm³, while the mean value before the treatment was 0.637 g/cm³. Statistical analysis showed that there is no significant difference (p=0.056) between density of heat treated and non treated subfossil oak. It would still be necessary to examine the effect of thermal treatment on the mechanical properties of this specific material.

Heat treatment process did not affect the colour change of subfossil oak wood ΔE = 0.3 – small difference (Tab. 2). This is partially expected, due to the already dark colour of the subfossil wood, and it differs from recent oak, because this wood significantly changes colour in thermal treatment. It is good that subfossil wood retains its natural colour, because this is, in addition to its age, the main factor contributing to the high value of this material.

Table 2. *Colour of subfossil oak before (kiln dried) and after heat treatment*

Subfossil oak wood colour	Air dried	Kiln dried	Heat treated
L*	36,2	36,0	35.9
a*	5,0	4,9	4.9
b*	11,5	11,4	11.3

4. CONCLUSIONS

In this paper the influence of heat treatment of subfossil oak wood was analyzed, regarding dimensional stability, density and colour. The idea was to decrease dimensional changes of subfossil oak wood, but also to retain its natural colour.

Heat treatment improved the dimensional stability of subfossil oak wood. Shrinkage was reduced by 25% in radial and 35% in tangential direction.

There is no difference in colour between non treated subfossil oak wood and the same wood after heat treatment at 180˚C for four hours.

Heat treatment at 180˚C did not cause the change in subfossil oak wood density. The density, as the main indicator of mechanical properties, remained unchanged, which could be an indicator that the heat treatment did not significantly change the mechanical properties. Future research should analyze the effect of the different applied temperatures and the duration of the heat treatment on the properties of subfossil oak.

Mechanical properties of heat treated subfossil oak should also be investigated in the future, but it should be noted that this wood, due to already reduced mechanical properties, is not used as construction timber or for other applications where good mechanical properties are required.

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