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Determination of color-changing effects of bleaching chemicals on some heat-treated woods

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Abstract

The aim of this study was to use bleaching chemicals to remove the discoloration occurring on the surface of wood after heat treatment in order to restore the natural color of the wood. For this purpose, samples prepared from Scots pine (*Pinus sylvestris* L.), sessile oak (*Quercus petraea* L.), Eastern beech (*Fagus orientalis* L.), and Uludağ fir (*Abies bornmuelleriana* Mattf.) were exposed to heat treatment at temperatures of 140 and 160 °C for time periods of 3, 5, and 7 h. Bleaching solutions S1 (NaOH + H₂O₂), S2 (NaSiO₃ + H₂O₂), and S3 (H₂C₂O₄) at a concentration of 18% were then applied to the surface of the materials and the color change was determined according to ASTM D 2244 standard. Depending on the heat treatment temperature and duration, an increase in total color change values was detected on the surfaces of the materials and the color of the samples became darker. The total color change values decreased after bleaching with the S2 solution in the heat-treated Scots pine and fir samples, with the S3 solution in the beech samples, and with the S1, S2, and S3 solutions in the oak samples. The findings showed that by using bleaching chemicals to lighten wood materials darkened after heat treatment, it is possible to obtain results close to the natural color values.

Keywords: Wood material, Heat treatment, Bleaching, Color measurement

Introduction

Although wood materials exhibit adequate natural resistance to some external influences, wood cannot maintain long-term tolerance against the outdoor environment. For this reason, wood materials are impregnated with preservatives or coated with a protective layer [1]. Due to the fact that the solvent-based protective coating products and traditional wood impregnation chemicals threaten the environment and human health, scientific research has focused on new environmentally friendly chemicals and methods and new products for wood protection. As a result of developing technology, protecting wooden materials with impregnation products and chemicals (including toxic substances) has either been limited or completely banned [2–4].

Heat treatment of wooden material in Europe, and more recently in North America has attracted attention as an environmentally friendly wood protection method. Heat treatment is a physical process that results in permanent changes in the chemical composition of the polymer components of wood cell wall [5]. Heat treatment of wood was researched in Germany for the first time by Stamm and Hansen in the 1930s. Other studies on the subject were conducted in the United States by White in the 1940s and Bavendam, Rundel, and Buro in the 1950s. Kollman and Schneider published their findings in the 1960s and Rusche and Burmester did so in the 1970. In the 1990s, research was carried out in France and the Netherlands [6]. In recent years, different commercial heat treatment processes have emerged: the Finnish process (ThermoWood) uses steam, the Dutch (Plato Wood) uses a combination of steam and heated air, the French (Rectification) uses an inert gas, and the German (OHT) uses heated oil [7]. Therefore, the thermal process of

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modification has been applied to improve and change the properties of wood for a long time. ThermoWood technology can be used for an extensive range of applications including garden fences, sauna furnishings, window frames, cladding, and decking [8].

The heat treatment process for wood modification has both the advantage of being evaluated as an environmentally friendly method and the disadvantage of reducing the durability of the wooden material. Deepening the color of the wood and negative after-effects on the gloss and hardness are also among the disadvantages of heat treatment modification [2, 9–11].

Sundqvist [12] performed heat treatment on Scots pine (*Pinus sylvestris*), Norway spruce (*Picea abies*), and birch (*Betula pubescens*) at temperatures of 65–95 °C for 0–6 days. As a result of the process, pine treated at 65 °C and 80 °C showed a red–yellow shift in the sapwood and a yellow–red shift in the heartwood. The color homogeneity was less for birch sapwood than for pine and spruce, and the homogeneity generally tended to decrease with increasing treatment temperature. According to Sehlstedt-Persson [13], the main reason for the color changes in the sapwoods of pine and spruce exposed to heat treatment at 60 °C – 95 °C arose from the compounds in the extractives of the materials. The color changes of the woods and extractives caused by heat treatment increased with increasing time and temperature. Tomak et al. [14] reported that red color values increased on all heat-treated wood material surfaces and that the coniferous species were more reddish than deciduous species. They stated that as a result of heat treatment, darker tones were dominant on all wood material surfaces because of the different amounts of lignin and hemicellulose content found in the wood materials used.

Each type of wood has its own variation of color, texture, and grain pattern. Some cuts of solid wood and flitches of veneer may be lighter or darker than others. To obtain a uniform color in furniture, the wood is either bleached or dyed. In other words, the only way to avoid this discoloration is to bleach the wood or use a bleaching toner on the wood before finishing. Bleaching is the removing of color pigments in the structure of wood using various bleaching chemicals and bleaching systems. While there are many bleaching agents available, the two most commonly used chemicals are sodium hydroxide (NaOH) and hydrogen peroxide (H₂O₂) [15]. Liang and Wang [16] found that peracetic acid had no effect on the structure of birch veneer samples, and that hydrogen peroxide and sodium hypochlorite bleaching chemicals removed some of the lignin from the coating surface. Yamamoto et al. [17], in their study examining color change values, in order to reduce the use of resin and improve the bonding resistance, treated birch

veneers at 80 °C with 5% H₂O₂, 5% H₂O₂ + alkali (1.4% sodium hydroxide + 5% sodium silicate + 0.5% magnesium sulfate), and water. As a result, they reported that the veneer surfaces were significantly whiter, resulting in a decrease in the yellow and red color values while increasing the color brightness value. Mononen et al. [18] in their study, treated ordinary birch (*Betula pendula* Roth) wood with H₂O₂ solution and determined the color and chemical changes that occurred. As a result of the study, they stated that there was a significant increase in whiteness, whereas the red color value decreased and the yellow color value increased. Chemical analysis of the birch samples treated with H₂O₂ indicated an increase in unconjugated carbonyl structures and a simultaneous degradation of aromatic structures. Budakci and Karamanoglu [19] in their study, applied bleaching chemicals, including 18% concentrations of S1 (NaOH + H₂O₂), S2 (NaOH + Ca (OH)₂), S3 (KMnO₄ + NaHSO₃ + H₂O₂), S4 (NaSiO₃ + H₂O₂), and the commercial product S5 [Cuprinol Decking Restorer- (H₂C₂O₄ + C₂H₄ (OH)₂] to samples of pine, Eastern beech, oak and chestnut that had been exposed to a 12-month aging process. At the end of the application, the highest color change value was obtained on the pine, beech and chestnut surfaces with the S4 (NaSiO₃ + H₂O₂) solution and on the oak surfaces with the S1 solution. Cheumani-Yona et al. [20] carried out the bleaching of liquefied black poplar wood with hydrogen peroxide. After the bleaching process, they found a remarkable increase in the color parameters L^* , a^* , b^* and ΔE^* .

The objective of this study was to use bleaching chemicals to remove the discoloration occurring on the surface of wooden material after heat treatment in order to restore the natural color to the surface of the wood.

Materials and methods

Wood materials

Scots pine (*Pinus sylvestris* L.), sessile oak (*Quercus petraea* L.), Eastern beech (*Fagus orientalis* L.), and Uludağ fir (*Abies bornmuelleriana* Mattf.), which are widely used in the furniture and decoration industry in Turkey, were chosen for this study. Samples were randomly selected from the sapwood section of first-grade timber, and all exhibited regular-fiber, with no variation in color or density, were knotless and crack-free, had annual rings perpendicular to the surface, and were cut to dimensions of 18 × 110 × 350 mm, with regard to the principles in TS 2470 [21].

Heat treatment

The air-dried samples were first kept at 103 ± 2 °C until they reached a constant weight. They were then exposed to heat treatment at temperatures of 140 °C and 160 °C

for 3, 5, and 7 h. Subsequently, they were stored in an air-conditioned cabinet at 20 ± 2 °C and $65 \pm 5\%$ relative humidity until a constant weight was reached.

Bleaching chemicals

Sodium hydroxide (NaOH), sodium silicate (NaSiO₃), hydrogen peroxide (H₂O₂), and oxalic acid (H₂C₂O₄) were used in the bleaching procedure shown in Table 1 as three different solution groups.

Application of the bleaching process

The chemical substances used for bleaching, depending on their properties, were prepared at 18% concentration by weight (M_g) or by volume (V_{mL}) [22]. For this purpose the following Eqs. (1), (2) were used:

$$\text{For the solid form : } M_g = \frac{M_s \cdot \% \frac{M}{M}}{\% S}, \tag{1}$$

$$\text{For the liquid form : } V_{mL} = \frac{V_s \cdot \% \frac{V}{V}}{\% S \cdot d}, \tag{2}$$

where M_g is the amount of the desired solution (g); M_s , the amount of the solution for the desired preparation; %S, the impurity rate of the substance; V_{mL} , the amount of the desired solution (mL); V_s , the amount of the solution for the desired preparation; V/V , the percentage by volume for the desired solution, and d is the density of the solution.

The prepared chemicals were applied with a sponge to the surface of the heat-treated samples, first parallel, then perpendicular, and then again parallel to the fibers at 100 ± 10 mL/m². Constitutive chemical solutions of different combinations were individually applied to the wood surfaces at 3-min intervals in order to allow interaction of the previously applied chemicals with the wood.

To improve the perpendicular penetration, after bleaching, the treated wood samples were allowed to dry at room temperature for 2 days, and then the neutralization process was performed with distilled water. Finally, the moisture content of the samples was adjusted to 12% and the sample surfaces were sanded slightly before taking the experimental measurements.

Color measurement

Color measurements were performed according to the ASTM D 2244 [23] using the BYK Gardner Spectro-Guide 45/0 at Duzce University. The 1976 International Commission on Illumination (Commission Internationale de l’Eclairage—CIE) system was used for measuring the L^* , a^* , and b^* color coordinates. The three CIE $L^*a^*b^*$ coordinates representing the lightness of the colors L^* [from 0% (black) to 100% (white)], a^* [from red (+) to green (-)], and b^* [from yellow (+) to blue (-)] were calculated automatically using Datacolor Quality software (Fig. 1). The L^* , a^* , and b^* values were used to calculate the color changes ΔL^* , Δa^* , and Δb^* . Finally, the total color change (ΔE^*) was calculated using Eq. (3). Before performing the color measurements, the device was calibrated to $a = -1.00 \pm 0.3$; $b = 0.58 \pm 0.3$; $L = 94.95 \pm 0.3$ [19, 24]:

$$\Delta E^* = \sqrt{(\Delta a^*)^2 + (\Delta b^*)^2 + (\Delta L^*)^2}, \tag{3}$$

where ΔL^* is $L_h^* - L_c^*$, Δa^* is $a_h^* - a_c^*$, Δb^* is $b_h^* - b_c^*$; c is the untreated control sample, and h is the heat-treated sample; where ΔL^* is $L_s^* - L_c^*$, Δa^* is $a_s^* - a_c^*$, Δb^* is $b_s^* - b_c^*$; c is the untreated control sample, and s is the bleached sample after the heat treatment process.

Evaluation of data

In the study, the Δa^* , Δb^* , ΔL^* and ΔE^* color change values of the untreated samples (controls) and heat-treated

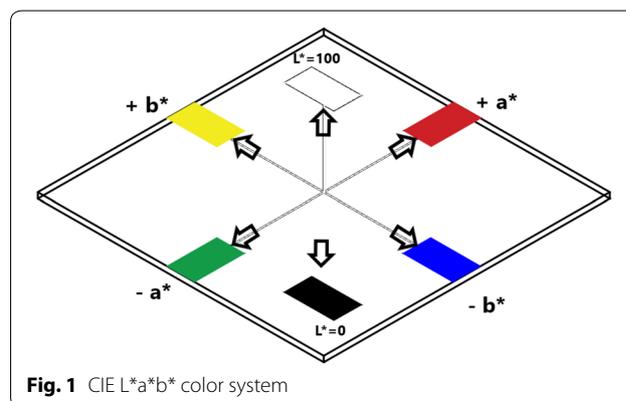


Fig. 1 CIE $L^*a^*b^*$ color system

Table 1 Solution groups used in bleaching process on the surfaces of wood

| Solution groups | Chemicals | pH level | Chemical amount to be applied (mL/m ²) | Neutralization agent |
|-----------------|--|----------|--|----------------------|
| S1 | NaOH + H ₂ O ₂ | 12.04 | 100 ± 10 | Distilled water |
| S2 | NaSiO ₃ + H ₂ O ₂ | 10.05 | | |
| S3 | H ₂ C ₂ O ₄ | 1.4 | | |

samples, were statistically compared with those of the samples bleached after heat treatment using the statistical package program. As a result of the analysis of variance (ANOVA) multiple-factor analysis, the bleaching effect of the wood species, the solution group, the heat treatment temperature, and the duration of heat treatment on the wooden material were determined. Comparisons were made via Duncan analysis and LSD (least significant difference).

Results and discussion

Color changes of Scots pine

Table 2 shows the Δa^* , Δb^* , ΔL^* , and ΔE^* color change results for Scots pine samples subjected to heat treatment and the heat-treated samples after bleaching treatment.

According to Table 2, the maximum difference in the red color value (Δa^*) was determined as 7.01 for the Scots pine samples heat-treated at 160 °C for 7 h and the minimum as 3.68 in those treated at 140 °C for 5 h (Fig. 2). When Table 2 is examined, the S1 and S2 bleaching solutions applied to the heat-treated Scots pine samples are seen to cause the red color value of the material to decrease and the results are close to the natural red color value of Scots pine. Within the scope of the study, the S3 solution caused the red color value of heat-treated Scots pine to increase more. It has been stated in the literature that heat treatment lowers the pH value of wood, leading to a more acidic structure and as a result, increasing the red color value of the material [25]. In this context, it was concluded that there was an inverse relationship between the pH of the wood material and the color darkening. The acidic structure of S3 used as a bleaching solution could be seen as the reason for the increase in the red color value.

The maximum difference in the yellow color value (Δb^*) was found as 8.20 in the samples heat-treated at 160° for 7 h and the minimum as 5.07 in the samples heat-treated at 160° for 3 h (Table 2). Tomak et al. [13] stated that the yellow color value of Scots pine increased after heat treatment. They emphasized that this could be caused by formation of secondary products in the wood material as a result of the heat treatment and/or decomposition due to the release of quinone and quinonemethide. All the bleaching solutions applied to the Scots pine surfaces after heat treatment resulted in a decrease in the yellow color value. However, the S2 and S3 solutions were more effective in decreasing the yellow color value than the S1 solution. Therefore, it was observed that application of the S2 and S3 solutions gave results closer to the natural yellow color value of the material (Fig. 3).

According to Table 2, the maximum difference in the lightness value (ΔL^*) in the Scots pine was obtained as -20.31 in the samples heat-treated at 160 °C for 7 h,

while the minimum was found as -9.22 in those treated at 140°C for 3 and 5 h. In the literature, it is reported that the lightness value of Scots pine decreased after heat treatment [14]. When Table 2 is examined, it can be seen that the lightness value of the Scots pine tended to decrease after application of the S1 and S3 bleaching solutions, while with the S2 solution application it increased.

The maximum total color change value (ΔE^*) of the Scots pine was determined as 23.01 in the samples heat-treated at 160 °C for 7 h, and the minimum as 12.46 at 140 °C for 5 h (Fig. 2). In the literature, it was stated that, as a result of heat treatment, redness, yellowness, and total color change values in Scots pine increased, while the lightness value was reduced [14]. It has been reported that higher drying temperatures lead not only to color changes and darkening of wood but also to an increase of resin flow, especially around knots in pine. We have observed the resin flow from the surfaces of pine after heat treatment processes [13]. According to Tarvainen et al. [26], color change in pine heartwood increases markedly at drying temperatures exceeding 70 °C, probably depending on the resin content. Pine sap also plays an important role in color change of pine wood surfaces after heat treatment. It is thought that these by-products formed in the material as a result of heat treatment interact with the lightening chemicals and affect the total color change values. In the study, the highest total color change value (21.70) was obtained in the heat-treated Scots pine samples bleached with the S3 solution, while the lowest value (8.03) was obtained in those bleached with the S2 solution after heat treatment at 140 °C for 5 h.

However, no significant difference was observed between samples that were bleached with the S2 solution after 5 h of heat treatment at 140 °C and those that were bleached with the S2 solution after heat treatment at 160 °C for 3 h or at 160 °C for 7 h ($p < 0.05$). In general, the S1 and S3 solutions applied to heat-treated Scots pine surfaces caused an increase in the total color change value while the S2 solution caused a decrease. This was attributed to the fact that the S2 solution had a low alkaline property.

Color changes of Uludağ fir

Table 3 shows the statistical comparisons of Δa^* , Δb^* , ΔL^* , and ΔE^* color change results for the fir specimens which were subjected to heat treatment and the heat-treated samples after bleaching treatment.

According to Table 3, the maximum difference in the red color value (Δa^*) in the fir was 2.80 for the samples exposed to heat treatment at 160 °C for 5 h, and the minimum as 0.20 in those treated at 140 °C for 3 h (Fig. 4). The S1 and S2 bleaching solutions applied to the fir surfaces

Table 2 Statistical comparison of color change values for Scots pine specimens subjected to heat treatment and heat-treated samples after bleaching treatment

| Temp (°C) | Duration (h) | Samples after heat treatment | | | Solution | | | Samples bleached after heat treatment | | | | | |
|-----------|--------------|------------------------------|---------------|----------------|---------------|----------------|-----------------|---------------------------------------|----------------|----------------|-----------------|-----------------|-----------------|
| | | Mean Δa* | Mean Δb* | Mean ΔE* | Mean Δa* | Mean Δb* | Mean ΔE* | Mean Δa* | Mean Δb* | Mean ΔE* | | | |
| 140 | 3 | 4.81 (0.38)j | 7.54 (0.76)mn | 14.51 (0.93)fg | S1 | 6.01 (0.75)hi | -13.84 (0.54)ef | 15.12 (0.47)g | 0.61 (0.15)de | 0.70 (1.38)ab | -9.41 (1.08)j | 9.56 (1.04)b | |
| | | | | | S2 | 3.07 (0.47)f | -14.93 (0.63)d | 17.08 (0.71)i | 7.70 (0.30)n | 5.10 (0.98)gh | -12.45 (1.22)g | 13.49 (1.32)e | |
| | | | | | S3 | 7.19 (1.30)jm | 12.46 (1.82)d | | 0.04 (0.38)c | 0.99 (1.61)b | -7.77 (0.96)k | 8.03 (1.12)g** | |
| | 5 | 3.68 (0.48)g | 7.19 (1.30)jm | 12.46 (1.82)d | S1 | 2.91 (1.00)ef | -14.00 (0.56)e | 16.03 (0.70)h | 7.18 (0.49)km | 5.80 (1.07)hi | -16.10 (0.64)bc | 17.17 (0.67)i | |
| | | | | | S2 | 7.37 (0.56)jmn | 13.29 (0.45)de | | 0.94 (0.35)e | 1.57 (1.42)bcd | -9.25 (0.93)j | 9.51 (0.93)b | |
| | | | | | S3 | 4.42 (0.22)h | 7.37 (0.56)jmn | 13.29 (0.45)de | 10.11 (0.50)ij | 7.76 (0.31)n | 3.36 (0.60)f | -15.02 (0.57)d | 17.23 (0.57)i |
| | 160 | 3 | 4.34 (0.37)h | 5.07 (0.89)gh | 13.39 (0.96)e | S1 | 4.55 (1.01)g | -13.05 (1.06)fg | 13.85 (1.25)ef | 0.43 (0.29)d | 4.55 (1.01)g | -13.05 (1.06)fg | 13.85 (1.25)ef |
| | | | | | | S2 | 6.46 (0.67)ij | 18.96 (0.91)j | | -1.13 (0.28)a | 1.37 (1.16)bc | -7.91 (0.55)k | 8.18 (0.60)a |
| | | | | | | S3 | 6.08 (0.32)j | 6.46 (0.67)ij | 18.96 (0.91)j | 16.74 (0.74)b | 7.05 (0.40)km | 2.43 (0.70)def | -15.42 (0.95)cd |
| | 5 | 7.01 (0.35)k | 8.20 (0.67)n | 23.01 (1.06)m | S1 | 1.58 (1.46)bcd | -16.83 (1.13)b | 18.42 (1.36)j | 0.67 (0.20)de | 4.50 (0.62)g | -16.15 (0.80)bc | 16.79 (0.82)hi | |
| | | | | | S2 | 8.20 (0.67)n | 20.31 (1.04)a | | -0.12 (0.35)c | -0.05 (0.53)a | -10.90 (0.99)hi | 10.92 (0.99)c | |
| | | | | | S3 | 4.81 (0.38)j | 7.54 (0.76)mn | 14.51 (0.93)fg | 9.22 (2.62)j | 7.18 (0.88)km | 1.58 (1.46)bcd | -16.83 (1.13)b | 18.42 (1.36)j |
| | 7 | 4.81 (0.38)j | 7.54 (0.76)mn | 14.51 (0.93)fg | S1 | 5.72 (1.15)hi | -20.72 (1.01)a | 21.59 (1.17)k | 1.77 (0.38)f | 5.72 (1.15)hi | -20.72 (1.01)a | 21.59 (1.17)k | |
| | | | | | S2 | 1.52 (1.63)bcd | -7.97 (0.75)k | 8.32 (0.83)g | | -1.00 (0.36)a | 1.52 (1.63)bcd | -7.97 (0.75)k | 8.32 (0.83)g |
| | | | | | S3 | 7.40 (0.27)mn | 2.04 (0.56)cde | 21.70 (0.61)k | | 7.40 (0.27)mn | 2.04 (0.56)cde | -20.28 (0.60)a | 21.70 (0.61)k |

Italic numbers indicate the highest and the lowest values of total color change (ΔE*)

** The values in parentheses are the standard deviations; different letters in the parameter of colour change column indicate statistical differences at the 95% confidence level

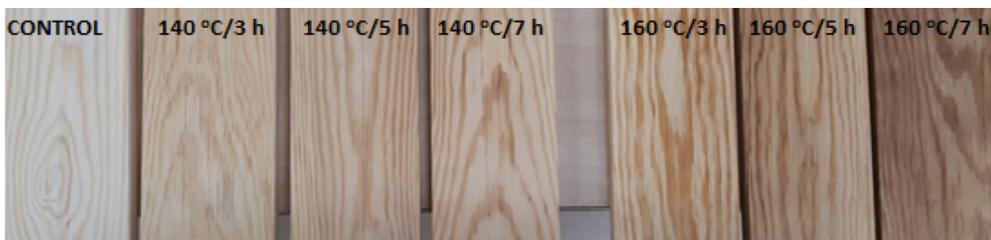


Fig. 2 Color change in Scots pine after heat treatment

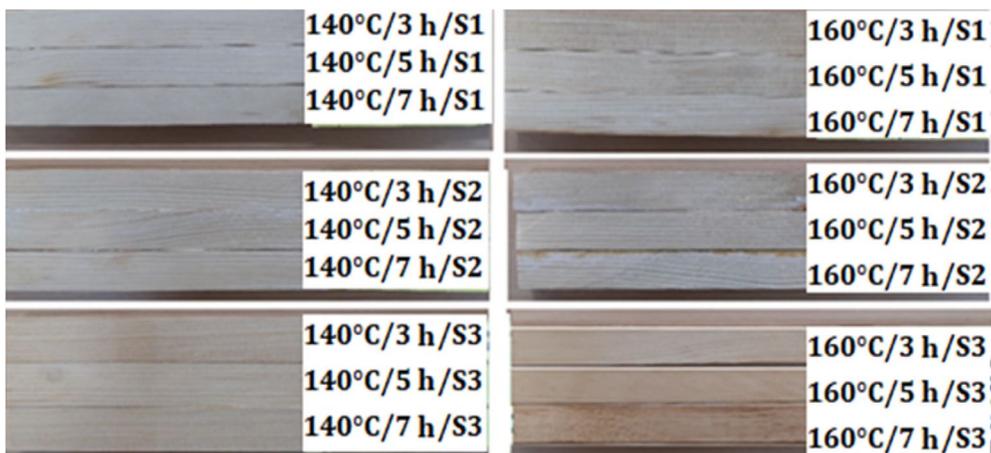


Fig. 3 Heat-treated Scots pine samples after bleaching treatment

after heat treatment had a negative effect on the red color value, while the S3 solution was found to affect it positively. The S1 and S2 solutions caused the heat-treated fir to have a more greenish appearance, while the S3 solution caused the fir to appear more reddish (Fig. 5), as in the Scots pine wood.

In Table 3, the maximum difference in the yellow color value (Δb^*) was determined as 6.9 in the samples heat-treated at 140 °C for 7 h, and the minimum as 1.05 in those treated at 160 °C for 3 h. In the fir samples, after the heat treatment, the S2 solution generally had a negative effect on the yellow color value, while the S1 and S3 solutions had a positive effect on the yellow color value. Thus, the S1 and S3 solutions caused the fir to appear more yellowish in color. The closest result to the natural yellow color value (-0.30) was obtained with the S2 solution on the fir surfaces heat-treated at 140 °C for 7 h.

According to Table 3, the maximum difference in the lightness value (ΔL^*) in the fir was obtained as -9.1 in the samples heat-treated at 160 °C for 5 h, and the minimum as -4.69 in those treated at 140 °C for 5 h. It was observed that the lightness value decreased in all heat treatment variations applied to the fir. The S1 and S3 bleaching

solutions applied after heat treatment generally reduced the lightness value, while the S2 solution increased it. Bleaching with the application of the S2 solution to the fir specimens subjected to heat treatment at 160 °C for 3 h gave the lightness value closest to the natural (untreated) samples.

The maximum total color change value (ΔE^*) in the fir wood was determined as 11.10 in the samples heat-treated at 160 °C for 5 h, and the minimum as 6.81 in those treated at 140 °C for 3 h (Fig. 4). In many studies, it is stated that as the heat treatment temperature increased, the color of the wood material darkened [5, 8, 14, 27–29]. The darkening of wood is one of the most visible effects of heat treatment. This effect is often explained as the result of the formation of colored degradation and oxidation products from hemicelluloses and extractives. The intensity of the discoloration depends on the severity of the treatment [30]. It is stated that heat treatment influences the surface color of different woods to varying extents. This phenomenon is probably related to the volatilization of color extracts as well as to the oxidation of some chemical constituents of wood, including lignin and polysaccharides [31].

Table 3 Statistical comparison of color change values for fir specimens subjected to heat treatment and heat-treated samples after bleaching treatment

| Temp (°C) | Duration (h) | Samples after heat treatment | | | Solution | | | Samples bleached after heat treatment | | |
|-----------|--------------|------------------------------|-------------------|-------------------|----------|--|----------------|---------------------------------------|-------------------|-------------------|
| | | Mean Δa^* | Mean Δb^* | Mean ΔE^* | | | | Mean Δa^* | Mean Δb^* | Mean ΔE^* |
| 140 | 3 | 0.20 (0.27)h | 1.93 (0.84)ef | 6.81 (0.88)hij | | | | 4.62 (1.42)ijm | – 7.18 (1.17)fgh | 8.99 (0.96)hi |
| | | | | – 6.48 (0.83)hij | | | | – 3.76 (1.03)a | – 1.70 (1.20)m | 5.09 (0.53)b |
| | | | | | | | | 5.45 (0.52)k | – 10.68 (0.85)b | 12.05 (0.94)m |
| | 5 | 1.73 (0.27)i | 5.49 (0.55)mnp | 7.44 (0.83)ef | | | | 5.41 (0.99)mnp | – 6.85 (0.88)ghi | 8.92 (0.92)hi |
| | | | | – 4.69 (0.72)k | | | | – 2.10 (0.96)b | – 1.66 (0.96)m | 3.85 (0.41)a |
| | | | | | | | | 7.69 (0.64)j | – 10.51 (0.92)b | 13.28 (1.12)n |
| 7 | 2.70 (0.20)j | 6.90 (0.43)r | 10.68 (0.66)j | | | | 3.41 (0.96)h | – 8.41 (0.81)de | 9.45 (0.76)i | |
| | | | – 7.68 (0.63)efg | | | | – 0.30 (1.10)c | – 4.72 (1.12)k | 5.77 (0.73)bc | |
| | | | | | | | 6.83 (0.39)m | – 12.52 (0.76)a | 14.45 (0.87)p | |
| 160 | 3 | 0.36 (0.32)h | 1.05 (0.68)d | 7.77 (0.80)fg | | | | 3.99 (1.08)hij | – 5.66 (0.90)j | 7.76 (0.74)fg |
| | | | | – 7.67 (0.71)efg | | | | 1.56 (1.71)de | – 1.63 (0.58)m | 5.17 (0.59)b |
| | | | | | | | | 0.67 (0.84)d | – 10.55 (1.08)b | 11.89 (1.22)km |
| | 5 | 2.80 (0.22)j | 5.69 (0.42)np | 11.10 (0.79)jk | | | | 5.84 (0.76)p | – 9.36 (0.75)c | 11.13 (0.88)jk |
| | | | | – 9.10 (0.71)cd | | | | 0.90 (1.04)d | – 5.60 (1.70)j | 6.51 (1.38)cd |
| | | | | | | | | 7.25 (0.43)n | – 11.90 (1.16)a | 14.33 (1.30)p |
| 7 | 1.65 (0.31)i | 3.86 (0.44)hi | 8.43 (0.83)gh | | | | 4.86 (0.70)jmn | – 6.18 (0.84)ij | 8.25 (0.76)fgh | |
| | | | – 7.30 (0.82)fgh | | | | – 0.59 (0.98)c | – 8.03 (1.17)ef | 8.42 (1.05)gh | |
| | | | | | | | 6.87 (0.35)m | – 10.38 (1.25)b | 12.71 (1.27)mn | |

Italic numbers indicate the highest and the lowest values of total color change (ΔE^)*

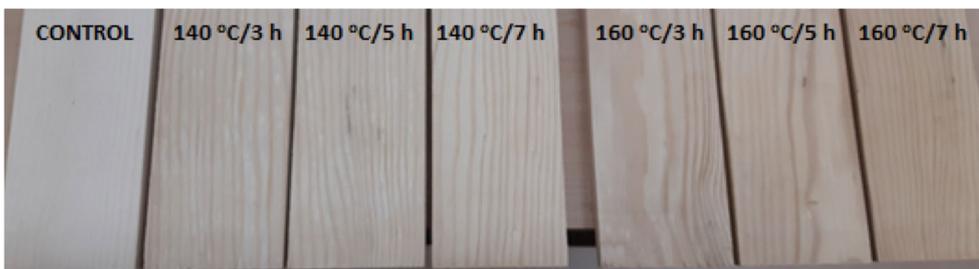


Fig. 4 Color change in fir after heat treatment



Fig. 5 Heat-treated fir samples after bleaching treatment

In this study, in the samples heat-treated and followed by the bleaching process with the S1 and S3 solutions, the total color change values were found to increase, while they decreased in those bleached with the S2 solution.

For the purpose of the study, the color value closest to the natural fir wood was obtained in samples heat-treated at 140 °C for 5 h followed by bleaching with the S2 solution. Bleaching chemicals act on the side compounds of the wood material to lighten the wood material [32, 33]. It is thought that applied chemical bleaching materials can cause color change in materials by affecting the acetic acid, free radicals and extractives released at the end of the heat treatment. In addition, the difference between the total color change values among the solutions and the different pH values of the solutions used may have been effective. In the literature, it was stated that as a result of the bleaching process using different solution groups, the Ca (OH)₂ + H₂O₂ solution increased the yellow color values in the samples and that this resulted from the pH of the bleaching solutions used [34].

Color changes of eastern beech

Table 4 shows the statistical comparisons of Δa^* , Δb^* , ΔL^* , and ΔE^* color change results for the beech specimens which were subjected to heat treatment and the heat-treated samples after bleaching treatment.

The maximum difference in the red color value (Δa^*) of the heat-treated beech was obtained as -1.05 at 160 °C for 3 h (Fig. 6). There was no statistically significant difference between heat treatment at 160 °C for 3 h or at 160 °C for 7 h ($p < 0.05$). The minimum difference in the red color value was found as -0.23 in the samples subjected to heat treatment at 140 °C for 5 h. In the literature it was emphasized that, although the natural state of beech material is reddish-white, after technical drying, the color changes to a brick-red color [35]. Depending on the heating application method, temperature, and duration, decreases in wood volume and mass are thought to occur as a result of water loss in the wood structure caused by increased fibrillation and the decreased presence of hydroxyl groups, material losses in the cell wall, and hemicellulose disintegration [3, 5]. The application of the S1 and S2 bleaching solutions

Table 4 Statistical comparison of color change values for beech specimens subjected to heat treatment and heat-treated samples after bleaching treatment

| Temp (°C) | Duration (h) | Samples after heat treatment | | | Samples bleached after heat treatment | | | | |
|-----------|--------------|------------------------------|----------------|----------------|---------------------------------------|-----------------|-----------------|-----------------|-----------------|
| | | Mean Δa* | Mean Δb* | Mean ΔE* | Mean Δa* | Mean Δb* | Mean ΔE* | | |
| 140 | 3 | -0.50 (0.63)gh | 0.52 (0.45)l | -3.72 (1.10)ef | 3.89 (1.00)a | -8.91 (1.45)c | 1.72 (2.52)j | 5.11 (1.41)hi | 10.77 (1.40)g |
| | | | | | | -10.82 (0.46)a | -7.47 (0.51)a | 10.78 (0.91)km | 17.02 (0.85)mn |
| | | | | | | 1.13 (0.88)jk | -1.15 (0.68)gh | -3.87 (1.22)ef | -3.87 (1.22)ef |
| 140 | 5 | -0.23 (0.66)ghi | 0.00 (0.40)l | -5.76 (0.97)cd | 5.82 (0.96)b | -6.32 (0.87)e | 5.14 (0.60)m | 4.12 (1.46)hi | 9.23 (1.12)ef |
| | | | | | | -10.04 (0.56)ab | -5.00 (0.81)c | 9.38 (1.28)k | 14.66 (1.24)ij |
| | | | | | | 1.13 (0.70)jk | -1.94 (0.66)efg | -5.05 (0.93)de | -5.05 (0.93)de |
| 140 | 7 | -0.73 (0.64)fgh | -0.39 (0.54)hi | -7.05 (1.43)c | 7.14 (1.43)c | -7.71 (1.21)d | 2.73 (1.18)k | 0.92 (2.12)g | 8.57 (0.99)de |
| | | | | | | -9.76 (0.45)b | -5.90 (1.18)b | 7.53 (0.94)j | 13.73 (0.72)l |
| | | | | | | 0.09 (0.72)hi | -2.74 (0.56)de | -6.11 (1.07)cd | -6.11 (1.07)cd |
| 160 | 3 | -1.05 (0.65)fg | -1.55 (0.47)fg | -6.08 (1.30)cd | 6.43 (1.18)bc | -6.65 (1.84)e | 4.86 (1.01)m | 4.72 (2.80)hi | 9.88 (2.00)fg |
| | | | | | | -10.03 (0.67)ab | -4.67 (2.06)c | 11.68 (1.74)m | 16.19 (2.00)km |
| | | | | | | 0.47 (0.49)ij | -2.46 (0.40)def | -3.51 (0.98)f | -3.51 (0.98)f |
| 160 | 5 | -0.54 (0.60)gh | -2.21 (0.52)ef | -6.48 (1.26)cd | 6.91 (1.25)bc | -6.89 (0.63)e | 4.55 (0.93)m | 4.19 (1.28)hi | 9.35 (1.07)ef |
| | | | | | | -10.13 (0.70)ab | -5.93 (0.99)b | 10.38 (2.31)km | 15.76 (1.88)jk |
| | | | | | | 1.53 (0.47)k | -2.30 (0.61)ef | -4.96 (1.22)def | -4.96 (1.22)def |
| 160 | 7 | -1.01 (0.63)fg | -3.33 (0.80)d | -17.65 (1.91)a | 18.01 (1.97)n | -6.37 (1.21)e | 0.41 (0.52)l | 3.94 (2.33)h | 7.66 (2.11)cd |
| | | | | | | -9.43 (0.67)bc | -4.83 (0.46)c | 5.50 (1.70)l | 12.01 (1.25)h |
| | | | | | | -1.43 (0.54)j | -4.63 (1.03)c | -13.22 (0.85)b | -13.22 (0.85)b |

Italic numbers indicate the highest and the lowest values of total color change (ΔE)*

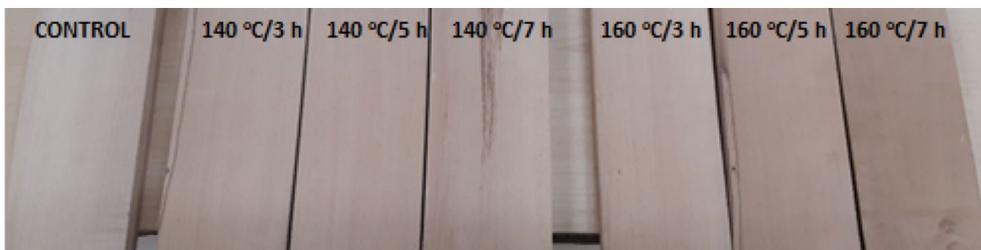


Fig. 6 Color change in beech after heat treatment

to the beech surfaces after heat treatment made them appear more greenish, while the S3 solution achieved values close to the natural red color tone (Fig. 7).

According to Table 4, the maximum difference in the yellow color value (Δb^*) in the beech was obtained as -3.33 in the samples heat-treated at 160 °C for 7 h. No difference (0.00) was detected among the heat-treated samples. In the beech samples subjected to heat treatment followed by bleaching, the S2 and S3 solutions caused a more bluish appearance by negatively affecting the yellow color value, whereas the S3 solution caused a more reddish appearance. It is stated in the literature that application of 5% H_2O_2 on birch veneer surfaces reduced the red color value of the material [17].

The maximum difference in the lightness value (ΔL^*) for the beech wood was determined as -17.65 in the samples heat-treated at 160 °C for 7 h, and the minimum as -3.72 in those treated at 140 °C for 3 h. The S1 and S2 solutions had a positive effect on the lightness value of the samples bleached after heat treatment, while the S3 solution had a negative effect.

According to Table 4, the maximum total color change value (ΔE^*) of the beech was obtained as 18.01 in the samples heat-treated at 160 °C for 7 h, and the minimum as 3.89 in those treated at 140 °C for 3 h (Fig. 6). In the study, as the heat treatment duration and temperature increased, the total color change value in the beech increased in parallel. It is stated that the sap and extractives of beech undergo a clearly visible color change after heat treatment, and lightness (L^*) decreases, saturation (C^*) increases, and the hue (h°) moves towards red. The color changes increase with time and temperature. This might be explained by the formation of colored degradation products deriving from the hydrolysis of hemicelluloses [13]. The S1 and S2 solutions increased the total color change value in all samples subjected to post-heat treatment bleaching except for the samples subjected to heat treatment at 160 °C for 7 h. The S3 solution resulted in a general decrease in the total color change value. The S1 and S2 bleaching solutions had an alkaline structure and this was thought to be an important factor in the increase of the total color change value. In the literature, the degradation of hemicellulose, lignin, and some



Fig. 7 Heat-treated beech samples after bleaching treatment

extractives in heat-treated wood is mentioned as the cause of color changes [36]. Substances such as quinone and quinone methide released in the wood material after the decomposition of these compounds interact according to the structure and pH values of the chemicals used and affect the total color change of the material. In a different study, birch veneer samples were treated with peracetic acid, hydrogen peroxide and sodium hypochlorite chemicals. According to the results, the peracetic acid did not cause any effect on the structure of the wood, whereas the alkaline hydrogen peroxide and sodium hypochlorite chemicals removed some of the lignin on the birch veneer surface [16].

Color changes of sessile oak

Table 5 shows the statistical comparisons of the Δa^* , Δb^* , ΔL^* , and ΔE^* color change results for the oak samples that were subjected to heat treatment and the heat-treated samples after bleaching treatment.

According to Table 5, the maximum difference in the red color value (Δa^*) for the oak was determined as 2.08 in the samples heat-treated at 160 °C for 7 h, and the minimum as 0.14 in those treated at 140 °C for 5 h (Fig. 8). The S1 and S2 bleaching solutions applied to the oak surfaces after heat treatment reduced the red color value negatively and created a more greenish appearance (Fig. 9). On the other hand, as in the pine and fir, the S3 solution increased the difference in the red color value for the oak.

In the oak, the maximum difference in the yellow color value (Δb^*) was found as 2.36 in the samples heat-treated at a 140 °C for 7 h. There was no difference in the samples heat-treated at 160 °C for 3 h when compared to the natural (untreated) samples. The S1 bleaching solution applied to the oak surfaces after heat treatment had a positive effect on the yellow color value difference, while the S2 and S3 solutions had a negative effect. In the study, the result closest to the yellow color value of the oak material was obtained from the samples bleached with the S3 solution (Fig. 9).

When Table 5 is examined, the maximum difference in the lightness value (ΔL^*) for the oak is found as -18.55 in the samples that were heat-treated at 160 °C for 7 h, while the minimum difference can be seen as -2.76 in the samples exposed to heat treatment at 140 °C for 5 h. In general, the results closest to the lightness value of natural (untreated) oak were recorded in the samples treated with the S1 and S2 solutions. The S3 solution negatively affected the lightness value.

The maximum total color change value (ΔE^*) of the oak wood was recorded as 18.72 in the samples heat-treated at 160 °C for 7 h, and the minimum as 8.03 in those treated at 140 °C for 3 h. In the study, it was determined

that the increase in the total color change value was related to the increase of heat treatment temperature and duration. The specimens became darker with increasing treatment time and temperature, as reported by several authors [37, 38]. Esteves et al. [39] reported that noticeable color changes could already be obtained for small mass losses of 2–4%, but the effect depended on the extent of treatment and was related to the chemical composition of the heat-treated woods. The darker tonality of heat-treated wood is often attributed to the formation of colored degradation products from hemicelluloses and to extractives that seem to participate in the color formation of heat-treated wood [40]. It is reported that FTIR analysis showed degradation of cell wall polymers resulting in the generation of structures which are responsible for the color darkening of thermally modified wood [31]. The highest total color change value (13.42) was obtained in the samples treated with the S2 solution following heat treatment at 140 °C for 5 h, while the lowest value (6.67) was in samples treated with the S1 solution after heat treatment at 160 °C for 5 h. In general, it was found that all the bleaching solutions applied to the surface of the heat-treated oak wood decreased the total color change value of the material. Although the arithmetic average of the total color change values for the heat-treated oak wood was 10.4, as a result of the application of the S1 solution it fell to 7.46, with the S2 solution to 9.95, and with the S3 solution to 8.32.

Conclusions

In this study, the aim was to use S1, S2, and S3 bleaching solutions to remove the negative color changes occurring in the Scots pine, fir, beech and oak woods from the effect of heat treatment. The goal was to achieve values close to the natural colors of the wood materials used in the experiments.

- The greatest red color change in the study occurred in the Scots pine. The red color change increased positively in the fir, Scots pine, and oak, but negatively in the beech.
- After heat treatment, the yellow color change was found to be greater in the coniferous pine and fir wood than in the deciduous oak and beech.
- As a result of the heat treatment, the total color change value occurred in the pine, oak, fir, and beech, respectively.
- After the heat treatment, the alkaline bleaching solutions applied to the wood had a positive effect on the recovery of the natural color of the material. However, it was observed that the different color tones could have been formed due to the heterogeneous

Table 5 Statistical comparison of color change values for oak specimens subjected to heat treatment and heat-treated samples after bleaching treatment

| Temp (°C) | Duration (h) | Samples after heat treatment | | | Solution | | | Samples bleached after heat treatment | | |
|-----------|--------------|------------------------------|-------------------|-------------------|----------|-------------------|-------------------|---------------------------------------|------------------|--|
| | | Mean Δa^* | Mean Δb^* | Mean ΔE^* | Solution | Mean Δa^* | Mean Δb^* | Mean ΔE^* | | |
| 140 | 3 | 0.98 (0.54)h | 1.58 (0.68)ij | 8.03 (1.06)cdef | S1 | -6.00 (0.43)c | 3.63 (0.51)n | 4.95 (0.91)hi | 8.62 (0.73)figh | |
| | | | | | S2 | -6.59 (0.74)b | -6.90 (0.47)a | 5.99 (2.38)l | 11.43 (1.54)k | |
| | | | | | S3 | 2.30 (0.34)kmn | -0.49 (0.70)ef | -7.78 (1.14)d | 7.94 (0.85)cdef | |
| | 5 | 0.14 (0.41)g | 1.22 (0.56)hi | 8.14 (0.76)defg | S1 | -6.57 (0.46)b | 2.02 (1.30)jm | 5.94 (1.61)l | 9.22 (1.31)ghij | |
| | | | | | S2 | -7.22 (0.41)a | -6.26 (1.11)ab | 9.30 (1.52)j | 13.42 (1.11)m | |
| | | | | | S3 | 2.55 (0.43)mn | -0.64 (0.88)ef | -3.86 (1.00)ef | 4.78 (0.87)a | |
| | 7 | 1.37 (0.27)hi | 2.36 (0.60)m | 8.19 (0.70)efg | S1 | -5.07 (0.36)d | 3.16 (0.51)n | 0.86 (1.67)g | 6.25 (0.48)b | |
| | | | | | S2 | -6.07 (0.61)c | -6.00 (0.60)b | 5.28 (2.60)hi | 10.25 (1.61)j | |
| | | | | | S3 | 2.63 (0.49)n | -0.60 (0.62)ef | -7.44 (0.94)d | 7.94 (0.97)cdef | |
| 160 | 3 | 0.98 (0.48)h | 0.00 (0.38)fg | 9.45 (1.34)hij | S1 | -5.72 (0.33)c | 3.01 (1.14)n | 1.71 (1.40)g | 6.89 (0.61)bc | |
| | | | | | S2 | -5.81 (0.69)c | -5.67 (0.66)b | 3.76 (1.64)h | 9.07 (1.05)fighi | |
| | | | | | S3 | 1.55 (0.47)ij | -1.76 (0.60)d | -6.59 (0.96)d | 7.03 (0.97)bcd | |
| | 5 | 1.19 (0.41)hi | 0.64 (0.72)gf | 9.87 (1.41)ij | S1 | -4.58 (0.34)e | 3.25 (0.67)n | -3.21 (1.71)ef | 6.67 (0.67)b | |
| | | | | | S2 | -5.64 (0.62)c | -3.19 (0.94)c | 0.82 (3.21)g | 7.17 (1.31)bcde | |
| | | | | | S3 | 1.90 (0.40)jk | -1.67 (0.58)d | -7.52 (1.57)d | 7.96 (1.57)cdef | |
| | 7 | 2.08 (0.47)km | -1.08 (0.64)de | 18.72 (0.80)n | S1 | -4.01 (0.63)f | 3.24 (0.77)n | -4.54 (2.01)e | 7.13 (0.94)bcde | |
| | | | | | S2 | -5.89 (0.61)c | -3.65 (0.67)c | 4.11 (2.84)h | 8.38 (1.72)figh | |
| | | | | | S3 | 1.09 (0.89)hi | -3.42 (0.75)c | -13.69 (1.05)b | 14.19 (1.06)m | |

Italic numbers indicate the highest and the lowest values of total color change (ΔE^)*

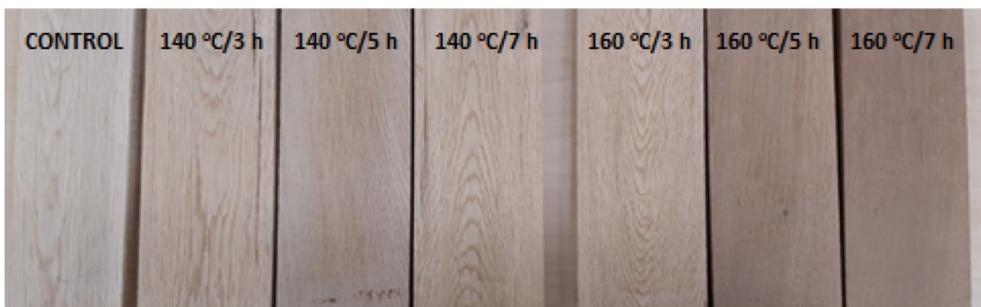


Fig. 8 Color change in oak after heat treatment



Fig. 9 Heat-treated oak samples after bleaching treatment

structure of the wood and the compounds found within it.

- The average total color change value of the Scots pine after heat treatment was determined as 15.93. As a result of the application of the S1 solution after heat treatment, the average total color change value of the Scots pine was 16.33, with the S2 solution 9.29, and with the S3 solution 17.93. Application of the S2 solution can be recommended to restore the natural color in heat-treated Scots pine wood.
- The average total color change in the fir after heat treatment was 8.7. As a result of the application of the S1 solution after heat treatment, the average total color change value in the fir was 9.08, with the S2 solution 5.8, and with the S3 solution 13.11. Application of the S2 solution can be recommended to restore the natural color in heat-treated fir wood.
- The average total color change value of the beech after heat treatment was 8.03. As a result of the application of the S1 solution after heat treatment,

the average total color change value of the beech was 9.24, with the S2 solution 14.89, and with the S3 solution 6.8. Application of the S3 solution can be recommended to restore the natural color in heat-treated beech wood.

- The average total color change value of oak after heat treatment was 10.4. As a result of the application of the S1 solution after heat treatment, the average total color change value in the oak was 7.46, with the S2 solution 9.95, and with the S3 solution 8.32. Application of the S1 and S3 solutions can be recommended to restore the natural color in heat-treated oak wood.

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Authors' contributions

MB designed and performed the experiments and analyzed the data. The heat treatment and bleaching process of wood materials carried out by MA. MA

wrote the manuscript in consultation with MB. All authors read and approved the final manuscript.

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Competing interest

The authors declare that they have no competing interests.

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