

## COLOUR AND GLOSS CHANGES OF SCOTS PINE AFTER HEAT MODIFICATION

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### ABSTRACT

Heat treatment of Scots pine (*Pinus sylvestris* L.) wood was carried out by hot air in an oven for 2, 4, and 8 h at 150, 175, and 200°C. After heat treatment, colour and gloss changes of wood specimens were measured. The colour parameters  $a^*$ ,  $b^*$ , and  $L^*$  were determined by the CIELAB method. Also, gloss values of wood specimens after heat treatments were determined according to ASTM D 523.

Results showed that oven heat-treated wood became darker tonality. While,  $a^*$  coordinate (red component) increased as temperature increases,  $b^*$  coordinate (yellow component) initially increased and then decreased at temperatures tested, 175°C and 200°C. Heat treatment caused decrease in gloss values of Scots pine.

KEYWORDS: Heat treatment, colour, gloss, Scots pine (*Pinus sylvestris* L.).

### INTRODUCTION

Among the construction materials which are used by people wood holds a special place because of its impressive range of attractive properties, including low thermal extension, low density and high enough mechanical strength (Bekhta and Niemz 2003). But, it has less desirable properties such as poor durability and poor dimensional stability. This problem can be reduced by using chemical treatments. Increased environmental awareness has raised the demand for more environmentally friendly methods. Heat treatment is an alternative method for improving these properties with no use of chemical additives (Johansson 2008). Wood is a complex polymeric material constituted mainly of cellulose, hemicelluloses and lignin, with a minor proportion of extractives. The exposure of wood to elevated temperatures causes thermal degradation of its structure, i.e., changes in composition, often accompanied by loss of mass, and thus the properties of wood are somewhat modified (Borrega and Kärenlampi 2008). Heat treatment of wood changes its chemical composition by degrading both cell wall compounds and extractive (Esteves et al.

2008). The main purpose of heat treatment is to achieve new material properties, rather than dry wood, material properties such as increased biological durability controllable colour changes. The disadvantages that have to be dealt with are reductions in various kinds of mechanical strength such as increased brittleness and decreased bending and tension strength (Sehlstedt-Persson et al. 2006). With heat treatment, the colour of wood is modified acquiring a darker tonality which is often justified by the formation of colour degradation produced from hemicelluloses (Sehlstedt-Persson 2003, Sundqvist 2004). Viitaniemi et al. (1997) noted that good correlations between lightness ( $L^*$ ) and treatment temperature for *Pinus sylvestris*. Schnabel et al. (2007) reported that with regard to all three colour parameters ( $L^*$ ,  $a^*$ , and  $b^*$ ), the colour change increased when higher temperatures in the modification process were set. Ahajji et al. (2003) focused on colour modification of torrefied wood of three species; beech, spruce and eucalyptus, and showed that torrefaction strongly affects wood colour with a large decrease of lightness for the three species. Schneider (1973) noted that both the temperature and the processing time have an effect on the wood colour.

In this study, it was aimed to determine colour and gloss changes of Scots pine after heat modification.

## MATERIAL AND METHODS

### Preparation of test specimens and chemicals

Wood specimens measuring 6 x 75 x 150 mm (radial by tangential by longitudinal) were prepared from air-dried sapwood of Scots pine (*Pinus sylvestris* L.). Wood specimens were oven dried at  $103 \pm 2^\circ\text{C}$  before and after treatment.

### Colour changes

The colour parameters  $a^*$ ,  $b^*$ , and  $L^*$  were determined by the CIELAB method. The  $L^*$  axis represents the lightness, whereas  $a^*$  and  $b^*$  are the chromaticity coordinates. The  $+a^*$  and  $-a^*$  parameters represent red and green, respectively. The  $+b^*$  parameter represents yellow, whereas  $-b^*$  represents blue.  $L^*$  can vary from 100 (white) to zero (black) (Zhang 2003). The colours of the specimens were measured by a colourmeter (X-Rite SP Series Spectrophotometer) before and after heat treatment. The measuring spot was adjusted to be equal or not more than one-third of the distance from the center of this area to the receptor field stops. The colour difference, ( $\Delta E^*$ ) was determined for each wood as follows (ASTM D 1536–58 T 1964):

$$\Delta a^* = a_f^* - a_i^* \quad (1)$$

$$\Delta b^* = b_f^* - b_i^* \quad (2)$$

$$\Delta L^* = L_f^* - L_i^* \quad (3)$$

$$(\Delta E^*) = [(\Delta a)_2 + (\Delta b)_2 + (\Delta L^*)^2]^{1/2} \quad (4)$$

where:  $\Delta a^*$ ,  $\Delta b^*$ , and  $\Delta L^*$  are the changes between the initial and final interval values.

### Gloss test

The gloss values of wood specimens were determined according to ASTM D 523 (1970) with a measuring device (Micro-TRI-Gloss). The chosen geometry was an incidence angle of  $60^\circ$ . Results were based on a specular gloss value of 100, which relates to the perfect condition under identical illuminating and viewing conditions of a highly polished, plane, black glass surface.

## Heat modification

Heat treatment was performed using a temperature-controlled laboratory oven. Three different temperatures (150°C, 175°C, and 200°C) and three treatment durations (2, 4, and 8 h) were applied to wood specimens under atmospheric pressure and in the presence of air.

## RESULTS AND DISCUSSION

Tab. 1 shows the changes of  $\Delta a^*$ ,  $\Delta b^*$ ,  $\Delta L^*$ , and  $\Delta E^*$  according to the treatment duration and temperature of treatment. The  $L^*$  values of Scots pine are given in Tab. 1 and Fig. 1. The decrease in  $L^*$  at all temperatures indicates that the specimens became darker with the treatment time. Earlier researchs showed similiar results (Matsuo et al. 2010, Esteves et al. 2008, Ahajji et al. 2009). The darkening of heat-treated Scots pine might be due to degradation of lignin and other non-cellulosic polysaccharides (Hon and Chang 1985, Grelier et al. 2000, Petric et al. 2004). Darkening with the heat treatment was clearly visible, increasing with treatment duration and temperature and this is consistent with earlier findings (Mitsui et al. 2003, Miltz 2002, Esteves et al. 2008). Lightness of heat-treated Scots pine wood decreased with treatment duration and temperature. For instance, lightness of Scots pine decreased 3.30 %, 16.61 %, and 24.05 %, within 2 h of treatment at 150°C, 175°C, and 200°C, respectively. The maximum reduction (41.28 %) was obtained within 8 h at 200°C. Esteves et al. (2008) reported a 52.9 % decrease in lightness of pine wood for a treatment at 200°C for 12 h. Another study, Bekhta and Niemz (2003) reported a 73 % decrease in lightness of spruce wood for a treatment at 200°C for 24 h. The  $a^*$  values of Scots pine are given in Tab. 1 and Fig. 2. Positive values of  $\Delta a^*$  indicate a tendency of wood surface to become reddish. The  $\Delta a^*$  of heat-treated Scots pine increased with treatment duration and temperature, it became more reddish. The increase of the chromaticity coordinate ( $\Delta a^*$ ) may be explained by the modification of some chromophoric groups of lignin (Grelier et al. 2000). For heat-treated Scots pine,  $\Delta a^*$  varied between 2.06 % and 50.64 %. Increasing of  $\Delta a^*$  for the less intensive 2 h treatment for Scots pine was 2.06 % at 150°C. With increasing treatment duration and temperature, increasing of  $\Delta a^*$  reached 50.64 % for 8 h at 200°C. The  $b^*$  values of Scots pine are given in Tab. 1 and Fig. 3. Positive values of  $\Delta b^*$  indicate a tendency of wood surface to yellowing while negative values mean a tendency to bluing. Colour of wood was made more in the tone of yellow at 150°C, and becomes blue at temperature 175°C and 200°C. The initial increase and following decrease of  $\Delta b^*$  indicate that the specimens initially became more yellow and then changed to blue. Bekhta and Niemz (2003) reported that the  $b^*$  value of spruce wood specimens increased with increase in treatment temperature up to 150°C (reaches a maximum value at this temperature), and afterwards began to decrease. Johansson and Morén (2006) investigated the effects of thermal treatment of birch with respect to colour. They found that the yellow  $b^*$  coordinate was fairly constant for the different times at 175°C. When treated at 200°C, the  $b^*$  decreased over time. Also, Esteves et al. (2008) reported similar results with pine and eucalypt wood. Our results are consistent with these researcher's findings. The total colour changes ( $\Delta E^*$ ) of heated Scots pine are given in Tab. 1. The total colour change ( $\Delta E^*$ ) of heated Scots pine was changed from 3.0 to 31.8. Our results showed that  $\Delta E^*$  were remarkably increased after heat treatment at 200°C. Also, it was found that the total colour change of heat-treated Scots pine wood increased with treatment duration and temperature. Gloss values of Scots pine before and after heat treatment were given in Tab. 2 and Fig. 4.

Tab. 1: Colour changes of Scots pine after heat modification<sup>a</sup>.

°C	Hours	Before exposure			After exposure			After exposure				Change (%)		
		L <sub>i</sub> <sup>*</sup>	a <sub>i</sub> <sup>*</sup>	b <sub>i</sub> <sup>*</sup>	L <sub>f</sub> <sup>*</sup>	a <sub>f</sub> <sup>*</sup>	b <sub>f</sub> <sup>*</sup>	ΔL <sup>*</sup>	Δa <sup>*</sup>	Δb <sup>*</sup>	ΔE <sup>*</sup>	ΔL <sup>*</sup>	Δa <sup>*</sup>	Δb <sup>*</sup>
150	2	72.7	9.7	31.9	70.30	9.9	33.90	-2.4	0.2	2.0	3.0	-3.30	2.06	6.26
	4	73.4	8.9	31.2	69.60	10.5	31.60	-3.8	1.6	0.4	4.3	-5.17	17.97	1.28
	8	74.3	7.3	27.8	69.40	8.9	29.00	-4.9	1.6	1.2	7.3	-6.59	21.91	4.31
175	2	71.0	9.1	29.3	59.20	10.7	28.50	-11.8	1.6	-0.8	12.2	-16.61	17.58	-2.73
	4	72.2	9.9	31.5	58.40	12.1	29.60	-13.8	2.2	-1.9	14.1	-19.11	22.22	-6.03
	8	74.2	7.9	29.0	59.90	11.0	28.80	-14.3	3.1	-0.2	15.6	-19.27	39.24	-0.68
200	2	74.0	8.1	29.5	56.20	10.6	29.00	-17.8	2.5	-0.5	18.8	-24.05	30.86	-1.69
	4	72.6	9.8	31.8	50.90	12.4	25.00	-21.7	2.6	-6.8	22.5	-29.88	26.53	-21.38
	8	74.6	7.7	28.0	43.80	11.6	22.10	-30.8	3.9	-5.9	31.8	-41.28	50.64	-21.07

<sup>a</sup> Five replication were made for each group

Tab. 2: Gloss values of Scots pine after heat modification<sup>a</sup>.

°C	Hours	Before exposure	After exposure	Change (%)
150	2	3.6 (0.33) <sup>b</sup>	3.4 (0.38)	-5.5
	4	4.2 (1.03)	3.8 (0.96)	-9.5
	8	4.3 (0.79)	3.9 (0.67)	-9.3
175	2	4.0 (0.51)	3.4 (0.35)	-15.0
	4	3.9 (0.25)	3.2 (0.26)	-17.9
	8	4.2 (0.57)	3.2 (0.55)	-23.8
200	2	4.5 (0.80)	3.5 (0.70)	-22.2
	4	4.1 (0.48)	2.6 (0.49)	-36.6
	8	4.4 (1.66)	3.0 (0.95)	-31.8

<sup>a</sup> Five replication were made for each group

<sup>b</sup> Values in parenthesis are standard deviations.

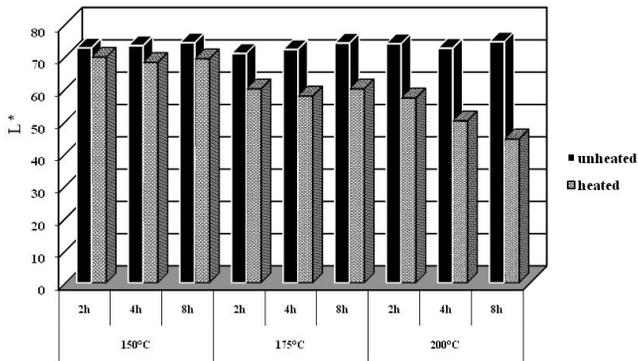


Fig. 1:  $L^*$  values of Scots pine before and after heat treatment.

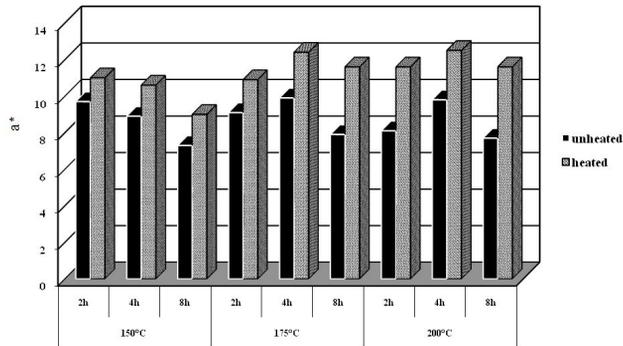


Fig. 2:  $a^*$  values of Scots pine before and after heat treatment.

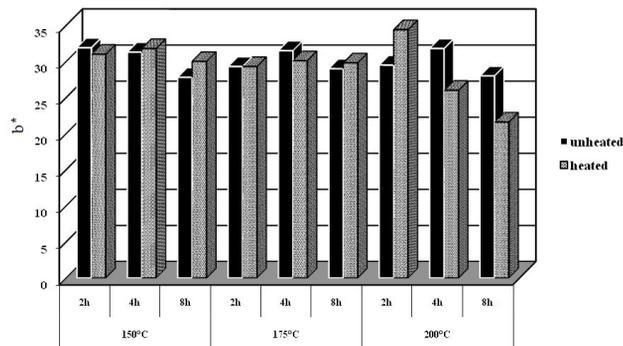


Fig. 3:  $b^*$  values of Scots pine before and after heat treatment.

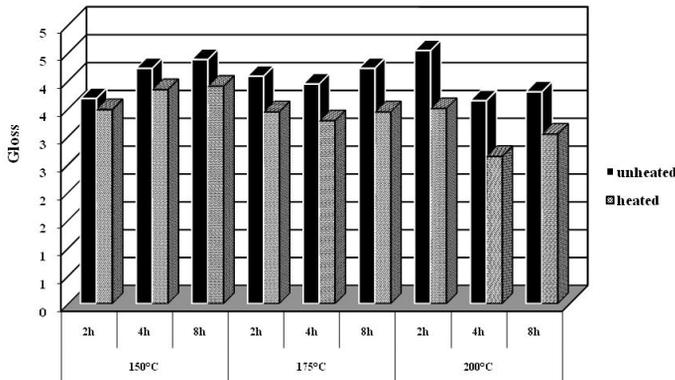


Fig. 4: Gloss values of Scots pine before and after heat treatment.

Heat treatments decreased gloss values of the Scots pine specimens to some extent. The gloss values of Scots pine decreased 5.5-36.6 % after heat treatments. In general, gloss values of heat-treated Scots pine specimens decreased with increasing treatment duration and temperature.

## CONCLUSION

Colour and gloss changes of Scots pine wood heated at 150, 175, and 200°C for 2, 4, 8 h were studied. For all samples, heat treatment induces a strong decrease of luminance (darkening). These variations are all more pronounced at higher treatment temperatures. While  $a^*$  parameter increased with the increase in treatment severity, the  $b^*$  increased initially and then decreased at temperatures tested, 175°C and 200°C. Heat treatments decreased gloss values of Scots pine in some extent. According to our results, with increasing treatment duration and temperature, the  $\Delta E^*$  of Scots pine increased.

## REFERENCES

1. Ahajji, A., Elbakali, I., George, B., Merlin, A., 2003: Analysis by electron paramagnetic resonance spectroscopy (EPR) of heat-treated wood exposed to solar-type radiation. 6<sup>th</sup> Scientific Meeting of the Forest and Wood - Epinal. Annals of wood GIS. (Analyse par spectroscopie de résonance paramagnétique électronique (RPE) de bois traités thermiquement exposés à un rayonnement de type solaire. 6<sup>èmes</sup> Journées Scientifiques de la Forêt et du Bois – Epinal. Annales du GIS bois/ environnement). Pp 175-182. (in French).
2. Ahajji, A., Diouf, P.N., Aloui, F., Elbakali, I., Perrin, D., Merlin, A., George, B., 2009: Influence of heat treatment on antioxidant properties and colour stability of beech and spruce wood and their extractives. Wood Sci. Technol. 43(1-2): 69-83.
3. ASTM D 1536-58, 1964: Tentative method of test color difference using the colormaster differential colourimeter.

4. ASTM-D 523, 1970: Standard method of test for specular gloss.
5. Bekhta, P., Niemz, P., 2003: Effect of high temperature on the change in colour, dimensional stability and mechanical properties of spruce. *Holzforschung* 57(5): 539-546.
6. Borrega, M., Kärenlampi, P.P., 2008: Mechanical behavior of heat-treated spruce (*Picea abies*) wood at constant moisture content and ambient humidity. (Mechanisches Verhalten von wärmebehandeltem Fichtenholz (*Picea abies*) bei konstanter Holz- und Luftfeuchte). *Holz als Roh- und Werkstoff* 66(1): 63-69. (in German).
7. Esteves, B., Marques, A.V., Domingos, I., Pereira, H., 2008: Heat-induced colour change of pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood. *Wood Sci. Technol.* 42(5): 369-384.
8. Grelier, S., Castellan, A., Kamdem, D.P., 2000: Photo-protection of copper amine treated wood. *Wood and Fiber Science* 32(2): 196-202.
9. Hon, D.N.S., Chang, S.T., 1985: Photoprotection of wood surfaces by wood-ion complexes. *Wood and Fiber Science* 17(1): 92-100.
10. Johansson, D., Morén T., 2006: The potential of colour measurement for strength prediction of thermally treated wood. *Holz als Roh- und Werkstoff* 64(2): 104-110.
11. Johansson, D., 2008: Heat treatment of solid wood: Effects on absorption, strength and colour. Doctoral Thesis. Luleå University of Technology LTU Skelleftea. Division of Wood Physics, 142 pp.
12. Matsuo, M., Yokoyama, M., Umemura, K., Gril, J., Yano, K., Kawai, S., 2010: Color changes in wood during heating: Kinetic analysis by applying a time-temperature superposition method. *Applied Physics A* 99(1): 47-52.
13. Militz, H., 2002: Thermal treatment of wood: European Processes and their background. In: International Research Group on Wood Preservation. Section 4-Processes, No. IRG/WP 02-40241.
14. Mitsui, K., Murata, A., Kohara, M., Tsuchikawa, S., 2003: Colour modification of wood by light-irradiation and heat treatment. In: Abstracts of the First European Conference on Wood Modification, Belgium.
15. Petric, M., Kricej, B., Humar, H., Pavlic, M., Tomazic, M., 2004: Patination of cherry wood and spruce wood with ethanolamine and surface finishes. *Surface Coat International, Part B, Coat Trans.* 87(B3): 95-201.
16. Schnabel, T., Zimmer, B., Petutschnigg, A.J., Schönberger, S., 2007: An approach to classify thermally modified hardwoods by color. *Forest Products Journal* 57(9): 105-110.
17. Schneider, A., 1973: Investigations on the convection drying of lumber at extremely high temperatures. Part II: Drying degrade, changes in sorption, colour and strength of pine sapwood and beechwood at drying temperatures from 110 to 180°C. (Zur Konvektionstrocknung von Schnittholz bei extrem hohen Temperaturen. 2. Mitt. Trocknungsschäden, Sorptions-, Farb- und Festigkeitsänderungen von Kiefern-, Splint- und Buchenholz bei Trocknungstemperaturen von 110 bis 180°C). *Holz als Roh- und Werkstoff* 31(5): 198-206. (in German).
18. Sehlstedt-Persson, M., 2003: Colour responses to heat treatment of extractives and sap from pine and spruce. In: 8<sup>th</sup> International IUFRO Wood Drying Conference. Brasov Romania. Pp 459-464.
19. Sehlstedt-Persson, M.S., Johansson, D., Morén, T., 2006: Effect of heat treatment on the microstructure of pine spruce and birch and the influence on capillary absorption. In: Proceedings of the 5<sup>th</sup> IUFRO Symposium "Wood Structure and Properties '06", Slovakia, Zvolen. Pp 251-255.

20. Sundqvist, B., 2004: Colour changes and acid formation in wood during heating. Doctoral Thesis, Lulea University of Technology, Sweden.
21. Viitaniemi, P., Jämsä, S., Viitanen, H., 1997: Method for improving biodegradation resistance and dimensional stability of cellulosic products. United States Patent No. 5678324 (US005678324).
22. Zhang, X., 2003: Photo-resistance of alkylammonium compound treated wood. MS.c. Thesis. The University of British Columbia Vancouver, Canada.

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