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COLOUR MODIFICATION OF *ROBINIA PSEUDOACACIA* L. DURING THE PROCESSES OF HEAT TREATMENT WITH SATURATED WATER STEAM

Ladislav Dzurenda

ABSTRACT

The results of the heat treatment process with saturated water steam of black locust wood in terms of the effect of the saturated water steam temperature and the exposure time on the colour modification of woodturning blanks of black locust with dimensions of 30 × 50 × 500 mm and on the heat consumption are presented in the paper. The colour of woodturning blanks of black locust was modified by modes at the temperatures: $t_I = 112.5 \pm 2.5\ ^{O}C$ for τ = 55 hours (mode I), $t_{II} = 127.5 \pm 2{,}5\ ^{O}C$ for τ = 6.5 hours (mode II) and $t_{III} = 137.5 \pm 2.5\ ^{O}C$ for τ = 7.5 hours. (mode III). The colour of wood was determined using Color Reader CR-10 and described with the coordinates of CIE-L*a*b*colour space. There is only a minimal change in the colour of black locust wood during the heat treatment by mode I, the wood becomes slightly brown. Brown-gray colour is achieved by mode II. Black locust wood thermally modified by mode III gets an original dark brown-gray colour. When the temperature of saturated water steam during the heat treatment process is increased and the exposure time of the colour modification of black locust wood is prolonged, the lightness of the hue is affected, wood darkens. Specific hues are achieved in the case of increasing values of red coordinate a* and decreasing values of yellow coordinate b*. Heat consumption depending on the temperature of saturated water steam and exposure time of colour modification of black locust woodturning blanks ranges from Q_{TZN} = 111.29 – 139.13 kWh/m^3. Irreversible changes in the colour of black locust wood achieved during some of modes of colour modification with saturated water steam widen the possibility for its use in the field of construction and carpentry, design as well as in the field of art.

Key words: wood, black locust, colour, heat treatment, saturated water steam.

INTRODUCTION

The colour of wood is an essential optical property and a typical feature of the sapwood or heartwood of individual wood species. Wide range of colours of native wood of commercially important wood species is used as a material in cabinet making and furniture manufacturing industry: from light white-gray-yellow colour of the wood species of Norway spruce (*Picea excelsa*), Silver fir (*Abies alba* L.), Small-leaved linden (*Tilia cordata*), European hornbeam (*Carpinus betulus* L.), through red-brown colour of heartwood of the wood species of English oak (*Quercus robur*), European ash (*Fraxinus excelsior* L.), Persian walnut (*Juglans regia*).

Using the coordinates of CIE-L*a*b*colour space is one of the ways to quantify the given optical wood property objectively. Lab colour space (according to CIE – Commission Internationale de Eclairage) in accordance with ISO 7724 is based on the measurement of three parameters: lightness L* represents the darkest black at L* = 0 and the brightest white at L* = 100. The value of a* is a measure of the red–green character of the colour, with positive values for red shades and negative values for green. The value of b* gives the yellow–blue character with positive values for yellow shades and negative for blues.

Heat treatment processes of wood, in addition to specific physico-mechanical and chemical changes of wood, are accompanied by a colour change as well KOLLMANN – GOTE (1968), SERGOVSKY – RASEV (1987), TREBULA (1986). In the past, colour changes when wood becoming darker during the steaming process were used to remove the undesirable colour differences between light coloured sapwood and dark coloured heartwood or to eliminate wood stain colours as a result of mould. At the present time, increased attention is paid to the issue of the change aimed at the colour of specific wood species during the heat processes MOLNAR – TOLVAJ (2002), MATUŠKOVA – KLEMENT (2009), TOLVAJ *et al*. (2010), FAN *et al*. (2010), DZURENDA – DELIISKI (2012), BEKHTA – NIEMZ (2013), DZURENDA (2013, 2018), BARCIK *et al*. (2015).

Black locust wood has narrow yellow-white sapwood and yellow-green or yellow-green and brown heartwood. According to the authors TREBULA (1986), TOLVAJ *et al*. (2010) black locust wood becomes darker during the steaming process and its final hue can be dark brown or dark brown-gray. The interest of wood specialists in black locust wood in darker brown colour emphasising the texture of wood suitable for manufacturing the furniture, floorings, toys as well as utility components and decorative objects results in the research into colour modification of black locust wood using the heat treatment process by saturated water steam.

Aim of the paper is to determine the colour of black locust (*Robinia pseudoacacia* L.) after the heat treatment processes with saturated water steam at the following temperatures: $t_I = 112.5 \pm 2.5$ °C for $\tau = 5.5$ hours (mode I), $t_{II} = 127.5 \pm 2.5$ °C for $\tau = 6.5$ hours (mode II) and $t_{III} = 137.5 \pm 2.5$ ° C for $\tau = 7.5$ hours (mode III) using the CIE-L*a*b* colour space and the assessment of the regimes in terms of energy intensity.

EXPERIMENT

Black locust wood in the form of woodturning blanks with dimensions of 30 x 55 x 500 mm and moisture content of Wp = 60.2 ± 4.5% was thermally treated with saturated steam in the pressure autoclave APDZ 240 (Himmasch AD, Haskovo, Bulgari) in the company Sundermann Ltd. in Banská Štiavnica. Heartwood was used to prepare woodturning blanks. The mode of color modification of black locust wood is given in Fig. 1. Optimized conditions of the heat treatment of woodturning blanks in terms of the colouring throughout the whole of the mass and process duration for individual modes of colour modification are shown in Tab. 1.

Thermally untreated as well as treated woodturning blanks of black locust wood were dried to report the moisture content of $W_p = 12 \pm 0.5\%$ in a conventional wood drying kiln KAD 1x6 by KATRES Ltd. Subsequently, dried woodturning blank surfaces were processed using Swivel spindle milling machine FS 200.

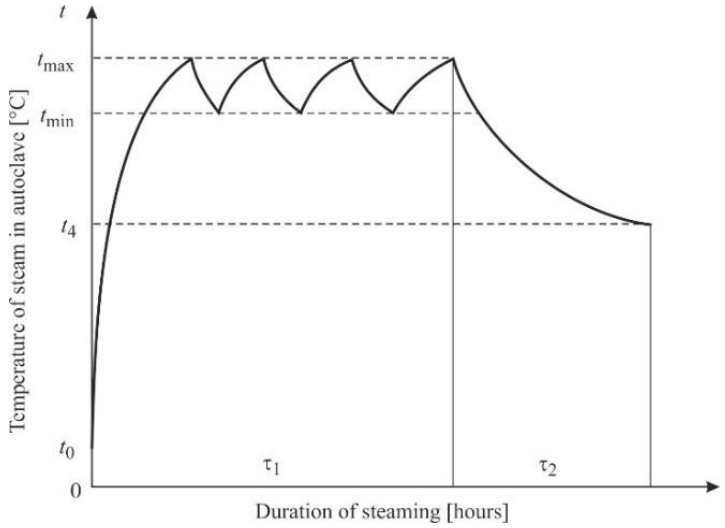

Fig. 1 Regime of color modification of black locust wood with saturated water steam.

Tab.1 Modes of colour modification of black locust wood with saturated water steam.

Modes	Temperature of the saturated water steam [°C]			Process duration [hours]		
	t_{min}	t_{max}	t_4	τ_1- stage I	τ_2-stage II	Total time $\tau_1 + \tau_2$
Mode I	110	115	100	4.5	1.0	5.5
Mode II	125	130	100	5.0	1.5	6.5
Mode III	135	140	100	5.5	2.0	7.5

Colour of black locust woodturning blanks was determined using the Color Reader CR-10 (Konica Minolta, Japan). The light source used was D65 with luminosity including a field of measurement of 8mm.

Lightness coordinates L* and coordinates a* and b* of CIE-L*a*b* colour space were measured using a sample unit of n = 183 black locust woodturning blanks. A sample unit of n = 185 black locust woodturning blanks was modified using mode I, a sample unit of n = 185 black locust woodturning blanks was thermally treated by mode II as well as a sample unit of n = 180 black locust woodturning blanks modified by mode III. Measurement of the coordinates of CIE-L*a*b*colour space using dried and planed woodturning blanks was carried out in the centre of the blank width and 250mm far from the face of processed dried woodturning blanks using Swivel spindle milling machine FS 200.

Colour coordinate values are introduced using a formula: $x = \bar{x} \pm s_x$, i.e. average measured value and standard deviation are mentioned. The extent of variation of set (measured) values in the CIE-L* a* b* colour space of thermally untreated as well as treated black locust wood is determined by the coefficient of variation.

Total colour difference ΔE* is determined according to Formula 2, in accordance with the standard ISO 11 664-4 (2008) as the result of the difference in the colour coordinates (ΔL*, Δa*, and Δb*) following the woodturning surface measurements before and after heat treatment.

$$\Delta E^* = \sqrt{\left(L_2^* - L_1^*\right)^2 + \left(a_2^* - a_1^*\right)^2 + \left(b_2^* - b_1^*\right)^2} \quad (1)$$

where: L_2*, a_2*, b_2* coordinate values of the wood colour space before the wood heat treatment,

L_1*, a_1*, b_1* coordinate values of the colour space of the thermally treated surface of black locust wood.

Rate of change in the wood colour and hues during the processes of heat treatment following the total colour difference ΔE* can be classified according to the chart shown in Tab. 2 (CIVIDINI *et al.* 2007).

Tab. 2 Classification of ΔE.

0.2 < ΔE*	Not visible difference
0.2 < ΔE* < 2	Small difference
2 < ΔE* < 3	Colour difference visible with low quality screen
3 < ΔE* < 6	Colour difference visible with medium quality screen
6 < ΔE* < 12	High colour difference
ΔE* > 12	Different colours

Energy consumption of the technological process of black locust woodturning blank colour modification is presented through the heat consumption standard Q_{TZN} and saturated water steam consumption per 1 m^3 of wood. Heat consumption standard of colour modification of 1 m^3 wood is according to the research of DZURENDA (2016) described by the formula:

$$Q_{TFS} = \frac{Q_A + Q_D + Q_I + Q_S + Q_P + Q_K}{V_D}, \qquad (kWh/m^3) \qquad (2)$$

where: Q_A – heat necessary to heat the construction material of the autoclave, kWh; Q_D – heat necessary to cause heat related to colour modification of wood, kWh; Q_I – heat necessary to heat the autoclave's insulation, kWh; Q_K – heat extracted as condensate from the pressure autoclave, kWh; Q_P – heat extracted by saturated steam after opening the autoclave, kWh; Q_S – heat necessary to cover heat losses from the surface of the pressure autoclave, kWh; V_D – volume of the colour modification wood in the pressure autoclave, m^3.

The consumption of saturated water steam necessary for colour modification of 1m^3 of black locust woodturning blanks is determined using the formula:

$$m'' = 3600 \cdot \frac{Q_{TZN}}{h'' - h'}, \qquad (kg/m^3) \qquad (3)$$

where: Q_{TZN} – heat consumption standard to modify 1 m^3 of black locust woodturning blanks, kWh/m^3; h´´ - enthalpy of saturated water steam at the temperature t_{max}, kJ/kg; h´ - enthalpy of saturated water steam at the temperature t_4, kJ/kg.

RESULTS AND DISCUSSION

The colour of black locust wood is described with the coordinate values of the CIE-L*a*b*colour space: L* = 71.8; a* = 5.3; b* = 25.0 according to the authors: BABIAK *et al.* (2004). These findings are confirmed by our measurements. We found out that the coordinate values of the dried black locust wood on the planed surface are: L* = 69.2 ± 2.9; a* = 4.7 ± 0.8; b* = 28.7 ± 2.4. Coefficients of variation of the individual sample units of measured colour coordinate values are: v_{L^*} = 4.1%, v_{a^*} = 17.0, v_{b^*} = 8.4.

The colour of dried planed black locust wood not thermally treated and hues resulting from the heat processes with saturated water steam by modes I, II, II are illustrated in Fig. 2.

a) b) c) d)

Fig. 2. The colour of dried and planed wood of black locust a) before and after heat treatment by b) mode I, c) mode II, d) mode III.

Original light yellow colour of black locust heartwood becomes light brown during the process of heat treatment by mode I. Black locust wood modified by mode II achieves brown-gray colour emphasizing the texture of black locust wood in tangential cut. The colour of thermally modified black locust wood is equivalent to the colour of Persian walnut (*Juglans regia*). Black locust wood thermally modified by mode III gets an original dark brown-gray colour.

The coordinate values of the CIE-L* a* b* colour space describing the colour of native black locust woodturning blanks after individual heat treatment modes and after drying are shown in Tab. 3.

Tab. 3 The coordinates of the CIE-L*a*b* colour space of thermally treated black locust with saturated water steam by modes I, II and III.

Colour modification mode	Colour coordinates	Parameter		
		number of measurements	coordinate value	coefficient of variation
		[-]	[-]	[%]
Mode I t = 112.5 ± 2.5 °C τ = 5.5 hours	L*	185	65.7 ± 2.0	5.0
	a*		7.6 ± 0.4	5.3
	b*		26.4 ± 1.4	5.3
Mode II t = 127.5 ± 2.5 °C τ = 6.5 hours	L*	185	59.4 ± 2.5	4.2
	a*		9.9 ± 0.4	4.0
	b*		23.8 ± 1.1	4.6
Mode III t = 137.5 ± 2.5 °C τ = 7.5 hours	L*	180	44.3 ± 1.6	3.6
	a*		8.9 ± 0.4	4,5
	b*		15.1 ± 0.9	5,9

The rate of change of values ΔL*, Δa*, Δb* for individual coordinates of the colour space of black locust wood resulting from the heat treatment process with saturated water steam is shown in bar graph in Fig. 3.

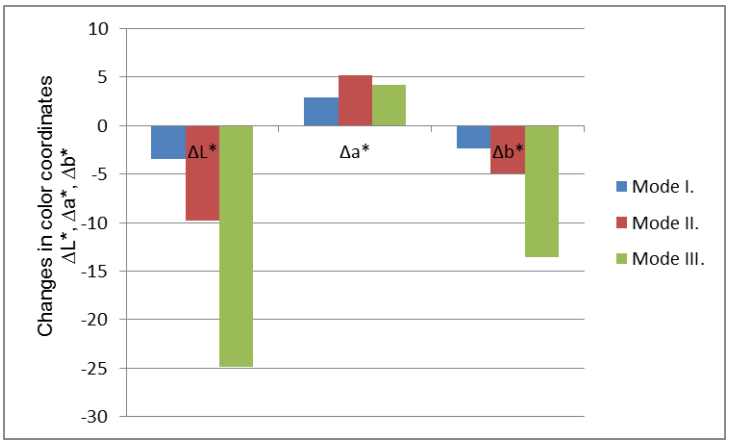

Fig. 3 The change of ΔL*, Δa*, Δb* values of the CIE-L*a* b* colour space of the thermally treated black locust wood resulting from the colour modification modes I, II, III.

Dependence of the lightness L*, red a* and yellow b* colour of black locust wood on temperature related to the heat treatment of woodturning blanks with saturated water steam at the temperature ranged from t = 112.5 ÷ 137.5°C is shown in Fig. 4.

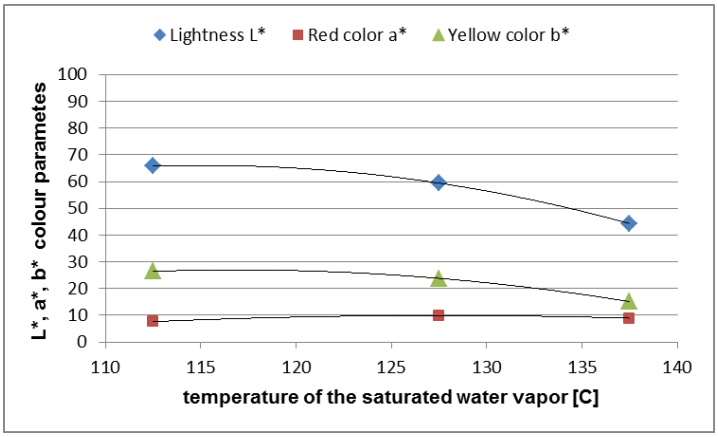

Fig. 4 Dependence of the lightness decrease and an increase in values of red and yellow colour coordinates of the CIE L* a* b* colour space of thermally treated black locust wood on the temperature of saturated water steam.

Total colour differences of black locust wood ΔE* achieved by the individual heat treatment modes with saturated water steam are shown in Fig. 5.

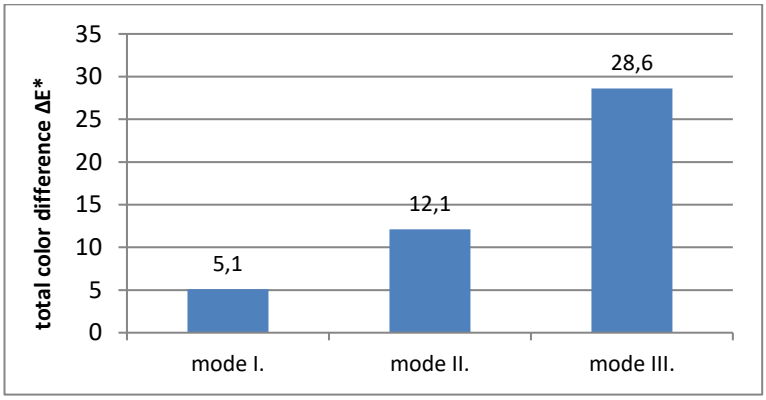

Fig. 5 Values of the total colour differences ΔE* of the thermally modified black locust wood achieved by the individual heat treatment modes with saturated water steam.

The change in black locust wood colour resulting from the conditions of the heat treatment by the individual modes of modification is reflected in changes in individual coordinate values of the CIE L*a*b* colour space. Lightness decrease due to modification mode I from $L^* = 69.2 \pm 2.9$ to $L_I^* = 65.7 \pm 2.0$, it means a decrease of $\Delta L^* = -3.5$ and an increase in red and yellow coordinate values from $a^* = 4.7 \pm 0.8$ to $a_I^* = 7.6 \pm 0.4$, it means a decrease of $\Delta a^* = +2.9$ is observed. At the same time a decrease in the value of yellow colour from $b^* = 28.7 \pm 2.4$ to $b_I^* = 23.8 \pm 1.1$, it means a decrease of $\Delta b^* = -4.9$ can be observed as well. The decrease in lightness of black locust wood and an increase in the values of red and, at the same time, the decrease in the yellow coordinate values reflects in mild browning of black locust wood quantified by the value of total colour difference $\Delta E^* = 5.1$.

More significant changes in the colour of black locust wood during the heat treatment process are achieved by modes II and III. Thermally treated black locust wood using mode II becomes brown to brown-gray in the colour expressed by the value of total colour difference $\Delta E^* = 12.1$. A decrease in lightness of $\Delta L^* = -24.9$, an increase in red colour coordinate value of $\Delta a^* = +4.2$ and, at the same time, a decrease in yellow colour coordinate value of $\Delta b^* = -13.6$ are achieved using mode III with saturated water steam at the temperature of $t = 137.5 \pm 2.5°C$ with the process duration of $\tau = 7.5$ hours. Wood becoming brown and dark in the colour creates an original dark brown-gray hue of the black locust woodturning blanks.

Homogeneity of achieved hues proven by the low values of the coefficients of variation of lightness L*, red colour a* and yellow colour b * coordinate values that do not exceed the value of $v_{x^*} \leq 5.9\%$ can be considered the benefit of heat treatment of black locust woodturning blanks by saturated water steam by modes I, II and III.

Total colour differences of the black locust wood ΔE^* resulting from the heat treatment processes by saturated water steam in at the temperature interval $t = 112.5°C \div 137.5°C$ ranges from the values of $\Delta E^* = 5.1 \div 28.6$. Within the colorimetric classification of the colour changes shown in Tab. 2, achieved hues can be defined as medium up to significant changes in wood colour.

Dependence of the growth of the total colour differences ΔE^* of thermally treated black locust wood on the temperature of saturated water steam in the CIE-L* a* b* colour space is consistent with the knowledge of wood colour changes during the heat treatment processes described by the authors: MOLNAR – TOLVAJ (2002), TOLVAJ *et al*. (2010), DZURENDA (2014, 2018), as well as of high temperature drying in the environment of superheated water steam KLEMENT – MARKO (2009), BARANSKI *et al*. (2016), or heat processes in the production of thermowood KAMPERIDOU *et al*. (2013), PLESCHBERGER *et al*. (2014), BARCIK *et al*. (2015).

According to the authors KOLLMANN – GOTE (1968), TREBULA (1996) in terms of the classification of changes in physical and chemical properties of wood during heat treatment process, the wood colour change belongs to irreversible changes. It is caused by partial hydrolysis of hemicelluloses in the lignin-saccharide wood matrix and by the extraction of water-soluble accessory substances. The findings are confirmed not only by the differences in ATR-FTIR spectroscopic analyses of thermally untreated as well as treated wood TIMAR *et al*. (2016), KUČEROVÁ *et al*. (2016), GEFFERT *et al*. (2017) but also by the presence of monosaccharide, organic acids and basic lignin units with guajacyl and syringyl structure in the condensate after pressure steaming of wood presented in the papers: DZURENDA – BUČKO (1998), KAČÍK (2001), KAČÍKOVÁ – KAČÍK (2011). The irreversible colour change and new brown, brown-gray hues of black locust wood achieved during the heat treatment modification with saturated water steam widen the possibility for the use of black locust wood in the field of construction and carpentry, design as well as in the field of art.

Heat consumption standard for individual modes of colour modification of black locust woodturning blanks and the consumption of saturated water steam necessary for the process of heat treatment are shown in Tab. 4.

Tab. 4 Heat consumption standards and the consumption of saturated water steam per 1 m^3 of modified black locust wood.

Colour modification mode	Heat consumption standards $Q_{TFS,}$ kWh/m^3	The consumption of saturated steam kg / m^3
Mode I	111.29	179,6
Mode II	127.73	206,2
Mode III	139.13	224,6

Short time and lower heat consumption can be considered positive features of mentioned modes used to modify the colour of black locust woodturning blanks by saturated water steam. Time necessary for the technological process of colour modification of black locust wood by saturated water steam is three-times shorter in comparison to the traditional technology for steaming treatment by saturated humid air at the temperature $t = 80 \div 95\ ^0$C in steam rooms for $\tau = 18 – 24$ hours TREBULA (1986), TOLVAJ *et al*. (2009).

Heat consumption depending on the temperature of saturated water steam and exposure time of colour modification of black locust woodturning blanks ranges from $Q_{TZN} = 111.29 – 139.13$ kWh/m^3 and saturated water steam m´´ = 179.6 – 224.6 kg/m^3. Mentioned values of heat consumption are by 1/3 lower compared to the values of heat consumption used for colour modification of black locust wood by humid air at the atmospheric pressure in steam rooms. Mentioned statement results not only from significantly shorter time of the technological process but also from the fact that ¾ of supplied heat is used for heating DZURENDA (2017).

CONCLUSION

The hues of black locust (*Robinia pseudoacacia* L.) obtained during the heat treatment process – colour modification by saturated water steam at the temperatures: $t_I = 112.5 \pm 2.5$ ^{o}C for $\tau = 5.5$ hours, $t_{II} = 127.5 \pm 2.5$ ^{o}C for $\tau = 6.5$ hours and $t_{III} = 137.5 \pm 2.5$ ^{o}C for $\tau = 7.5$ hours are presented in the paper. Black locust wood colour changes from light yellow-green to a pale brown with coordinates of the CIE-L*a*b*colour space as follows: $L^* = 65.7 \pm 2.0$; $a^* = 7.6 \pm 0.4$; $b^* = 26.4 \pm 1.4$ during the process of heat treatment by mode I. Subsequently, the wood colour changes to brown or brown-gray with colour coordinates: $L^* = 59.4 \pm 2.5$; $a^* = 9.9 \pm 0.4$; $b^* = 23.8 \pm 1.1$ during the heat treatment by mode II and to genuine dark brown-gray with colour coordinates: $L^* = 44.3 \pm 1.6$; $a^* = 8.9 \pm 0.4$; $b^* = 15.1 \pm 1.6$ using mode III.

When the temperature of saturated water steam during the heat treatment process is increased and the exposure time of the colour modification of black locust wood is prolonged, the lightness of the hue is affected, wood darkens. Specific new brown to dark brown-gray hues are achieved in the case of increasing values of red coordinate a* of CIE-L*a*b*colour space and decreasing values of yellow coordinate b* of CIE-L*a*b*colour space.

Mentioned modes used to modify the colour of black locust woodturning blanks by saturated water steam are more effective in terms of time and heat consumption in comparison to the traditional technology for steaming treatment by saturated humid air at the atmospheric pressure in steam rooms.

Irreversible changes in the black locust wood colour achieved by some of the heat treatment modes with the saturated water steam and new hues resulting from the processes widen the possibility of its use in the field of construction and carpentry, design as well as in the field of art.

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Author address

Prof. Ing. Ladislav Dzurenda, PhD.
Technical University in Zvolen
T. G. Masaryka 24
960 53 Zvolen
Slovakia
dzurenda@tuzvo.sk