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MODIFICATION OF MAPLE WOOD COLOUR DURING THE PROCESS OF THERMAL TREATMENT WITH SATURATED WATER STEAM

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ABSTRACT

The technological process of colour modification and the associated parallel processes of changing the acidity and density of maple wood modified in pressure autoclave with saturated water steam is evaluated in the paper. Maple wood was thermally treatment at the temperatures of: t_I = 105 ± 2.5 °C, t_{II} = 125 ± 2.5 °C and t_{III} = 135 ± 2.5 °C for τ = 3, 6, 9 and 12 hours. Direct *pH* measurement of maple wood with the moisture content above the fiber saturation point (FSP) was performed using SI 600 pH meter with LanceFET + H puncture probe. The acidity values of maple wood ranged from *pH* = 5.1 in the case of native wood, and the acidity of the wood changed to *pH* = 3.3 due to the temperature and time of modification. The density of maple wood was determined using the RADWAG KIT 128 instruments. The total change in the density of wood during the modification process was 5.3 % lower than the density of native wood. Statistical evaluation of the measured results showed the correlations between the total colour difference and *pH.*

Key words: maple wood, colour modification, acidity, wood density, saturated water steam.

INTRODUCTION

Wet wood placed in the environment of hot water, saturated water steam, or saturated humid air is heated and its physical, mechanical, as well as chemical properties, are changed.

Thermal treatment of wood, besides physical and mechanical changes applied in the process of manufacturing veneers, plywood, bentwood furniture or pressed wood are accompanied with the changes in chemical properties and colour of the wood (KOLLMANN and GOTE 1968, TREBULA 1986, MOLNAR and TOLVAJ 2002, TOLVAJ *et al*. 2010, DZURENDA and ORLOWSKI 2011, DZURENDA 2013, BARANSKI *et al.* 2017).

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 mold. In recent times, research into thermally modified wood has been focused on the issue of the colour change of specific tree species into more or less bright hues or wood imitation of domestic or exotic tree species (MOLNAR and TOLVAJ 2002, TOLVAJ *et al*. 2009, DZURENDA 2014, 2018a, b, c, BARCIK *et al*. 2015, BARANSKI *et al*. 2017).

The effect of heat on wet wood is also initiated by chemical changes in wood. The first chemical reactions include partial hydrolysis of hemicelluloses and extraction of water-soluble substances (FENGEL and WEGENER 1984, BUČKO 1995, SOLÁR 2004, SUNDQVIST *et al*. 2006, SAMEŠOVÁ *et al.* 2018). Depending on the temperature and time of the activity of the hydrolysis products, e.g. acetic acid and formic acid, degradation of polysaccharides occurs. During the thermal treatment of wood, dehydration of pentoses to 2-furaldehyde as well as oxidation of carbohydrates also occurs. New chromophoric groups begin to form in lignin, causing the colour change of wood (FENGEL and WEGENER 1984, BUČKO 1995, HON and SHIRAISHI 2001, SOLÁR 2004, SUNDQVIST *et al.* 2006, GEFFERT *et al.* 2019).

The aim of this paper is to present changes in the properties of maple wood, such as density, acidity, and colour in the process of thermal treatment with saturated water steam in the temperature ranging from t = 105 °C to 135 °C and for the time from τ = 3 to 12 hours.

MATERIAL AND METHODS

The wood of *Acer pseudoplatanus* in the form of blanks with dimensions: the thickness of 38 mm, the width of 90 mm and the length of 750 mm in 260 pieces were divided into 13 groups of 20 pieces in one group. The initial moisture content of wet maple wood was in the range between w = 57.2 ± 3.1 %. Blanks in the group 1 were not thermally treated. The other blanks were divided into 12 groups of 20 pieces and thermally treated with saturated water steam at the temperatures t = 105 °C, t = 125 °C, and t = 135 °C for 3, 6, 9, and 12 h. Thermal treatment of maple wood with saturated water steam was carried out in a pressure autoclave APDZ 240 (Himmasch AD, Haskovo, Bulgaria) installed in the company Sundermann s.r.o. Banská Štiavnica (Slovakia).

The mode of thermal treatment in order to modify the colour of maple wood with saturated water steam is illustrated in Fig. 1. The conditions of thermal treatment of individual modes of colour modification are described in Tab. 1.

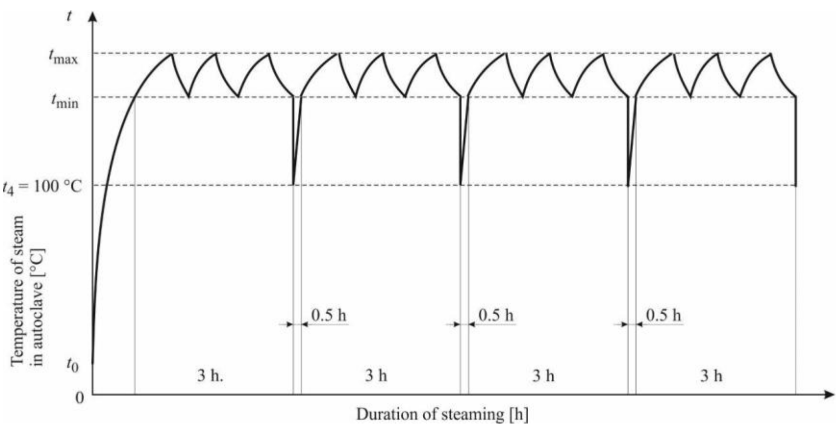

Fig. 1 Mode of colour modification of maple wood with saturated water steam.

The thermal process of maple wood colour modification was performed in the pressure autoclave at a higher saturated water steam pressure than atmospheric pressure. Saturated water steam temperatures in individual colour modes are given in Table 1. The temperatures t_{max} and t_{min} are the temperature intervals at which saturated water steam is fed into the autoclave to carry out the technological process. The temperature t_4 is the temperature of the saturated water steam in the autoclave after reducing the water steam pressure in the autoclave to the atmospheric pressure to allow the safe opening of the pressure equipment and sampling after the time of thermal treatment.

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

Temperature of saturated water steam	t_{min}	t_{max}	t_4	Length of time the wood is exposed to colour modification			
Mode *I*	102.5	107.5	100	τ_1 = 3 h	τ_2 = 6* h	τ_3 = 9* h	τ_4 = 12* h
Mode *II*	122.5	127.5	100				
Mode *III*	132.5	137.5	100				

Note: * After individual time of colour modification, exactly according to the determined diagram (course), 0.5 hour must be added to the given time of thermal modification of maple wood colour. This time between the individual sections of the modification serves to reduce the pressure for the safe opening of the autoclave and handling-selection of one group of modified blanks from the autoclave-closing and pressurization of the autoclave with saturated steam.

The moisture content of maple wood before entering the technological process of thermal treatment was determined by a random selection of 5 samples. Similarly, the moisture of thermally treated blanks was determined in individual modes and times after their selection from the autoclave and cooling to ambient temperature. The moisture content of wet maple wood was determined by the gravimetric method according to the standard STN EN 13183-1 (2003).

The acidity measurement of wet maple native and thermal treatment wood was performed using SI 600 pH meter with a functional Lance FET+H probe (Sentron, Roden, The Netherlands). Three measurements were performed on all samples in the middle of the thickness of the blank and 100 mm from the front of the blank. Using an accu drill (DeWalt DCD791NT, Germany), 12 mm diameter hole was made in the measuring point. The drilled wet sawdust was pushed back into the hole with a glass rod, where the LanceFET+H sensor head was inserted (GEFFERT *et al.* 2019). After about 60 seconds of stabilization, the *pH* value was read on SI 600 pH meter.

Dried colour-modified maple wood is used as a material for manufacturing the furniture, flooring, or interior tiling in the dry state. For this reason, the samples of untreated and thermally treated maple wood were dried in the convection hot air dryer KAD 1x6 (KATRES s.r.o. Czech Republic) with mild drying regime to the final moisture content w = 12 ± 0.5 % (DZURENDA 2020). Subsequently, the surface of dry blanks was machined using the FS 200 milling machine (BENET Trading, Kvasiny, Slovakia).

The colour of both thermal treatment and untreated maple wood in the *CIE L*a*b** colour space was determined using Color Reader CR-10 colorimeter (Konica Minolta, Japan). The measurement was performed on the loading and side surfaces at a distance of 300 mm. A D65 light source with an illuminated area of 8 mm was used. The colour was evaluated based on the changes in the colour space *CIE L*a*b** on the luminance coordinate L^*, the red colour a^*, the yellow colour b^*, and the total colour difference ΔE^*.

The value of the total colour difference is described with the equation:

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

where: L^*_1, a^*_1, b^*_1 are the values of the coordinates of the colour space of the dried milled thermally untreated maple wood surface,
L^*_2, a^*_2, b^*_2 are the values of the coordinates of the colour space of the dried milled thermally treated maple wood surface.

Test samples for measuring density with the following dimensions were prepared from native and thermally treated blanks in individual modes and times: the thickness of 15 mm, the width of 50 mm, and the length of 100 mm. The produced samples were dried in the laboratory oven (MEMMERT UM110m, Germany) at the temperature of t = 103 ± 2 °C to constant weight. The density of maple wood in each mode and time of treatment was determined using the instrument RADWAG KIT 128.

DISCUSSION

Physical properties of maple wood, such as wood moisture and acidity of wet wood before the heat treatment process, as well as wood density, colour space coordinates *CIE L*a*b** in the dry state are given in Table 2.

Tab. 2 Measured values of density, colour space coordinates *CIE L*a*b of dry wood, moisture and acidity of wet maple wood.**

Wood	Dry maple wood				Wet maple wood	
	Wood density	Coordinate colour space *CIE L*a*b**			Moisture content	Acidity
	ρ_0 [kg·m^{-3}]	*L**	*a**	*b**	*w* [%]	*pH*
Acer pseudo-platanus	600.1 ± 50.1	86.6 ± 1.2	5.9 ± 0.5	16.4 ± 0.5	57.2 ± 3.1	5.1 ± 0.2

The measured values of the density of maple wood in the dry state are the average values of the density of healthy maple wood not damaged by fungi or molds. Similar values of maple wood density for the territory of Central Europe are given in the works: POŽGAJ *et al*. 1997, MAKOVÍNY 2010, KURJATKO *et al.* 2010. Based on the above statement, it is possible to mention the analyzed changes in the properties of maple wood achieved by thermal treatment in individual modes as representative (standard).

The results of laboratory work determining the density of thermally treated maple wood by individual modes in the dry state are given in Table 3.

Tab. 3 The densities of the dry thermally treated maple.

Temperature of saturated water steam	Time of thermal modification of maple wood [h]			
	3	6	9	12
	ρ_0[kg/m^3]	ρ_0 [kg/m^3]	ρ_0 [kg/m^3]	ρ_0 [kg/m^3]
t_I = 105 ± 2.5 °C	590.5 ± 27.8	586.4 ± 25.2	584.1 ± 29.3	582.9 ± 22.8
t_{II} = 125 ± 2.5 °C	587.9 ± 29.1	585.2 ± 22.6	580.4 ± 25.9	577.1 ± 20.9
t_{III} = 135 ± 2.5 °C	581.4 ± 21.9	577.1 ± 24.5	573.2 ± 21.0	568.4 ± 20.9

During the process of thermal modification of maple wood colour, weight loss occurs depending on the temperature of saturated water steam and the time of modification. The weight loss is mainly due to the process of hydrolysis of polysaccharides and extraction of water-soluble substances in wood (LAUROVÁ *et al.* 2004, SOLÁR 2014, VÝBOHOVÁ *et al.* 2018, GEFFERT *et al.* 2020). The reduction of the density of maple wood in the given modification process represents the value of by $\rho_0 \leq 5.3$ %. The changes in the density of colour-modified maple wood do not exceed the limits of the natural tolerance of maple wood densities, which is in the Central European area: ρ_0 = 480-590-750 kg.m^{-3}.

The moisture thermally treated maple wood for 3, 6, 9, 12 h measured after cooling the wood to ambient temperature is given in Table 4.

Tab. 4 Average values of maple wood moisture during the thermal treatment process

Temperature of saturated water steam	Time of thermal modification of maple wood [h]			
	3	6	9	12
	w [%]	*w* [%]	*w* [%]	*w* [%]
t_I = 105 ± 2.5 °C	51.6 ± 1.9	51.2 ± 1.2	49.5 ± 1.0	48.9 ± 1.1
t_{II} = 125 ± 2.5 °C	52.3 ± 1.2	50.9 ± 1.4	50.2 ± 1.2	48.2 ± 1.1
t_{III} = 135 ± 2.5 °C	51.8 ± 1.6	51.0 ± 1.1	49.4 ± 1.1	47.9 ± 0.9

Reducing the maple wood moisture by $\Delta w \approx 13.9$ % resulting from the thermal treatment process is caused by evaporation of water from the wood into the environment in the autoclave during cooling to t = 100 °C. The source of heat for vaporization and evaporation of water from wood is the heat accumulated during the heating of wood to the required technological temperature (DZURENDA and DELIISKI 2000, DZURENDA 2018c).

Acidity values measured by the direct pH measurement method during the thermal treatment of maple wood at regular time intervals, after 3, 6, 9, 12 h are mentioned in Table 5.

Tab. 5 Measured average *pH* values of maple wood during the process of thermal treatment

Temperature of saturated water steam	Time of thermal modification of maple wood [h]			
	3	6	9	12
	pH [-]	*pH* [-]	*pH* [-]	*pH* [-]
t_I = 105 ± 2.5 °C	4.8 ± 0.3	4.7 ± 0.1	4.6 ± 0.2	4.2 ± 0.2
t_{II} = 125 ± 2.5 °C	4.1 ± 0.1	3.9 ± 0.2	3.5 ± 0.3	3.3 ± 0.2
t_{III} = 135 ± 2.5 °C	3.9 ± 0.1	3.6 ± 0.2	3.3 ± 0.2	3.3 ± 0.1

The wet wood acidity in the case of most temperate tree species ranges from pH = 3.3 to 6.4 (SANDERMANN and ROTHKAMM 1956, IRLE 2012, SOLAR 2014, GEFFERT *et al.* 2019). The acidic reaction of most woody plants is caused by free acids and acidic groups found in an aqueous solution of dilute sugars, organic acids, and water-soluble inorganic substances fed by the root system to the tree in cell lumens (ČUDINOV 1968, BLAŽEJ *et al.* 1975, ZEVENHOVEN 2001, PŇAKOVIČ and DZURENDA 2015). The measured acidity values of wet untreated maple wood do not differ from the acidity values of maple wood with the moisture content above the saturated fiber point reported by the authors (SOLAR 2004, GEFFERT *et al.* 2019).

A decrease in the *pH* values of thermally modified maple wood by individual colour modification modes confirms the known fact about the course of hydrolysis of hemicelluloses in wet hardwood wood by the action of heat, as reported by MELCER *et al.* 1989, LAUROVÁ *et al.* 2004, SUNDQVIST *et al.* 2006, SAMEŠOVÁ *et al.* 2018, VÝBOHOVÁ *et al.* 2018, GEFFERT *et al.* 2019, DZURENDA *et al.* 2019).

The colour of dried, planed untreated maple wood and colour hues obtained during thermal treatment modes with saturated water steam is shown in Fig. 2. EPSON PERFECTION V850 PRO scanner with the quality of the created scan of maple wood colour samples of 1200 dpi was used in order to visually evaluate the colour change in the process of thermal treatment of maple wood by individual modes and for specific times.

Mode *I* t = 105 °C

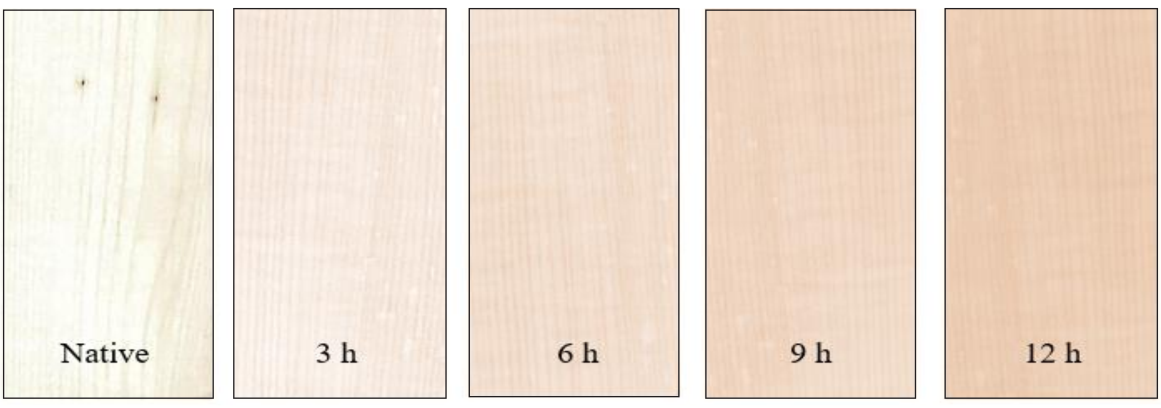

Fig. 2 Changes in the colour of maple wood during the process of thermal treatment

A light white colour with yellow tinge of dry untreated maple wood was identified in the colour space *CIE L*a*b** by the coordinates $L^* = 86.6 \pm 1.2$; $a^* = 5.9 \pm 0.5$; $b^* = 16.4 \pm 0.5$. The values given are comparable to the values of colour coordinates given for maple wood by the authors BABIAK *et al.* 2004, MEINTS *et al.* 2017.

The coordinate values describing the colour of the maple wood before and after the thermal treatment by the individual modes in the colour space *CIE L*a*b** measured by colorimeter Color Reader CR-10 and the total colour difference ΔE^* are given in Table 6.

Tab 6. Measured values of the coordinates *L*, a*, b in the colour space *CIE L*a*b**, values of the total colour difference ΔE^* of maple wood during the process of thermal modification.**

Temperature of saturated water steam	Coordinates *CIE L*a*b**	Time of thermal modification of maple wood [h]			
		3	6	9	12
$t_I = 105 \pm 2.5$ °C	L^*	81.0 ± 0.4	79.1 ± 0.3	77.4 ± 0.6	76.2 ± 0.9
	a^*	8.5 ± 0.3	8.9 ± 0.4	10.6 ± 0.6	10.9 ± 0.5
	b^*	17.6 ± 0.5	17.7 ± 0.6	18.3 ± 0.6	18.9 ± 0.4
	ΔE^*	5.5	7.4	9.7	10.9
$t_{II} = 125 \pm 2.5$ °C	L^*	75.6 ± 0.6	73.0 ± 0.6	69.7 ± 0.8	69.3 ± 1.0
	a^*	12.0 ± 0.4	12.0 ± 0.4	12.3 ± 0.5	12.4 ± 0.3
	b^*	16.0 ± 0.5	16.1 ± 0.4	16.6 ± 0.5	17.1 ± 0.7
	ΔE^*	11.3	13.3	16.3	17.4
$t_{III} = 135 \pm 2.5$ °C	L^*	69.3 ± 0.8	65.3 ± 0.8	61.8 ± 0.7	59.1 ± 0.6
	a^*	11.9 ± 0.5	11.5 ± 0.4	11.6 ± 0.2	11.8 ± 0.2
	b^*	16.3 ± 0.3	17.0 ± 0.5	17.3 ± 0.4	17.8 ± 0.4
	ΔE^*	17.2	21.2	24.2	27.3

From a decrease in the value of lightness L_0* = 86.6 of thermally untreated wood to the value of L_1* = 76.2 of thermally treated maple wood with saturated water steam with a temperature t_I = 105 ± 2.5 °C, to the value of L_2* = 69.3 during the treatment of wood with water steam at the temperature of t_{II} = 125 ± 2.5 °C, to the value of L_3* = 59.1 at saturated water steam temperature t_{III} = 135 ± 2.5 °C, it is clear that increasing wood temperature results in the change in the brightness of the thermally treated maple wood, ΔL^* increases and the wood darkens. A decrease in brightness is not uniform during the technological process of thermal modification of maple wood. A decrease in the brightness of thermally treated wet maple wood is in line with the knowledge about wood darkening in technological processes, such as wood steaming declared in the works of TOLVAJ *et al.* 2009, 2010, DZURENDA, 2018b.

Changes in the red chromatic coordinate a^* have an increasing tendency. The red values of maple wood reach higher values during the modification depending on the temperature and time, while the modified wood acquires a redder tinge. The magnitudes of the changes in the red coordinate are significantly smaller compared to the changes in the luminosity coordinate. In the case of the yellow chromatic coordinate b^*, the changes are slightly similar to the changes in brightness or changes of the colour coordinate a^*.

The changes in the total colour difference ΔE^* depending on the acidity of wet maple wood during the process of thermal treatment at the temperatures ranging between t = 105–135 °C and the time of the technological process τ = 3–12 h is shown in Fig. 3.

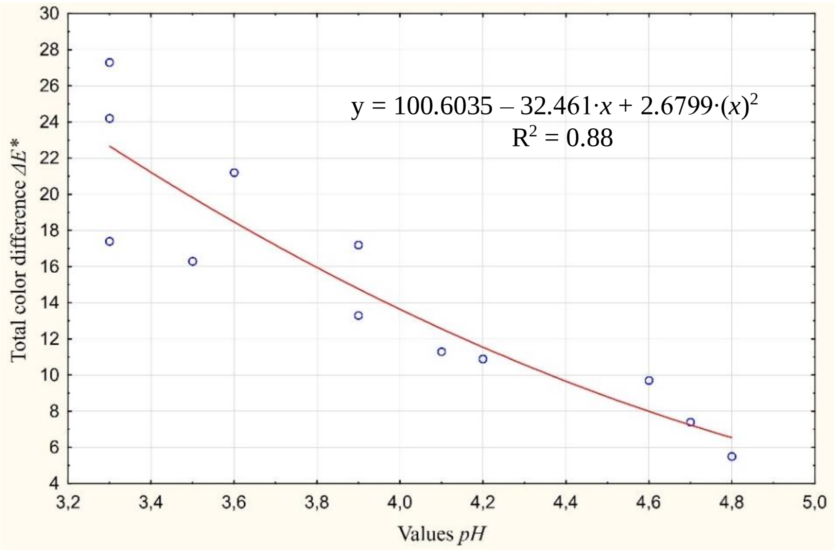

Fig. 3 Dependence of the total colour difference of maple wood ΔE^* on the value of acidity of maple wood *pH* during the process of thermal treatment.

The dependence of the total colour difference of maple wood on the value of acidity is mathematically described by the equation:

$$\Delta E^* = 100.6035 - 32.461 \cdot pH + 2.6799 \cdot (pH)^2 \qquad (2)$$

where: *pH* - acidity value of wet maple wood.

The dependence of the total colour difference ΔE^* on the change in acidity of maple wood in the thermal process is a suitable tool for evaluating the achieved colour change based on the *pH* of maple wood in the technological process.

CONCLUSION

1. The results of colour change, acidity and density of maple wood during the process of thermal modification of wood with saturated water steam at the temperatures: $t_I = 105 \pm 2.5$ °C, $t_{II} = 125 \pm 2.5$ °C and $t_{III} = 135 \pm 2.5$ °C for $\tau = 3–12$ hours are presented in the paper .
2. The wood colour changes as a result of the process of thermal modification. The change of the original white to light white-yellow colour, pale brown to dark brown colour was observed. More or less deep colour hues depending on the temperature and time of modification occured. The colour changes achieved during the modification process are described using the coordinates in the colour space *CIE L*a*b**.
3. During the technological process of wood modification, the acidity of the wood changes, which is more pronounced at higher steam temperature and for a longer time. The change is from $pH = 5.1$ in the case of native wood to $pH = 3.3$ in mode *III* after 12 hours of modification. Simultaneously, with the change in the acidity of the wood, there is also a decrease in the total weight of the wood due to acid hydrolysis of the chemical components of the wood and extraction of water-soluble substances, which represents a total decrease in density by up to 5.3 %.
4. The dependence of the total colour change ΔE^* of maple wood on the acidity of maple wood in the range between $pH = 3.3 – 5.1$ is described by Eq. 2.

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