Original scientific paper
Received:19.6.2019
Accepted:6.12.2019
UDK: 674.031.632.1:620.251

THE EFFECT OF TIME AND TEMPERATURE OF SATURATED WATER STEAM ON ACIDITY AND WOOD COLOUR IN THE PROCESS OF THERMAL MODIFICATION OF SILVER BIRCH WOOD COLOUR

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ABSTRACT

The aim of this paper is to determine the correlation between change in acidity and colour of wood species *Betula pendula Rot.* in the CIE-L*a*b* colour space in the process of heat treatment of woodturning blanks with dimensions of 40 x 90 x 800 mm, and saturated water steam in the range of temperatures from t = 105 to 135 °C, as well as the time of heat treatment from τ = 3 up to 12 hours. Wet silver birch wood changes pH in the range of pH = 5.3 ÷ 3.2 due to partial hydrolysis of hemicelluloses and extraction of water-soluble substances, and it loses whiteness (gets darker). Increment in the value of the coordinate of the red colour a* and slight changes in the coordinate of yellow colour b * lead to colour levels of varying intensity of the brown colour.

Colour coordinates of birch wood in the CIE L*a*b* colour space with dependence on temperature of saturated water steam *t* and the time of heat treatment *τ* are described using the equations:

$$L^* = 83.6232+0.4815\cdot t-1.9377\cdot\tau -0.0041\cdot t^2-0.0068\cdot t\cdot\tau +0.1091\cdot\tau^2,$$
$$a^* = 6.7847-0.0795\cdot t+1.2265\cdot\tau+0.0007\cdot t^2-0.0026\cdot t\cdot\tau-0.0511\cdot\tau^2,$$
$$b^* = 19.8107-0.0014\cdot t +0.7326\cdot\tau-9.3472E\text{-}5\cdot t^2-0.0027\cdot t\cdot\tau-0.0255\cdot\tau^2.$$

Key words: wood, silver birch, CIE-L*a*b* colour space, heat treatment, saturated water steam

1. INTRODUCTION

Wood located in the environment of hot water, saturated water steam or saturated humid air is heated and its physical, mechanical and chemical properties change. Already established facts are used in technology of steam while bending and boiling veneers and plywood, as well as in the manufacturing processes of bent furniture or pressed wood. *Kollmann and Gote (1968), Nikolov, Rajčev and Deliiski (1980), Sergovsky and Rase (1987), Lawnniczak (1995), Trebula (1996), Deliiski and Dzurenda (2010).*

Thermal treatment processes of wood with saturated water steam, in addition to specific physical-mechanical changes of wood, are accompanied by chemical reactions such as partial hydrolysis and extraction, leading to colour change as well. (*Melcer et al. (1989), Bučko (1995), Kačík (2001), Geffert, Vybohova and Geffertova, (2018).* In the past, colour modification, especially wood darkening, was used to remove undesirable differences in colour of lighter sapwood and darker heartwood, or to remove wood stains resulting from steaming or moulding. Recently, research has aimed at colour change of specific wood species to more or less distinctive hues, or imitation of the exotic wood species (*Tolvaj et al. (2009), Dzurenda (2014), Barcik, Gašparík and Razumov (2015), Baranski et al. (2017) Hadjiski and Deliiski (2016), (Dzurenda 2018).*

Using the coordinates of the 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* representing the darkest black at L* = 0 and the brightest white at L* = 100. The value of a* measures the red–green character of the colour, with positive values for red shades (+a*), and negative values for green (-a*). The value of b* gives the yellow–blue character, with positive values for yellow shades (+b*) and negative ones for blue shades (-b*).

The aim of this paper is to determine the dependence of the acidity and the colour of the wood species *Betula pendula Rot*. in the CIE-L*a*b*colour space resulting from the processes of heat treatment – modification of wood with saturated water steam at the following range of temperatures: from t = 105 to 135 ^{O}C for τ = 3 to 12 hours.

2. MATERIALS AND WORK METHODS

Silver birch wood in form of woodturning blanks with dimensions 40 x 90 x 800 mm and moisture content w = 40 - 55 % was thermally treated with saturated steam in the pressure autoclave APDZ 240 (Himmasch AD, Haskovo, Bululharsko) in the company Sundermann s.r.o. Banská Štiavnica). Modes of colour modification with saturated water steam are shown in Figure 1. Description of modes of thermal colour modification of silver birch wood with saturated water steam is provided in Table 1.

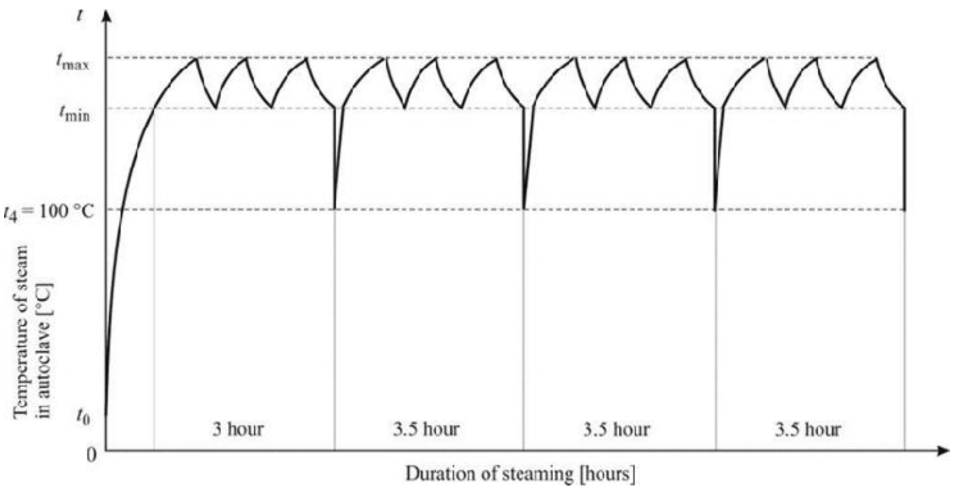

Figure 1. *Modes of colour modification of silver birch wood with saturated water steam*

Table 1. *Modes of colour modification of silver birch wood with saturated water steam*

<table>
<tr><th>Temperature of saturated water steam</th><th>t_{min}</th><th>t_{max}</th><th>t_4</th><th colspan="4">Time of thermal colour modification of wood</th></tr>
<tr><td>Mode I</td><td>102.5</td><td>107.5</td><td>100</td><td rowspan="3">τ_1 = 3 h</td><td rowspan="3">τ_2 = 6 (+0.5[a]) h</td><td rowspan="3">τ_3 = 9 (+0.5[a]) h</td><td rowspan="3">τ_4 = 12 (+0.5[a]) h</td></tr>
<tr><td>Mode II</td><td>122.5</td><td>127.5</td><td>100</td></tr>
<tr><td>Mode III</td><td>132.5</td><td>137.5</td><td>100</td></tr>
</table>

Note: [a] technological pause

Before heat treatment, as well as after its finishing and cooling the wood, the pH value of wet wood was measured. Since the diameter of the sensor head of the potentiometer used to measure pH is d = 10 mm, and it cannot be put into solid material, a hole with diameter of 12 mm was formed in the place of measurement with the accu drilling machine. Sawdust resulting from drilling was poured into the hole and the sensor head LenceFET+H 22704-010 of the pH meter SENTRON SI 6OO was inserted into the wet sawdust.

Thermally treated wood of silver birch woodturning blanks, as well as thermally untreated samples, were dried to report the moisture content of w = 12 ± 0.5 % in a conventional wood drying kiln KAD 1x6 (KATRES Ltd.). Subsequently, surfaces and edges were processed using Swivel spindle milling machine FS 200.

The colour of silver birch woodturning blanks in the CIE-L*a*b*colour space was determined using the Colour Reader CR-10 (Konica Minolta, Japan). Light source D65 with lit area of 8 mm was used.

Measurement of values colour sliver birch, lightness coordinate L* and coordinates a* and b* in the CIE-L*a*b* colour space on the samples of heat-treated as well as non-heat-treated wood was carried out after drying on the planed surface in the middle of the side and loading surfaces at a distance of 300 mm from the forehead. The measurement was performed on 75 pieces of blanks.

Values of colour coordinates of thermally treated as well as untreated silver birch wood are presented using a formula $x = \bar{x} \pm s_x$, i.e. average measured value and standard deviation.

Total colour difference ΔE* is determined according to Formula 1, in accordance with the standard *ISO 11 664-4* as a result of the difference in colour coordinates ΔL*, Δa*, and Δb* following the measurements of the wood colour of treated as well as untreated birch woodturning blanks:

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

where: L^*_1, a^*_1, b^*_1 are values of the coordinates in the wood colour space of the surface of dried milled thermally treated silver birch wood

$L^*_{2,}$ a^*_2, b^*_2 are values of the coordinates in the wood colour space of the surface of dried milled thermally treated silver birch wood

Rate of change in wood colour and hues during the processes of thermal treatment following the total colour difference ΔE* is classified according to the chart mentioned by the authors: *Cividini et al (2007)* shown in Tab. 2.

Table 2. *Classification of the total colour difference 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

3. RESULTS

According to the authors *Perelygin (1965), Makoviny (2010), Klement, Réh and Detvaj (2010)*, the wood colour of the wood species *Betula pendula Rot*. is light white-brown. The colour of silver birch in the CIE-$L^*a^*b^*$ colour space is described by the authors: *Babiak, Kubovský and Mamoňova (2004)*, with the coordinates: L^* = 78.07; a^* = 5.92; b^* = 20.02. In our research, L* = 83.7 ± 1.3; a* = 6.8 ± 0.6; b* = 19.8 ±0.9 were the values of coordinates in CIE L*a*b* colour space measured on the planed surface of silver birch wood when moisture content was w ≈ 12 %.

Acidity of wet thermally untreated birch wood at moisture content of 53.9% was pH = 5.3 ± 0.2. The pH values of the wet heat treated silver birch after cooling are shown in Tab. 3. Table 3 also presents the coordinates L*, a*, b*, which describe the colour of the dried thermally treated birch wood on the planed surface.

***Table 3**. Values of moisture content, acidity and coordinates L*, a*, b* of silver birch wood in the process of thermal treatment.*

Temperature of saturated water steam	Moisture content of wood	Time of thermal colour modification of birch wood			
		3 hours	6 hours	9 hours	12 hours
t_I = 105 ± 2.5 °C	w ≈ 45.5 %	pH = 4.9 ± 0.1	pH = 4.7 ± 0.1	pH = 4.6 ± 0.2	pH = 4.4 ± 0.3
	w ≈ 11.5 %	L* = 80.7 ± 1.2	L* = 75.8 ± 1.2	L* = 74.7 ± 0.8	L* = 71.3 ± 1.2
		a* = 8.5 ± 0.8	a* = 10.7 ± 0.7	a* = 10.5 ± 0.7	a* = 10.5 ± 0.8
		b* = 19.2 ± 0.6	b* = 21.1 ± 0.5	b* = 21.4 ± 0.6	b* = 19.8 ± 0.4
		ΔE* = 3.5	ΔE* = 8.9	ΔE* = 9.8	ΔE* = 12.9
t_{II} = 125 ± 2.5 °C	w ≈ 43.8 %	pH = 3.9 ± 0.1	pH = 3.8 ± 0.2	pH = 3.7 ± 0.3	pH = 3.6 ± 0.2
	w ≈ 11.8 %	L* = 73.9 ± 1.1	L* = 66.6 ± 1.2	L* = 64.3 ± 1.2	L* = 63.8 ± 1.2
		a* = 10.3 ± 0.8	a* = 11.9 ± 0.7	a* = 12.5 ± 0.7	a* = 12.2 ± 0.8
		b* = 19.8 ± 0.9	b* = 18.9 ± 0.5	b* = 18.5 ± 0.5	b* = 19.8 ± 0.4
		ΔE* = 10.4	ΔE* = 18.8	ΔE* = 20.2	ΔE* = 20.6
Temperature of saturated water steam	Moisture content of wood	Time of thermal colour modification of birch wood			
		3 hours	6 hours	9 hours	12 hours
t_{III} = 135 ± 2.5 °C	w ≈ 46.5 %	pH = 3.6 ± 0.1	pH = 3.4 ± 0.2	pH = 3.2 ± 0.1	pH = 3.2 ± 0.1
	w ≈ 11.7 %	L* = 65.1 ± 1.9	L* = 59.9 ± 1.5	L* = 55.6 ± 0.9	L* = 53.5 ± 0.7
		a* = 11.6 ± 0.6	a* = 12.5 ± 0.4	a* = 12.5 ± 0.3	a* = 12.1 ± 0.4
		b* = 18.7 ± 0.6	b* = 19.4 ± 0.5	b* = 19.5 ± 0.6	b* = 18.8 ± 0.4
		ΔE* = 19.2	ΔE* = 24.4	ΔE* = 28.8	ΔE* = 30.6

The correlation between the values pH of silver birch wood when the temperatures ranged from t = 105 °C to 135 ° and when the length of the thermal treatment process was up to $\tau \leq 12$ hours is presented using the 3D diagram in the following graph:

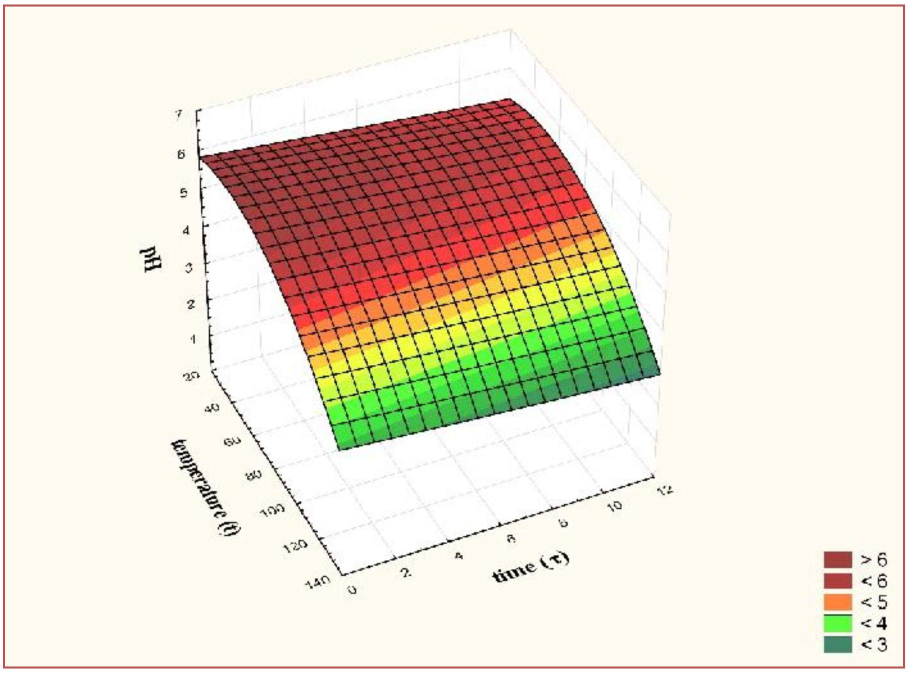

***Figure 2.** 3D diagram pH – t – τ*

The changes in lightness coordinate L^* in the CIE-L*a*b* colour space of silver birch woodturning blanks resulted from the process of thermal modification of colour with saturated water steam at temperatures of t_I = 105 ± 2.5 °C, t_{II} = 125 ± 2.5 °C, t_{III} = 135 ± 2.5 °C for τ = 3, 6, 9, 12 hours, and they are shown in Figure 3.

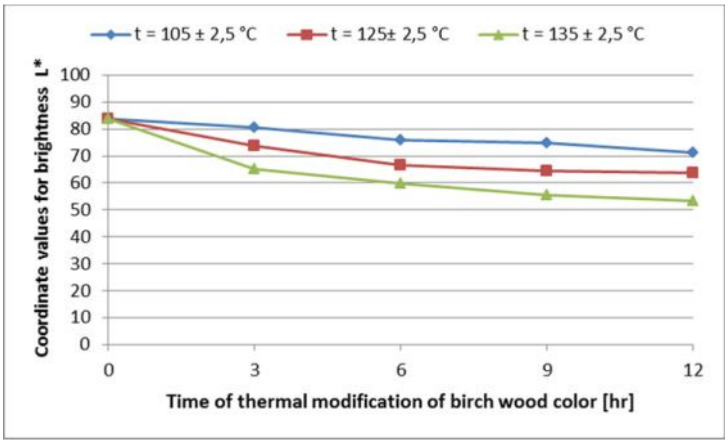

Figure 3. *Values of coordinate L* in the process of thermal treatment of birch wood*

Dependence of lightness L* in the CIE L*a*b* colour space of thermally treated silver birch wood in the form of sawn timber and woodturning blanks with h ≤ 40 mm, with the temperature of saturated water steam ranging from t = 105 to 135 °C and the time of the process of thermal treatment to τ ≤ 12 hours, is described using the equation:

$$L^*=83.6232+0.4815\cdot t-1.9377\cdot \tau-0.0041\cdot t^2-0.0068\cdot t\cdot \tau+0.1091\cdot \tau^2 \quad (2)$$

The changes in chromatic coordinates: red colour a^* and yellow one b^* in the CIE-L*a*b* colour space of the silver birch wood resulting from the thermal modification of wood colour with saturated water steam, at temperatures of t_I = 105 ± 2.5 °C, t_{II} = 125 ± 2.5 °C, t_{III} = 135 ± 2.5 °C for τ = 3, 6, 9, 12 hours, are illustrated in Fig. 4.

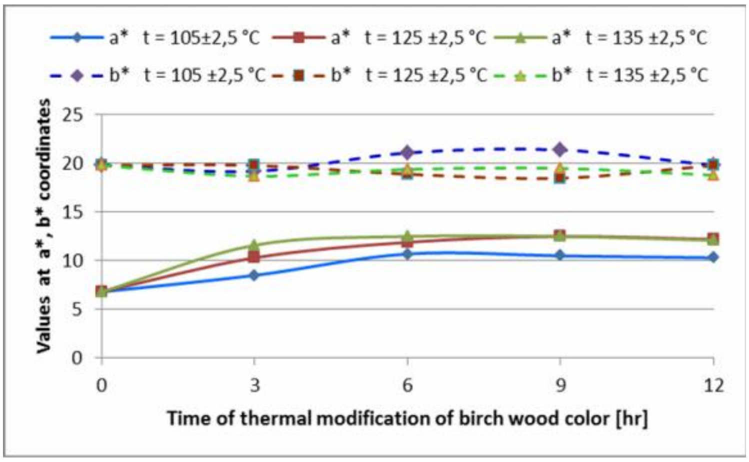

Figure 4. *Coordinates of red colour a* and yellow colour b* in the process of thermal treatment of birch wood*

Dependence of red colour a* and yellow colour b* in the CIE L*a*b* colour space of thermally treated silver birch wood in the form of sawn timber and woodturning blanks with h ≤ 40 mm on temperature of saturated water steam ranging from t = 105 to 135 °C and the time of heat treatment process of τ ≤ 12 hours is described using the equation:

$$a^* = 6.7847-0.0795\cdot t+1.2265\cdot \tau+0.0007\cdot t^2-0.0026\cdot t\cdot \tau-0.0511\cdot \tau^2 \quad (3)$$
$$b^* = 19.8107-0.0014\cdot t +0.7326\cdot \tau-9.3472E-5\cdot t^2-0.0027\cdot t\cdot \tau-0.0255\cdot \tau^2 \quad (4)$$

Changes in colour of thermally treated silver birch wood by the modes with saturated water steam with temperatures of t_I = 105 ± 2.5 °C, t_{II} = 125 ± 2.5 °C, t_{III} = 135 ± 2.5 °C for τ = 3, 6, 9, 12 hours are illustrated using total colour difference ΔE* in Fig. 5.

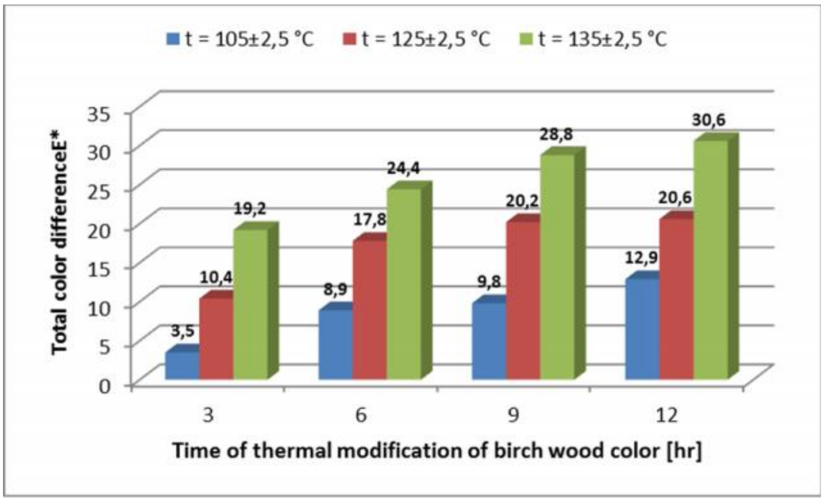

Figure 5. *Values of total colour difference of birch wood in the process of thermal treatment*

4. DISCUSSION

Measured pH values of silver birch in the process of heat treatment confirm the data known about hydrolysis of polysaccharides in wet wood during application of heat. Products of hydrolysis and extraction after wood boiling or other heat treatment processes mentioned in works: *Melcer et al (1989), Kačík (2001), Laurova et al (2004), Samešova, Dzurenda and Jurkovič (2018)* were quantified using hydromodulus or created condensate. Measuring the pH value of wet wood using the potentiometer SENTRON SI 600 with the sensor head LenceFET+H 22704-010 can be considered unique. This way, wood hydrolysis and its effect on wood colour change can be monitored. Following the measured values of acidity of wet wood resulting from given modes of heat treatment of silver birch wood, one can state that temperature affects hydrolysis of hemicelluloses and the change in the chromophore system of wood more significantly than the time of heat treatment process.

The colour of silver birch in the process of heat treatment with saturated water steam changes from light white-brown to light brown at temperature of saturated water steam of t = 105±2,5 °C and time of heat treatment of τ = 6 – 12 hours, through brown hues resulting from application of saturated water steam with temperature of t = 125±2.5 °C for 6 to 12 hours, up to brown colour of silver birch wood at temperature of t = 135± 2.5 °C for τ = 9 – 12 hours of heat treatment.

Following the visual control of wood colour on the edges of silver birch woodturning blanks, as well as following the measurement of individual chromatic coordinates, one can establish the fact that the colour throughout the thickness of woodturning blanks is coloured equally. Hydrolysis and wood colour modification processes can occur especially during relatively fast heat treatment process of woodturning blanks or sawn timber with thickness of h ≤ 40 mm with saturated water steam to required temperature throughout the thickness of the wood *Deliiski (2003, 2007, 2011), Dzurenda (2018).* Following the mentioned fact, thermally treated sawmill products can be used to produce lamellae for flooring or other 3D processing of solid timber without any change in colour in the cross section of wood emerged.

Colour changes in silver birch wood ΔE* resulting from heat treatment with saturated water steam with temperature of t = 105±2.5 °C at time of thermal modification for up to 3 hours are considered not significant changes in colour, not visible with naked eye. Following the conditions of heat treatment of silver birch at temperature of saturated water steam of t = 125± 2.5 °C with colour difference: ΔE* = 17.8 ÷ 20.6, silver birch wood is classified as wood changing the colour to brown hues visible with naked eye. The colour of silver birch resulting from heat treatment with saturated water steam with temperature of t = 135±2.5 °C for more than 6 hours is unique rich brown.

A decrease in lightness of silver birch wood thermally treated with saturated water steam and an increase in the values of total colour differences ΔE* in the CIE-$L^*a^*b^*$ colour space is in compliance with the knowledge about colour changes of wood in heat treatment processes by steaming presented in the works: *Molnar and Tolvaj (2002), Dzurenda (2014, 2018),* as well as by high temperature drying in the environment of overheated water steam *Klement and Marko (2009),* or by heat treatment processes during thermowood manufacturing (*Barcik, Gašparík and Razumov (2015).*

Irreversible colour change of silver birch wood resulting from the process of thermal treatment – colour modification of wood with saturated water steam, increases the possibility for using birch wood in the field of construction for production of flooring, panelling and solid wood furniture or toys.

5. CONCLUSION

This paper presents the results of experimental monitoring acidity and color change of birch wood in the process of thermal treatment of wood by saturated water vapor in temperature range T = 105-135 ° C for τ 3-12 hours.

Wet birch wood changes pH in the range of pH = 5.3 ÷ 3.2 due to partial hydrolysis of hemicelluloses . Wood acidity measurement was performed with a SENTRON SI 600 potentiometer with LenceFET + H probe 22704-010. The values measured indicate that the temperature factor has a more pronounced influence on the size of hemicellulose hydrolysis and the change of the chromophore system of the wood than the thermal treatment time.

Birch wood in the process of thermal treatment loses whiteness (gets darker) and changes colours from pale brown to brown. The coordinates of the colour of birch wood in the coloor space CIE L * a * b * depending on the temperature of saturated water vapor t and the time of thermal modification of the colour τ describe the equations:

$$L^* = 83{,}6232+0{,}4815\cdot t-1{,}9377\cdot \tau -0{,}0041\cdot t^2-0{,}0068\cdot t\cdot \tau +0{,}1091\cdot \tau^2,$$
$$a^* = 6{,}7847-0{,}0795\cdot t+1{,}2265\cdot \tau+0{,}0007\cdot t^2-0{,}0026\cdot t\cdot \tau-0{,}0511\cdot \tau^2,$$
$$b^* = 19{,}8107-0{,}0014\cdot t +0{,}7326\cdot \tau-9{,}3472E\text{-}5\cdot t^2-0{,}0027\cdot t\cdot \tau-0{,}0255\cdot \tau^2.$$

The aforementioned modifications of the color of birch wood achieved by the thermal process of saturated water vapor extend the possibilities of using colour scales birchwood in the construction-firearm area for production of flooring, cladding, as well as structural-artistic design in the manufacture of solid wood furniture, toys or for other usages and decorative objects.

ACKNOWLEDGMENTS
This paper was prepared within the grant project: APVV 17-0456, as a result of work of the authors and considerable assistance by the APVV agency.
We also thank the IPA TUZVO project agency, which has contributed to some extent to the creation of this contribution through the project: IPA TUZVO 15/2019.

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