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SELECTED PHYSICAL PROPERTIES OF THERMALLY MODIFIED SPRUCE WOOD

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Abstract:

Thermal modification of wood does not only change the color of the wood, it also changes wood properties that are closely related to its reaction to water (changes in dimensions, weight, density,...). These properties of thermally modified wood play an important role, limiting the use of thermally modified wood itself. These changes may be positive or negative depending on the wood species, thermal modification mode, the temperature used during the modification and other factors.

The aim of this article is to broaden and collect knowledge about the effect of different thermal modification temperatures (160°C, 180°C and 210°C) on selected physical characteristics of spruce wood.

The results of the study show that thermal modification at a temperature range of 160°C – 210°C has a statistically significant effect on the absorbability of thermally modified wood, as well as on tangential and volumetric swelling. Conversely, the effect of thermal modification on longitudinal swelling and a change in density during absorption was not proven.

Key words: *absorption; density; thermally modified wood.*

INTRODUCTION

Modifying wood with high temperatures is currently considered a suitable way to regulate the resulting physical properties of wood. Thermal modification eliminates the application of toxic agents necessary to improve wood resistance, and it improves dimensional stability, thereby reducing the hygroscopic behavior of the material, but the density of the wood also decreases. High-temperature treatment of material can be considered a method of wood modification in which the wood's color can be modified (Romagnoli et al. 2007).

In terms of the practical use of thermally modified wood, its resulting physical properties are considered to be an important factor (Barcík 2015), and the color as such can be an indicator of the quality of the thermally modified wood, determining its final use in the market (Boonstra 2008). During thermal modification, the wood darkens and acquires a different shade. In the past, the thermal modification process has been applied to remove unwanted color differences between the sapwood and the heartwood, or to remove stains that form during wood steaming (Tolvaj and Faix 1996). The color characteristics depend mostly on the specific chemical composition of the wood in interaction with light (Hon and Shiraishi 2001). Dry spruce wood is chemically composed of cellulose (40-50%), hemicelluloses (25-30%), lignin (25-30%), and extractives (3-10%). These proportions are only approximate, and they are influenced by multiple factors (Fengel and Wegener 2003). Initial changes in physical properties due to thermal modification begin to manifest themselves at 150°C (Gunduz et al. 2008). Changes in wood properties are primarily due to the

degradation of hemicelluloses and the production of water, carbon dioxide, formic acid, acetic acid, and other substances that may be involved in condensation reactions, thereby forming chromophore groups (Hakkou et al. 2005). These chemical reactions resulting from high temperatures significantly reduce tangential and radial swelling (Gunduz et al. 2009), reduce the moisture content and water absorption of the wood (Kortelainen 2011), and also affect the fire resistance of the spruce wood (Čekovská et al. 2017).

Significant changes in all physical properties of thermally modified wood are observed at temperatures around 180°C – 250°C (Patzelt et al. 2003). At these high temperatures, a high dimensional stability of 55 - 90% was found. In spruce wood, a weight loss of 16.1% was found at a temperature of 210°C (Gunduz et al. 2009). The change in color, moisture absorption, weight and dimensions is not only affected by the applied temperature, it is also greatly influenced by the period over which the wood is subjected to the temperature (Patzelt et al. 2003). At 250°C, the process of wood carbonization begins, resulting in the formation of carbon oxides along with other substances (Kačíková et al. 2008).

OBJECTIVE

The objective of the study was to determine the effect of the thermal modification temperature the weight, volume, density and dimensional changes during absorption at various thermal modification temperatures.

MATERIAL, METHOD, EQUIPMENT

Spruce test specimens (Picea abies L.) were used for the research. The wood came from the Poľana region in Slovakia. The research consisted of two sets of test specimens:

- A set of test specimens not subjected to thermal modification (S-20°C).
- A set of thermally modified test specimens (S-160°C, S-180°C, S-210°C).

The following effects were monitored:

- The effect of the thermal modification on dimensional and volumetric swelling, absorbability and density of the wood during the absorption process.

To assess the swelling, we used 20x20x30 (h x w x l) test specimens (Fig.1).

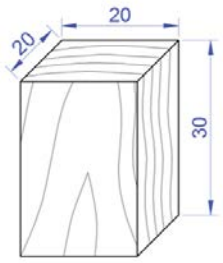

Fig. 1.
Test specimens for swelling evaluation.

The thermal modification process was carried out according to the following steps:

1. Heating and drying – In this stage, the temperature increases rapidly in an oven at about 100°C to support the action of the steam. Then, the pitch decreases and increases to a level of 130°C. The drying medium is hot air or hot steam. Throughout this phase, the wood is dried to approximately zero moisture content.
2. Thermal modification – In the second stage, the temperature is raised to 185-230°C for 2-3 hours. The height of the temperature and duration of action are determined by the requirements for the class of THERMOWOOD products (Thermo-S and Thermo-D)
3. Refrigeration and air conditioning – In the third phase, the thermally modified wood is gradually cooled to a temperature of 80-90°C, and the humidity is stabilized so that the final moisture level is in a normal range of 4 - 7%.

The parameters of the thermal modification of the test specimens are shown in Table 1, and the thermal modification mode is shown in Fig. 2.

Table 1

Input technological parameters and thermal modification process

	Input technological parameters		
Wood moisture content	2 to 4 %		
Filled kiln capacity	0.8 m^3		
Maximum temperature achieved	210 °C		
	Thermal modification process		
	160 ° C	**180° C**	**210° C**
Heating	6.3 Hours	4.5 Hours	4.8 Hours
Thermisation	4.4 Hours	5.3 Hours	6.7 Hours
Cooling	1.7 Hours	2.4 Hours	4.2 Hours
Total modification time	**12.4 Hours**	**12.2 Hours**	**15.7 Hours**

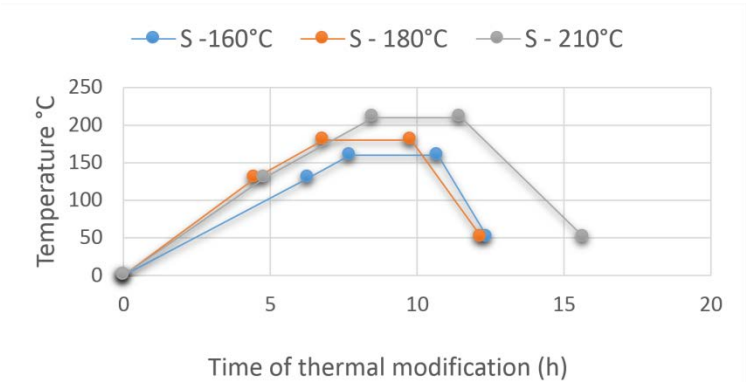

Fig. 2.
The thermal modification process specimens (S-210°C, S-180°C, S-160°C).

METHODS

Absorption

The absorption indicators were values of selected characteristics measured on test specimens subjected to thermal modification. These measured values were compared with the values measured on test specimens that were not subjected to thermal modification. The following characteristics were monitored:

- Dimensional gain in individual wood directions (tangential, radial and longitudinal).
- Volumetric gain
- Weight gain
- Density gain over time, from dry state to full water saturation of the wood.
- Wood moisture content over time, from absolute dry state to full water saturation of the wood.

We determined the increase in individual monitored characteristics by measuring them at certain time intervals (0hr., 0.06hr., 0.2hr., 1hr., 3hrs., 6hrs., 12hrs., 24hrs., 48hrs., 72hrs., 96hrs.). The density of the measured materials is higher at the beginning of the measurement due to the faster reaction of the material to the moisture change (Fig. 4).

EVALUATION AND CALCULATION

Increase in monitored characteristics

The total and partial swelling of the wood over time was carried out in accordance with **ČSN 49 0104:** 1988. In order to determine the wood swelling values, the specimens were submerged in buckets filled with water, weighed down and left for 96 hours.

$$\beta_t = \frac{\beta_o - \beta_{wt}}{\beta_o} (\%) \quad (1)$$

where: β_t indicates the gain percentage in each direction depending on the monitored time, β_o is the dimension of the specimen in absolutely dry state, and β_{wt} specifies the dimensions of specimens in individual directions after each interval has elapsed.

We recorded the weight and dimensions in each direction (radial, tangential and longitudinal) at the given time intervals. We then used the measured data to calculate the increase in the monitored value over time Eq. 2,

For the completeness of the values needed to calculate the swelling over time and the overall swelling, the results from the density measurements at 0% moisture content were used. Eq. X.

$$P_1 = Ch_{t2} - Ch_{t1} \quad (2)$$
$$P_2 = Ch_{t3} - Ch_{t1}$$
$$P_3 = Ch_{t4} - Ch_{t1}$$

where: $P_{(1,2,3,.....)}$ is the increase in the monitored characteristic over time t_1, t_2, t_3, $Ch_{t(1, 2, 3, ...)}$ is the value of the monitored characteristic over time t_1, t_2, t_3, and Ch_{t1} is the value of the monitored characteristic at the beginning of the measurement, i.e. at t_1. The increments were measured until the limit of hygroscopicity was reached - the increments were measured at predetermined intervals for 96 hours.

We used this method to measure and calculate:

1. The weight gain (g) after 96 hours of soaking.
2. The volumetric gain (mm3) after 96 hours of soaking.
3. The density gain (kg.m3) after 96 hours of soaking.
4. The radial gain (mm) after 96 hours of soaking.
5. The tangential gain (mm) after 96 hours of soaking.
6. The longitudinal gain (mm) after 96 hours of soaking.

ANOVA (Fisher's F – test) and Duncan's multiple comparison tests were used to evaluate the statistical significance of individual factors. A 0.05% level of significance was used for all statistical analyses. Statistical analyses were performed using STATISTICA 12 (Statsoft Inc., USA).

The wood density was determined according to ISO 13061-2 (2014) after drying and after 96 hours. Eq. 3:

$$\rho_w = \frac{m_w}{a_w * b_w * l_w} = \frac{m_w}{V_w}, \quad (3)$$

where: ρ_w is the sample density at a certain moisture content w (kg/m^3), m_w is the sample mass at a certain moisture content, w (kg), a_w, b_w, and l_w are the sample dimensions at a certain moisture content w (m), and V_w is the sample volume at a certain moisture content w (m^3).

The moisture content in the samples was determined according to ISO 13061-1 (2014) and Eq. 4,

$$w = \frac{m_w - m_0}{m_0} * 100 \quad (4)$$

where: w is the moisture content of the sample (%), m_w is the sample mass at a certain moisture content w (kg), and m_0 is the sample mass in a dry state (kg).

RESULTS AND DISCUSSION

Increase in monitored characteristics

Table 2 shows the average values of increments in the monitored characteristics over time, from dry state to their complete saturation with water.

Table 2

Mean values of increments in monitored characteristics after 96 hours

Wood Species	Thermal treatment (°C)	Weight gain (g)	Volumetric gain (mm^3)	Density gain (kg.m3)	Radial gain (mm)	Tangential gain (mm)	Longitudinal gain (mm)	Density (Kg/m^3)
Spruce	**20**	5.6 (7,1)	2208.1 (4.5)	326 (10.3)	2.1 (7.2)	3.3 (4.5)	1.1 (8.0)	445 (5.39)
Spruce	**160**	6.1 (18,7)	2515.7 (9.6)	336 (21.6)	2.2 (6.4)	3.4 (4.3)	1.4 (3.3)	449 (4.45)
Spruce	**180**	4.8 (12,9)	1956.2 (12.8)	313 (22.0)	1.9 (11.6)	2.9 (4.5)	1.2 (2.8)	467 (1.96)
Spruce	**210**	4.3 (12,2)	1663.3 (14.6)	268 (11.2)	1.9 (16.8)	2.5 (5.6)	1.1 (3.6)	461 (4.94)

Values in parentheses are coefficients of variation (CV) in %

Based on the significance level "p", we can say that a statistically significant effect of the thermal modification can be observed in the following characteristics: weight gain, volumetric gain, radial and tangential dimensional gain (Tab. 3). The density gain and the longitudinal dimensional gain are characteristics on which the thermal modification has no statistically significant effect.

Table 3

Statistical evaluation of the effect of thermal modification on the increase in monitored characteristics

Weight gain (g) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	1088.058	1	1088.058	2050.755	***
Temperature of Thermal treatment (TT)	19.468	3	6.489	12.231	***
Error	19.100	36	0.531		
Volumetric gain (mm^3) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	174030679	1	174030679	3668.397	***
Temperature of Thermal treatment (TT)	3951034	3	1317011	27.761	***
Error	1707859	36	47441		
Density gain ($kg.m^3$) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	3866491	1	3866491	890.6155	***
Temperature of Thermal treatment (TT)	27580	3	9193	2.1176	NS
Error	156289	36	4341		
Radial gain (mm) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	164.187	1	164.187	3451.443	***
Temperature of Thermal treatment (TT)	0.798	3	0.266	5.590	***
Error	1.713	36	0.048		
Tangential gain (mm) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	370.090	1	370.090	18214.322	***
Temperature of Thermal treatment (TT)	5.019	3	1.673	82.340	***
Error	0.731	36	0.020		
Longitudinal gain (mm) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	57.701	1	57.701	1115.184	***
Temperature of Thermal treatment (TT)	0.170	3	0.057	1.098	NS
Error	1.863	36	0.052		

NS- not significant, ***- significant, Significance was accepted at P < 0.01

For the purpose of a deeper analysis of the effect of thermal modification on the monitored characteristics, we used Duncan's multiple comparison test; the results are shown in Table 4.

It is clear from the results that no statistically significant difference was found between the values of weight gain measured in the set of specimens modified at 160°C and the set of test specimens not subjected to thermal modification (20°C) (P = 0.172). A statistically insignificant difference (P = 0.170) in the measured weight gain values was also found between the values measured in sets of test specimens subjected to thermal modification at 180°C and 210°C.

The analysis of the effect of the thermal modification on volumetric gain values showed a statistically significant difference between all the monitored degrees of thermal modification (Tab. 4).

The results of the one-factor analysis of variance evaluating the effect of the thermal modification on the density gain listed in Table 3 show that the thermal modification does not have a statistically significant effect on the values of this characteristic. The results of Duncan's test shown in Table 4 indicate that there is

a statistically significant difference between sets of test specimens modified at 160°C and 210°C, with a significance level of p = 0.038.

The results of Duncan's test show that there is no statistically significant difference between the radial gain values measured in sets of test specimens modified at 160°C, 180°C and without thermal modification (20). A statistically significant difference was found between the values of untreated specimens and sets of test specimens modified at 210°C (p = 0.014). A significant difference was also confirmed between the values measured in different sets of test specimens (160°C – 180°C, p = 0.011; 160°C – 210°C, p = 0.002).

Thermal modification has a statistically very significant effect on the tangential gain; a statistically significant difference between all the sets of test specimens was found, with a significance level of p = 0.02 or less.

The longitudinal gain was not affected by the thermal modification in any of the monitored cases of thermal modification.

Table 4

Comparison of the effects of individual factors on the values of the gain of monitored characteristics using Duncan's test

Weight gain (g) after 96 hours					
Thermal treatment (°C)		**(1) 5.6480**	**(2) 6.1020**	**(3) 4.7840**	**(4) 4.3280**
S	**20**		0.172	0.012	0.000
S	**160**	0.172		0.000	0.000
S	**180**	0.012	0.000		0.170
S	**210**	0.000	0.000	0.170	
Volumetric gain (mm^3) after 96 hours					
Thermal treatment (°C)		**(1) 2208.1**	**(2) 2515.7**	**(3) 1956.2**	**(4) 1663.3**
S	**20**		0.003	0.014	0.000
S	**160**	0.003		0.000	0.000
S	**180**	0.014	0.000		0.005
S	**210**	0.000	0.000	0.005	
Density gain ($kg.m^3$) after 96 hours					
Thermal treatment (°C)		**(1) 326.44**	**(2) 336.47**	**(3) 312.91**	**(4) 267.79**
S	**20**		0.736	0.649	0.067
S	**160**	0.736		0.458	0.038
S	**180**	0.649	0.458		0.135
S	**210**	0.067	0.038	0.135	
Radial gain (mm) after 96 hours					
Thermal treatment (°C)		**(1) 2.1150**	**(2) 2.2060**	**(3) 1.9320**	**(4) 1.8510**
S	**20**		0.357	0.069	0.014
S	**160**	0.357		0.011	0.002
S	**180**	0.069	0.011		0.412
S	**210**	0.014	0.002	0.412	
Tangential gain (mm) after 96 hours					
Thermal treatment (°C)		**(1) 3.2770**	**(2) 3.4330**	**(3) 2.9490**	**(4) 2.5080**
S	**20**		0.020	0.000	0.000
S	**160**	0.020		0.000	0.000
S	**180**	0.000	0.000		0.000
S	**210**	0.000	0.000	0.000	
Longitudinal gain (mm) after 96 hours					
Thermal treatment (°C)		**(1) 1.1010**	**(2) 1.2830**	**(3) 1.2190**	**(4) 1.2012**
S	**20**		0.110	0.282	0.331
S	**160**	0.110		0.533	0.455
S	**180**	0.282	0.533		0.862
S	**210**	0.331	0.455	0.862	

The weight gain of test specimens with different thermal treatment is shown in Fig. 3. The graph clearly shows that there was a slight statistically insignificant increase in values by 9%, due to the effect of thermal treatment at 160°C, compared to the values measured in the set of specimens that were not subjected to thermal modification. In contrast, Li et al. (2011) achieved a slight increase in the weight gain of

wood thermally modified at 180°C, which was also statistically insignificant. Thermal modification at 180°C reduced the weight gain by 14.3% in comparison with untreated test specimens, and in specimens thermally modified at 210°C the weight gain values decreased by up to 23.3%. Increasing temperatures during thermal modification leads to a decrease in the water absorption capacity of spruce wood (Metsä-Kortelainen et al. 2006). The lowest weight values at temperatures higher than 200°C have been achieved by many authors (Li *et al.* 2011, Korkut and Guller 2008, Chaouch *et al.* (2010) a Brito *et al.* 2008).

The volumetric gain values (Fig. 4) measured in the monitored sets of test specimens have a similar course to that of the weight gain (Fig. 3). A statistically significant difference between all the sets of test specimens was confirmed. In comparison with the set of untreated test specimens (20°C), we found a 13.7% increase in the values of the monitored characteristic due to the application of 160°C. A decrease of 13.6% was caused by the application of 180°C, and a decrease of up to 22% was caused by the application of 210°C. Dimensional stability is one of the most important properties of wood materials, especially when they are exposed to high-performance conditions. A statistically significant difference in the volumetric gain of thermally modified wood was confirmed by Guller (2012).

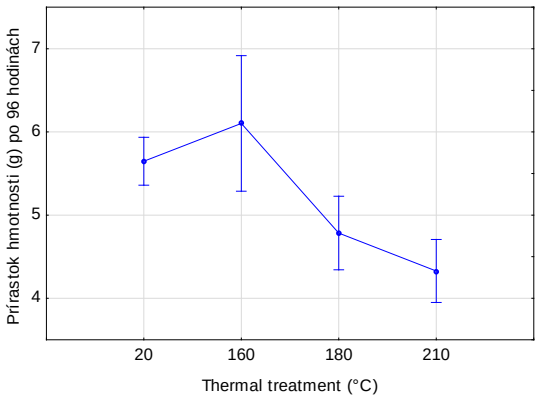

Fig. 3.
The effect of thermal modification on the weight gain during soaking.

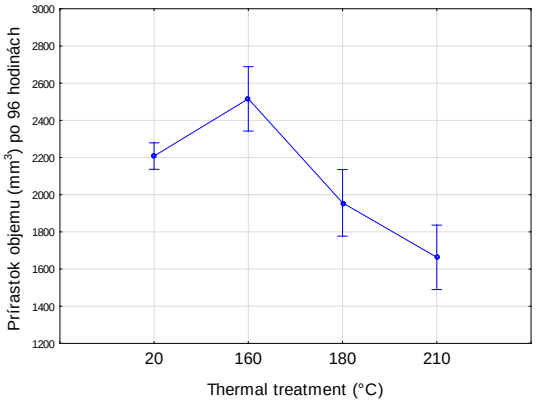

Fig. 4.
The effect of thermal modification on the volumetric gain during soaking.

The results showing the effect of the thermal modification on the density gain are listed in Fig. 5. The values in the graph confirm the results listed in Table 9, as well as the results of Duncan's test (Tab. 4), on the basis of which we can conclude that the density gain is not affected by the thermal modification. Wood density significantly affects the properties of wood (Tsoumis 1991). Other monitored properties shown in Figs. 6, 7 and 8 also have a similar density trend. Guller (2012), Kamdem et al. (2002) and Chaouch et al. (2010) reported a decrease in wood density values with the application of temperatures higher than 200°C.

Fig. 6 shows a decrease in the radial gain values, which is, however, not significant between untreated sets of specimens (20°C) and specimens thermally modified at 160°C, 180°C, based on Duncan's test results. A significant difference was confirmed between 20°C and 210°C sets of specimens. Thermal modification of wood significantly affects the radial swelling of wood. This effect was confirmed by Korkut and Guller (2008), Gündüz et al. (2008).

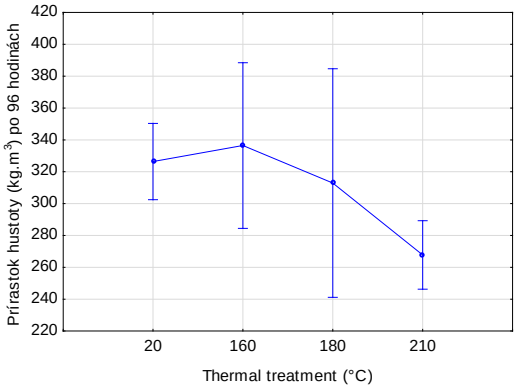

Fig. 5.
The effect of thermal modification on the density gain during soaking.

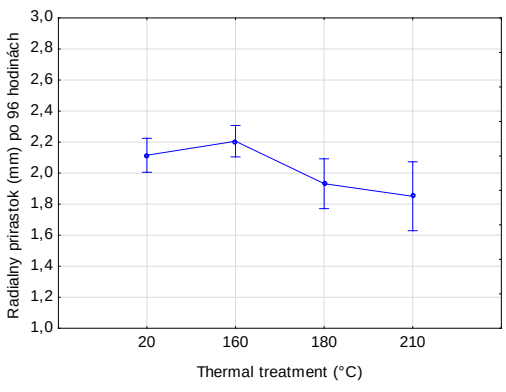

Fig. 6.
The effect of thermal modification on the radial gain during soaking.

Among the monitored characteristics, the tangential gain is most affected by the thermal modification (Fig. 7), which is evident from the results of the one-factor analysis of variance (Tab. 3), as well as the results of Duncan's test (Tab. 4). From a comparison of the tangential swelling and the radial swelling, it is evident that higher swelling values were achieved in the tangential direction. Higher tangential values were achieved by Gündüz et al. (2008). It was also found that as the temperature increases, with the exception of 180°C, the swelling in the tangential direction decreases (Korkut and Guller 2008).

The thermal modification temperature does not affect the values of the longitudinal gain during soaking (Fig. 8). Slight differences in swelling values in the longitudinal direction at different temperatures were also achieved by Gunduz *et al.* (2009).

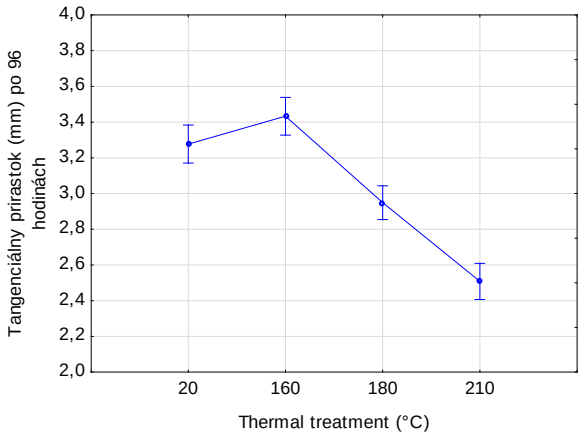

Fig. 7.
The effect of thermal modification on the tangential gain during soaking.

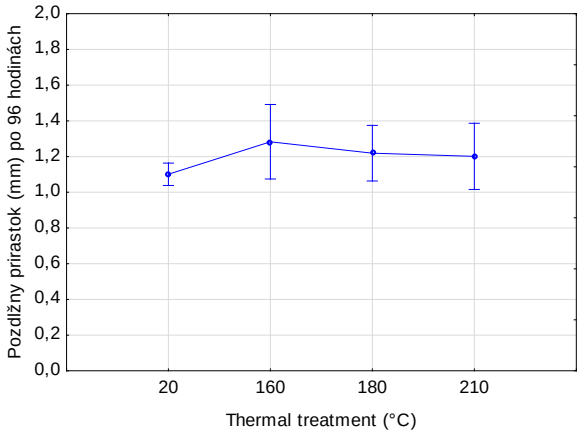

Fig. 8.
The effect of thermal modification on the longitudinal gain during soaking.

Fig. 9 shows the effect of thermal modification on the moisture absorption of wood. The graph shows a clear declining trend in the water absorption in correlation with the increasing temperature of the modification. The highest water absorption values were found in untreated wood, and the lowest moisture content was measured in a set of spruce specimens treated at 210°C. A decrease in water absorption with the increasing temperature of thermally modified wood was also confirmed by Li *et al.* (2011) and Metsä-Kortelainen et al. (2006).

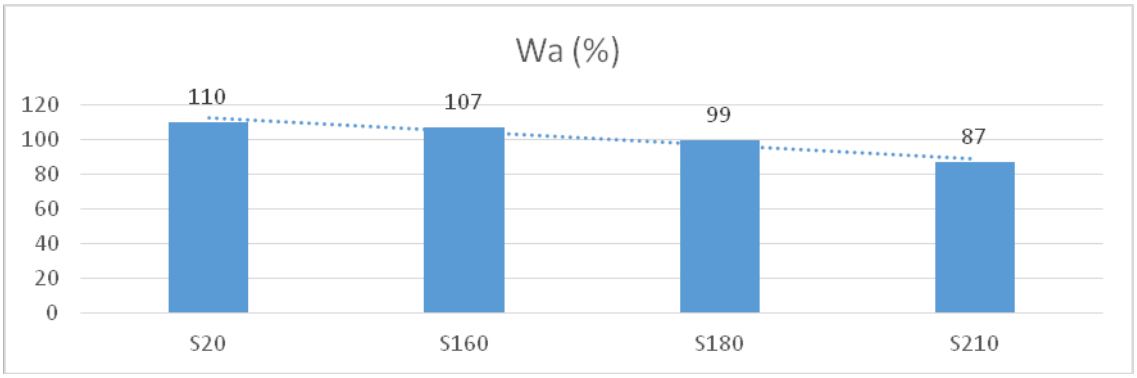

Fig. 9.
The effect of thermal modification on the water absorption.

CONCLUSIONS

1. Different thermal modification temperatures have a statistically significant effect on radial and tangential swelling, but they do not have a statistically significant effect on longitudinal swelling. The effect of thermal modification on volumetric swelling was also confirmed.
2. The effect of the thermal modification on the wood density during the soaking process was not confirmed.

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