Fire Resistance of Thermally Modified Spruce Wood

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The risk of possible ignition and burning is one of the greatest disadvantages of using wood as a construction material. An environmentally appropriate method of improving the fire-resistant properties of wood is available *via* thermal treatment. In this study, spruce wood (*Picea abies* L.) was thermally modified at 160 °C, 180 °C, and 210 °C. The effect of thermal modification on the fire performance of the wood, including weight loss and burn rate, was evaluated. A new testing method was designed to be sufficiently sensitive to monitor fire performance. The results showed that the thermally modified spruce wood had a lower weight loss than untreated wood. The burn rate of wood that was thermally modified at 160 °C was similar to that of untreated wood. Higher thermal treatment temperatures caused a higher burn rate. After the flame was removed, the burning process was rapidly stopped in thermally treated wood.

Keywords: Flammability of construction materials; Burn rate; Spruce; Thermal modification of wood; Burning process

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INTRODUCTION

Wood is currently utilized in many areas, including but not limited to construction and cladding materials, furniture production, as fuel, and for chemical treatments (pulp, paper, bioethanol, *etc.*). Of all the construction materials available, wood has the best weight/bearing ratio, making it possible to build wooden structures in places that are difficult to access. With wooden structures, this material is used in both the exterior and interior. The main disadvantage of wood is its flammability; many attempts have been made to increase its fire resistance. Treatments that change the basic properties of a material are considered modifications. Depending on the purpose of the treatment, the modification can be classified as mechanical (wood densification), physical (different types of radiation (plastification VF, VVF, IR), layering, increasing the moisture content in interaction with heat, plasticization, or chemical (anhydrides, carboxylic acids, isocyanates, epoxides, *etc.*). One popular method of modification is the thermal treatment of wood, which is considered environmentally friendly (Hill *et al.* 2006; Kubovsky and Babiak 2009; Hrčka and Babiak 2012; Gašparík and Barcík 2013, 2014; Svoboda *et al*. 2015; Miftieva *et al*. 2016).

The interest in thermally modified wood has recently increased due to the decreased production of durable raw wood material, the increased interest in durable construction materials, and legislative changes that restrict the use of toxic substances.

Five different modifications that have the greatest commercial significance: Thermowood (Finland), Plato Wood (Netherlands), oil heat treatment wood (OHT; Germany), Bois Perdure (France), and rectification (France). There are new methods being developed in Denmark (Wood Treatment Technology) and in Austria (Huber Holz) (Reinprecht and Vidholdová 2008). While some of these processes are being introduced, others are already in full operation. Multiple wood species are used under different operating conditions, which depend on the wood species and the end product. The treatment temperatures range between 160 °C to 260 °C, but the differences between the individual modifications are the use of a gaseous environment (*i.e.*, nitrogen, steam), ambient humidity, use of oils, *etc.* Wood modified in this way has more favorable properties for its use in exteriors and interiors, such as its dimensional stability, durability, color change, *etc.* (Reinprecht and Vidholdová 2008; Esteves and Pereira 2009; Baysal *et al.* 2014).

The main disadvantage of thermally modified wood is the deterioration of its mechanical properties, which limits its use in certain applications, particularly as a construction material. The thermal changes depend on the wood species and modification conditions. Some properties deteriorate (impact strength, rigidity, hardness, *etc.*), and some improve (modulus of elasticity, hardness). The degradation of hemicellulose is probably the most important factor affecting the mechanical properties of thermally modified wood, particularly the flexural and tensile strength. However, changes in the crystalline and amorphous proportion of cellulose can also greatly affect the mechanical properties of treated wood. After thermal treatment, spruce wood exhibits decreased mechanical properties and cellulose, especially hemicellulose (Yildiz *et al*. 2006; Kacik *et al*. 2015). Multiple authors have published the correlation between the hemicellulose content and the flexural strength (Winandy and Lebow 2001; Esteves *et al*. 2008). There are correlations between strength properties, hemicellulose content, degree of polymerization, and cellulose crystallinity in thermally treated spruce wood (Kacikova *et al*. 2013).

The effect of thermal treatment on the anatomical, mechanical, physical, biological, and chemical properties of wood has been the subject of many studies; however, knowledge about the fire performance of this material are always beneficial for scientists interested in the topic. The fire performance of wood is especially important in wood constructions (Tewarson 1994; Yinodotlgor and Kartal 2010). Although there are studies dealing with the burning of Thermowood (Martinka *et al.* 2013a), information about the fire-resistant treatment of Thermowood and the effect of these modifications on its properties is missing. There are standard procedures specified in European standards for testing construction materials and their reaction to fire. Before universal testing methods for the EU were introduced, each country used its own regulations such as BS 476.6 (1968), DIN 4102 (1998), and CSN 73 0862 (2003). Each testing method used different test parameters, including different sizes of samples, different exposure times to the heat source, and different types of heat sources (flame, radiant, or a combination thereof). These differences made it difficult to compare the results. The current methods for determining the reaction to fire are valid for EU member states. They are necessary to determine the reaction to fire, but for experimental studies, they are quite difficult and impractical for monitoring the parameters of differently treated wood.

In this study, a simple apparatus that gives meaningful results with the selected evaluation criteria has been developed to create a new way of evaluating thermally modified wood. This method simulates the natural burning process of wood with a flame source, a sustained air flow, and free flow of flue gas. The aim of this study was to verify a new method determining the fire performance of thermally modified spruce wood.

EXPERIMENTAL

Materials

The research consisted of two sets of test samples. The first set consisted of samples of spruce (*Picea abies* L.) treated at 160 °C, 180 °C, and 210 °C (samples S-160, S-180, and S-210); the second set of test specimens consisted of untreated spruce wood (labelled S-20). The thermal modification process was performed according to Fig. 1. First the samples were heated and dried. In this stage, the temperature increases rapidly in an oven at about 100 °C to support the action of steam. Then, the temperature increases to 130 °C. The drying medium was hot air or hot steam. Throughout this phase, the wood was dried to approximately to zero moisture. The next stage was thermal modification. The temperature was raised to 185 °C to 230 °C for 2 h to 3 h. The temperature and duration of action are determined by the requirements of the Thermowood products (Thermo-S and Thermo-D) (Czech University of Life Sciences, Prague, Czech Republic). The final phase was refrigeration and air conditioning. The thermally modified wood was gradually cooled to 80 °C to 90 °C, and the humidity was stabilized so that the final moisture level was 4% to 7%. The technological parameters are shown in Table 1. Specimens measuring 200 mm × 100 mm × 20 mm (length × width × thickness) were used in the experiment (Figure 2). The average density of the test specimens is shown in Table 2. For each set of test samples were used six test samples.

Table 1. Input Technological Parameters and Thermal Modification Process

Input Technological Parameter	**Value**		
Wood Moisture	2% to 4 %		
Filled Kiln Capacity	0.8 m^3		
Maximum Temperature Achieved	210 °C		
Thermal Modification Cycle	**Process Time (h)**		
	160 °C	180 °C	210 °C
Heating	6.3	4.5	4.8
Thermal treatment	4.4	5.3	6.7 H
Cooling	1.7	2.4	4.2
Total Modification Time	**12.4**	**12.2**	**15.7**

Table 2. Average Density Values

Before Thermal Modification				
Test Samples	**S 20**	**S 160**	**S 180**	**S 210**
Density (kg m^{-3})	447	443	447	442
Moisture Content (%)	2.9	7.3	8.3	8.4
After Thermal Modification				
Test Samples	**S 20**	**S 160**	**S 180**	**S 210**
Density (kg m^{-3})	447	645	452	430
Moisture Content (%)	2.9	2.8	2.8	2.8

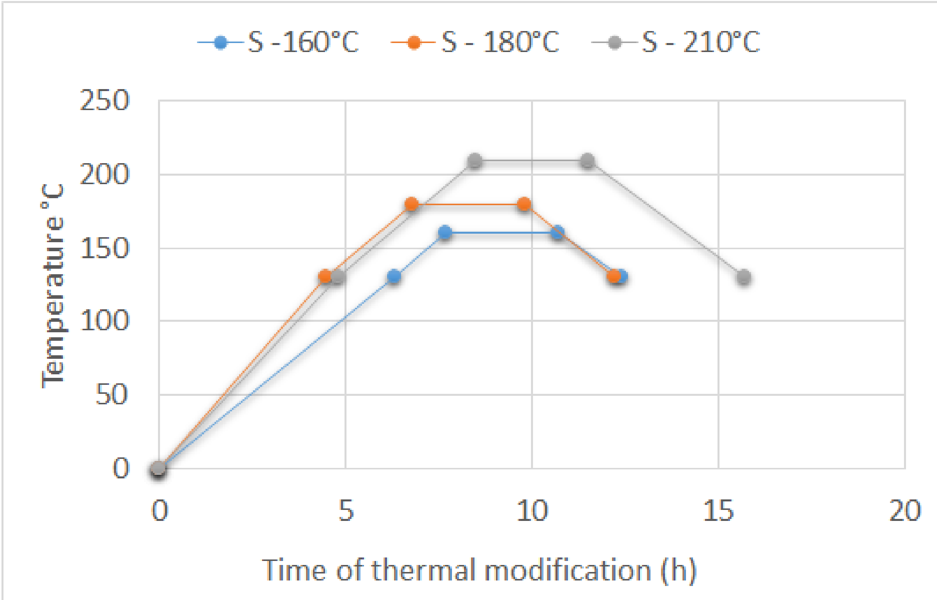

Fig. 1. The thermal modification process specimens (S-210, S-180, S-160, and S-20)

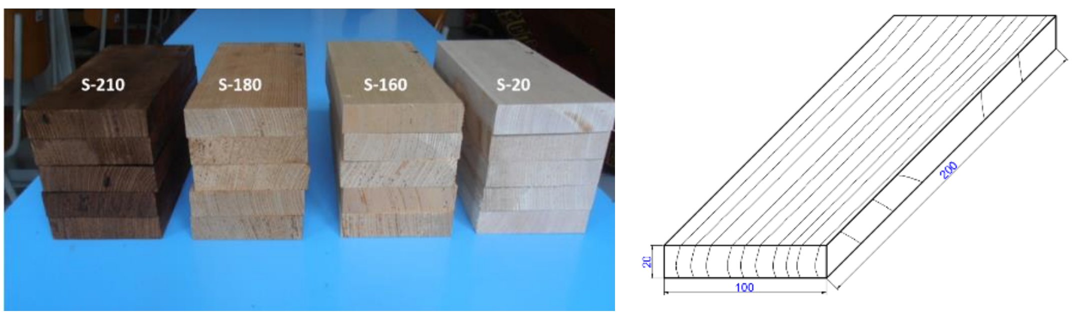

Fig. 2. The test specimens (S-210, S-180, S-160, and S-20)

Methods

A new evaluation method to determine the fire performance of thermally treated wood was designed. In this method each test specimen was directly exposed to a propane gas burner for 10 min, as shown in Fig. 3. The test specimen was placed at a 45° angle to the horizontal plane. The size of the flame was 10 cm from the mouth of the burner, and it is placed at the center of the test specimen on the underside. The baseline measurement was carried out for 10 min.

In this experiment, this method was continued for the second part of the test. In the second part the flame was moved further away from the sample, and the weight loss and burn rate were recorded for 5 min. The burning progression was examined after extending the measurement time by 5 min. It was assumed that the degradation of the wood would continue in the red-hot layer of the wood and that re-ignition of the wood was also possible. The method simulates the natural wood burning process with a flame source, a free flow of air, and free flow of flue gases.

A USBEC 1011/1 propane burner was used for the flame source (DIN-DVGW-Reg. Mr. NG-2211AN0133, blasting, Dresden, German) 1,7 kW. The weight was determined on Mettler Toledo scales (MS 1602S/MO1, Mettler Toledo, Geneva, Switzerland). BalanceLink 4.2.0.1 was used (Mettler Toledo, Switzerland) to record the wood weight.

During the fire test, the weight loss was recorded at an interval of 10 s with continuous weighing over the course of 15 min. The weight loss (Eq. 3) and average burn rate (Eq. 4) were determined.

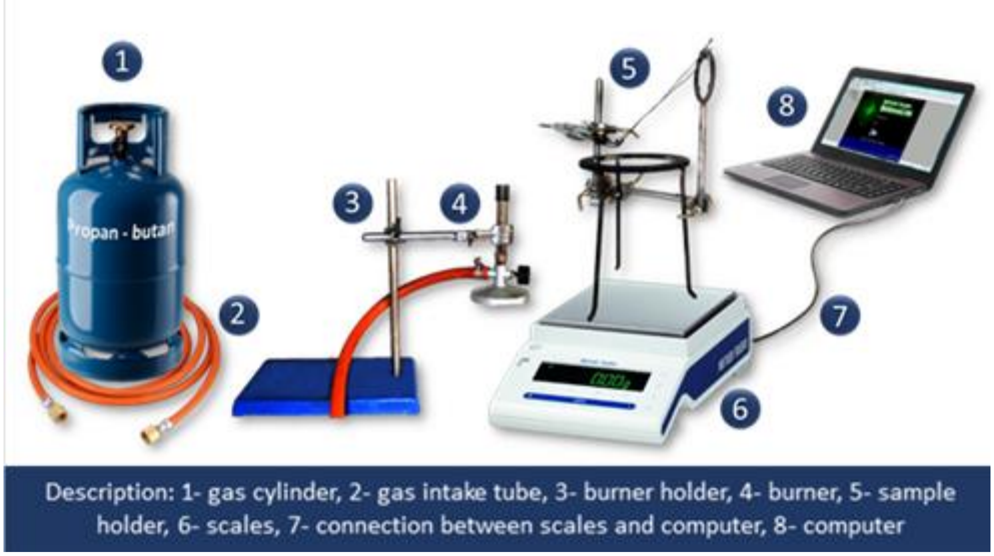

Fig. 3. Fire test device

Evaluation and Calculation

To determine the effect of the individual factors on the bending characteristics, an analysis of variance (ANOVA) and the Fisher F-test were performed using Statistica 12 software (Statsoft Inc., Tulsa, OK, USA).

The wood density was determined before and after testing according to ISO 13061-2 (2014). The moisture content of the samples was determined and verified before and after testing according to ISO 13061-1 (2014). Drying to an oven-dry state was also performed according to ISO 13061-1 (2014).

The main assessment criterion was the weight loss of test samples, which was calculated according to Eq. 1,

$$\Delta m = \frac{m_1 - m_2}{m_1} x100 \tag{1}$$

where Δm is the weight loss (%), m_1 is the sample's weight before the test (g), and m_2 is the sample's weight after the test (g). Based on the percentages calculated using Eq. 3, the materials were placed in different flammability classes, as shown in Table 2. The burn rate was calculated according to Eq. 2,

$$v = \frac{m_t - m_{t+10}}{m_{t0} x10} x100 \tag{2}$$

where v is the burn rate (%/s), m_t is the weight (g) at time t, m_{t+10} is the weight (g) of the sample 10 s later, and m_{t0} is the weight (g) of the sample at time 0.

Additional monitoring characteristic for the assessment of material properties relating to the fire is the ratio of the maximum speed of burning and the time at which the maximum speed reached. If there is a higher ratio value in terms of fire protection value worse. The ratio of the maximum speed was calculated According to Eq. 3,

$$P = \frac{v_{max}}{T_{max}} \tag{3}$$

where P is the ratio of the maximum speed of burning (%), v_{max} is the maximum speed of burning (%.s^{-1}), and T_{max} is the max time to reach the maximum speed of burning (s).

RESULTS AND DISCUSSION

As shown in Table 3, the thermally modified spruce wood had a lower weight loss after burning for 10 min than the untreated spruce wood.

Table 3. Weight Loss at 0 s to 600 s

Time (s)	S-20 Δ *m* (%)	S -160 Δ *m* (%)	S-180 Δ *m* (%)	S-210 Δ *m* (%)	Time (s)	S-20 Δ *m* (%)	S-160 Δ *m* (%)	S-180 Δ *m* (%)	S-210 Δ *m* (%)
0	0.00	0.00	0.00	0.00	310	2.95	2.51	3.27	2.72
10	0.08	0.07	0.08	0.18	320	3.01	2.65	3.33	2.76
20	0.15	0.09	0.10	0.24	330	3.07	2.78	3.37	2.79
30	0.19	0.10	0.11	0.28	340	3.15	2.88	3.42	2.83
40	0.23	0.12	0.13	0.31	350	3.24	2.98	3.48	2.87
50	0.27	0.14	0.14	0.34	360	3.31	3.04	3.54	2.92
60	0.30	0.15	0.16	0.35	370	3.37	3.10	3.60	2.95
70	0.36	0.18	0.17	0.37	380	3.42	3.14	3.64	2.98
80	0,54	0.20	0.19	0.40	390	3.48	3.18	3.68	3.02
90	0.73	0.22	0.23	0.42	400	3.52	3.22	3.72	3.07
100	0.90	0.24	0.36	0.44	410	3.57	3.26	3.74	3.10
110	0.97	0.32	0.54	0.46	420	3.62	3.30	3.78	3.15
120	1.05	0.43	0.71	0.52	430	3.67	3.33	3.81	3.22
130	1.17	0.59	1.00	0.59	440	3.73	3.37	3.83	3.31
140	1.34	0.77	1.34	0.66	450	3.79	3.40	3.86	3.41
150	1.50	0.91	1.67	0.75	460	3.84	3.44	3.89	3.50
160	1.59	1.00	1.92	0.78	470	3.89	3.47	3.91	3.58
170	1.68	1.07	2.06	0.90	480	3.95	3.50	3.94	3.68
180	1.79	1.13	2.18	1.04	490	4.02	3.54	3.97	3.76
190	1.89	1.19	2.29	1.17	500	4.11	3.57	3.99	3.83
200	1.95	1.25	2.39	1.25	510	4.20	3.61	4.02	3.90
210	2.10	1.31	2.46	1.35	520	4.27	3.64	4.05	3.96
220	2.25	1.40	2.51	1.44	530	4.32	3.68	4.07	4.01
230	2.35	1.51	2.55	1.58	540	4.39	3.72	4.10	4.06
240	2.44	1.56	2.66	1.74	550	4.44	3.75	4.14	4.11
250	2.51	1.67	2.79	2.10	560	4.50	3.79	4.19	4.17
260	2.56	1.78	2.90	2.29	570	4.56	3.84	4.25	4.20
270	2.62	1.93	3.01	2.42	580	4,61	3.88	4.32	4.23
280	2.68	2.09	3.08	2.52	590	4.67	3.93	4.40	4.26
290	2.75	2.24	3.15	2.60	600	4.72	3.98	4.48	4.31
300	2.86	2.37	3.21	2.67	-	-	-	-	-

The lowest weight loss (3.98%) was recorded in the spruce wood that was thermally modified at 160 °C, followed by 210 °C with a weight loss of 4.31%, and 180 °C with a weight loss of 4.48%. The unmodified spruce had a weight loss of 4.72% after 10 min of testing. During the test, the weight loss changed irregularly or non-linearly. S-180 exhibited the greatest weight loss during the test; however, the weight loss was eventually lower than in the untreated spruce.

Table 4 shows the second part of the test. From the 10 min to the 15 min mark, there was no after-flame, and the weight loss was minimal. The thermally modified wood had a lower weight loss rate than the untreated wood. The differences in the weight loss between 10 min to 15 min were 0.33% in S-210, 0.46% in S-180, 0.53% in S-160, and 0.54% in the untreated spruce. The assumption that there would be an after-flame in thermally treated spruce was not confirmed.

Table 4. Spruce Weight Loss from 600 s to 900 s

Time (s)	S-20 Δ *m* (%)	S-160 Δ *m* (%)	S-180 Δ *m* (%)	S-210 Δ *m* (%)	Time (s)	S-20 Δ *m* (%)	S-160 Δ *m* (%)	S-180 Δ *m* (%)	S-210 Δ *m* (%)
600	4.72	3.98	4.48	4.31	760	5.16	4.45	4.89	4.62
610	4.76	4.04	4.56	4.36	770	5.17	4.46	4.90	4.62
620	4.81	4.10	4.63	4.40	780	5.18	4.46	4.90	4.63
630	4.87	4.17	4.68	4.44	790	5.19	4.47	4.90	4.63
640	4.92	4.23	4.74	4.49	800	5.20	4.47	4.91	4.63
650	4.95	4.28	4.79	4.54	810	5.21	4.48	4.91	4.63
660	5.00	4.32	4.84	4.58	820	5.21	4.48	4.91	4.63
670	5.00	4.32	4.82	4.56	830	5.22	4.49	4.92	4.63
680	5.04	4.33	4.84	4.58	840	5.23	4.49	4.92	4.63
690	5.07	4.36	4.84	4.59	850	5.23	4.49	4.92	4.64
700	5.09	4.38	4.86	4.59	860	5.24	4.50	4.92	4.64
710	5.10	4.40	4.87	4.60	870	5.25	4.50	4.93	4.64
720	5.11	4.41	4.87	4.61	880	5.25	4.50	4.93	4.64
730	5.12	4.43	4.88	4.61	890	5.26	4.50	4.93	4.64
740	5.14	4.44	4.89	4.61	900	5.26	4.51	4.93	4.64
750	5.15	4.44	4.89	4.62	-	-	-	-	-

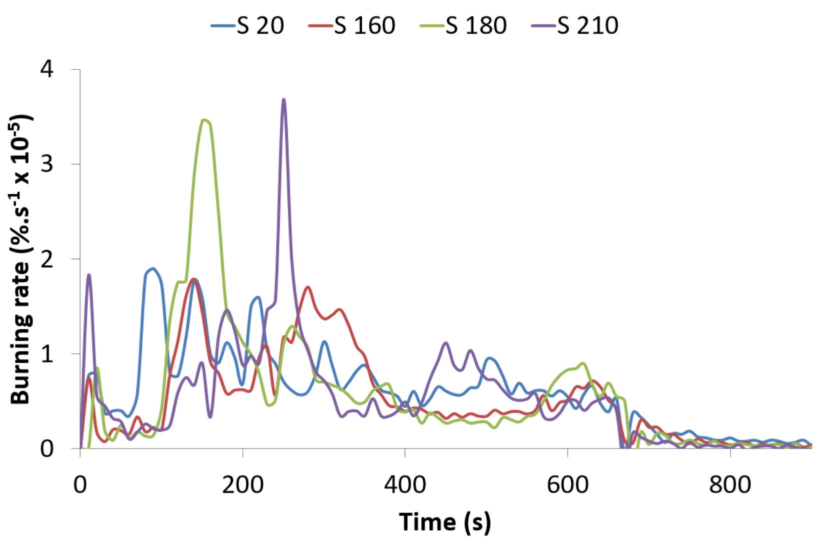

Fig. 4. Graph of burn rate from 0 s to 900 s

The burn rate was determined according to Eq. 4 using the measured weight of the test samples while burning. The highest burn rate was in thermally modified spruce at 210 °C, which started to burn vigorously at 30 s and again at 250 s; Fig. 4 shows two significant peaks.

The highest burn rate in spruce thermally modified at 180 °C was exhibited between 140 s and 160 s of the test. The spruce thermally modified at 160 °C exhibited a burn rate similar to that of the untreated spruce at 20 °C. The last increase in the burn rate took place at 10 min before the flame source was removed, and then the burn rate declined. At 11 min into the test, the burn rate did not change significantly, which means that the wood stopped burning and flaming (Fig. 5).

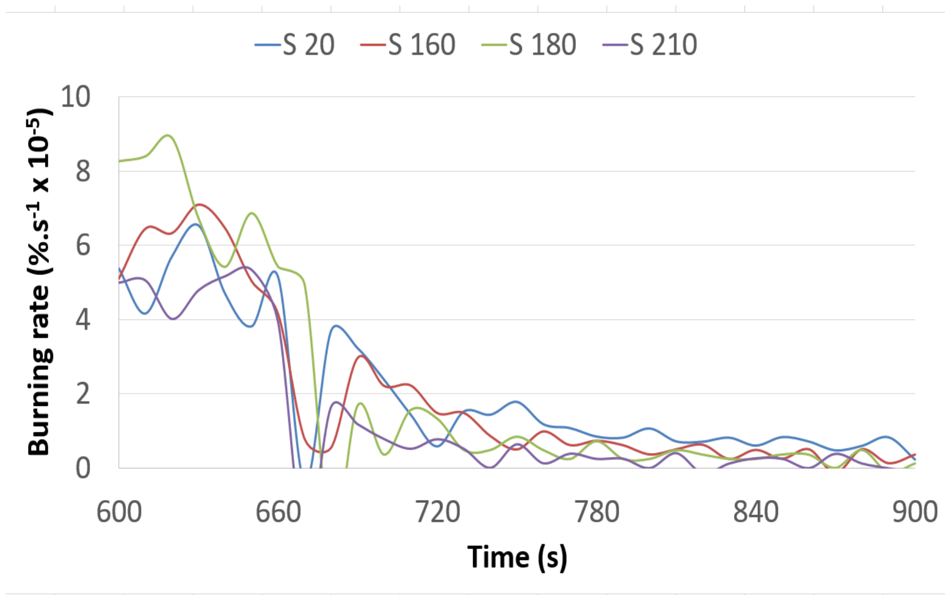

Fig. 5. Graph of burn rate from 600 s to 900 s

The experiment confirmed that the thermal treatment of wood did not have a negative effect on wood properties that were assessed relative to fire protection needs. The measurements displayed in the graphs were the average values from 10 measurements (10 samples). The variability of the average values did not exceed the values that were characteristic of untreated wood.

The present results are consistent with findings of other authors. Martinka *et al.* (2013b) found that thermally modified spruce wood showed a lower maximum heat release rate and also lower average heat release rate, although mentioned parameters were dependent on external heat flux from cone heater.

According to Xing and Li (2014), heat treatment of *Larix* spp. showed positive effects on the fire safety of wood, weakening the intensity of its combustion and also reducing the ignition time.

Table 5 shows the results of ANOVA evaluating the effect of thermal modification of wood on the monitored characteristics. Based on the significance level "P" it can be concluded that the degree of thermal treatment of wood no significant effect on any of the monitored characteristics.

Table 5. Basic Statistical Characteristics Evaluation the Effect of Thermal Treatment on the Values of the Monitored Characteristics

Effect	Sum of squares	Degrees of freedom	Variance	Fisher's F - Test	Significance level P
Weight Loss - 600 s (%)					
Free term	382.042	1	382.042	93.892	0.000
Thermal modification	1.460	3	0.487	0.120	0.947
Error	65.103	16	4.069		
Weight Loss 900 s (%)					
Free term	467.500	1	467.500	105.447	0.000
Thermal modification	1.688	3	0.563	0.127	0.943
Error	70.936	16	4.433		
Burning rate - 600 s (%.s^{-1} x 10^{-5})					
Free term	713.023	1	713.023	21.309	0.000
Thermal modification	39.480	3	13.160	0.393	0.760
Error	535.375	16	33.461		
Burning rate - 900 s (%.s^{-1} x 10^{-5})					
Free term	0.825	1	0.825	8.244	0.011
Thermal modification	0.804	3	0.268	2.678	0.082
Error	1.601	16	0.100		
The maximum speed of burning (%.s^{-1})					
Free term	0.000	1	0.000	44.419	0.000
Thermal modification	0.000	3	0.000	0.713	0.559
Error	0.000	15	0.000		
The time to reach the maximum speed of burning (s)					
Free term	714420.074	1	714420.074	55.136	0.000
Thermal modification	8259.996	3	2753.332	0.212	0.886
Error	207319.931	16	12957.496		
Ratio of the maximum speed of burning (%)					
Free term	0.109	1	0.109	1.000	0.332
Thermal modification	0.328	3	0.109	1.000	0.418
Error	1.747	16	0.109		

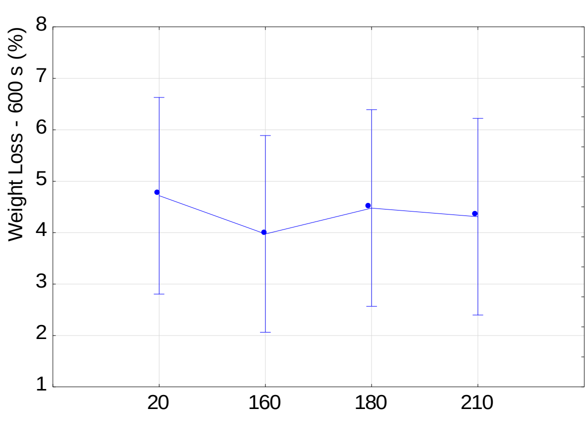

Fig. 6. The effect of the thermal modification on the weight loss in 600 s

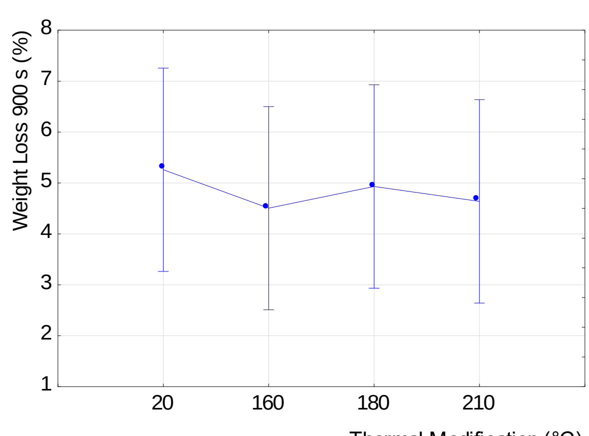

Fig. 7. The effect of the thermal modification on the weight loss in 900 s

Figures 6 to 12 shows the results of thermal impact adjusted to the monitored characteristics. From the results it is clear that none of the monitored performance characteristic is not affected by the thermal treatment of wood.

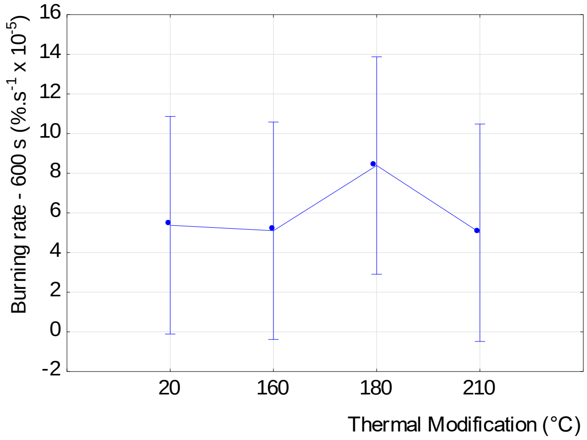

Fig. 8. The effect of the thermal modification on the burning rate in 600 s

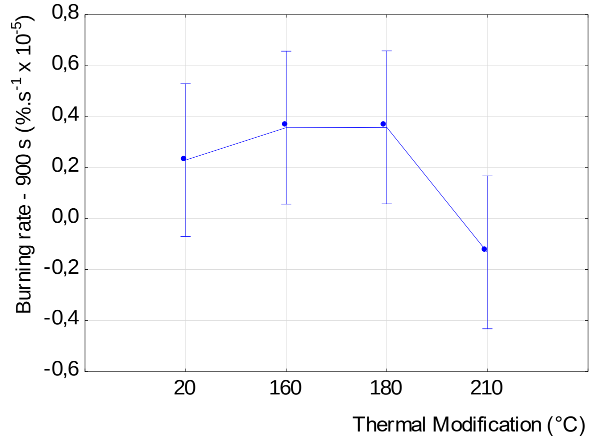

Fig. 9. The effect of the thermal modification on the burning rate in 900 s

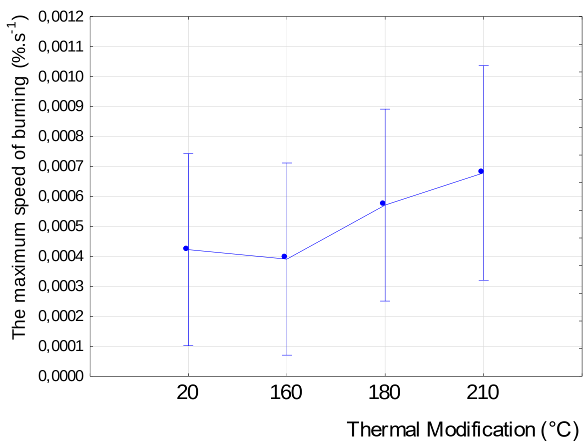

Fig. 10. The effect of the thermal modification on the maximum speed of burning (%.s^{-1})

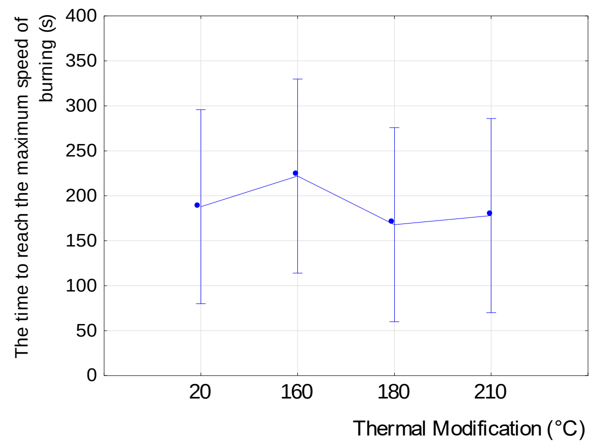

Fig. 11. The effect of the thermal modification on the time to reach the maximum speed of burning (s^{-1})

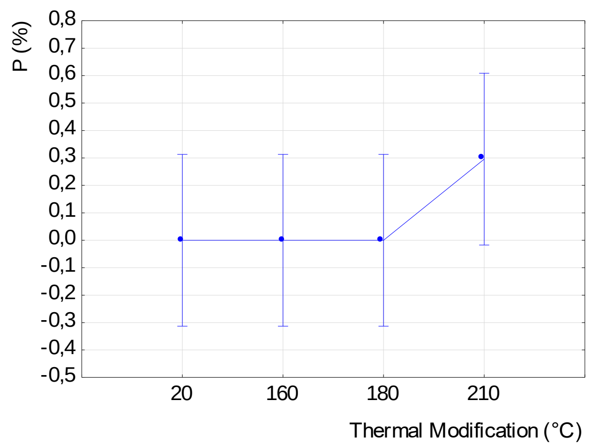

Fig. 12. The effect of the thermal modification on the ratio of the maximum speed of burning (%)

CONCLUSIONS

1. The experiment confirmed the suitability of the laboratory equipment and the method for testing thermally treated spruce wood. This method has only been used to evaluate retardant treatments. The method proved to be sensitive enough to assess the changes that occurred in the thermal treatment of the wood.
2. Open-flame burning caused a lower weight loss in thermally modified spruce wood than in wood that had not been thermally treated. After the direct flame was removed from the wood 10 min to 15 min into the test, there was no after-flame, and the weight loss was minimal, up to 0.54%. Spruce wood thermally modified at 180 °C and 210 °C exhibited the highest burn rate at 2 min to 6 min of burning.
3. The thermally modified spruce wood had a lower weight loss than the untreated wood at all temperatures. The burn rate was higher in thermally treated wood, and it was manifested by vigorous burning 1 min into the test, and again 2 min and 6 min. Higher thermal treatment temperatures resulted in a higher burn rate. After the removal of the flame source, the burning stopped rapidly; the assumption that thermally treated wood would continue to burn was not confirmed. The method used confirmed the adequacy of the laboratory equipment, as well as the method for testing the thermal treatment of spruce wood.
4. Based on the results of statistical analysis, it can be concluded that the thermal treatment does not affect the observed characteristics of fire. Thus, the thermally modified wood is allowed increased flammability and is therefore also suitable for use in timber construction, such as wood that has not undergone thermal prepared.

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