Influence of Temperature of Thermal Modification on the Fire-technical Characteristics of Spruce Wood

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Thermal modification is a widely used wood protection method. This method has attracted attention because there are no toxic chemicals used in the process. The influence of thermal modification was investigated relative to the ignitability and the mass burning rate of Norway spruce wood (*Picea abies*). The spruce wood samples were subjected to temperatures of 100 °C, 150 °C, 200 °C, 220 °C, 240 °C, and 260 °C for durations of 1 h, 3 h, and 5 h. The treatment at temperatures higher than 200 °C resulted in a lower mass loss at 600 s and a lower average relative burning rate, but it did not influence ignition time, the flame-died-out time, and maximum relative burning rate. The class of reaction to fire of the spruce wood samples was not changed due to the treatment. Therefore, it can be stated that the thermal treatment at temperatures below 200 °C does not influence the fire safety of an important class of wooden products.

Keywords: Thermal modification; Norway spruce wood; Mass burning rate; Ignitability

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INTRODUCTION

Unprotected wood after its exposure to outdoor conditions undergoes a variety of degradation reactions caused by diverse environmental factors, such as light, moisture, heat, oxygen, pollutants, pests, *etc*. (Evans *et al.* 1992; Hon 1994; Teacă *et al.* 2013). There are several methods of wood treatment to improve wood properties.

Thermal modification has attracted increased interest because the environmental impact of this process is low (Palanti *et al.* 2011). Heat is introduced to the treatment system and smoke from wood degradation can be retrieved, condensed, and purified (Pétrissans *et al.* 2007). At the end of its life cycle, heat-treated wood can be recycled without a detrimental impact on the environment in contrast to chemically treated wood impregnated with biocidal active ingredients (Candelier *et al.* 2016). It is also known that the environmental credentials of thermally modified wood in terms of ecotoxicity are superior to that of untreated wood and may surpass that of several man-made materials (González-Peña *et al.* 2009). Among the benefits of thermally modified wood are an improved decay resistance (Hakkou *et al.* 2006; Shi *et al.* 2007a; Calonego *et al.* 2010), dimensional stability (Tjeerdsma *et al.* 1998), surface hardness (Gündüz *et al.* 2009; Bakar *et al.* 2013), lower equilibrium moisture content (Esteves *et al.* 2007; Gündüz *et al.* 2008), and darker decorative colour (Bekhta and Niemz 2003; Brischke *et al.* 2007; González-Peña and Hale 2009). The disadvantages of thermally modified wood include the deterioration of some of the mechanical properties, such as the bending and compression strengths (Unsal and

Ayrilmis 2005; Yildiz *et al.* 2006), stiffness, shear strength (Bakar *et al.* 2013), modulus of rupture, and modulus of elasticity (Shi *et al.* 2007b; Esteves *et al.* 2008; Kačíková *et al.* 2013), and the occurrence of mass loss (Alén *et al.* 2002; Esteves *et al.* 2007; Kučerová *et al.* 2016).

According to the International ThermoWood Association (2003), the rate of the heat release level of the heat-treated pine was approximately 10 kW greater than that of the untreated pine, the total heat rate increased approximately 15%, the smoke production was roughly doubled, and the ignition time was shortened 30%. Additionally, it was stated that ThermoWood does not differ remarkably from normal wood when it comes to the fire safety and that ThermoWood has a fire class of D. Changes to all of these characteristics depend on the changes in the chemical composition of the wood (Kačík *et al.* 2015, 2016, 2017; Luptáková *et al.* 2018).

Research concerning the influence of thermal modification on fire-technical characteristics is scarce. Therefore, the aim of this study is to investigate the influence of thermal modification on the ignitability and mass burning rate of spruce wood.

EXPERIMENTAL

Materials

Norway spruce wood (*Picea abies*) samples were obtained from the University Forest Entreprise (Zvolen, Slovakia). Samples measuring 50 mm \times 40 mm \times 10 mm (length \times width \times thickness) were used to determine the mass burning rate. To determine the ignitability, samples measuring 250 mm \times 90 mm \times 2 mm (length \times width \times thickness) were used. The wood samples were thermally treated at temperatures of 100 °C, 150 °C, 200 °C, 220 °C, 240 °C, and 260 °C for 1 h, 3 h, and 5 h. Measurements of the mass burning rate were performed on five replicates per each treatment condition, and the ignitability was determined on three replicates per each treatment condition.

Methods

Heat treatment

The heat treatment was applied to the experimental Norway spruce wood samples in a laboratory type heating oven (Memmert UNB 200, Fisher Scientific, Loughborough, UK), which had an accuracy of 1 °C, under atmospheric pressure at temperatures of 100 °C, 150 °C, 200 °C, 220 °C, 240 °C, and 260 °C. Samples were placed in the oven preheated to the target temperature and kept there for 1 h, 3 h, or 5 h. Temperature was kept constant during the treatment. Static air atmosphere was used.

Fire-technical characteristics

The class of reaction to fire was determined according to EN ISO 11925-2 (2011). The mass burning rate was measured using an apparatus shown in Fig. 1 (Zachar *et al.* 2012) consisting of an electronic weight (4) with an accuracy of two decimal places, weight protection unit (3), metal holder (6) for placing the sample (5), metal loading frame (2) for placing the radiant heat source, and infrared thermal heater with an input of 1000 W (1). The sample was placed 30 mm (h) from the heat source and the weight was recorded every 10 s for 10 min



Fig. 1. Testing apparatus for the determination of mass burning rate (Zachar et al. 2012)

Statistical analysis

For all of the parameters, several of the comparisons were first subjected to an analysis of variance (ANOVA), and the significant differences between the average values of the control and treated samples were determined by Duncan's multiple range test with a p-value of 0.05. The data were analysed using Statistica software (StatSoft, version 12.0, Tulsa, OK, USA)

RESULTS AND DISCUSSION

The mass loss of the samples, logically, gradually increased during the exposure to the radiant heat (Figs. 2 to 4). Until the ignition (at 26 s to 53 s) and after the flames died out (at 288 s to 364 s), the increase was less sharp than during flame burning.

The mass loss of the samples treated for 1 h after exposure to the radiant heat source for 10 min decreased at 100 °C, increased at 150 °C, and then decreased in the rest of the investigated temperature range (Fig. 2). However, only the changes in the samples treated at temperatures over 220 °C were significant. The samples treated for 3 h showed the same trend as the samples treated for 1 h (Fig. 3). In samples treated for 3 h, the changes in the samples treated at the temperatures between 200 °C and 240 °C were significant. The samples treated for 5 h showed a similar trend as the samples treated for 3 h (Fig. 4), except that the mass loss in the samples treated at 260 °C increased, but this change was insignificant. Changes in these samples were also significant in the temperature range of 200 °C to 240 °C (Table 1 and Fig. 5). Linear regression of the data showed that generally, the mass loss at 600 s decreased with the treatment temperature. The decrease can be caused by the degradation of the less thermally stable hemicelluloses and amorphous fraction of cellulose due to the heat treatment (Kačíková *et al.* 2013), and by the increase of more thermally stable lignin content, which is more condensed in thermally-treated wood than in the natural wood (Kačíková *et al.* 2008).







Fig. 3. Mass loss of the spruce wood samples after the thermal treatment for 3 h while subjected to radiant heat



Fig. 4. Mass loss of the spruce wood samples after the thermal treatment for 5 h while subjected to radiant heat

Table 1. Selected	Fire-technical	Characteristics	of the	Spruce	Wood \$	Samples
after the Thermal	Treatment					

Temperature (°C)	Time (h)	Mass Loss at 600 s (%)	Ignition Time (s)	Flames Died Out Time (s)	Average Relative Burning Rate (%/s)	Maximum Relative Burning Rate (%/s)
20	0	94.13 ± 0.70	45 ± 6	343 ± 16	0.154 ± 0.001	0.444 ± 0.018
100	1	90.33 ± 5.23	42 ± 7	360 ± 33	0.148 ± 0.009	0.459 ± 0.056
150	1	93.04 ± 1.56	50 ± 10	364 ± 36	0.153 ± 0.003	0.486 ± 0.116
200	1	91.13 ± 5.07	48 ± 29	334 ± 34	0.149 ± 0.008	0.508 ± 0.102
220	1	90.37 ± 4.53	37 ± 7	308 ± 33	0.148 ± 0.007	0.542 ± 0.053
240	1	84.18 ± 3.95	32 ± 5	355 ± 30	0.138 ± 0.006	0.474 ± 0.064
260	1	79.25 ± 5.41	47 ± 4	348 ± 22	0.130 ± 0.009	0.454 ± 0.044
100	3	91.97 ± 4.70	37 ± 3	336 ± 50	0.151 ± 0.008	0.503 ± 0.065
150	3	93.22 ± 0.75	45 ± 9	347 ± 38	0.153 ± 0.001	0.448 ± 0.066
200	3	89.59 ± 5.14	35 ± 8	335 ± 31	0.147 ± 0.008	0.520 ± 0.070
220	3	83.85 ± 1.60	30 ± 8	353 ± 35	0.137 ± 0.003	0.476 ± 0.043
240	3	78.64 ± 7.04	48 ± 16	310 ± 35	0.129 ± 0.012	0.451 ± 0.084
260	3	75.26 ± 1.30	53 ± 15	288 ± 9	0.123 ± 0.002	0.458 ± 0.106
100	5	93.63 ± 1.16	35 ± 4	323 ± 21	0.153 ± 0.002	0.444 ± 0.027
150	5	94.42 ± 0.70	45 ± 7	321 ± 12	0.155 ± 0.001	0.484 ± 0.041
200	5	91.62 ± 5.02	28 ± 7	285 ± 40	0.150 ± 0.008	0.496 ± 0.064
220	5	85.60 ± 3.81	26 ± 11	302 ± 26	0.140 ± 0.006	0.489 ± 0.133
240	5	77.76 ± 0.42	49 ± 12	316 ± 20	0.127 ± 0.001	0.474 ± 0.079
260	5	78.98 ± 3.17	50 ± 4	285 ± 26	0.129 ± 0.005	0.455 ± 0.059

Note: Data represents the mean ± standard deviation



Fig. 5. Influence of the temperature on the mass loss at 600 s



Fig. 6. Influence of the temperature on the ignition time

The ignition time decreased in the samples treated at 100 °C for all of the treatment times, increased at 150 °C, and then decreased again. Then, the ignition time increased for the samples treated at 260 °C for 1 h and for the samples treated at temperatures over 240 °C for 3 h and 5 h. However, most of the changes were insignificant. Significant changes were only found between the samples treated at 220 °C and 240 °C for 3 h and 5 h and at 150 °C and 200 °C for 5 h (Table 1 and Fig. 6). This means that the thermal treatment does not influence the ignition time of the samples. Linear regression of the data showed almost constant lines.

The time when the flames died out on their own in the samples treated for 1 h increased until 150 °C, decreased until 220 °C, increased again at 240 °C, and then decreased. In the samples treated for 3 h, the flame-die-out time decreased at 100 °C, increased at 150 °C, decreased at 200 °C, increased at 220 °C, and then decreased in the rest of the investigated temperature range. In the samples treated for 5 h, the flame-die-out time decreased until 200 °C, increased until 240 °C, and then decreased again. Only one of these changes, between the samples treated at 220 °C and 240 °C for 1 h, was significant (Table 1 and Fig. 7). From these results, it can be concluded that the thermal modification does not influence the flame-die-out time. Linear regression of the data showed weak or very weak correlation between flame-die-out time and the temperature treatment.



Fig. 7. Influence of the temperature on the time it took for the flames to die out with 95% confidence intervals

The average relative burning rate of the samples treated for 1 h after the exposure to the radiant heat source for 10 min decreased at 100 °C, increased at 150 °C, and then decreased in the rest of the investigated temperature range. However, only the changes in the samples treated at temperatures higher than 220 °C were significant. The samples treated for 3 h showed the same trend as the samples treated for 1 h. The changes in the

samples treated in the temperature range of 200 °C to 240 °C were significant. The samples treated for 5 h showed a similar trend as the samples treated for 3 h, except the average relative burning rate increased in the samples treated at 260 °C. However, this change was insignificant. The changes in these samples in the temperature range of 200 °C to 240 °C were also significant (Table 1, Fig. 8). Linear regression of the data showed that generally, the mass loss at 600 s decreased with the treatment temperature. Reasons for the decrease in average relative burning rate are the same as for the mass loss at 600 s, since these two properties are interlinked.



Fig. 8. Influence of the temperature on the average relative burning rate

There were no significant changes in the maximum relative burning rate. However, it increased until 220 °C in the samples treated for 1 h and until 200 °C in the samples treated for 3 h and 5 h. After that, the maximum relative burning rate decreased (Table 1, Fig. 9). Thermal modification does not influence the maximum relative burning rate. Linear regression of the data showed almost constant lines.

Similar results were found by Čekovská *et al.* (2017a) after subjecting the spruce wood samples to direct flame. Thermally-modified samples of the teak wood had a greater mass loss after the exposure to a direct flame and higher burning rates than the untreated samples (Čekovská *et al.* 2017b).

The ignitability determined according to EN ISO 11925-2 (2011) (Tables 2 and 3) showed that all of the tested samples met the conditions for the class of reaction to fire E during testing on both the edge and the surface. The samples treated at 100 °C and 200 °C for 3 h and at 220 °C for 5 h during the surface test for a possible higher classification failed, and all of the tested samples failed during the edge test for a higher classification. However, to obtain a higher classification, a test according to EN 13823 (2010) would have to be conducted.

Table 2. Ignitability of Spruce Wood Samples After Thermal Treatment Test of the Surface

Treatment Temperature (°C)	Treatment Time (h)	Time of Flame Impingement (s)	Ignition (Yes / No)	<i>F</i> _s ≤ 150 mm / 20 s (Yes / No)	Time of Flame Impingement (s)	Ignition (Yes / No)	<i>F</i> s ≤ 150 mm / 60 s (Yes / No)
20	0	15	Yes	Yes	30	Yes	Yes
100	1	15	No	Yes	30	Yes	Yes
150	1	15	No	Yes	30	Yes	Yes
200	1	15	No	Yes	30	Yes	Yes
220	1	15	No	Yes	30	Yes	Yes
240	1	15	No	Yes	30	Yes	Yes
260	1	15	No	Yes	30	Yes	Yes
100	3	15	No	Yes	30	Yes	No (58)
150	3	15	Yes	Yes	30	Yes	Yes
200	3	15	Yes	Yes	30	Yes	No (51)
220	3	15	Yes	Yes	30	Yes	Yes
240	3	15	No	Yes	30	Yes	Yes
100	5	15	No	Yes	30	No	Yes
150	5	15	No	Yes	30	Yes	Yes
200	5	15	Yes	Yes	30	Yes	Yes
220	5	15	Yes	Yes	30	Yes	No (50)
240	5	15	No	Yes	30	Yes	Yes

Note: Numbers in parentheses represent the time in s when $F_s > 150 \text{ mm}$

Table 3. Ignitability of Spruce	Wood Samples After Th	nermal Treatment Test of
the Edge		

Treatment Temperature (°C)	Treatment Time (h)	Time of Flame Impingement (s)	Ignition (Yes / No)	F _s ≤ 150 mm / 20 s (Yes / No)	Time of Flame Impingement (s)	Ignition (Yes / No)	<i>F</i> s ≤ 150 mm / 60 s (Yes / No)
20	0	15	Yes	Yes	30	Yes	No (28)
100	1	15	Yes	Yes	30	Yes	No (32)
150	1	15	Yes	Yes	30	Yes	No (27)
200	1	15	Yes	Yes	30	Yes	No (30)
220	1	15	Yes	Yes	30	Yes	No (23)
240	1	15	Yes	Yes	30	Yes	No (22)
260	1	15	Yes	Yes	30	Yes	No (25)
100	3	15	Yes	Yes	30	Yes	No (28)
150	3	15	Yes	Yes	30	Yes	No (31)
200	3	15	Yes	Yes	30	Yes	No (27)
220	3	15	Yes	Yes	30	Yes	No (25)
240	3	15	Yes	Yes	30	Yes	No (27)
100	5	15	Yes	Yes	30	Yes	No (32)
150	5	15	Yes	Yes	30	Yes	No (35)
200	5	15	Yes	Yes	30	Yes	No (27)
220	5	15	Yes	Yes	30	Yes	No (21)
240	5	15	Yes	Yes	30	Yes	No (37)

Note: Numbers in parentheses represent the time in s when $F_s > 150 \text{ mm}$



Fig. 9. Influence of the temperature on the maximum relative burning rate with 95% confidence intervals

The samples treated at 260 °C for 3 h and 5 h were not subjected to the test because they broke when attempting to place them in the sample holder. The tested samples showed no unexpected physical behaviour. According to the International ThermoWood Association (2003), the results of the single burning item (SBI) test showed that the thermally modified wood can have a fire reaction classification of the class of reaction to fire D.

CONCLUSIONS

- 1. Heat treatment did not influence the ignition time, the time required for the flames to die out, or the maximum relative burning rate.
- 2. The heat treatment at higher temperatures resulted in a lower mass loss at 600 s and a lower average relative burning rate.
- 3. The heat treatment below 200 $^{\circ}$ C did not influence the fire safety of the wooden products.
- 4. The class of reaction to fire of the spruce wood was not changed due to the thermal treatment.

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