



Article Impact of Heat Treatment of Spruce Wood on Its Fire-Technical Characteristics Based on Density and the Side Exposed to Fire

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Abstract: The paper assessed the impact of the heat treatment of spruce wood, the (radial and tangential) side of the specimens exposed to fire, and the type of material (prism-higher density, floor—lower density) on the combustion process and the rate of fire spread. Five groups of specimens were used—untreated spruce wood specimens, two groups of heat-treated spruce wood specimens from the prism (higher density specimens), and two groups of heat-treated spruce wood specimens from the floor (lower density specimens). In one group, the flame was applied to the radial side, and in the other group to the tangential side of the specimens. The effect on the combustion process was assessed based on the parameters of mass loss and mass loss rate over time. The effect on the rate of fire spread across the specimens was assessed by the parameter fire spread rate. These parameters were determined using a simple test method where the specimens were exposed to a direct flame at an angle of 45° . To complement the results and to assess the processes involved, the temperatures at the specimen surfaces were also measured during the experiment. The main achieved results of the study are the findings on how the heat treatment, the density, and the side of the wood along which the fire spreads affect the burning process of the wood. The results indicated a significant effect of the density of the spruce thermowood on its combustion process. The higher density radial specimens exhibited a higher mass loss rate, and the overall average mass loss of the higher density samples was 27% of the original mass higher than that of the lower density samples. Additionally, the results suggested that the heat treatment of lower-density spruce wood (floor) does not significantly affect the mass loss and the mass loss rate. The difference in the overall average mass loss of the thermowood of floor and untreated wood samples was less than 2%, which is statistically insignificant. It was also found that for thermowood, fire will spread faster on the tangential side, where the fire spread rate is 29% higher compared with the radial side (for the floor samples). Based on the findings of other authors in a similar field, the results confirm that heat-treated spruce wood is more easily ignitable than untreated wood, which was proven by the spontaneous combustion of most of the thermowood samples during the experiment compared with the untreated wood samples.

Keywords: mass loss; mass loss rate; thermowood; fire resistance; density; exposed side

1. Introduction

Currently, we are observing an increase in the production of thermally treated wood thermowood, which partially replaces tropical woods in terms of its properties. Thermowood production has been steadily increasing over the last 20 years [1]. It is a product that combines the advantages of indigenous and exotic woods while eliminating their disadvantages, which are mainly the relatively high cost and the higher toxicity of the dusts produced during processing, causing the carcinogenic effects. Thermowood applications have rapidly expanded and are used, for example, in cladding, interior design products, for the construction of patios and gardens, and in the carpentry industry [1].

Thermowood is produced by heat treatment in the standard thermal range of 160–215 $^{\circ}$ C that changes the internal wood structure and its physical and mechanical properties. The



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). heat treatment process lasts several hours. Depending on the maximum temperature at which the wood is treated, its properties also change. Studies demonstrate that heat-treated wood has several positive properties compared with untreated wood, such as increased biological resistance and resistance to weather and sunlight [2–4], reduction of equilibrium moisture content [4–6], reduction of the heat transfer coefficient [7], improved dimensional stability [8–10], reduction of some undesirable substances (resins, polysac-charides) [11,12], thermal insulation [6,13,14], and aesthetic properties in the form of a darker decorative colour [15–19]. The disadvantage is the deterioration of its mechanical properties [3,4,20–23].

Relatively few studies have addressed the change in the fire performance characteristics of thermally treated wood. Thermowood Association [1] reports that the fire load of thermowood is lower due to the lower material density [24] and the lower content of wood constituents and extractives. From a fire engineering point of view, it is also possible to achieve a better sealing of thermowood cladding as it shrinks less due to moisture. The Thermowood Association [25] also states that thermowood (spruce and pine) reacts to fire class D, according to the SBI test (EN 13823) [26]; thus, there is no change in the reaction to fire class compared with untreated wood. According to Reinprecht and Vidholdová [4], thermally treated wood demonstrates lower smoke generation compared with untreated wood. Martinka et al. [27] evaluated the impact of heat treatment of spruce wood on the determination of ignition time, heat release rate, total heat released, and carbon monoxide yield using the cone calorimeter method [28]. The results indicated that heat treatment of spruce wood causes a reduction in the ignition time, heat release rate, and total heat released. The reduction of the heat release rate of thermowood was demonstrated in another study by Martinka et al. [29]. Zachar et al. [30] pointed out that the differences in the required activation energy for spontaneous ignition of untreated spruce wood and thermowood are minimal, as well as the differences in the minimum flashpoint. Luptakova et al. [31] investigated the effect of spruce wood treatment temperature on mass loss and ignitability after exposure to a radiant heat source. Heat treatments higher than 200 °C demonstrated lower mass loss and a lower relative mass loss rate, and the ignition time was not affected. Treatment of spruce wood at temperatures below 200 °C did not significantly affect the observed parameters compared to untreated specimens.

Changes in the fire performance of wood can be assessed by several methods [32]. One of them monitors the continuous mass loss and relative mass loss rate when specimens are subjected to thermal stresses from direct flames [33]. This method assesses the changes in the above parameters by the thermal treatment of wood with sufficient sensitivity [34–36]. Mass loss rate is also a significant parameter for fire resistance modelling of wooden structures [37,38] and mathematical models of forest fire spread [39]. This research is concerned with the effects of heat treatment of spruce wood on some fire characteristics, particularly continuous mass loss and relative mass loss rate. To complement this, the temperature of the specimen on the reverse side of the flame exposure was also monitored, tracking the heat transfer to the opposite specimen during the experiment. In addition, the experiment also assessed the differences in the fire-technical parameters of thermowood depending on the (radial, tangential) side of the specimen exposed to the flame.

2. Materials and Methods

2.1. Materials

For the experiment, spruce (Picea abies) wood specimens with dimensions of 82 mm \times 82 mm \times 26 mm (length \times width \times thickness) were used. The specimens were extracted from finished products and divided into five groups according to the heat treatment method, the exposed side (radial, tangential), and, for thermowood, the intended use of the product (base prism, flooring). Each group comprised three specimens. The first group of specimens contained untreated wood with an exposed radial side. The second and third specimen groups were extracted from thermally treated timber destined for use as a base prism, with an exposed radial or tangential side. The fourth and fifth specimen

groups were taken from thermally treated timber for terrace floors, with an exposed radial and tangential side. The designation of each group of specimens is specified in Table 1. The method of exposing the radial and tangential sides to flame during the experiment is demonstrated in Figure 1.

Table 1. Designation of individual specimen groups.

| | Thermowood from Prism | Thermowood from the Floor | Untreated Wood |
|----------------------------|--------------------------|------------------------------|----------------|
| Exposed side-Radial | TP-R | TF-R | U-R |
| Exposed side-Tangential | TP-T | TF-T | - |







All heat-treated specimens were treated with the Thermo-D thermal programme. First, high-temperature drying was conducted using steam, whereby the temperature was rapidly raised to 100 °C and then gradually increased to 130 °C. This phase lasted approximately 15 h. The next stage was the actual heat treatment, where the temperature rose to 212 °C (\pm 3 °C) over a period of 5.5 h. This temperature was maintained for 2–3 h. The wood was then cooled in a controlled manner for approximately 10 h. Finally, the wood was stabilised after heat treatment for 24 to 48 h in a warm, pressurised, roofed area.

The moisture content of the delivered untreated wood specimens was about 12%, and the moisture content of the thermowood specimens—treated wood was about 6% (declared by the supplier). Prior to the experiment, all specimens were placed in the laboratory for two weeks at a temperature of 21 °C and a humidity of approximately 40%.

2.2. Test Equipment and the Procedure

The testing method allows for continuous mass measurement of the test specimens during the experiment that generates parameters such as mass loss and relative mass loss rate over time. The test equipment consists of the parts depicted in Figure 2.

The specimens were attached horizontally. During the experiment they were exposed to a flame heat source applied perpendicularly from below. The used fuel was propanebutane gas. The flame height on the burner was set to 5 cm. The mouth of the burner was positioned at a distance of 4 cm from the specimen (1 cm of flame extending into the specimen).



Figure 2. Test equipment scheme (legend: 1—gas bomb, 2—gas supply to the burner, 3—Bunsen burner, 4—burner holder, 5—specimen holder, 6—scales, 7—connection of the scales to the computer, 8—computer).

During the experiment, the mass was continuously recorded at 10 s intervals. The accuracy of the mass measurement was ensured by Mettler Toledo MS1602S/M01 (Greifensee, Switzerland) scales to the hundredths of grams. Accurate measurement times at set intervals and automatic recording of masses were provided by BalanceLink 4.2.0.1 (Mettler Toledo, Greifensee, Switzerland). The exposure time of the specimens to the flame was 10 min. Afterwards, the flame was extinguished while the mass was still continuously recorded for 3 min after its extinguishing.

In addition, the temperatures on the unexposed specimen sides were recorded with a digital thermometer at times of 2, 4, 6, 8, 10, and 13 min during the experiment.

2.3. Evaluation and Calculation

The main assessment criteria are the mass loss of the tested specimens calculated according to Formula (1) and the relative mass loss rate calculated according to Formula (2).

$$\delta_{mp}(\tau) = \frac{m - m(\tau)}{m} \cdot 100 \tag{1}$$

where $\delta_{mp}(\tau)$ is the mass loss at time (τ) (%), *m* is the original specimen mass before the experiment (g), and *m*(τ) is the specimen mass at time (τ) (g).

$$v_r = \frac{m(\tau) - m(\tau + \Delta \tau)}{m(\tau) \cdot \Delta \tau} \cdot 100$$
⁽²⁾

where v_r is the relative mass loss rate (%·s⁻¹), $m(\tau)$ is the specimen mass at time (τ) (g), $m(\tau + \Delta \tau)$ is the specimen mass at time ($\tau + \Delta \tau$) (g), and $\Delta \tau$ is the time interval at which the masses are read (s). The time interval for reading the mass is 10 s in our case.

Another significant parameter is the ratio of the maximum value of the mass loss rate and the time to reach this value. As the value grows, the rate of flame spread increases [40]. This value is determined as an average only for the first peak of the mass loss rate according to Formula (3).

$$R_{fs} = \frac{v_r}{\tau(v_r)} \tag{3}$$

where R_{fs} is the fire spread rate (%·s⁻²), v_r is the relative mass loss rate (%·s⁻¹), and $\tau(v_r)$ is the time to reach the first peak of the maximum mass loss rate (s).

The results in the graphs and tables are specified as average values.

For the mass loss parameter, several comparisons were subjected to one- and twofactor analysis of variance (ANOVA) using the statistical software R (The R Project for Statistical Computing) version 4.1.2.

3. Results and Analyses

This section focuses on the specimen assessment based on mass loss and mass loss rate. We consider these to be essential for evaluating the behaviour of individual materials under the direct flame [41,42]. The results indicated differences in the observed fire characteristics not only between thermowood and untreated wood but also between thermowood specimens according to the intended use (base prism, floor) and according to the side exposed to the flame (radial, tangential). The Discussion section includes assessment based on the measured temperatures on the unexposed side of the specimens that explain the combustion processes of the particular specimens in greater detail.

As can be noticed in Figure 3, the mass loss of all specimens increased from the beginning of the flame exposure until the end of the test duration. In the first seconds, an increase in mass loss occurred for all specimens. From approx. one minute to three minutes, the time-dependent mass loss was steady for all specimens. After three minutes, however, there was a sudden mass loss change from specimen to specimen. While the TF-R and U-R specimens demonstrated a steady linear trend in mass loss, the TP-R, TP-T, and TF-T specimens experienced a higher mass loss increase. There was a gradual decrease in mass loss from the 600 s time onwards that was expected since the flame affecting the specimens was extinguished after this time.



Figure 3. Average percentage mass loss of individual specimens over time.

The variability of the final mass loss over the 780 s time of the experiment provides us with crucial data for establishing the dependence of the total mass loss between the different specimen groups. Figure 4 demonstrates the lowest variability of the measured total mass loss for specimens U-R, TF-R, and TF-T. On the contrary, we observed a broad variation range for the TP-R and TP-T specimens, i.e., the thermowood specimens extracted from the base prism. The mass loss dependence between the U-R and TF-R specimens is not important, in contrast to the significant dependence between the U-R and TF-T and the TF-R and TF-T specimens. We did not observe a significant dependence between TP-R and TP-T specimens but between the TP-R and TF-R specimens. It can be concluded that the wood flammability does not only depend on its heat treatment compared to untreated wood but also on the specimen material in terms of its intended use and, also, on the side of the specimen exposed to the flame. Therefore, we further focus on the assessment of the observed parameters of the different specimen groups and their comparison based on the studied groups.

Wood has different properties in different (radial, tangential) directions, so it is also assumed that the fire spreads differently on the radial side compared with the tangential side. Figure 5 depicts the dependence of the mass loss and the mass loss rate on the side (radial, tangential) exposed to the flame. For the thermowood prism specimens (Figure 5A,B) the courses of the mass loss and the mass loss rate for both radial and



tangential specimens were highly similar. In this case, we did not observe a significant dependence between the specimens.

Figure 4. Boxplots of the mass loss of each specimen group at 780 s testing time.



Figure 5. Mass loss and mass loss rate over time of thermowood specimens. (**A**) Mass loss of specimens TP–R and TP–T; (**B**) Mass loss rate of specimens TP–R and TP–T; (**C**) Mass loss of specimens TF–R and TF–T; (**D**) Mass loss rate of specimens TF–R and TF–T.

However, with the thermowood floor specimens, there is a significant effect of the specimen side on the mass loss and mass loss rate (Figure 5C,D). The mass loss rate of

the TF-T specimen started to increase significantly from time 180 s until the flame was extinguished at time 600 s. On the other hand, the TF-R specimen's mass loss rate indicated a predominantly decreasing trend and started to increase slightly at time 400 s. The TP-R specimens reached the highest mass loss rate of $0.207\% \cdot s^{-1}$ at 600 s, followed by the TP-T specimens (0.196%) at 470 s, the TF-T specimens (0.135%) at 390 s, and, finally, the TF-R specimens (0.082%) at 30 s.

The dependence of the total mass loss of the thermowood on the type of material in terms of the intended use and on the side of the specimen to which the flame was applied is statistically analysed in Table 2. The ANOVA test results indicated statistically significant differences between all specimen groups concerning mass loss. Specimens exposed to flame on the radial side were statistically different from specimens exposed to flame on the tangential side; *p*-value = 0.095. Significant dependence related to mass loss was confirmed between the base prism and floor specimens, *p* = 0.0025.

Table 2. Multivariate analysis of variance (ANOVA) of mass loss of thermowood specimens TP-R, TP-T, TF-R, TF-T at 780 s.

| | Df | Sum Sq | Mean Sq | F Value | Pr (>F) | Influence |
|--------------------------|----|--------|---------|---------|---------|-----------|
| Exposed side | 1 | 150.0 | 150.0 | 3.596 | 0.09451 | • |
| Density | 1 | 779.8 | 779.8 | 18.688 | 0.00254 | ** |
| Exposed side: Density | 1 | 243.4 | 243.4 | 5.833 | 0.04217 | * |
| Residuals | 8 | 333.8 | 41.7 | | | |

Signif. codes: 'influence' - 'p-value to': '**' - '0.01'; '*' - '0.05'; '.' - '0.1'; '-' - '1'.

The ratio of the maximum value of the mass loss rate at the first peak and the time to reach this value (hereafter referred to as the fire spread rate) was highest for the TF-T specimens, with a significantly higher difference compared with the other specimens. The TP-T specimens demonstrated the second-highest R_{fs} value. The TP-R and TF-R specimens had similar values (Table 3). All specimens reached the first peak of the mass loss rate in the same time of the 30 s. Based on the above results, it can be assumed that the thermowood specimens will have the highest fire spread rate on the tangential side, regardless of the type of material used (prism, floor).

Table 3. Proportion of the maximum value of the first peak mass loss rate and the time to reach this.

| Specimens | TP-R | TP-T | TF-R | TF-T | U–R |
|-----------|----------|----------|----------|----------|---------|
| Rfs | 0.002736 | 0.003077 | 0.002749 | 0.003546 | 0.00288 |

To further assess the effect of heat treatment on the mass loss and the mass loss rate, we need to consider which side will be exposed to the flame. Therefore, we were further concerned with monitoring the parameters for specimens exposed to the flame from the radial side. In this case, the mass loss and the mass loss rate for the thermally treated timber from the prism (TP-R) were significantly different for the thermally treated timber from the floor (TF-R) and the untreated timber (U-R) (Figure 6). The TF-R and U-R specimens demonstrated nearly identical mass loss and mass loss rate behaviour. For the mass loss parameter, the TP-R specimens were statistically significantly different from the U-R specimens (p = 0.01), but the TF-R specimens were not different from the U-R specimens (p = 0.31) (Tables 4 and 5).



Figure 6. Mass loss and relative mass loss rate over time for TP–R, TF–R, and U–R specimens. (A) Mass loss; (**B**) mass loss rate.

| Table 4. ANOVA of mass loss of $TP-R$, $U-R$ specimens at 78 |
|--|
|--|

| | Df | Sum Sq | Mean Sq | F Value | Pr (>F) | Influence |
|--------------------|----|---------------------|---------|---------|---------|-----------|
| Specimens | 1 | 1100 | 1100.0 | 19.91 | 0.0111 | * |
| Residuals | 4 | 221 | 55.2 | | | |
| Ciamif and an limf | | alua ta'. (*' (0.05 | 1 | | | |

Signif. codes: 'influence' - 'p-value to': '*' - '0.05

Table 5. ANOVA of mass loss of TF-R, U-R specimens at 780 s.

| | Df | Sum Sq | Mean Sq | F Value | Pr (>F) | Influence |
|----------------------|----|------------------|---------|---------|---------|-----------|
| Specimens | 1 | 5.704 | 5.704 | 1.347 | 0.31 | - |
| Residuals | 4 | 16.936 | 4.234 | | | |
| Ciamif and any finfl | | alua ta' (1 / 11 | | | | |

Signif. codes: 'influence' - 'p-value to': '-' - '1'.

The above results demonstrate the differences in mass loss and mass loss rate of individual specimens. In particular, the TP-R and TP-T (thermowood prisms) specimens differed significantly from the other specimens. The mass loss rate of these specimens started to increase significantly at a specific time (after approx. the third minute) compared with the other specimens. It subsequently caused a higher mass loss. The reason for the sudden increase in the mass loss rate of TP-R, TP-T, and TF-T specimens is the spontaneous combustion of the specimens that also burn through the sides after a certain time. Either only the lateral specimen side burned, or in some cases, the entire specimen. The specimens all started to combust spontaneously during the experiment. The average time of spontaneous combustion was: TP-R: 5 min 40 s; TP-T: 4 min 43 s; TF-T: 5 min 3 s (Table 6). In each of these groups, there was one specimen that spontaneously combusted at about 3.5 min. This occurrence explains the rapid increase in the average mass loss rate after the above-mentioned time, which increased further. In the other specimens (TF-R and U-R), we did not observe a significant increase in the average mass loss rate as only one specimen in the TF-R group spontaneously combusted and one specimen in the U-R group only at the end of the experiment. A study [30,36] reported that for thermowood, lower energy is required for spontaneous combustion than for untreated wood. Martinka et al. [27], 2013 pointed to findings that heat treatment of spruce wood causes a reduction in ignition time. The above statements explain why the majority of the thermally treated wood specimens ignited earlier than untreated wood.

Heat treatment of spruce wood increases its mass loss rate, as observed in the majority of specimens. This claim is consistent with the findings of the authors of ref. [34], who demonstrated that higher temperatures of heat treatment of spruce wood caused a higher burning rate.

| | TP-R | TP-T | TF-R | TF-T | U-R |
|---|------------------|----------------|------------|------------------|------------|
| Time of spontaneous combustion of specimens | 7:00; 6:30; 3:30 | 6:00-4:50-3:20 | 4:30; -; - | 6:30; 5:00; 3:40 | 9:50; -; - |
| Interval | 3:30 | 2:40 | _ | 2:50 | - |
| Average | 5:40 | 4:43 | - | 5:03 | - |

Table 6. Time of spontaneous combustion of individual specimens.

On the other hand, we did not observe a difference in the mass loss rate for TF-R specimens compared to U-R specimens. It suggests that heat treatment of spruce wood will not affect this value unless direct flame burning of the specimens occurs. A similar result was demonstrated by a study [31] claiming that the exposure to a flameless radiant heat source does not affect the maximum burning rate of heat-treated spruce wood compared to untreated wood.

We further assume that the higher mass loss rate of TP-R and TP-T specimens (thermowood prism) compared with TF-R and TF-T specimens (thermowood floor) is influenced by the specimen density, which according to several authors [43,44] influences the mass loss rate. The average densities of the prism thermowood specimens were approximately $50 \text{ kg} \cdot \text{m}^{-3}$ greater than the other specimens (Table 7). Density differences between specimens were also observed in the density of the annual rings in Figure 1. Specimens TP-R and TP-T feature denser annual rings that affect the wood density [45]. The spruce wood density increases with decreasing annual ring width [46] and decreases as a result of heat treatment [47]. In this case, higher density resulted in a higher mass loss rate and higher mass loss. The causes of this phenomenon have not been clarified yet. One reason could be the changes in the chemical composition of heat-treated wood, containing more lignin than untreated wood [24,48–50]. On the one hand, higher lignin content has been confirmed to have an effect on reducing the mass loss rate [37,51,52]. On the other hand, lignin has a significantly higher calorific value than cellulose and hemicellulose [53] that are subject to decomposition during heat treatment [8,54]. Thus, thermowood also has a higher calorific value, as confirmed by Todaro et al. [55]. After the specimens spontaneously combusted and burned through from the sides, large mass loss rates occurred (Figure 6) because the higher density specimens contain more lignin and cause higher burning temperatures, as can also be observed in Figure 7. The average surface temperatures of TP-R and TP-T specimens reached maximum temperatures of 230 and 190 °C, respectively, and TF-R and TF-T specimens only 94 and 118 °C, respectively. Higher temperatures may have caused faster decomposition and release of combustion products, thus increasing the mass loss rate.

Table 7. Average specimen densities (kg·m⁻³).

| | TP-R | TP-T | TF-R | TF-T | U-R |
|--------------------|--------|--------|--------|--------|--------|
| Average density | 424.47 | 422.23 | 371.50 | 367.61 | 380.03 |
| Variation interval | 12.55 | 6.54 | 16.76 | 9.29 | 41.77 |

Temperatures between radial and tangential specimens did not differ significantly, especially when considering possible measurement variations. In some cases, it was difficult to determine exact temperatures due to their constant fluctuations, or the temperature failed to be read at all. Nevertheless, the differences in the mass loss rate on the radial and tangential sides of the floor thermowood specimens (Figure 5), the statistical ANOVA analysis of mass loss (Table 2), and the R_{fs} values (Table 3) indicated that the mass loss rate and fire spread is faster on the tangential side. During the wood pyrolysis, a charred surface layer is produced. It creates an insulating barrier and prevents fire penetration into the inner layers [56,57]. However, during combustion, due to shrinkage, cracks form in this layer, increasing heat transfer and allowing the passage of combustible gases to the surface [58].



Figure 7. Average temperatures on the specimen surface during the experiment. (**A**) samples TP-R and TP-T; (**B**) Samples TF-R and TF-T.

In tangential specimens, these cracks form more frequently than in radial specimens; hence, there is a higher release of combustible gases that causes an increase in the mass loss rate. These findings agree with refs. [59,60], who tested the fire resistance of radial and tangential wood specimens with a charred layer formed during direct flame exposure. The tangential specimens showed a higher mass loss, and the formed charred layer prevented combustion less than in radial specimens.

4. Conclusions

In the study, the fire-technical properties of thermally treated spruce wood such as mass loss rate and mass loss were evaluated. Samples with different densities were used. The fire performance was investigated when a direct flame was applied perpendicularly to the radial and tangential sides of the specimen.

According to the average mass loss rate and average mass loss achieved when direct flame was applied to the samples using our proposed test method, conclusions here drawn. Thermal treated spruce wood with higher density has a higher mass loss and a higher mass loss rate than thermally treated spruce wood with lower density and untreated wood. The difference in densities was 50 kg·m⁻³. The average mass loss of the higher-density thermowood samples ($423 \text{ kg} \cdot \text{m}^{-3}$) was 55%, and that of the lower-density samples ($370 \text{ kg} \cdot \text{m}^{-3}$) was 39%. The radial samples (radial side exposed to flame) of the higher-density spruce thermowood had a significantly higher mass loss and mass loss rate compared with the floor and untreated wood samples (difference in weight loss of 25% and 27%, respectively). The higher-density samples had the highest mass loss rate (radial 0.207%·s⁻¹, tangential 0.196%·s⁻¹) compared with the lower-density samples (radial 0.082%·s⁻¹). According to the results, the fire resistance of spruce thermowood deteriorates with increasing density.

Based on the "fire spread rate" parameter, we can conclude that a thermowood fire will spread faster on the tangential side. The R_{fs} values of the tangential floor samples were 29% higher compared with the radial samples.

The dependence of the mass loss on the density of the thermowood and the side exposed to the flame was also confirmed by analysis of variance with p value = 0.00254 resp. 0.09451.

The results also showed that the mass loss of radial floor spruce thermowood samples (lower density samples) was not statistically significantly different from the untreated samples (*p*-value = 0.31). From the above it can be concluded that the thermal treatment of spruce wood does not have a significant effect on the selected fire characteristics of the wood (mass loss rate and mass loss). However, thermally treated wood is easier to ignite as confirmed by the spontaneous combustion times. While for thermowood, 10 samples out of 12 burned in an average time of 5 min from the start of experiment, on the contrary for untreated wood burned, in which only 1 sample out of 3 burned by the end of the experiment at almost the 10th min.

Our research, aiming to improve the quality of research in this area, confirms the results of a study by several authors who evaluated the fire resistance of thermowood. It provides new findings on the differences in burning of thermowood on the radial and tangential sides and the differences in burning as a function of the specimen density. So far, however, research has only assessed the fire behaviour of thermowood based on laboratory tests. Therefore, research in this area needs to be further focused on conducting large-scale tests to determine the actual behaviour of thermowool under realistic fire conditions. Because thermowood is often used for wall cladding, research also needs to focus on monitoring fire spread in the vertical direction where it is most prominent.

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References

- 1. Thermowood Association. ThermoWood Handbook; Finish International ThermoWood Association: Helsinky, Finland, 2021; p. 56.
- 2. Huang, X.; Kocaefe, D.; Kocaefe, Y.; Boluk, Y.; Pichette, A. Study of the degradation behavior of heat-treated jack pine (Pinus banksiana) under artificial sunlight irradiation. *Polym. Degrad. Stab.* **2012**, *97*, 1197–1214. [CrossRef]
- Reinprecht, L. Ochrana Dreva, 1st ed.; Technická Univerzita vo Zvolene: Zvolen, Slovakia, 2008; p. 453. ISBN 978-80-228-1863-6.
 Reinprecht, L.; Vidholdová, Z. Thermowood; Šmíra Print: Ostrava, Czech Republic, 2011; p. 89. ISBN 978-80-87427-05-7.
- Reinprecht, L.; Vidholdová, Z. *Thermowood*; Šmíra Print: Ostrava, Czech Republic, 2011; p. 89. ISBN 978-80-87427-05-7.
 Metsä-Kortelainen, S.; Antikainen, T.; Viitaniemi, P. The water absorption of sapwood and heartwood of Scots pine and Norway
- spruce heat-treated at 170°C, 190°C, 210°C and 230°C. Holz Als Roh-Und Werkst. **2006**, 64, 192–197. [CrossRef]
- Borůvka, V.; Šedivka, P.; Novák, D.; Holeček, T.; Turek, J. Haptic and Aesthetic Properties of Heat-Treated Modified Birch Wood. Forests 2021, 12, 1081. [CrossRef]
- Hortobágyi, Á.; Pivarčiová, E.; Koleda, P. Holographic Interferometry for Measuring the Effect of Thermal Modification on Wood Thermal Properties. *Appl. Sci.* 2021, *11*, 2516. [CrossRef]
- Tjeerdsma, B.F.; Boonstra, M.; Pizzi, A.; Tekely, P.; Militz, H. Characterisation of thermally modified wood: Molecular reasons for wood performance improvement. *Holz Als Roh-Und Werkst.* 1998, 56, 149–153. [CrossRef]
- Hill, C.; Altgen, M.; Rautkari, L. Thermal modification of wood—A review: Chemical changes and hygroscopicity. J. Mater. Sci. 2021, 56, 6581–6614. [CrossRef]
- Herrera-Builes, J.F.; Sepúlveda-Villarroel, V.; Osorio, J.A.; Salvo-Sepúlveda, L.; Ananías, R.A. Effect of Thermal Modification Treatment on Some Physical and Mechanical Properties of *Pinus oocarpa* Wood. *Forests* 2021, 12, 249. [CrossRef]
- González-Peña, M.M.; Curling, S.F.; Hale, M.D.C. On the effect of heat on the chemical composition and dimensions of thermallymodified wood. *Polym. Degrad. Stab.* 2009, 94, 2184–2193. [CrossRef]
- Candelier, K.; Dumarçay, S.; Pétrissans, A.; Desharnais, L.; Gérardin, P.; Pétrissans, M. Comparison of chemical composition and decay durability of heat treated wood cured under different inert atmospheres: Nitrogen or vacuum. *Polym. Degrad. Stab.* 2013, 98, 677–681. [CrossRef]
- 13. Hrčka, R.; Babiak, M. Some non-traditional factors influencing thermal properties of wood. Wood Res. 2012, 57, 367–374.
- Osvaldova, L.M.; Gaspercova, S.; Petho, M. Natural Fiber Thermal Insulation Materials from Fire Prevention Point of View. In Proceedings of the 2015 International Symposium on Material, Energy and Environment Engineering (ISM3E), Changsha, China, 28–29 November 2015; pp. 58–60. [CrossRef]
- Nasir, V.; Nourian, S.; Avramidis, S.; Cool, J. Prediction of physical and mechanical properties of thermally modified wood based on color change evaluated by means of "group method of data handling" (GMDH) neural network. *Holzforschung* 2019, 73, 381–392. [CrossRef]

- Dudík, R.; Borůvka, V.; Zeidler, A.; Holeček, T.; Riedl, M. Influence of Site Conditions and Quality of Birch Wood on Its Properties and Utilization after Heat Treatment. Part II—Surface Properties and Marketing Evaluation of the Effect of the Treatment on Final Usage of Such Wood. Forests 2020, 11, 556. [CrossRef]
- 17. Bekhta, P.; Niemz, P. Effect of high temperature on the change in color, dimensional stability and mechanical properties of spruce wood. *Holzforschung* **2003**, *57*, 539–546. [CrossRef]
- González-Peňa, M.M.; Hale, M.D.C. Colour in thermally modified wood of beech, Norway spruce and scots pine. Part 1: Colour evolution and colour changes. *Holzforschung* 2009, 63, 385–393. [CrossRef]
- 19. De Angelis, M.; Humar, M.; Kržišnik, D.; Tamantini, S.; Romagnoli, M. Influence of Thermal Modification and Impregnation with Biocides on Physical Properties of Italian Stone Pine Wood (*Pinus pinea* L.). *Appl. Sci.* **2022**, *12*, 3801. [CrossRef]
- 20. Percin, O.; Peker, H.; Atilgan, A. The Effect of Heat Treatment on The Some Physical and Mechanical Properties of Beech (Fagus Orientalis Lipsky) Wood. *Wood Res.* **2016**, *61*, 443–456.
- Akyurek, S.; Akman, M.; Ozalp, M. Effects of Heat Treatment on Some Chemical Compound and Mechanical Properties of Black Pine Wood. Wood Res. 2021, 66, 621–629. [CrossRef]
- 22. Torniainen, P.; Popescu, C.-M.; Jones, D.; Scharf, A.; Sandberg, D. Correlation of Studies between Colour, Structure and Mechanical Properties of Commercially Produced ThermoWood[®] Treated Norway Spruce and Scots Pine. *Forests* **2021**, *12*, 1165. [CrossRef]
- Borůvka, V.; Dudík, R.; Zeidler, A.; Holeček, T. Influence of Site Conditions and Quality of Birch Wood on Its Properties and Utilization after Heat Treatment. Part I—Elastic and Strength Properties, Relationship to Water and Dimensional Stability. *Forests* 2019, 10, 189. [CrossRef]
- Čabalová, I.; Výbohová, E.; Igaz, R.; Kristak, L.; Kačík, F.; Antov, P.; Papadopoulos, A. Effect of oxidizing thermal modification on the chemical properties and thermal conductivity of Norway spruce (*Picea abies* L.) wood. *Wood Mater. Sci. Eng.* 2021. [CrossRef]
 Thermowood Association. *ThermoWood Handbook*; Finish ThermoWood Association: Helsinky, Finland, 2003; p. 66.
- EN 13823; Reaction to Fire Tests for Building Products-Building Products Excluding Floorings Exposed to the Thermal Attack by a Single Burning Item. CEN: Brussels, Belgium, 2002.
- 27. Martinka, J.; Chrebet, T.; Král, J.; Balog, K. An examination of behaviour of thermally treated spruce wood under fire conditions. *Wood Res.* **2013**, *58*, 599–606.
- ISO 5660-1; Reaction-to-fire Tests—Heat Release, Smoke Production, and Mass Loss Rates—Part 1: Heat Release (Cone Calorimeter Method), ISO 5660-1. International Standards Organization: Geneva, Switzerland, 2002.
- 29. Martinka, J.; Kačíková, D.; Rantuch, P.; Balog, K. Investigation of the influence of spruce and oak wood heat treatment upon heat release rate and propensity for fire propagation in the flashover phase. *Acta Fac. Xylologiae Zvolen* **2016**, *58*, 5–14. [CrossRef]
- Zachar, M.; Majlingová, A.; Šišulák, S.; Baksa, J. Comparison of the activation energy required for spontaneous ignition and flash point of the Norway spruce wood and thermowood specimens. *Acta Fac. Xylologiae Zvolen* 2017, 59, 79–90. [CrossRef]
- Luptakova, J.; Kacik, F.; Mitterova, I.; Zachar, M. Influence of temperature of thermal modification on the fire-technical characteristics of spruce wood. *BioResources* 2019, 14, 3795–3807. [CrossRef]
- Gaspercova, S.; Makovická Osvaldova, L. Fire Protection in Various Types of Wooden Structures. Civ. Environ. Eng. 2015, 11, 57. [CrossRef]
- 33. Gaspercova, S.; Makovicka Osvaldova, L. Influence of surface treatment of wood to the flame length and weight loss under load single-flame source. *Key Eng. Mater.* **2017**, *755*, 353–359. [CrossRef]
- Čekovská, H.; Gaff, M.; Osvald, A.; Kačík, F.; Kubš, J.; Kaplan, L. Fire resistance of thermally modified spruce wood. *BioResources* 2017, 12, 947–959. [CrossRef]
- 35. Turekova, I.; Markova, I. Ignition of Deposited Wood Dust Layer by Selected Sources. Appl. Sci. 2020, 10, 5779. [CrossRef]
- Osvaldova, L.M.; Gaspercova, S.; Mitrenga, P.; Osvald, A. The influence of density of test specimens on the quality assessment of retarding effects of fire retardants. *Wood Res.* 2016, *61*, 35–42.
- 37. Janssens, M.L. Modeling of the thermal degradation of structural wood members exposed to fire. *Fire Mater.* **1998**, *28*, 199–207. Available online: ww3.interscience.willey.com (accessed on 17 June 2015).
- Vandlickova, M.; Markova, I.; Makovicka-Osvaldova, L.; Gaspercova, S.; Svetlik, J. Evaluation of African Padauk (Pterocarpus soyauxii) Explosion Dust. *BioResources* 2020, 15, 401–414. [CrossRef]
- MCKenzie, D.; Hessl, A.E.; Kellog, L.K. Using neutral models to identify constraints on low-severity fire regions. *Landsc. Ecol.* 1996, 21, 139–152. [CrossRef]
- Kačíková, D.; Makovická-Osvaldová, L. Wood burning rate of various tree parts from selected softwoods. *Acta Fac. Xylologiae* 2009, 51, 27–32.
- Makovická Osvaldová, L.; Markova, I.; Jochim, S.; Bares, J. Experimental Study of Straw-Based Eco-Panel Using a Small Ignition Initiator. *Polymers* 2021, 13, 1344. [CrossRef] [PubMed]
- 42. Čekovská, H.; Gaff, M.; Osvaldová, L.M.; Kačík, F.; Kaplan, L.; Kubš, J. Tectona grandis Linn. and its fire characteristics affected by the thermal modification of wood. *BioResources* **2017**, *12*, 2805–2817. [CrossRef]
- 43. Babrauskas, V. Charring rate of wood as a tool for fire investigations. Fire Saf. J. 2005, 40, 528–554. [CrossRef]
- Hostikka, S.; McGrattan, K.B. Large Eddy Simmulation of Wood Combustion. In *Interflam* 2001; Interscience Communications Ltd.: London, UK, 2001; Volume 1, pp. 755–762.
- 45. Bouriaud, O.; Bréda, N.; Le Moguédec, G.; Nepveu, G. Modelling variability of wood density in beech as affected by ring age, radial growth and climate. *Trees* **2004**, *18*, 264–276. [CrossRef]

- 46. Bouriaud, O.; Teodosiu, M.; Kirdyanov, A.V.; Wirth, C. Influence of wood density in tree-ring-based annual productivity assessments and its errors in Norway spruce. *Biogeosciences* **2015**, *12*, 6205–6217. [CrossRef]
- Cermák, P.; Hess, D.; Suchomelová, P. Mass loss kinetics of thermally modified wood species as a time-temperature function. *Eur. J. Woodand Wood Prod.* 2021, 79, 547–555. [CrossRef]
- Kačíková, D.; Kubovský, I.; Ulbriková, N.; Kačík, F. The Impact of Thermal Treatment on Structural Changes of Teak and Iroko Wood Lignins. *Appl. Sci.* 2020, 10, 5021. [CrossRef]
- Zachar, M.; Majlingová, A.; Mitterová, I.; Čabalová, I. Influence of an age and damage of the oak wood in its fire risk. Wood Res. 2017, 62, 495–504.
- Zachar, M.; Čabalová, I.; Kačíková, D.; Zacharová, L. The Effect of Heat Flux to the Fire-Technical and Chemical Properties of Spruce Wood (*Picea abies* L.). *Materials* 2021, 14, 4989. [CrossRef] [PubMed]
- 51. Rowell, R.M.; Susott, A.R.; Degroot, F.W.; Shafizadeh, F. Bonding fire retardants to wood. Part I. Thermal behavior of chemical bonding agents. *Wood Fiber Sci.* **1984**, *16*, 214–223.
- 52. Tran, H.C.; White, R.H. Burning Rate of Solid Wood Measured in Heat Release Rate Calorimeter. *Fire Mater.* **1992**, *16*, 197–206. [CrossRef]
- 53. Kaltschmitt, M.; Hartmann, H.; Hofbauer, H. Energie aus Biomasse. Grundlagen, Techniken und Verfahren, 2nd ed.; Springer: Berlin/Heidelberg, Germany, 2009; p. 1867. ISBN 978-3-662-47437-2.
- Nuopponen, M.; Vuorinen, T.; Jamsä, S.; Viitaniemi, P. Thermal modifications in softwood studied by FT-IR and UV resonance Raman spectroscopies. J. Wood Chem. Technol. 2004, 24, 13–26. [CrossRef]
- 55. Todaro, L.; Rita, A.; Cetera, P.; D'Auria, M. Thermal treatment modifies the calorific value and ash content in some wood species. *Fuel* **2015**, *140*, 1–3. [CrossRef]
- Sweet, M.S. Fire Performance of Wood: Test Methods and Fire Retardant Treatments. In Proceedings of the 4th Annual BCC Conference on Flame Retadancy, Norwalk, CT, USA, 18–20 May 1993; pp. 36–43.
- 57. Gan, W.; Chen, C.; Wang, Z.; Song, J.; Kuang, Y.; He, S.; Mi, R.; Sunderland, P.B.; Hu, L. Dense, self-formed char layer enables afire-retardant wood structural material. *Adv. Funct. Mater.* **2019**, *29*, 1807444. [CrossRef]
- 58. Buchanan, A.H. Fire performance of timber construction. Prog. Struct. Eng. Mater. 2000, 2, 278–289. [CrossRef]
- Machová, D.; Oberle, A.; Zárybnická, L.; Dohnal, J.; Šeda, V.; Dömény, J.; Vacenovská, V.; Kloiber, M.; Pěnčík, J.; Tippner, J.; et al. Surface Characteristics of One-Sided Charred Beech Wood. *Polymers* 2021, 13, 1551. [CrossRef]
- 60. Makovicka Osvaldova, L.; Kadlicova, P.; Rychly, J. Fire characteristics of selected tropical woods without and with fire retardant. *Coatings* **2020**, *10*, 527. [CrossRef]