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Effect of Spruce Wood Density on Selected Fire-Technical Parameters during Thermal Loading

Patrik Mitrenga *, Linda Makovická Osvaldová and Milan Konárik

Department of Fire Engineering, Faculty of Security Engineering, University of Žilina, 010 26 Žilina, Slovakia; linda.makovicka@uniza.sk (L.M.O.); milan.konarik@uniza.sk (M.K.)
* Correspondence: patrik.mitrenga@uniza.sk; Tel.: +421-415136752

Abstract: The paper evaluates the effect of spruce wood density on the parameters of mass loss and mass loss rate during exposure to thermal load. The intention was to determine whether the effect of density is still evident after the application of flame retardants to the test samples. Groups of samples with different densities under the same retardant treatment were compared. The differences in densities of the compared groups of samples were different for each flame retardant. Water-soluble flame retardants based on inorganic salts were used. For testing, a simple test method was used in which the samples were exposed to direct flame from a Bunsen burner. The results of the study are the findings of how wood density affects the burning process of the samples treated with flame retardants. Statistical evaluation of the experimental results shows a significant effect of wood density on the monitored parameters even when flame retardants are used. For a difference in sample densities of 244 kg·m^{-3}, there was a density dependence of the mass loss rate, with the lower density samples having a higher mass loss rate (0.158%·s^{-1}) over the whole experimental period compared to the higher density samples (0.077%·s^{-1}). The ANOVA test also demonstrated the influence of density on the mass loss of the samples at the above density difference. At lower density differences (51 kg·m^{-3} and below), the effect of sample density on the observed parameters was no longer evident. The fire spread rate parameter was also investigated. Here, a linear correlation between the difference in sample densities and the difference in the values of the above parameter at high and low densities is observed with a reliability coefficient R^2 = 0.99.

Keywords: spruce wood; density; mass loss; mass loss rate; flame retardant

Citation: Mitrenga, P.; Osvaldová, L.M.; Konárik, M. Effect of Spruce Wood Density on Selected Fire-Technical Parameters during Thermal Loading. *Appl. Sci.* **2024**, *14*, 170. https://doi.org/10.3390/app14010170

Academic Editor: Giuseppe Lazzara

Received: 5 December 2023
Revised: 15 December 2023
Accepted: 21 December 2023
Published: 24 December 2023

1. Introduction

Wood is a widely used construction material for both exterior and interior applications [1], and its global production has been steadily increasing, surpassing 800 million tonnes per year in 2015. In Europe, the wood industry comprises approximately 400,000 enterprises, representing approximately one fifth of all manufacturing businesses [2]. One of the main drawbacks of wood and other wood-based materials in all forms, when used as a building material, is its flammability [3–10], limiting its fire resistance [11–13].

To address this concern, efforts are being made to develop protective measures and flame retardants [14]. However, accurately testing these flame retardants is crucial to detect even minor differences in their effectiveness. In Europe, several testing methods [15–19] are applied to classify products into reaction-to-fire classes [20]. However, these are insufficient for assessing flame retardants. Therefore, other, much more sensitive methods are also used to compare the effects of flame retardants. Thermal analysis methods are used to study the properties of materials as temperature changes. Of these, differential scanning calorimetry (DSC), which measures the heat released or absorbed during the heating process, is used in particular. Differential thermal analysis (DTA) can be used to determine the oxidation process. Thermo-gravimetric analysis (TGA) can be used to measure the change in mass of the sample during the heating or cooling process [21–23]. For the analysis of gases

produced under heat load, the determination of heat release and mass loss rate can be performed using the cone calorimeter method [24]. There are other non-normal methods for the evaluation of flame retardants that are sensitive enough to investigate the combustion behaviour of these materials. Several authors [25–32] have already evaluated some flame retardants using different methods [9,33].

Among the suitable test methods, one involves exposing test samples to direct flame [34,35]. In this procedure, actual sample masses are recorded over time, and subsequently, the mass loss rate can be calculated. It is one of the most significant fire engineering parameters used in the past in mathematical models of forest fire spread [36] or in modelling the fire resistance of wooden structures [7,37]. It is also possible to determine the heat release rate HRR from this parameter under certain conditions [38]. The specific testing conditions were already set by the researchers according to the type and kind of material. To compare the effectiveness of different wood retardant treatments with sufficient accuracy, it is necessary to minimise other factors that affect wood combustion. These are mainly the chemical composition, structure, density, material surface, moisture content and geometric shape of the wood [39–42]. Chemical composition and structure are mainly determined by the type of wood. Experimental tests are always conducted on the same species of wood when the retardation effects are compared. Also, the shape of the samples (dimensions), the moisture content of the wood and the material treatment must be identical. The above factors (species, moisture content, dimensions and treatment of the samples) shall be minimised in the experiments.

However, a significant, frequently neglected, factor is the density of the wood. Wood density indicates the mass per unit volume of wood [43], usually given at a specific moisture content because this directly influences it [44,45]. It is calculated as the ratio of the mass and the volume of wood at the same moisture content. According to several authors, wood density influences the combustion process in addition to physical and mechanical properties [46,47]. Wood with a higher density is more difficult to ignite than wood with a lower density because it has better thermal conductivity and more intense heat dissipation from the material surface to the internal zones. This effect on ignitability is particularly pronounced in large wood-based materials [48,49]. In order to achieve better fire-resistant properties in the wood, wood densification is carried out, which removes the intercellular space. Delignification, i.e., the reduction in lignin and hemicellulose content, is also part of the densification process [50,51]. According to several studies, wood densification significantly increases fire retardation [52–54]. The use of flame retardants in the densification process further increases the flame resistance of wood [55–57]. However, there are no scientific studies yet that provide relevant evidence on the extent to which wood density affects the combustion process of undensified wood treated with flame retardants.

This paper aims to investigate how the density of spruce wood influences the burning process of wood samples treated with flame retardants. In particular, the study is applicable in the evaluation of the effectiveness of different wood retardant treatments. As already indicated, the experiments are performed by a test method in which the samples are exposed to a direct flame. The criteria evaluated will be mainly the rate of flame retardancy, mass loss and other additional parameters. We also chose this method because of the simplicity of the test equipment, and we designed the test procedure to reflect the conditions of a real fire. The samples for the experiment will be selected from spruce wood, as it is the most widely used wood species in the construction industry [58,59].

2. Materials and Methods

2.1. Materials

The samples of spruce wood (*Picea abies*) with dimensions 200 × 100 × 10 mm were used for the experiment. They were selected to be free of lumps, resins and other defects that could affect the combustion process. Before the experiment and the application of flame retardants, all samples were placed in the laboratory for two weeks at a temperature of 21 °C and a humidity of approximately 40% to ensure that the samples' moisture content

matched the end-use conditions. The final moisture content of the samples before applying the flame retardants was approximately 12%.

For the first experiment, untreated samples were first used. Due to the high variability in the measured data caused by the samples being burnt through, samples treated with flame retardants were used for further experiments to see if the wood density would affect the experimental results with such samples.

Three different wood flame retardants were applied to three groups of samples. The applied retardants were transparent, water-soluble, colourless coatings based on inorganic salts. Their specific composition is not specified here as the aim of this paper is not to evaluate the retardants themselves. They were applied to all sides of the samples, including their edges, in one coat using a brush. The average amount of coating varied between 60 and 80 g·m^{-2} depending on the specific retardant (according to the instructions of the manufacturers of the individual flame retardants). The retardants on the samples were dried for two months in a room with a temperature of 21 °C and a humidity of about 40%.

The samples were supplied by an external contractor. When measuring the densities of the individual samples, we found a very large variability. The aim was to take advantage of this variability and create groups of samples with various density differences. To investigate the effect of density on the mass loss rate and mass loss, each studied sample group included ten higher- and ten lower-density samples when one type of flame retardant was applied. The designations of each sample group are specified in Table 1. The average sample densities used in the experiment and the range of variation of the densities of the individual samples within a test set are depicted in Table 2.

Table 1. Designation of individual sample groups.

Samples	High-Density Wood Samples (ρ > 450 kg·m^{-3})	Low-Density Wood Samples (ρ < 450 kg·m^{-3})
Samples treated with Retardant 1	R1-HD	R1-LD
Samples treated with Retardant 2	R2-HD	R2-LD
Samples treated with Retardant 3	R3-HD	R3-LD
Untreated samples	U-HD	U-LD

Table 2. Average sample densities and their variation range before flame retardant application.

Samples	Average Density (ρ) [kg·m^{-3}]	Variation Range [kg·m^{-3}]	Difference in Density (HD-LD) ($\Delta\rho$) [kg·m^{-3}]
R1-HD	560.69	47.86	560.69 − 317.00 = 243.69 * (218.92–281.51)
R1-LD	317.00	14.72	
R2-HD	462.12	5.79	462.12 − 411.47 = 50.65 * (44.87–57.32)
R2-LD	411.47	6.66	
R3-HD	455.58	4.62	455.58 − 447.14 = 8.44 * (4.62–12.48)
R3-LD	447.14	12.48	
U-HD	480.20	8.75	480.20 − 378.54 = 101.66 * (84.79–115.02)
U-LD	378.54	21.47	

* Variation range of density difference.

As demonstrated in Tables 1 and 2, the samples were intentionally selected to exhibit varying mean densities within each group. For each retardant coating, the samples comprised both higher (>450 kg·m^{-3}) and lower (<450 kg·m^{-3}) densities, with the magnitude of density differences differing for each coating. The boundary between high and low densities was determined approximately as the median of the densities of all samples used for the research. This design enabled us to establish the relationship between the difference in sample densities and the resulting testing parameters.

The densities of each sample were calculated based on the exact masses and volumes of the samples. The volume of the samples was calculated based on the exact dimensions of the samples. The length of the samples was measured in mm at the centre of the sample to the nearest 0.5 mm. Width was measured with a calliper to hundredths of millimetres at both ends and then averaged. Thickness was measured with a calliper at three points of the specimen to hundredths of a millimetre and then averaged.

2.2. Test Equipment and the Procedure

The test apparatus consists of a gas cylinder, a flow valve, a flow regulator, a burner, a burner holder, a sample holder, precision scales weighing in grams to two decimal places and a computer. Figure 1 demonstrates a schematic diagram with the actual elements of the apparatus.

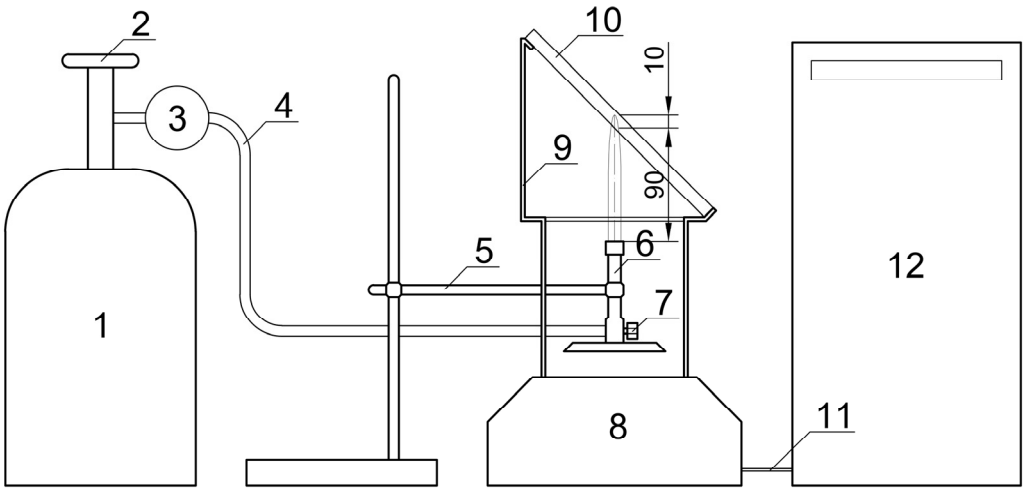

Figure 1. Schematic diagram of the test equipment (description: 1—gas cylinder, 2—shut-off valve, 3—flow regulator, 4—gas intake tube, 5—burner holder, 6—burner, 7—gas flow regulator on the burner, 8—scales, 9—sample holder, 10—sample, 11—connection between scales and computer, 12—computer).

The flow regulator on the cylinder (3) ensures a constant flow of 30 mbar, without interruptions and fluctuations. The burner also has a gas flow regulator (7) to allow precise adjustment of the flame height, which in our case is 10 cm. The samples (10) are fixed in the holder (9) at an angle of 45° to the horizontal plane. With this setup, we also captured the flame propagation. During the experiment, the samples were exposed to a flame heat source applied from the underside, with 1 cm of flame in direct contact with the sample. Propane–butane gas was used as the fuel. The flame temperature of the propane–butane used is 1950 °C, and its calorific value is 44 MJ·kg^{-1} [60]. Scales (8) recorded the actual sample mass at 10 s intervals. Measurement accuracy was ensured by a Mettler Toledo MS1602S/M01 scale (Greifensee, Switzerland) weighing hundredths of grams. Automatic mass recording at set intervals was provided by the BalanceLink 4.2.0.1 programme (Mettler Toledo, Switzerland). The total testing time per sample (exposure to the flame heat source and mass recording) was 10 min. After this time, the mass recording was stopped. Figure 2 is a real photo of the main part of the test equipment during the experiment. The variability of the average values did not exceed the values that were characteristic of untreated wood.

Figure 2. Main part of the test equipment during the experiment.

The study [34] confirms the suitability of this method for testing spruce wood and its treatments. The variability of the values of the parameters tested by this method does not exceed the characteristic variability values for untreated spruce wood.

2.3. Evaluation and Calculation

The main evaluation criterion was the relative mass loss rate, calculated from the measured mass data over time according to the Formula (1).

$$v_r = \frac{m(\tau) - m(\tau + \Delta\tau)}{m(\tau) \cdot \Delta\tau} \cdot 100, \quad (1)$$

where v_r is the relative mass loss rate [%·s^{-1}], $m(\tau)$ is the mass of the sample at a time (τ) [g], $m(\tau + \Delta\tau)$ is the sample mass at a time ($\tau + \Delta\tau$) [g] and $\Delta\tau$ is the time interval at which the mass is read [s]. The time interval for reading the mass was 10 s in our case.

Another datum significant for the statistical detection of the dependence of different average sample densities on the experimental results is the mass loss calculated by the Formula (2).

$$\delta_{mp}(\tau) = \frac{m - m(\tau)}{m} \cdot 100, \quad (2)$$

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

Next, we investigated the fire spread rate parameter, which is the ratio of the maximum value of the mass loss rate and the time to reach this value. This value is determined as the average for the first peak of the mass loss rate according to Formula (3) [61].

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

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].

As this value increases, the fire spread rate also increases [61].

The results presented below are specified as average values.

The dependence of mass loss on sample density was verified by one-factor analysis of variance (ANOVA) using the statistical software "R" (The R Project for Statistical Computing) version 4.1.2.

3. Results and Discussion

In our case, the fundamental parameters for monitoring the influence of the density of spruce wood samples on the combustion process are the mass loss and the mass loss rate. The tested samples were carefully selected to ensure consistency, free of knots, visible resins and any visible deformations or cracks. They displayed identical wood texture and moisture content before coating and were processed by the same method—smooth planning. However, during the experiment, it was observed that some samples burned through from the sides to the top, resulting in a significant increase in the mass loss and, consequently, the mass loss rate. The flame retardants used dilute the combustible gases released from the wood by heat and thus make it more difficult to ignite. In the above cases, the effect of the retardants was apparently insufficient, which may have caused the spontaneous combustion of the samples. This may be due to the application of less retardant to the samples or an overall lower concentration of active substance in the individual retardants combined with a higher flame intensity on the samples, where after a certain time the retardant failed to produce sufficient non-flammable gases to prevent spontaneous combustion. Why this was the case for only some samples is not entirely clear. It may be due to the different properties of the individual samples (chemical composition, resin, porosity, etc.) or possible variations in the testing.

To prevent these outliers from significantly affecting the average values, they were excluded from the overall evaluation. The identification of these extremes was performed statistically using box plots displaying the values of the total mass loss over the 600 s testing time for each group of samples. These box plots were constructed using the statistical software "R" (The R Project for Statistical Computing) version 4.1.2 and are presented in Figure 3.

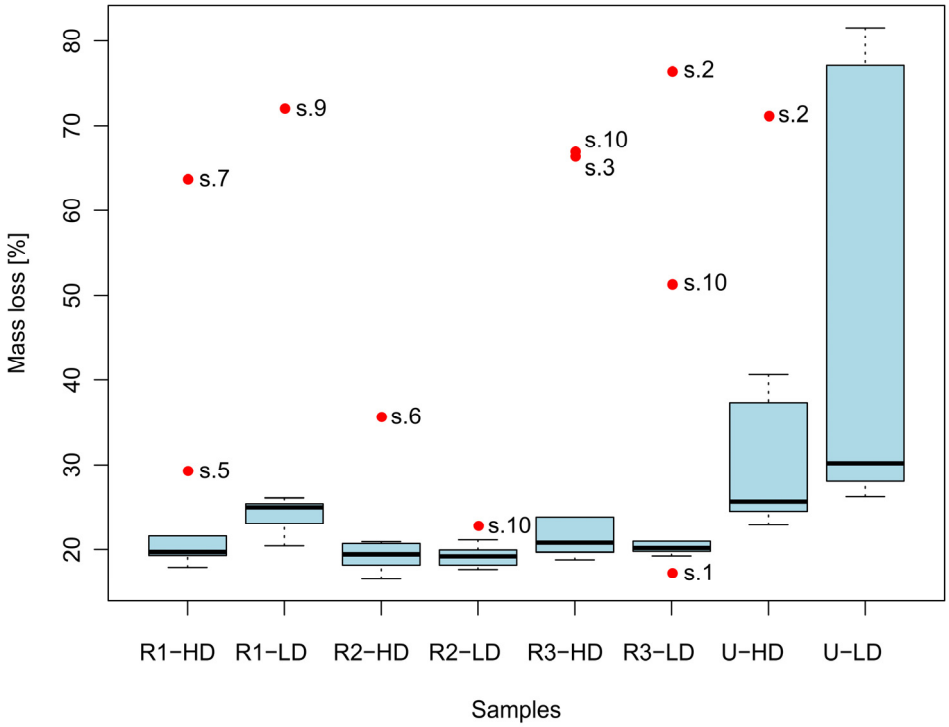

Figure 3. Box plots of the mass loss of each group of samples at 600 s testing time with outliers indicated (red dots).

From the plot in Figure 3, we can see that there were outliers in the total mass loss for each group of samples. The highest number of outliers (three) occurred in the lower-density samples with the application of retardant 3 (R3-LD). It was mainly due to the low variability in the other measured data from that group. The number of outliers for each group of samples was random and did not depend on the treatment applied or the sample density. We then excluded the extreme values (marked by red dots) from further evaluations to minimise their undesired influence on the overall evaluation of the experiment.

The mean values of the mass loss of the samples after the experiment, including the standard deviation, and the maximum mass loss rate are given in Table 3.

Table 3. Average values of mass loss of samples after the experiment and the maximum mass loss rate achieved (indicating the standard deviation).

Specimens	Total Mass Loss (δ) [%]	Standard Deviation of Total Mass Loss (σ_δ) [%]	Max Mass Loss Rate ($v_{r\,max}$) [%·s^{-1}]	Standard Deviation of Max Mass Loss Rate ($\sigma_{vr\,max}$) [%·s^{-1}]
R1-HD	19.47	±1.16	0.0816	±0.0072
R1-LD	23.88	±2.06	0.1650	±0.0196
R2-HD	19.05	±1.50	0.0953	±0.0081
R2-LD	19.09	±1.10	0.1016	±0.0148
R3-HD	20.62	±1.63	0.1173	±0.0182
R3-LD	20.10	±0.53	0.1262	±0.0088
U-HD	28.32	±6.40	0.1699	±0.3986
U-LD	44.12	±24.80	0.4036	±0.2933

The untreated samples exhibited a variability of up to 18% mass loss for the higher-density samples and approximately 55% mass loss for the lower-density samples. This is because the untreated samples experienced spontaneous combustion, with some cases even burning through the side edges to their top.

From the beginning of the experiment to the testing time of 300 s, we monitored significant differences in the mass loss rate of the samples as a function of their density. This can be seen in Figure 4. The lower-density samples displayed a higher mass loss rate than the higher-density samples from the beginning of testing. The highest mass loss rate of the U-LD samples (up to a time of 300 s) was 0.130%·s^{-1}, reached in 60 s. The U-HD samples had the highest mass loss rate (up to a time of 300 s) of 0.100%·s^{-1}, reached in 70 s. The highest mass loss rate of the U-LD samples during the whole testing period was 0.22%·s^{-1} at a testing time of 450 s due to spontaneous sample combustion.

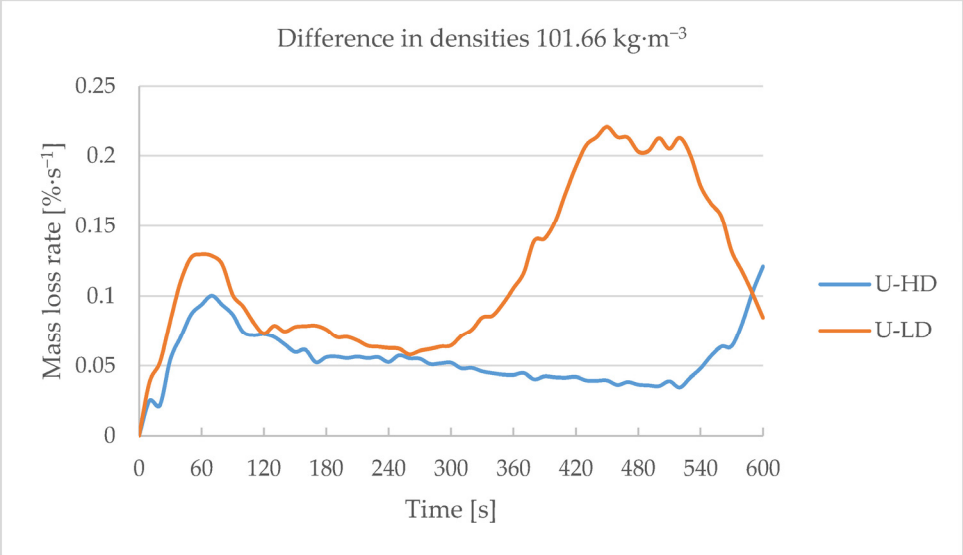

Figure 4. Average mass loss rate of untreated samples over time.

The sudden increase in the mass loss rate of untreated samples occurs at a testing time of about 300 s. In this case, it was the second peak of the maximum mass loss rate, which, according to several authors [62–64], occurred in untreated wood. It caused spontaneous combustion or ignition of the samples. It had a significant telling value. The later the ignition occurs, the safer the material is from a fire protection point of view.

In contrast, untreated samples with higher density (480 kg·m^{-3}) did not exhibit a second peak in the mass loss rate during the experiment. If the testing time were extended, it would probably appear since, from a testing time of 540 s, mass loss rate started to increase significantly until the end of the experiment. However, the second peak in the maximum mass loss rate would occur much later than for the lower-density samples. Thus, it can be argued that the lower-density samples are easier to ignite, in line with [48,49]. Additionally, throughout the experiment, lower-density samples consistently displayed higher mass loss rates compared to higher-density samples.

It was also because of the significant variability in the measured mass loss due to the combustion of untreated samples that we decided to perform experiments using flame retardants on the samples. The assumption was that the retarding effect would prevent overburning of the samples, which would contribute to an objective evaluation of the influence of sample density on the experimental results.

We can conclude that for all groups of flame retardant-treated samples, the variability of the final mass loss over the 600 s experiment time is relatively low (up to 5% mass loss) (see Figure 3). The retardant treatment had a significant retarding effect on the combustion process and, in most cases, prevented their spontaneous combustion.

A one-factor analysis of variance (ANOVA) was performed to determine the mass loss dependence on the density of the wood samples. The analysis was always performed between two sample groups with the same retardant treatment but different densities. The

results are presented in Table 4 for the 300 s test time mass loss and Table 5 for the 600 s test time mass loss.

Table 4. ANOVA of sample mass loss at 300 s testing time.

Samples Treated with Retardant 1, Difference in Density 243.69 kg·m^{-3}						
	Df	Sum Sq	Mean Sq	F value	Pr (>F)	Influence
Density (ρ)	1	107.64	107.64	39.05	1.56×10^{-5}	Strong influence
Residuals	15	41.34	2.76			
Samples Treated with Retardant 2, Difference in Density 50.65 kg·m^{-3}						
	Df	Sum Sq	Mean Sq	F value	Pr (>F)	Influence
Density (ρ)	1	0.424	0.4245	0.453	0.51	No influence
Residuals	16	14.982	0.9364			
Samples Treated with Retardant 3, Difference in Density 8.44 kg·m^{-3}						
	Df	Sum Sq	Mean Sq	F value	Pr (>F)	Influence
Density (ρ)	1	0.012	0.0124	0.011	0.919	No influence
Residuals	13	15.175	1.1673			

Table 5. ANOVA of sample mass loss at 600 s testing time.

Samples Treated with Retardant 1, Difference in Density 243.69 kg·m^{-3}						
	Df	Sum Sq	Mean Sq	F value	Pr (>F)	Influence
Density (ρ)	1	82.23	82.23	28.38	8.46×10^{-5}	Strong influence
Residuals	15	43.46	2.90			
Samples Treated with Retardant 2, Difference in Density 50.65 kg·m^{-3}						
	Df	Sum Sq	Mean Sq	F value	Pr (>F)	Influence
Density (ρ)	1	0.011	0.0105	0.006	0.939	No influence
Residuals	16	27.743	1.7340			
Samples Treated with Retardant 3, Difference in Density 8.44 kg·m^{-3}						
	Df	Sum Sq	Mean Sq	F value	Pr (>F)	Influence
Density (ρ)	1	1.011	1.011	0.645	0.436	No influence
Residuals	13	20.372	1.567			

The results of the ANOVA test at 300 s test time indicated statistically significant differences in mass loss as a function of density for retardant 1-treated samples. Samples with retardant treatment 1 demonstrated a significant dependence with a p-value of $p = 1.56 \times 10^{-5}$. However, for the sample groups treated with retardants 2 and 3, there was no longer any mass loss dependence on sample density. For these sample groups, the average difference in densities was within 50 kg·m^{-3}. Similar results were also found at a testing time of 600 s (Table 5).

For the samples treated with retardant 1, we observed a significant difference in the mass loss rates among the higher- and lower-density samples (Figure 5). The average difference in densities here was approximately 244 kg·m^{-3}. We also saw the highest difference in the mass loss rate in the first few seconds of the experiment (up to a testing time of about 90 s), where the lower density samples (R1-LD) displayed twice the maximum mass loss rate as the higher density samples. Although this phenomenon only occurred in the first 90 s of the experiment, it resulted in a significant difference in the final mass loss, as indicated by the dependence of the mass loss on the density of the samples in Tables 4 and 5. The maximum mass loss rate of the R1-LD samples was reached at a testing time of 50 s with a value of 0.158%·s^{-1}, while the R1-HD samples had their highest mass loss rate at a testing time of 70 s with a value of 0.077%·s^{-1}. However, differences in the mass loss rates were still observed up to a testing

time of about 300 s (R1-LD samples had a noticeably higher one). After this time, the mass loss rates of both the higher- and lower-density samples were virtually identical due to the retardant treatment of the samples, which prevented them from spontaneous combustion and had a significant effect on the mass loss rate.

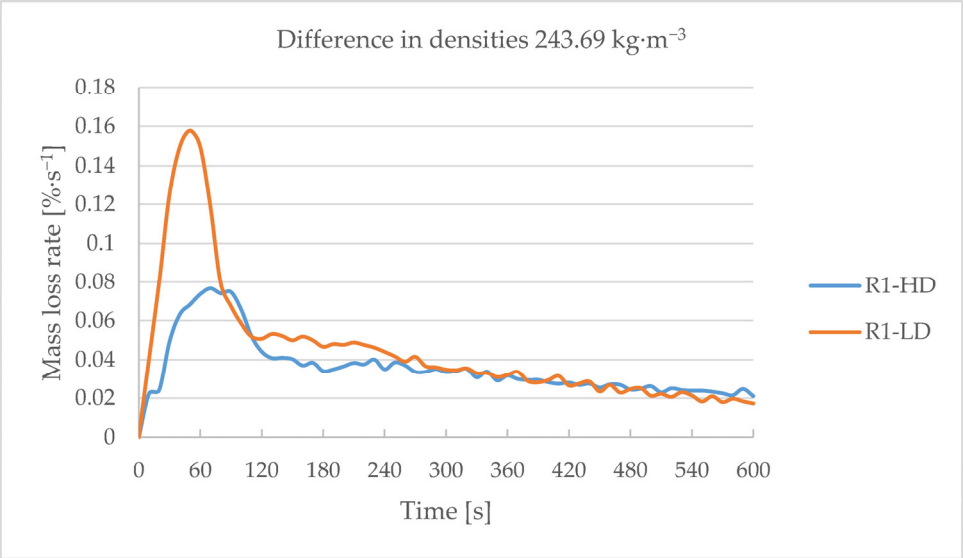

Figure 5. Average mass loss rate of samples treated with retardant 1 over time.

Samples treated with retardant 2, which had a difference in densities of approximately 50 kg·m^{-3}, and samples treated with retardant 3, with a difference in densities of about 8.5 kg·m^{-3}, showed significantly lower variations in the mass loss rates (Figures 6 and 7), which were negligible. The differences primarily occurred in the maximum mass loss rate within the initial seconds, with a difference of 0.002%·s^{-1} for samples treated with retardant 2 and 0.010%·s^{-1} for samples treated with retardant 3. Consequently, the average mass loss rate of the higher- and lower-density samples was virtually identical throughout the experiment. These differences also had no significant effect on the total mass loss, as confirmed by Tables 4 and 5, where the dependence of the mass loss on the sample density was not evident for samples R2 and R3.

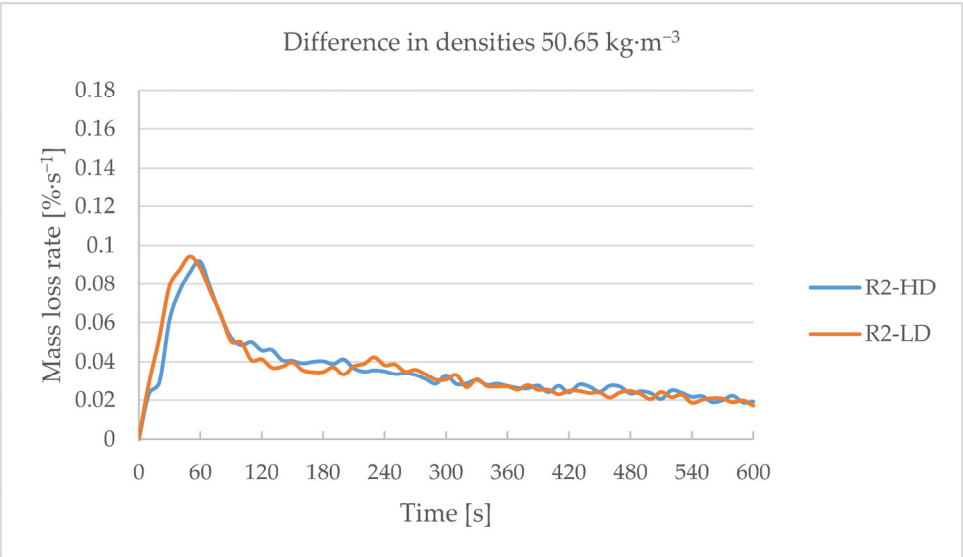

Figure 6. Average mass loss rate of samples treated with retardant 2 over time.

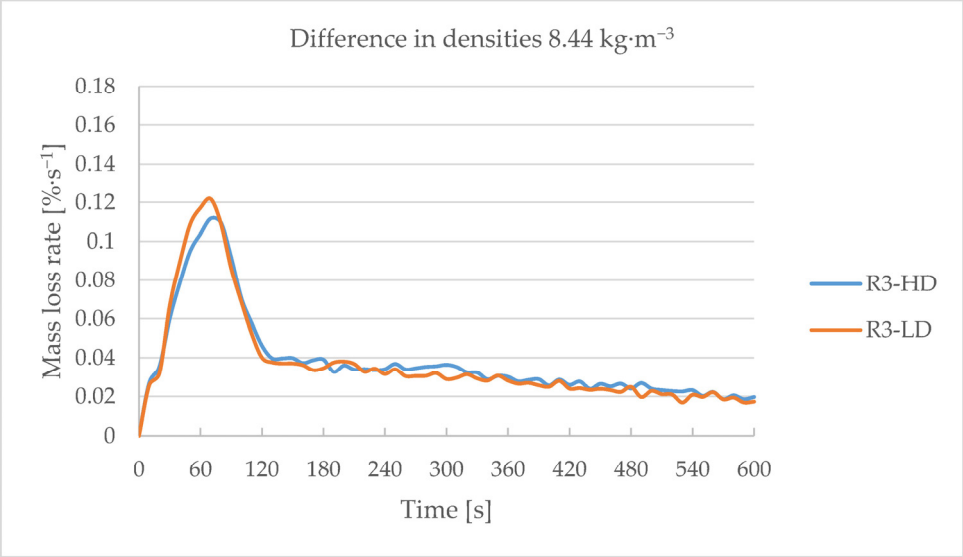

Figure 7. Average mass loss rate of samples treated with retardant 3 over time.

From the point of view of fire spread, not only the maximum value of the mass loss rate was significant, but also the time to reach this value. The ratio of these two parameters (Equation (3)) provided us with the rate of fire spread. The higher the value, the more the material contributed to the fire spread [61]. Table 6 demonstrates the fire spread rate (R_{fs}) values for each group of samples. These values were averaged for the first peak of the mass loss rate (up to a time of 100 s). Here, we observed that as the difference in densities between the sample groups increased, the difference in R_{fs} values also increased. The fire spread rate between two sample groups with identical treatment was always higher for the lower-density (LD) samples. For example, the samples with the highest density difference R1 exhibited a fire spread rate R_{fs} difference of 0.00206, whereas the samples with the lowest density difference (R3) had a difference in R_{fs} values of 0.00014.

Table 6. Fire spread rate (proportion of the maximum value of the first peak mass loss rate and the time to reach this).

Samples	**R1-HD**	**R1-LD**	**R2-HD**	**R2-LD**	**R3-HD**	**R3-LD**
R_{fs} [%·s^{-2}]	0.00109	0.00316	0.00153	0.00189	0.00160	0.00174
Difference in R_{fs} [%·s^{-2}]	0.00206		0.00036		0.00014	

Higher differences in densities are logically associated with the higher differences in fire spread rates, as demonstrated by the graph in Figure 8. The strong linear dependence of the difference in fire spread rate on the difference in sample densities with a reliability coefficient of R^2 = 0.99 supports this assumption. Based on the above results in Figure 8 and Table 6, it is evident that spruce wood with lower density exhibits a higher fire spread rate, which has significant implications for testing wood flame retardants.

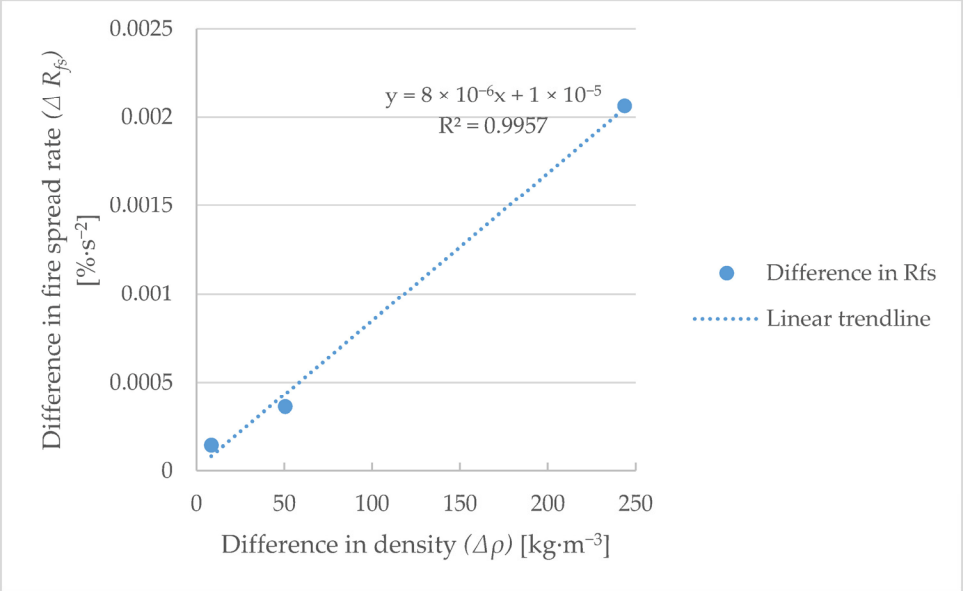

Figure 8. Dependence of the difference of fire spread rate on the difference of sample densities.

The above experimental findings demonstrate the significant impact of spruce wood sample density on test results, specifically the evaluation criteria of mass loss rate, mass loss and fire spread rate.

For samples treated with retardants, the experimental outcomes were likewise influenced by sample density, depending on the density difference. Samples with an approximate density difference of 244 kg·m^{-3} exhibited significantly different maximum mass loss rates, with lower-density samples showing higher rates. However, when flame retardants were applied, there was no second peak in the mass loss rate, confirming the retardants' effect in prolonging ignition time, as reported by [62], effectively preventing combustion initiation.

The mass loss parameter, which is dependent on sample density, was also significantly affected (confirmed by ANOVA tests in Tables 4 and 5). As the density difference decreased, the dependence of mass loss rate and mass loss on sample density also decreased. Figures 6 and 7 reveal that with density differences up to approx. 50 kg·m^{-3}, there were no significant differences in the mass loss rate, and the dependence of mass loss on sample density was also not confirmed (Tables 4 and 5).

These findings underscore the vital role of sample density in wood testing and the effectiveness of wood retardant treatments. However, the results are subject to the variability of sample densities selected for testing. Experiments revealed that lower variability in sample densities minimised the impact on test results. For precise testing, a difference in sample densities of up to 50 kg·m^{-3} is optimal, as supported by ANOVA tests (Tables 4 and 5) and fire spread rate versus time plots (Figures 5–7). Nevertheless, it is challenging, if not impossible, to select samples with identical density. In addition, other properties (e.g., material processing) and defects or deformations (excessive resin content, bulges, cracks, etc.) that can also affect testing results should be considered when selecting samples [65].

4. Conclusions

The study evaluated various fire-technical parameters of spruce wood with different densities, including some samples treated with wood flame retardants. The spruce wood samples were divided into several groups with identical treatments (or no treatment) but different average densities, gradually reducing the density difference to establish a limit for the dependence of experimental results on the difference in sample densities. The experiment utilized a simple method where the samples were exposed to a direct flame at a 45° angle. The monitored parameters were mass loss and mass loss rate, with an

additional parameter being the fire spread rate. The conclusions from the experiment can be summarised as follows:

- Untreated samples exhibited significant variability in the measured data (mass loss variability of untreated samples with low density was 55%, with higher-density samples at 18%), unlike samples treated with retardants, which showed a lower variability of mass loss, approximately 5%. This significant variability in mass loss for untreated samples is mainly attributed to their ignition after a specific time, resulting in a subsequent rapid increase in the mass loss rate. The results confirm, among other findings, that flame retardants significantly prolong the ignition time.
- Untreated samples with a density difference of about 100 kg·m^{-3} displayed a clear density dependence on the mass loss. Samples with a lower density have a significantly higher average mass loss rate over the entire duration of the experiment.
- For samples treated with retardant 1 with a density difference of approximately 244 kg·m^{-3}, a dependence of the mass loss rate on the sample density was observed. The most significant difference was in achieving the maximum mass loss rate, which was twice as high for the lower-density samples compared to the higher-density samples. Also, the mass loss dependence on the sample density was statistically confirmed by the ANOVA test (p-value = 8.46 × 10^{-5} at a testing time of 600 s).
- Lower-density samples exhibited higher mass loss and mass loss rate. The study results further indicated that the sample density also affected the fire spread rate parameter (R_{fs}). As the sample density decreased, the value of R_{fs} increased, showing a linear relationship between the difference in R_{fs} values and sample densities with a reliability coefficient of R^2 = 0.99.
- For samples treated with retardants with a density difference of up to 50 kg·m^{-3}, no dependence of the mass loss rate on sample density was observed. The ANOVA statistical analysis did not confirm the mass loss dependence on the sample density. Therefore, the difference in densities of up to 50 kg·m^{-3} no longer significantly affected experimental results, particularly parameters such as the mass loss rate and mass loss. Consequently, selecting the test samples with such a variation range of densities can be considered optimal without impacting the test results.

The research was aimed at investigating the influence of spruce wood density on mass loss and mass loss rate when using flame retardants. The research was carried out on groups of samples with different densities using three flame retardants. In order to refine the research, it would be advisable to complement the experiments so that several flame retardants were applied for each density respective of the difference in densities. However, such research would be very challenging, especially due to the selection of samples with different densities. In our case of "simplified research", we had hundreds of samples available to us, from which we selected only 80 suitable for testing.

Author Contributions: Conceptualization, P.M. and L.M.O.; methodology, P.M. and L.M.O.; software, P.M.; validation, M.K.; formal analysis, M.K.; resources, P.M. and L.M.O.; writing—original draft preparation, P.M.; writing—review and editing, L.M.O. and M.K.; visualization, P.M.; supervision, L.M.O. and M.K.; project administration, P.M.; funding acquisition, P.M. All authors have read and agreed to the published version of the manuscript.

Funding: This article was funded by the Grant System of University of Zilina of the project: Experimental Determination of Fire-technical Parameters of Alternative Building Materials and Evaluation of its Fire Safety. Project No. 16961.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article.

Acknowledgments: This work was supported by the Grant System of University of Zilina No. 1/2022 (17324).

Conflicts of Interest: The authors declare no conflict of interest.

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