

Article

The Influence of Different Types of Expandable Graphite on the Thermal Resistance of Spruce Wood

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Abstract: In order to not limit the possibilities of using wood due to its flammability, the implementation of fire protection is an essential requirement. An integral part of the research on the fire protection of wood is the determination of the effectiveness of the applied protective means and their effect on changing its behavior under fire conditions. In this work, samples of spruce wood (*Picea abies* (L.) H. Karst) (50 mm × 40 mm × 10 mm) were treated with an aqueous solution of sodium silicate and different types of expandable graphite flakes that were applied to the surface of the samples. The fire characteristics of the samples were studied using a non-standard test method, the radiant heat source test, which was used to determine the mass loss, burning rate, and ignition time of the test samples, and the measurement was carried out via visual recording with a thermal camera. The results of the laboratory test method showed a significant positive effect of the application of all types of expandable graphite flakes. The main insight is that a suitable wood modification using expandable graphite flakes in combination with water glass has the potential to reduce mass loss by at least 73 ± 3% and reduce the temperature rise on the surface of the sample. The lower temperature of the surface of the burning material reduces the possibility of heat transfer to the surrounding materials and thus reduces the rate of fire spread.

Keywords: flame retardant; expandable graphite; water glass; mass loss; burning rate; surface temperature



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1. Introduction

Wood has been used for many years for fuel; as a construction material; and for making various tools, furniture, pulps, and paper. Wood (and plant biomass in general) is currently also used as a carbon source to produce many monomers and biopolymers (including biodegradable ones) that are used in various industries, from energy to pharmaceuticals [1–3]. Wood is composed of three main components, cellulose, hemicelluloses, and lignin, that form unique and complex structures. Softwoods include 33%–42% cellulose, 22%–40% hemicelluloses, 27%–32% lignin, and 2%–3.5% extractives. Hardwood species contain 38%–51% cellulose, 17%–38% hemicelluloses, 21%–31% lignin, and 3% extractives [4–6]. These substances have different resistance to heat and fire, which is why wood is flammable if exposed to heat, flames, or sparks. In addition to the chemical composition, the flammability of wood depends on various factors, such as its density, moisture, and the presence of other substances [7]. Compounds used to protect wood against fire reduce the flammability of the surface. They protect the wooden material from significant damage, especially to its mechanical properties, by acting as a barrier between the fire source and the substrate. The mechanisms to reduce burning include changing the process of the pyrolysis of wood, isolating the surface layer, changing the wood thermal properties, and diluting pyrolysis gases [8,9].

Flame retardants are chemicals that are used to slow or prevent the spread of fire. They are commonly used in wood products to reduce the risk of fire. There are various types of wood flame retardants, including halogenated, phosphorus-based, and nitrogen-based compounds. Halogenated flame retardants are the most widely used type of flame retardants for wood. They include chemicals such as bromine and chlorine, which can be effective at reducing the flammability of wood. However, they can also have negative environmental and health effects, such as being persistent in the environment and potentially toxic. Some phosphorus compounds have high retardant efficiency, good stability, and wide application, including wood protection. They can be effective at reducing the flammability of wood and are generally less toxic and more environmentally friendly than halogenated flame retardants. Nitrogen-containing flame retardants are a newer type of flame retardant for wood. They can be effective at reducing the flammability of wood, and some are more ecological and less toxic than halogenated flame retardants [10–12]. Some conventional retarders no longer comply with legislative requirements; therefore, a lot of effort is devoted to the application of new, eco-friendly preparations. Nanocomposites are the new generation of fire retardants which improve the flammability of wood. Nanocomposite fire retardants induce the formation of char and prevent char oxidation. It forms a thermal insulation layer on the wood and prevents the release of volatiles for further combustion [13–16].

Expandable graphite (EG) is a commonly used material in the field of fire retardancy because of its ability to prevent or slow the spread of fire. It is a form of graphite that has been chemically treated to expand when exposed to high temperatures or fire, creating a barrier that blocks oxygen from reaching the flames. When expandable graphite is heated, it expands and forms a layer of insulating char that prevents further oxidation of the material underneath. This layer acts as a physical barrier that inhibits the transfer of heat and the spread of flames. A similar effect was observed in precharred wood. The char also releases carbon dioxide, which further helps to suppress a fire. In addition to its fire-retardant properties, expandable graphite also has good thermal and electrical conductivity, as well as high strength and stiffness. These properties make it a versatile material that can be used in a wide range of applications beyond fire retardancy. Therefore, expandable graphite can be used in a variety of applications, including as a fire retardant in building materials, textiles, plastics, and electronics. It is particularly useful in applications where fire safety is critical, such as in aircrafts and other transportation vehicles [17–22].

In the field of reducing the flammability of various materials, expandable graphite has very promising possibilities, but its applications in the protection of wood from fires have not been rigorously investigated yet, although some works on this topic have been published. Its retardation effect is influenced by the method of application to the wood surface, particle size, expansion temperature, etc. [23–26].

Therefore, the aim of this work was to evaluate different types of expandable graphite in combination with water glass, Na_2SiO_3 , to enhance the fire resistance of spruce wood.

2. Materials and Methods

2.1. Wood Treatment

For this work, specimens of Norway spruce (*Picea abies* (L.) H. Karst) with dimensions of 50 mm × 40 mm × 10 mm (length × width × thickness) and moisture content of approximately 12% were used. Specimens were partitioned into 8 sets; each set contained 5 pieces. The 1st set was an unmodified sample of sound spruce wood; the 2nd set (WG) was treated with a concentrate aqueous solution of water glass (sodium silicate). Sets 3 to 8 were firstly treated with concentrate aqueous solutions of WG, and then covered by the corresponding designation of EG flakes (Table 1), and finally, they were sprayed with a 50% aqueous solution of WG. The wood specimens were covered on the top with concentrated WG using a brush, and the surface was then sprinkled with EG flakes and subsequently sprayed with a 50% WG. All three components, WG, EG, and 50% WG, were weighed before application, and the ratio of WG/EG/50% WG was 1:1:2. The quantity of all used

layers of retardants was $250 \text{ g}\cdot\text{m}^{-2}$. The modified specimens were then dried to a constant weight at ambient temperature.

Table 1. Specification of used EG flakes.

Product	Company (Country)	Particle Size (μm)	pH	Onset Temperature ($^{\circ}\text{C}$)	Expansion Volume (mL/g)
GG-210-200N	Graf Guard (US)	90	5–8.5	210	80
GG-200-100N	Graf Guard (US)	150	5–8.5	200	175
Thermographite 10 F-150	Epsomite (CZ)	150	5–9	180–220	100
Thermographite 25 K + 180	Epsomite (CZ)	180	5–9	180–220	250
Thermographite 20 K + 300 HST 260	Epsomite (CZ)	300	5–9	260	200
Thermographite 40 D + 500 LST	Epsomite (CZ)	500	5–9	180–220	400

2.2. Samples Analyses

We used the non-standard test method—the radiant heat source test. The test conditions were the same as those used by Kmeťová et al. [25]. The total time of the experiment was 600 s; then, we calculated the relative mass loss and relative burning rate from the obtained data.

2.3. Surface Temperature Measurement

With the thermal camera Fluke RSE600 (Fluke Corporation, Everett, WA, USA), images were taken during the test using the Smart View R&D software IRSoft2 at selected points—A, B, C, and D (Figure 1). Point A was at the top of the sample, point B was at the top edge of the front side of the sample, point C was in the middle of the front side, and point D was at the bottom edge of the front side of the sample. All samples had the same orientation in relation to anatomical directions.

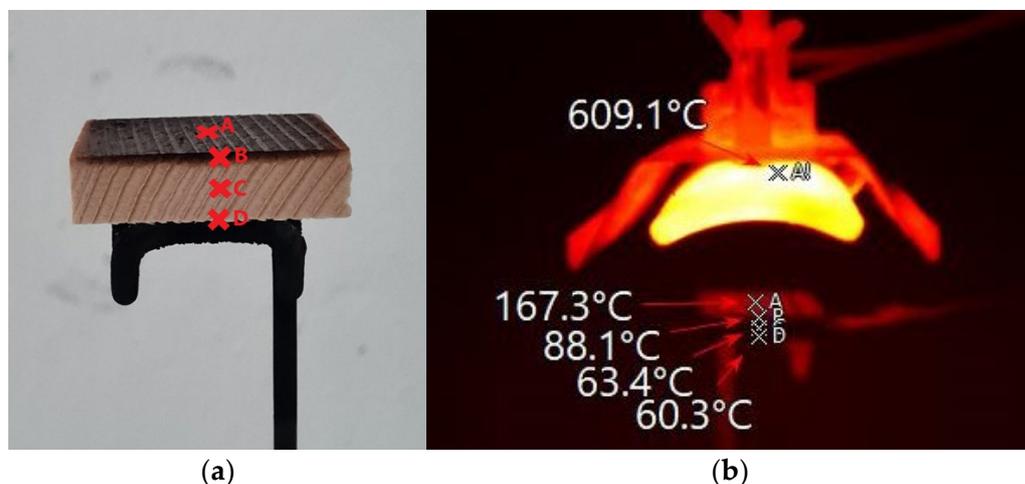


Figure 1. Locations of points for thermal camera measurement (a) and infrared snapshot (b).

3. Results and Discussion

In the materials and methodology section, series of experiments were introduced to monitor the relative mass loss, relative burning rate, and time to ignition of the test specimens after exposure to the thermal infrared heater. The results of the monitored evaluation criteria are shown in Figures 2–11.

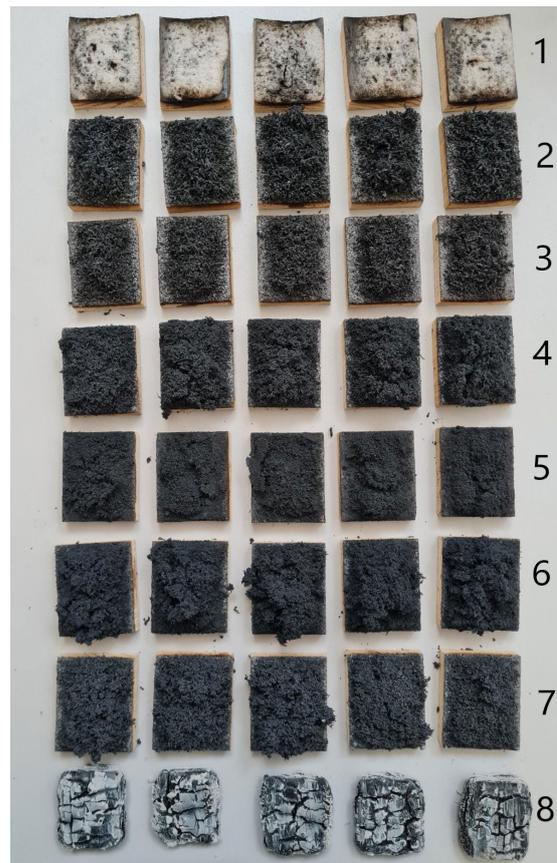


Figure 2. Visual appearance of samples after the experiment (1—WG; 2—40 D + 500 LST; 3—20 K + 300 HST 260; 4—25 K + 180; 5—10 F-150; 6—GG-200; 7—GG-210; 8—untreated).

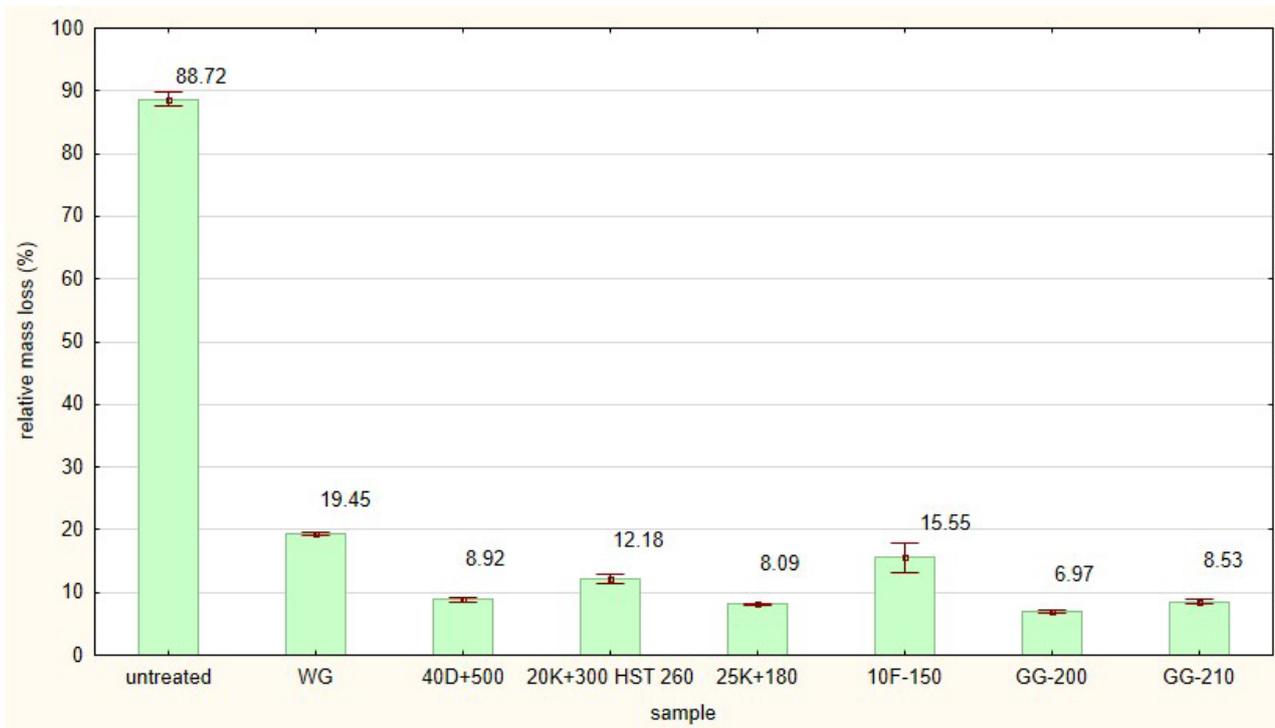


Figure 3. Relative mass loss of tested samples.

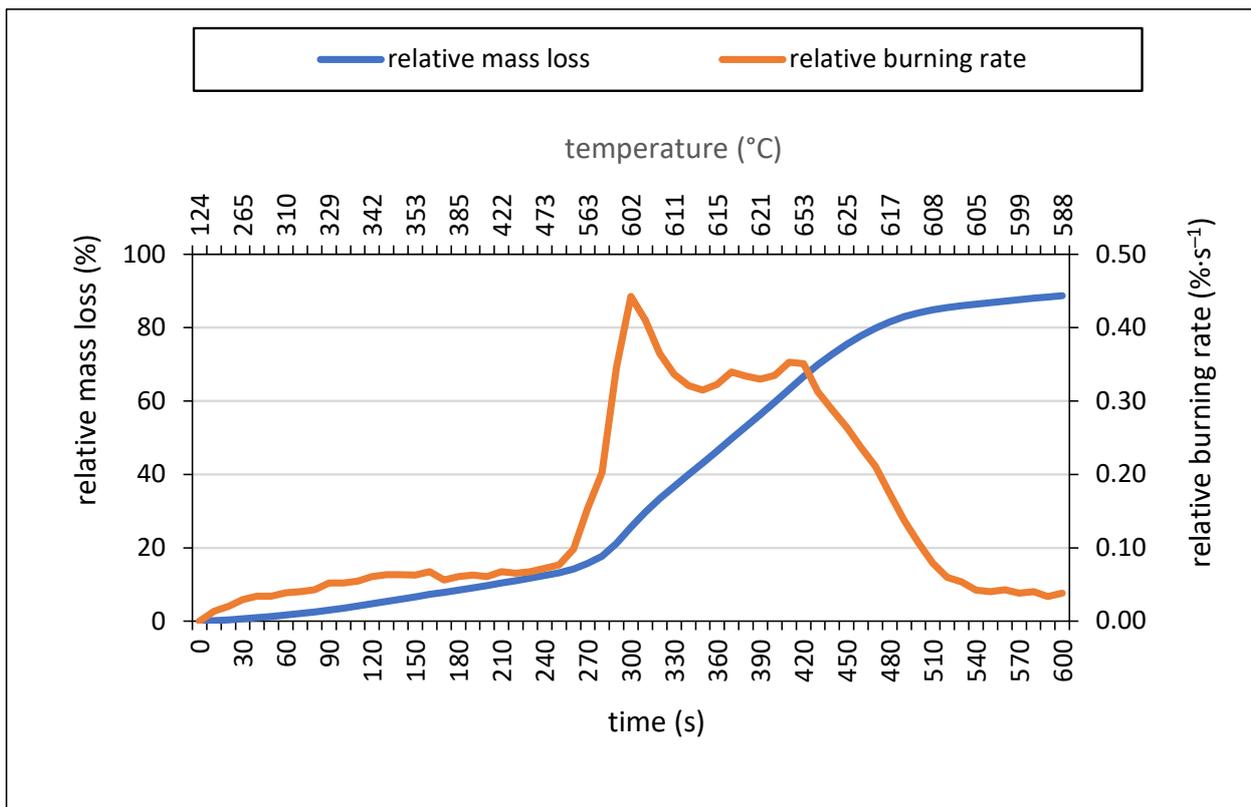


Figure 4. Untreated sample.

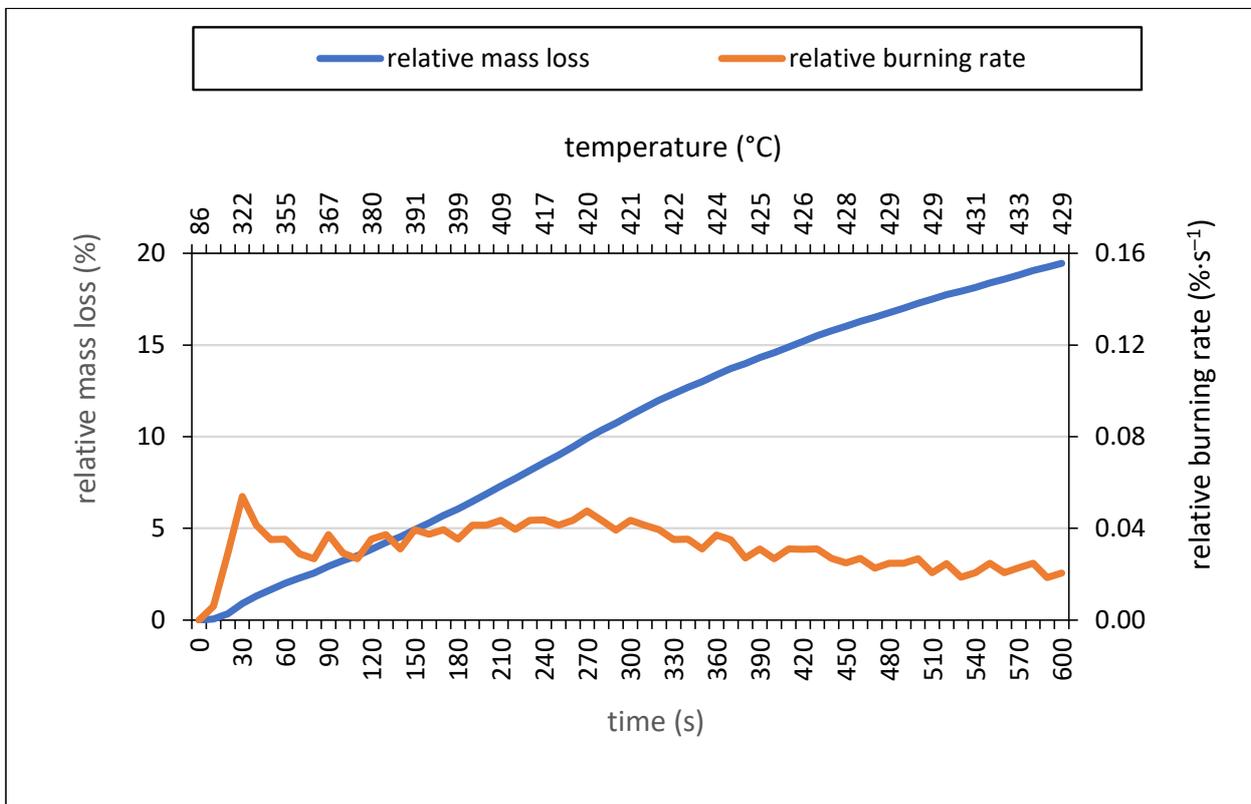


Figure 5. WG sample.

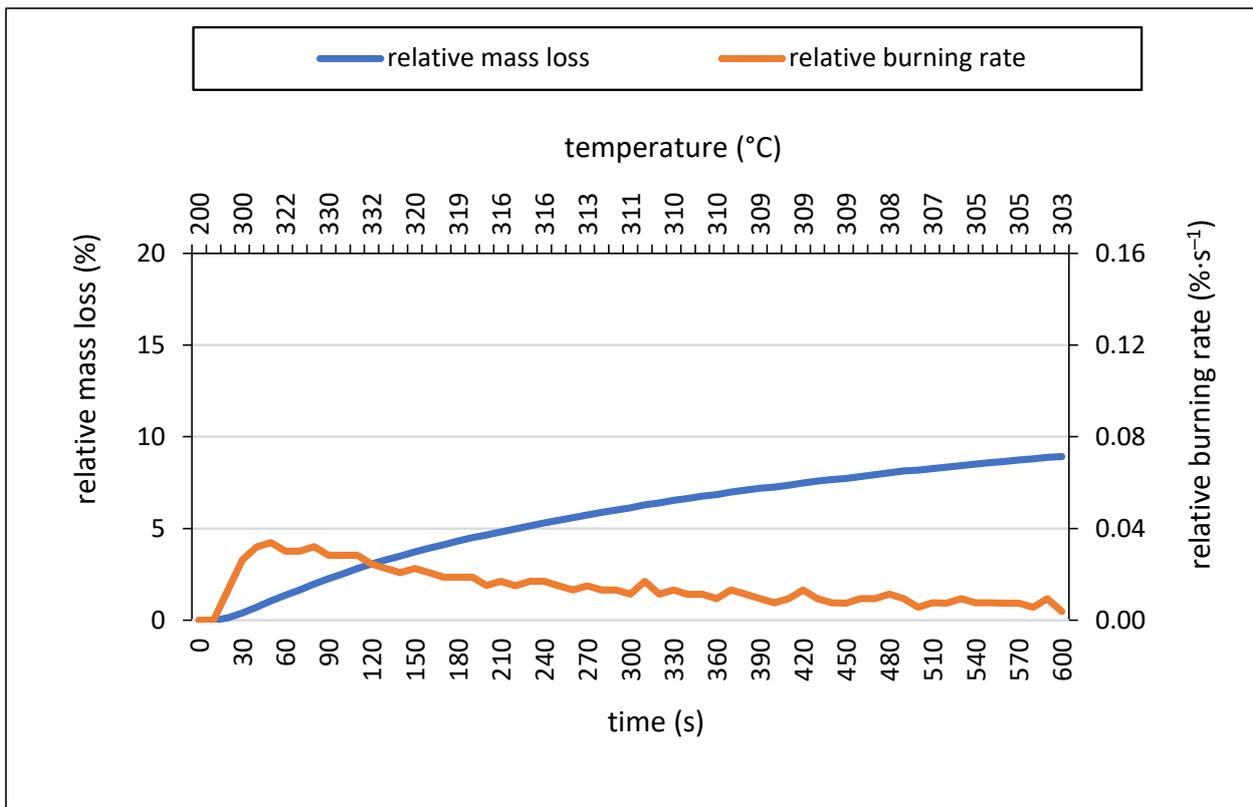


Figure 6. The 40 D + 500 LST sample.

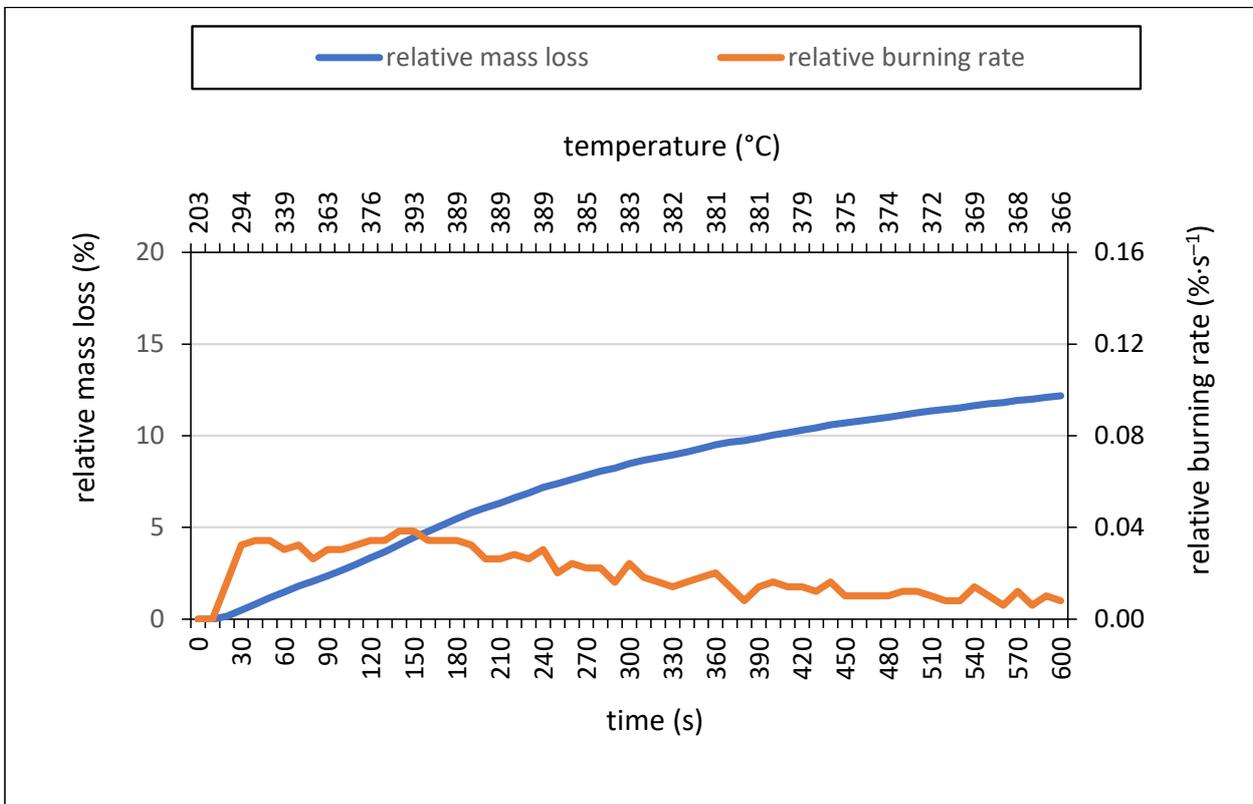


Figure 7. The 20 K + 300 HST 260 sample.

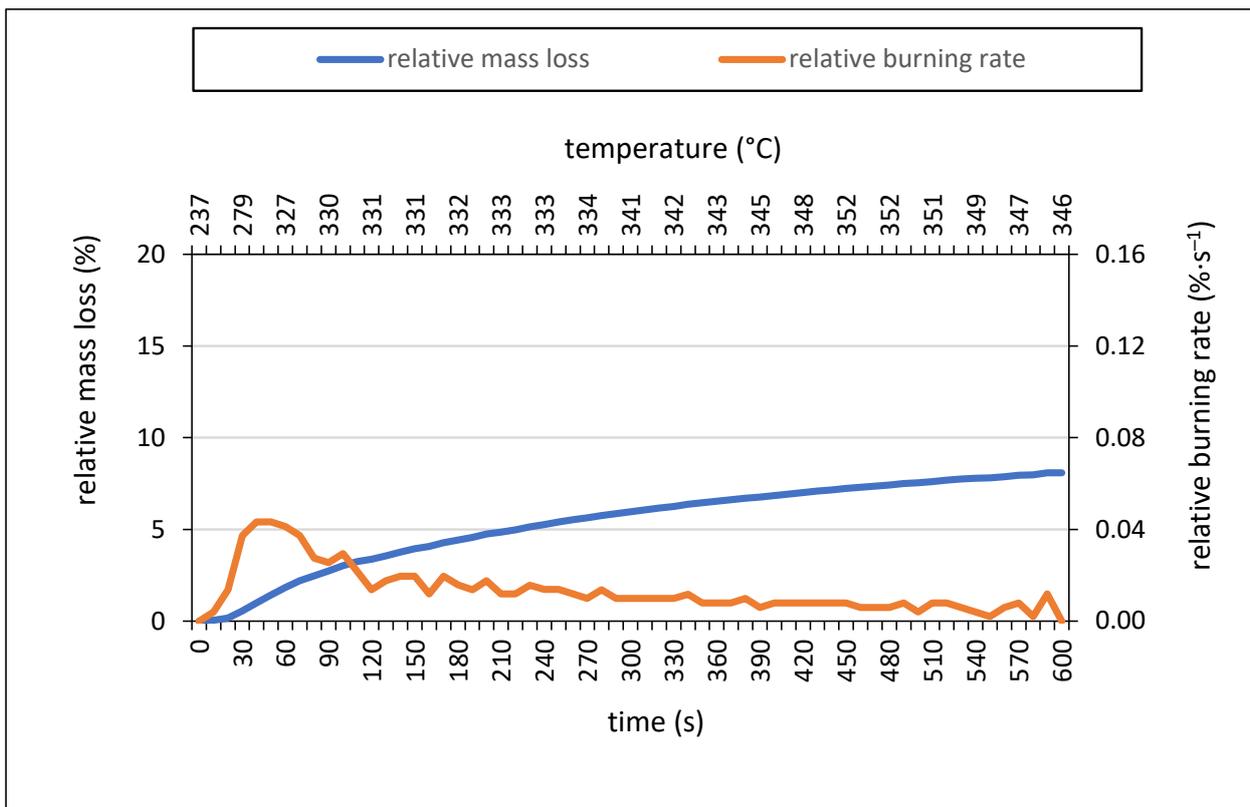


Figure 8. The 25 K + 180 sample.

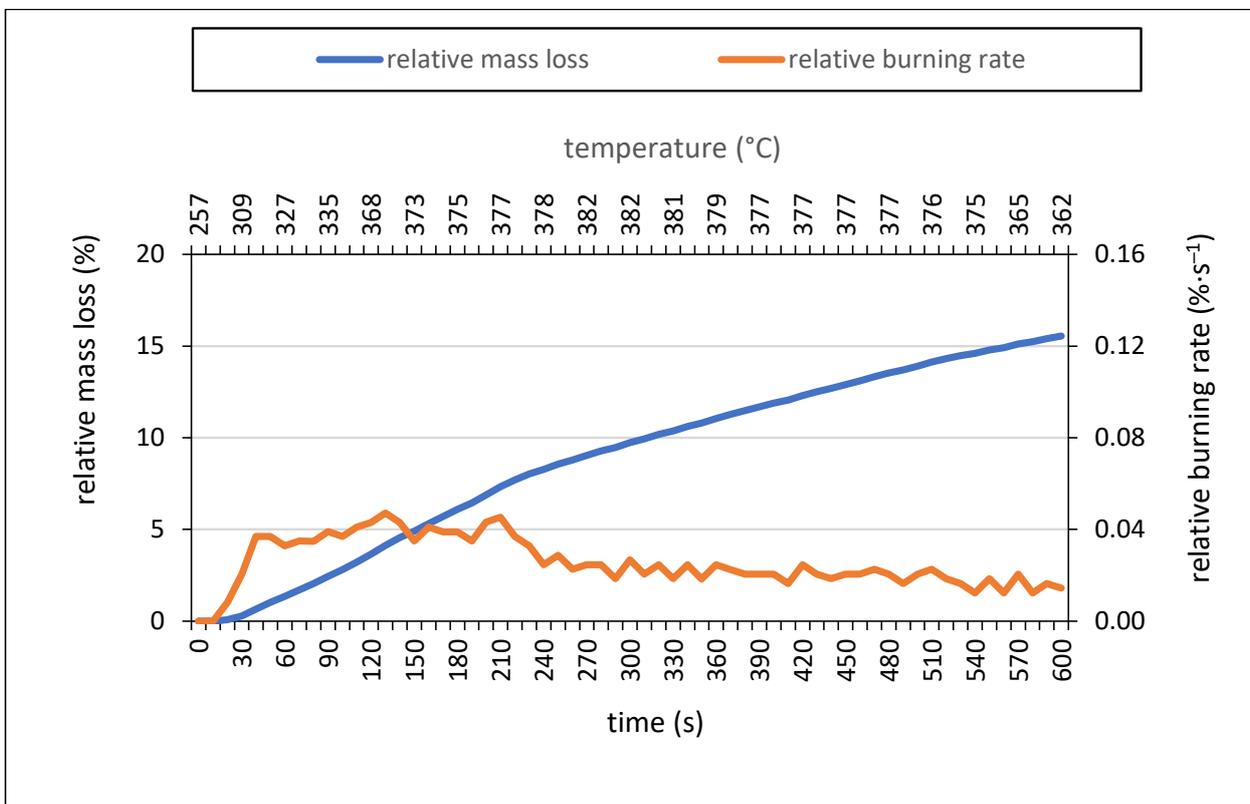


Figure 9. The 10 F-150 sample.

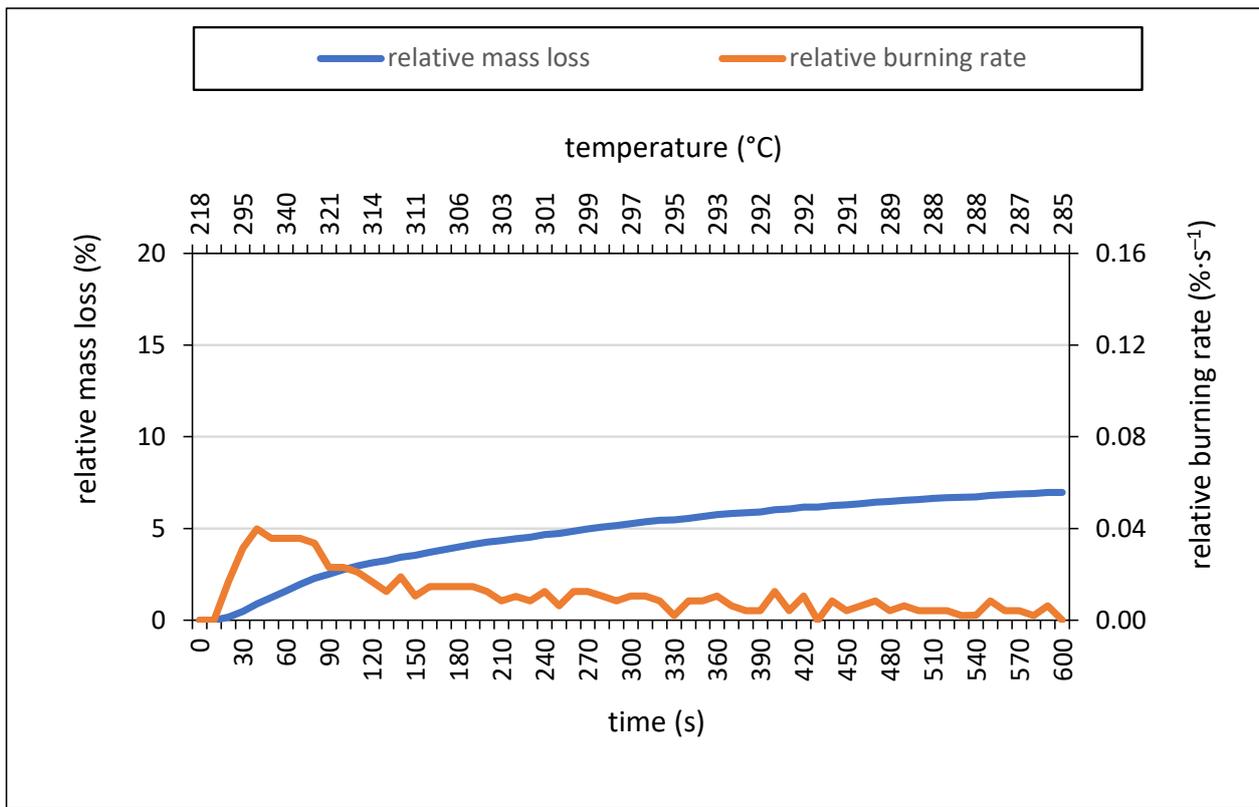


Figure 10. GG-200 sample.

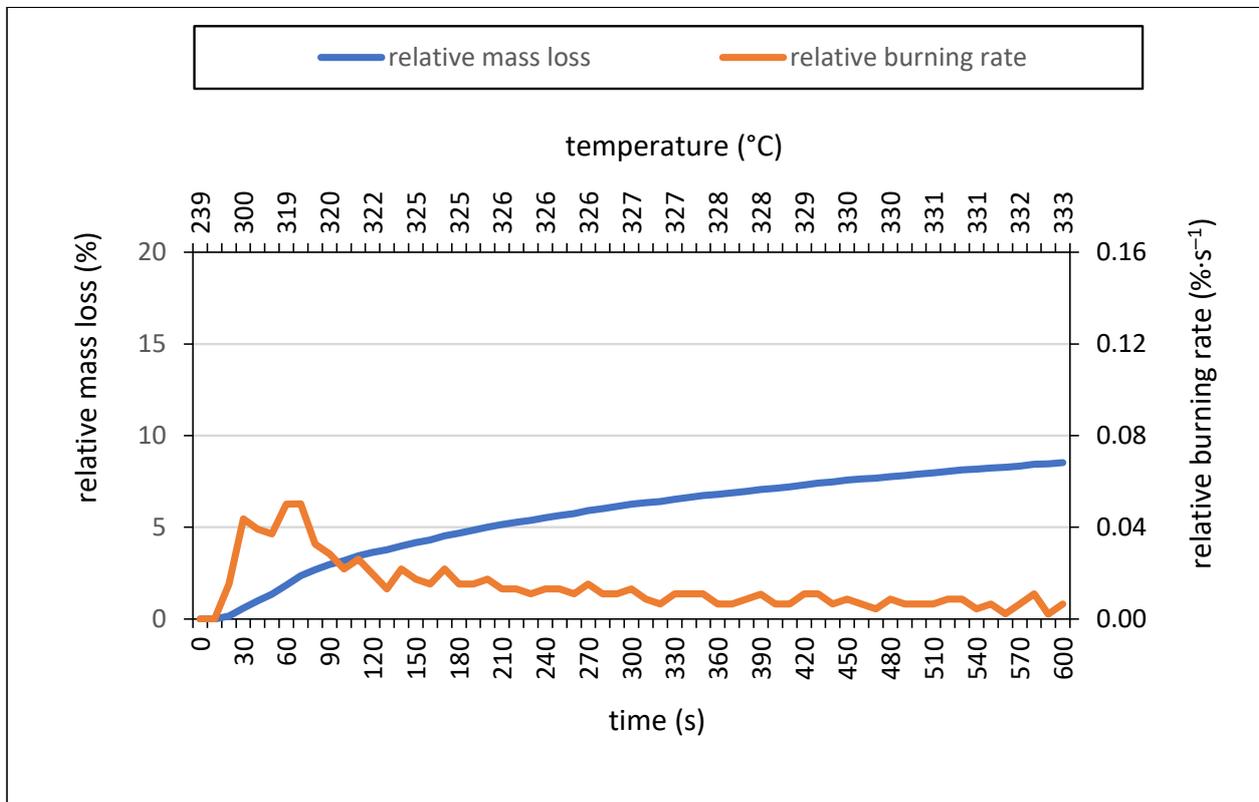


Figure 11. GG-210 sample.

From the measurements, we calculated the relative mass loss and the relative burning rate for all seven types of treatment and for untreated spruce wood samples. Untreated wood samples achieved a significant difference in terms of relative mass loss compared to treated samples, which lost up to $88.72 \pm 1.10\%$ of their original weight, which is the highest relative mass loss among the tested samples. The treatment of samples with only WG, without the addition of EG, reduced the relative mass loss by $69.27 \pm 1.32\%$ compared to the untreated samples. The treatment of the samples with WG together with EG further improved the results from the point of view of the mentioned evaluation criterion. The relative mass loss of samples treated with a combination of EG and WG was in the range of $6.97\text{--}15.55 \pm 2.63\%$. Similar values were achieved by the samples GG-210, 25 K + 180, and 40 D + 500, even though they were treated with EG with a significantly different fraction—90, 180, and 500. If we compare the samples based on their relative mass loss, we recorded the best results in the case of the GG-200 samples; worse results were obtained by samples 25 K + 180, GG-210, and 40 D + 500 LST, followed by samples of 20 K + 300 HST 260. Of the samples treated with a combination of EG and WG, the worst in terms of relative mass loss was 10 F-150. They were followed by samples treated only with water glass. Overall, the samples of untreated spruce wood performed worst. A certain percentage of the weight loss of the treated samples was probably also caused by the decomposition of some components of the flame retardant.

In a study of the fire resistance of oak wood with an aqueous solution of sodium silicate and EG flakes [25], the difference between the mass loss of untreated oak wood samples and samples treated with the combination of EG (expansion ratio (X:1): 270 to 325; supplied by Sigma-Aldrich (Saint Louis, MI, USA)) and the WG on the top side of the sample was 79%. These results indicated that the proper treatment of wood with WG and EG has the potential to improve flame-retardant properties.

In the following Figures 4–11, we separately present mutual comparisons of the relative mass loss, the relative burning rate, and the temperature of the samples on the surface during the entire duration of the test for untreated samples and for all seven types of retardation treatments.

If we compare samples based on the relative burning rate, the ranking is different than when comparing samples based on the relative mass loss. Overall, the highest relative burning rate of $0.443\% \cdot s^{-1}$ was recorded for untreated spruce wood samples at the 300th s at a surface temperature of $600\text{ }^{\circ}\text{C}$. Among the treated samples, the highest burning rate was recorded for the WG samples: $0.054\% \cdot s^{-1}$ at 30th s. For the 40D + 500 LST samples, we measured the lowest value of the maximum burning rate of $0.034\% \cdot s^{-1}$ in the 50th s when the surface temperature was $320\text{ }^{\circ}\text{C}$. This were closely followed by the 20 K + 300 HST 260 samples with a maximum relative burning rate of $0.038\% \cdot s^{-1}$ at the 150th s. The maximum relative burning rate for the other treated samples was in the range of $0.040\% \text{--} 0.050\% \cdot s^{-1}$. It is important to note that with the treatment of wood with flame retardants, we managed to significantly reduce the burning rate.

No correlation was found between particle size and mass loss, and similarly, no dependence was found between particle size and burning rate. In contrast, a very strong correlation between burning rate and weight loss was confirmed, which is a logical finding. Wang et al. [26] studied the effect of the particle size of flaky graphite (1, 2.6, 4.8, 9.5, and $15\text{ }\mu\text{m}$) on the flame resistance of intumescent flame retardant coatings. The results of the cone calorimeter showed that graphite with an average particle size of $9.5\text{ }\mu\text{m}$ had the highest flame-retarding efficiency due to the formation of continuous laminar char residue. However, the finer graphite particles showed worse fire-resistant properties, as evidenced by the loose and porous char residue. This indicates that EG with appropriate particle size certainly contributes to the improvement in flame resistance while the physical barrier prevails [26].

The temperature tendency on point A (at the specimen surface) was approximately the same for all treated specimens. Specimens covered with EG + WG had a steeper temperature rise during the first 60 s of the test. Samples treated only with WG reached an

approximately 40 °C higher temperature compared to samples treated with EG + WG. As for the untreated sample, at the 240th s, a sharper increase in temperature began to occur. The maximum temperature of 653 °C was reached in these samples in the 420th s. And after the flame ended, the temperature started to drop again, whereas for samples treated with EG + WG, the maximum surface temperature reached was around 390 °C.

In the following Figures 12–14, we show the course of temperatures on points B, C, and D during the test.

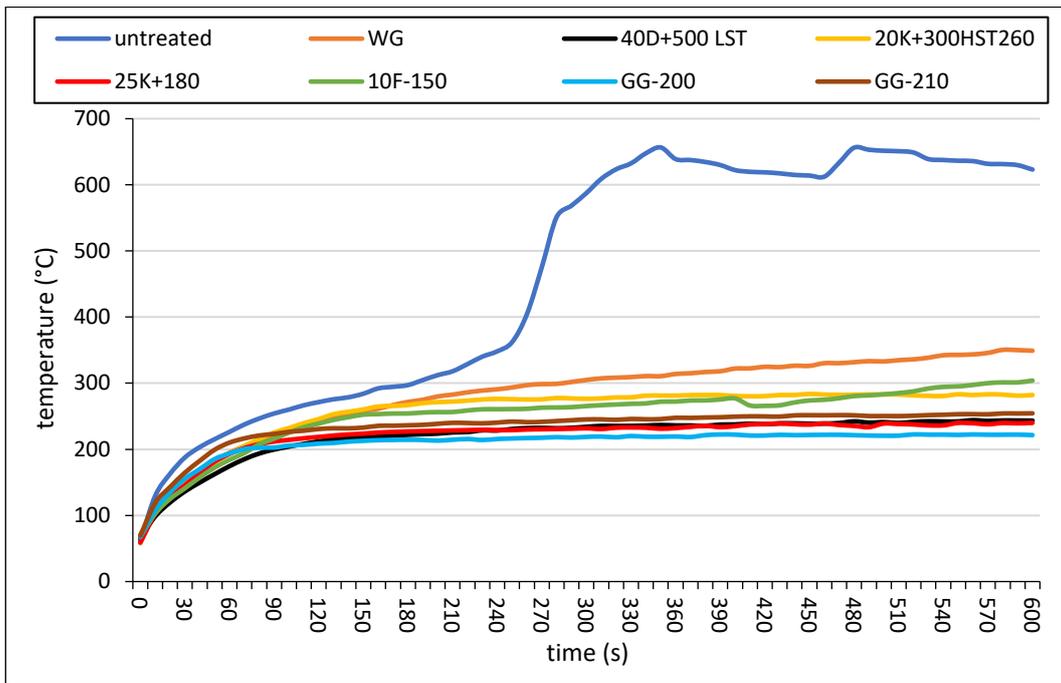


Figure 12. Temperature course of the samples on point B.

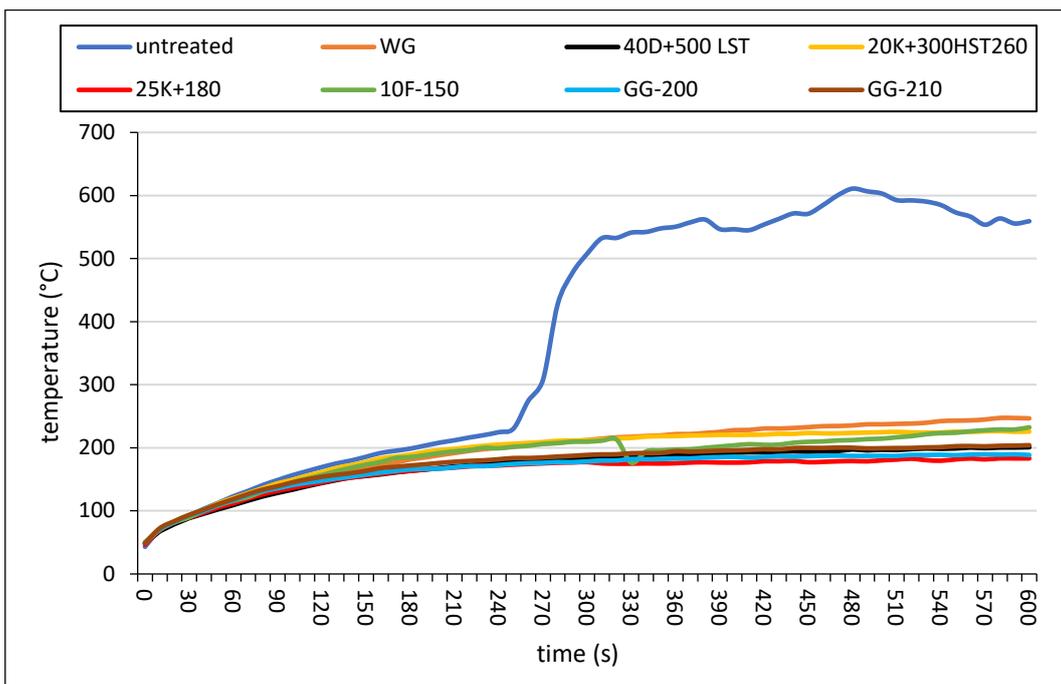


Figure 13. Temperature course of the samples on point C.

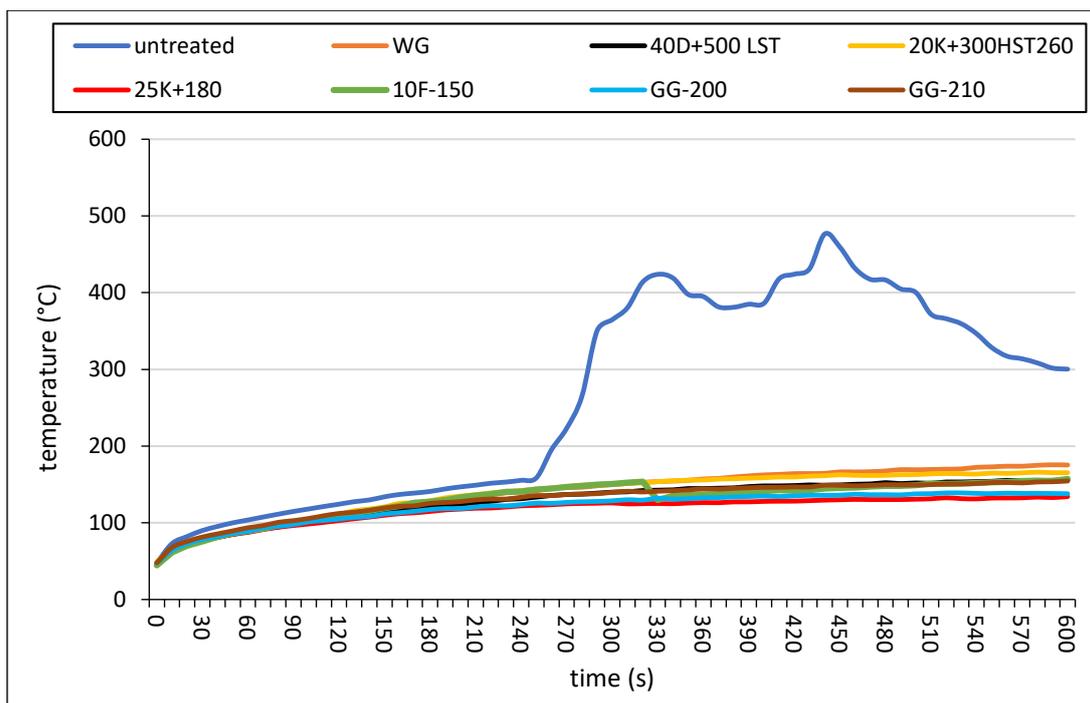


Figure 14. Temperature course of the samples on point D.

The temperature tendency on point B (at the upper edge of the sample front side) was approximately identical for all treated samples. Only with samples 10 F-150 was there a slight decrease in temperature by 30 °C in the 400th s and then an increase up to the value of 304 °C. The samples treated with EG and WG had a sharper increase in temperature during the first 60 s of the test, and subsequently, the temperature was almost constant; the highest temperature was reached at the end of the test (in the 600th s) in the range of 220–304 °C. The samples treated with WG alone reached an at least 45 °C higher temperature compared to the samples treated with EG + WG. As for the untreated sample, a sharper increase in temperature began to occur at the 250th s. The maximum temperature of 656 °C was reached in these samples in the 350th s. And after the flame stopped burning, the temperature started to drop again.

The temperature tendency on point C (in the middle of the sample front side) was approximately identical for all treated samples. In the case of samples 10 F-150, there was a slight decrease in temperature again, and then an increase up to a value of 232 °C. In the samples treated with EG and WG, the temperature increased during the first 120 s of the test, and subsequently, the temperature was almost constant; the highest temperature was reached at the end of the test (in the 600th s) in the range of 188–232 °C. Of the treated samples, the WG samples reached the highest temperature, namely 246 °C at the end of the test. As for the original samples, a sharper rise in temperature started to occur at the 250th s. The maximum temperature of 606 °C was reached in these samples in the 490th s.

The temperature trend on point D (at the lower edge of the sample front side) was approximately the same for all treated samples. In the case of samples 10 F-150, there was a slight decrease in temperature again, and then an increase up to a value of 232 °C. For the treated samples, the highest temperature was reached at the end of the test (in the 600th s) in the range of 137–175 °C. As for the untreated samples, a sharper increase in temperature started to occur at the 250th s. The maximum temperature of 476 °C was reached in these samples in the 440th s.

Therefore, if we compare the course of temperatures for individual samples, all samples reached their maximum temperatures precisely on point B—at the upper edge of the sample front side. With the location of the points lower on the sample, there was a decrease in the maximum temperature during the experiment. The maximum temperatures were

reached by the untreated samples on all points. Of the treated samples, the WG samples had the highest temperature values.

The ignition time was another evaluation criterion. In the case of treated samples, their ignition did not occur during the experiment. Untreated specimens ignited at a time of 269 ± 10 s, at a surface temperature of approx. 530 °C. None of the samples sustained flame burning until the end of the experiment (600 s).

Wang et al. [27] investigated the burning characteristics of wood. They reported that the time to ignition is the most important characteristic of flammable materials from the aspect of fire prevention. This value can be used to evaluate and compare the fire resistance of materials. The longer the ignition time, the better the fire resistance of the material. Increasing the ignition time of wood is a significant characteristic of the fire resistance of wood materials. Chun et al. [22] used a different method to apply EG. They mixed EG (approx. 180 μm size) and wood flour (approx. 150 – 180 μm size) in various proportions up to 50 wt% of EG and prepared samples (100 mm \times 100 mm \times 10 mm) from these mixtures and tested their physical and thermal properties. They found out that both the total heat released and the thermal conductivity decreased 15-fold (MJ/m^2) and 3-fold ($\text{W}/\text{m}\cdot\text{K}$), respectively. The expandable graphite added to the composite materials adopted worm-like shapes because of combustion, and it formed a fine lattice layer structure that could reduce the thermal conductivity.

Water glass is formed from an aqueous solution of sodium silicate. When the water in the solution evaporates, the solute is a glassy solid. Depending on its form, water glass is ready for myriad uses in industries across the world [28,29].

A coating of water glass on wood and other lignocellulosics surfaces improves their passive fire safety. As an adhesive for paper and cardboard, it is specifically used in situations involving high heat, such as baking, or in situations where contact with an open flame is common. When heated to drive off the water, the result is a hard translucent substance called silica gel, widely used as a desiccant. It can withstand temperatures up to 1100 °C [29,30]. Among the significant advantages of alkaline silicates for wood treatment, beyond those previously mentioned like the high efficiency against fire, are the low thermal expansion, the generation of smoke of low toxicity during conflagration, and their low cost, which constitute other excellent factors [31].

During the preparation of fire-resistant coatings, water glass becomes a deflocculant of graphite flakes in the form of an intumescent additive, ensuring an even suspension of graphite and an increase in the strength and stiffness of the applied formulation on wood surfaces.

This was a pilot study of the combination of these two components with the assumption of their fire resistance capacity. In the next phase, it is possible to work on improving the resistance of the protective layer against water, which would enable its use in exterior applications as well.

The alkaline silicate solutions spread on glass allowed the inference to be made that with higher silica content in their composition, the films show a higher curing rate as well as a smaller water dissolution rate [32]. Reducing the solubility from wood can be also improved by acid treatment, when silanols chemically react with hydrogen ions liberated from, e.g., phosphoric acid protonating the oxygen of the silanol. This intermediate reacts with the silanol, forming a stable siloxane bond. Another option for strengthening adhesion to the wood surface could be to ensure a reaction with cations when the silanol groups of alkaline silicates react with di- and tri-valent cations, forming metal silanol heterobridges, which then react to give metal silicate polymers, liberating hydrogen gas [31].

4. Conclusions

Seven different treatments to increase the fire resistance of spruce wood were studied—samples only with an aqueous solution of water glass and samples with EG flakes from different companies in combination with an aqueous solution of water glass. In addition, the results were also compared with a sample of untreated wood. The best results in terms

of mass loss were obtained with the GG-200 samples, followed by the 25 K + 180 samples. Untreated samples achieved the worst results. The difference between the mass loss of untreated and treated samples was $69 \pm 1\%$, with the addition of EG to WG up to $73 \pm 3\%$. It is important to note that when treating the wood with flame retardants, we also managed to significantly reduce the burning rate. In terms of temperature course, the maximum temperatures were reached at the top of the samples (on point A). As the points were placed lower on the sample, there was a decrease in the maximum temperature during the experiment. In the case of the treated samples, their ignition did not occur during the experiment. It is therefore evident that monitoring the surface temperature of the sample is important from this perspective. The surface temperature at ignition depends not only on the material properties but also strongly on the experimental conditions (irradiation and sample configuration). The results showed that the proper treatment of wood with WG and EG has the potential to improve fire performance and is necessary and addressed by the correct selection of EG. EG flakes can also be used in multiple layers of the composite, or simply as the reactive substance in the fire protective layer. In combination with WG, these are fire-reactive coatings that have very specific functions and provide improved functionality through their synergistic interactions. In addition, these are good sources for the effective design of environmentally friendly intumescent systems for wooden architectural and construction features to attain durability without using harmful chemicals. The subject of further research will therefore be to search for the most effective combination of these two components and their improved preparation technology.

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