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The Effect of Intumescent Coating Containing Expandable Graphite onto Spruce Wood

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Abstract: Wood, one of the materials predominantly employed in construction, possesses various advantageous properties alongside certain drawbacks, such as susceptibility to thermal degradation. To enhance wood fire resistance, one approach involves the application of flame retardants. This study compared the fire-retardant effectiveness of expandable graphite, bonded with water glass, as a coating for spruce wood against commercially available fire-retardant treatments. Spruce wood samples (*Picea abies* (L.) H. Karst) underwent treatment with three distinct retardants: expandable graphite in combination with water glass, Bochemit Antiflash, and Bochemit Pyro. The fire-technical characteristics of the samples were examined by a non-standard test method—a test with a radiant heat source. The experiment evaluated the fire-retardant properties by recording changes in sample mass, burning rate, and temperature difference. The best results among all flame retardants were achieved by expandable graphite in combination with water glass, in all evaluation criteria. Among all the flame retardants used, expandable graphite in combination with water glass achieved the best results in all evaluation criteria.

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

Citation: Kmeťová, E.; Kačíková, D.; Kačík, F. The Effect of Intumescent Coating Containing Expandable Graphite onto Spruce Wood. *Coatings* **2024**, *14*, 490. https://doi.org/10.3390/coatings14040490

Academic Editor: Marko Petric

Received: 22 March 2024
Revised: 11 April 2024
Accepted: 15 April 2024
Published: 16 April 2024

1. Introduction

Wood, a highly sustainable and renewable lignocellulosic material, offers a versatile range of architectural applications, from structural components of walls and roofs to doors, windows, flooring, and furniture. Wood is a renewable, ecological material with many excellent mechanical, physical, and aesthetic properties. However, its use in construction is questioned due to its flammability [1–4].

Wood is composed of three main components, cellulose, hemicelluloses, and lignin, which form unique and complex structures. Coniferous wood species contain 33%–42% cellulose, 22%–40% hemicelluloses, 27%–32% lignin, and 2%–3.5% extractives. Broadleaf wood species contain 38%–51% cellulose, 17%–38% hemicelluloses, 21%–31% lignin, and 3% extractives. These substances have different resistance to heat and fire. 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 [5–8].

Flame retardants are substances that reduce the flammability of materials. They are used to slow down or stop the spread of fire and protect the material from significant damage. Different types of flame retardants are based on different principles. Halogen flame retardants are the most common type used on wood. They contain chemicals like bromine and chlorine that can help reduce the flammability of wood. However, they can also have a negative impact on the environment and health, such as their durability in the environment and potential toxicity [9–11]. There are also many groups of important flame retardants based on different phosphorus containing compounds used in the field of flame retardants, with huge importance [12]. The methods for enhancing the flame

retardancy of polymeric materials are usually based on flame-retardant modifications either in the bulk polymer matrix or on the polymer surfaces. Adding flame retardants into the polymer matrix by physical blending, copolymerization of flame retardant with the polymer chains, and surface treatment are the fundamental flame-retardant methods for polymeric materials [13,14].

Based on chemical composition and mechanism of action, flame retardants can be categorized into organic flame retardants and inorganic flame retardants. Among them, inorganic flame retardants are known for their excellent thermal stability and smoke suppression, while organic flame retardants and polymers have good compatibility. Combining the advantages of both to obtain low-toxicity and high-efficiency flame retardants is a hot spot for future research [15].

The research and development of new flame retardants for wood protection is receiving a lot of attention from ecological, economic, and legislative points of view. In addition to commercially available flame retardants, such as Bochemit Antiflash or Bochemit Pyro, we also use promising new flame retardant alternatives. One of them is expandable graphite. Expandable graphite (EG) is used due to its ability to prevent or slow down the spread of flame. It is a form of graphite that has been chemically modified to expand when exposed to high temperatures or fire. When EG is heated, it expands and forms an insulating layer of carbon that prevents further oxidation of the material underneath. This layer acts as a physical barrier that prevents the transfer of heat and the spread of flames. The charcoal also releases carbon dioxide, which further helps slow down the burning [16,17]. Specifically, composites of EG with various materials such as polypropylene, ground tire rubber, erythritol, epoxy resin, paraffin, polypropylene/wax blends, and high-density polyethylene exhibit improved flame-retardant properties [17–19].

The main component of Bochemit Antiflash is boric acid and 2-aminoethanol. This flame retardant ensures a reduction in the flammability of wood by reducing the speed of its burning and the spread of the flame on the surface of the wood. If wood treated with Bochemit Antiflash is exposed to flame in the prescribed amount, the active substances begin to decompose into non-flammable gaseous substances when heated, which are released from the treated surface, and they release the wood into the surroundings and dilute the oxygen necessary for the burning of wood to such an extent that is not sufficient for further flame propagation. Additionally, the heat activates the fire retardant, causing it to form a foamed insulating layer on the wood's surface. This layer acts as a barrier, preventing the flame from directly contacting the wood. Absorbing a significant portion of the flame's heat hinders heat transfer and protects the underlying wood. This results in slowing down burning and accelerating the formation of a charred surface layer of wood. This layer has a significant thermal insulation effect and prevents the flame's further spread [20].

The main component of Bochemit Pyro is potassium carbonate. This flame retardant reduces the flammability of wood by reducing its burning speed and flame spread on the surface of the wood. If wood treated with Bochemit Pyro is exposed in the prescribed amount under the action of a flame, the active substances begin to break down into non-flammable gaseous substances upon heating, which are released from the surface of the treated wood into the surroundings and dilute the oxygen necessary for the burning of the wood material to such an extent that it is not sufficient for the further spread of the flame. At the same time, by heating the treated wood, a foamed insulating layer is formed on the surface, which prevents direct contact of the flame with the surface of the wood, thereby preferentially absorbing the heat of the flame and preventing its access to the surface of the wood. This has the effect of slowing down the burning again and accelerating the formation of a charred surface layer of wood. This layer has a significant thermal insulation effect and prevents the further spread of the flame [21].

Expandable graphite (EG) is a flame-retardant intumescent material; when heated, it forms worm-like shapes, and it forms a fine lattice layer hexagonal structure. A distinctive flame-retardant characteristic of EG is that sulfuric acid is intercalated between its

crystalline structure (carbon layers). At elevated temperatures, the EG becomes oxidized due to a redox process in reaction with sulfuric acid and forms evolved gases including carbon dioxide, sulfur dioxide, and water. These gases help dilute the heat and oxygen concentrated near the fire zone [18,22,23]. The blowing effect of evolved gases that are escaping through the EG leads to an increase in the volume of EG. This char suffocates the flame and hinders the transfer of heat and mass from the underlying substrate. The char can also act as a physical barrier to diffuse oxygen and heat from the burning zone. Due to a low oxygen concentration, flame spread becomes difficult [21,24]. EG has not only the flame-retardant effect but also has minimal smoke production and low toxicity [25–27]. In addition to its properties as a flame retardant, EG also has good thermal and electrical conductivity, high strength, and stiffness [28,29]. These properties make it a universal material that can be used in a wide range of applications, e.g., flame retardant, conductive additive, absorbent, covering product, graphene precursor, and graphite foils. Therefore, EG can be used in various fields including fire protection of wood, and the flame-retardant property of the composite of wood and EG improved as the total heat release rate (HRR) decreased [17,18,29]. The Bochemit Antiflash, based on boric acid and 2-aminoethanol, acts mainly chemically; its usage is limited (due to ammonia gas). The potassium carbonate-based Bochemit Pyro has multiple fire-fighting mechanisms. At lower temperatures, it dehydrates the burning material (cooling effect). At higher temperatures, it decomposes, releasing potassium oxide that dilutes oxygen and melts to form a glaze, limiting flammable gas release and isolating the surface. The expandable graphite with water glass mainly generates a physical barrier. Expandable graphite expands when heated, creating an insulating layer that slows fire spread. Water glass binds everything together and additionally hinders flammable gas release.

This work aimed to compare the fire-retardant effectiveness of expandable graphite, bonded with water glass, as a coating for spruce wood against commercially available fire-retardant treatments after exposure to a radiant heat source.

2. Materials and Methods

2.1. Wood Treatment

Samples of Norway spruce (*Picea abies* (L.) H. Karst) were prepared from trunk wood to the size of 50 × 40 × 10 mm (tangential × radial × longitudinal) and had a moisture content of 10 ± 0.5%, while the density was 443.61 kg·m^{-3}. These samples were divided into four groups, each group containing five samples. The groups were as follows: untreated samples (reference), samples treated with a combination of aqueous solution of sodium silicate (water glass—WG) (LARO v.o.s, Krupina, Slovakia), and expandable graphite (EG), samples treated with Bochemit Antiflash (Bochemie a.s, Bohumín, Czech Republic), and samples treated with Bochemit Pyro (Bochemie a.s, Bohumín, Czech Republic). The samples treated with expandable graphite in combination with water glass were first coated with a solution of concentrated WG, then EG (+50 mesh; >300 µm; expansion ratio (X:1): 270 to 325; supplied by Sigma-Aldrich, Burlington, MA, USA) was sprinkled on this layer (formula: $C_{24}(HSO_4)(H_2SO_4)_2$)), which was also sprayed with a 50% WG solution. The ratio of WG:EG:50%WG was 1:1:2, while the amount of applied substance per component was 250 g·m^{-2}. Bochemit Antiflash was diluted with water in a ratio of 2:1, while the amount of retardant was 300 g·m^{-2}, and Bochemit Pyro was also diluted with water in a ratio of 2:1, while the amount of retardant was 400 g·m^{-2}. Bochemit Antiflash and Bochemit Pyro were applied to the samples in three-layer coats using a flat brush, according to the manufacturer's recommendations. After treatment, the samples were dried to constant weight at room temperature.

2.2. Sample Analyses

We used a non-standard test method with a ceramic thermal infrared heater (Ceramicx, Cork, Ireland) with an electric power of 1000 W. The duration of heating was 600 s. The distance of the samples from the surface of the heater was 40 mm, and we tested five

samples for each group, because of wood density variability. In the experiment, we used an electronic scale (PS 3500.R2, Radwag, Radom, Poland); the mass loss was recorded every 10 s (using the RLAB program). Any ignition of the samples was visually checked with a time record if this phenomenon occurred. Subsequently, we calculated the relative mass loss of wood from the measured values [3].

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 (Testo, West Chester, PA, USA) at selected points—A, B, C, and D. The points where the temperature was measured were distributed in the same way as by Kmeťová et al. [30]. All wood samples had the same grain direction. The heat source was applied to the radial surface of the sample. (See Figure 1).

(**a**) (**b**)

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

3. Results and Discussion

Research on flame retardants is aimed at evaluating their effectiveness in improving the thermal resistance of spruce wood, using a test with a radiant heat source. This is a non-standard test method used in model burning tests. In Figure 2, we present for comparison photo documentation of the samples before and after the experiment.

The experimental results are presented in Figures 3–8. The figures show the average values for each group of tested samples.

From the measurements, we recalculated the relative mass loss of wood and the relative burning rate for untreated control samples and all three types of treatment. Regarding relative mass loss, similar values were achieved by Bochemit Antiflash- and Pyro-treated samples, with approximately 1.7% greater relative mass loss observed for Bochemit Pyro-treated samples. These samples achieved a relative mass loss of 35.59 ± 2.32%. A more significant difference in terms of the treated samples compared to the other two types of treatment was observed in the samples treated with expandable graphite in combination with water glass, which lost only 10.52 ± 0.63% of their original weight. We recorded the highest relative mass loss in untreated samples, which lost up to 84% of their original weight.

If we compare the samples based on their relative mass loss, the best results were obtained with the samples treated with expandable graphite, worse results were obtained with the samples treated with Bochemit Antiflash, closely followed by the samples treated with Bochemit Pyro, and the worst results were with the untreated spruce wood samples. A certain % of the mass loss of the treated samples is probably also caused by the decomposition of some components of the used flame retardant.

The relative mass loss results correlate with the relative burn rate results. If we compare the samples based on relative burning rate, the ranking remains the same as when

compared in terms of relative weight loss. Overall, the highest relative burning rate of 0.34%·s^{-1} was recorded for samples of untreated spruce wood at the 310th s. The highest relative burning rate was for samples treated with Bochemit Pyro 0.11%·s^{-1} at 80 s, for samples treated with Bochemit Antiflash 0.07% s^{-1} at 40 s, and for samples treated with expandable graphite in combination with water glass only 0.04%·s^{-1} at the 60th s. It is important to note that by treating the wood with flame retardants, we were able to reduce the burning rate by more than 0.20%·s^{-1}.

Figure 2. Photo documentation of samples after testing with a radiant heat source (1—untreated sample, 2—graphite with water glass, 3—Bochemit Antiflash, 4—Bochemit Pyro).

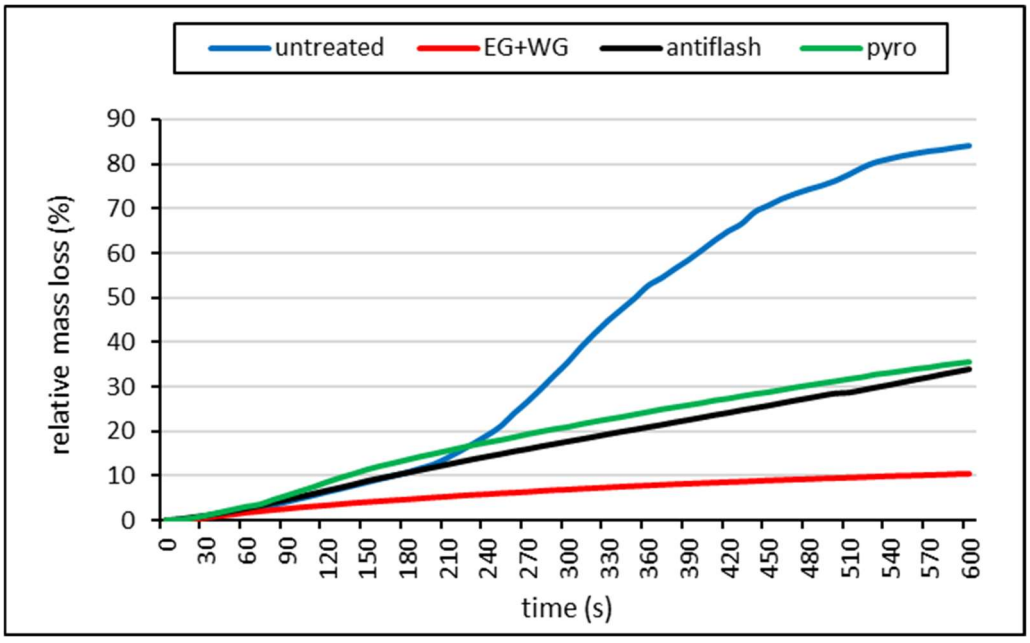

Figure 3. Relative mass loss of the samples during the test with a radiant heat source (untreated = reference samples, EG + WG = expandable graphite in combination with water glass, antiflash = Bochemit Antiflash, pyro = Bochemit Pyro).

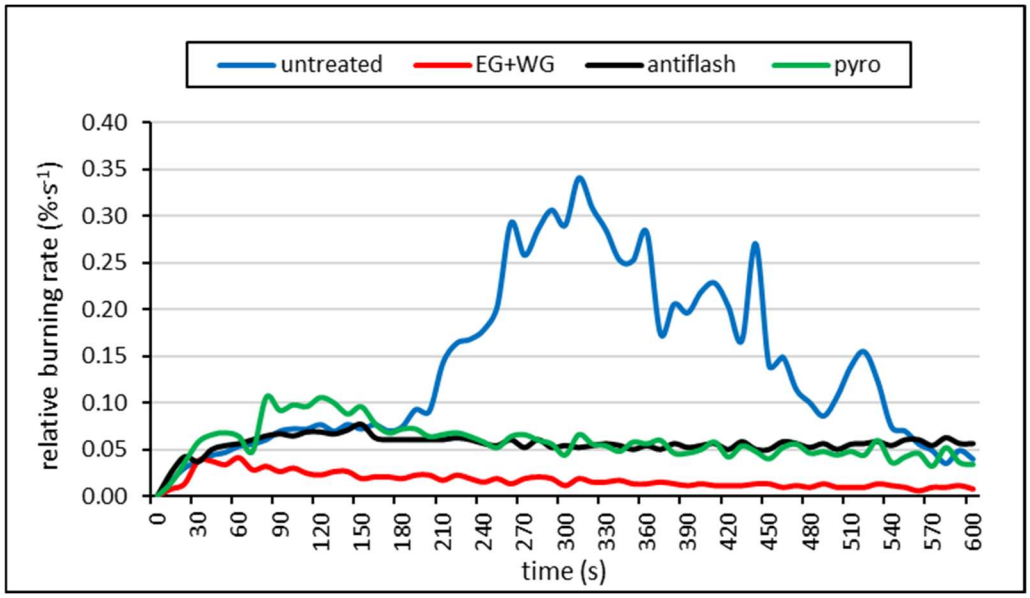

Figure 4. Relative burning rate of the samples during the test with a radiant heat source (untreated = reference samples, EG + WG = expandable graphite in combination with water glass, antiflash = Bochemit Antiflash, pyro = Bochemit Pyro).

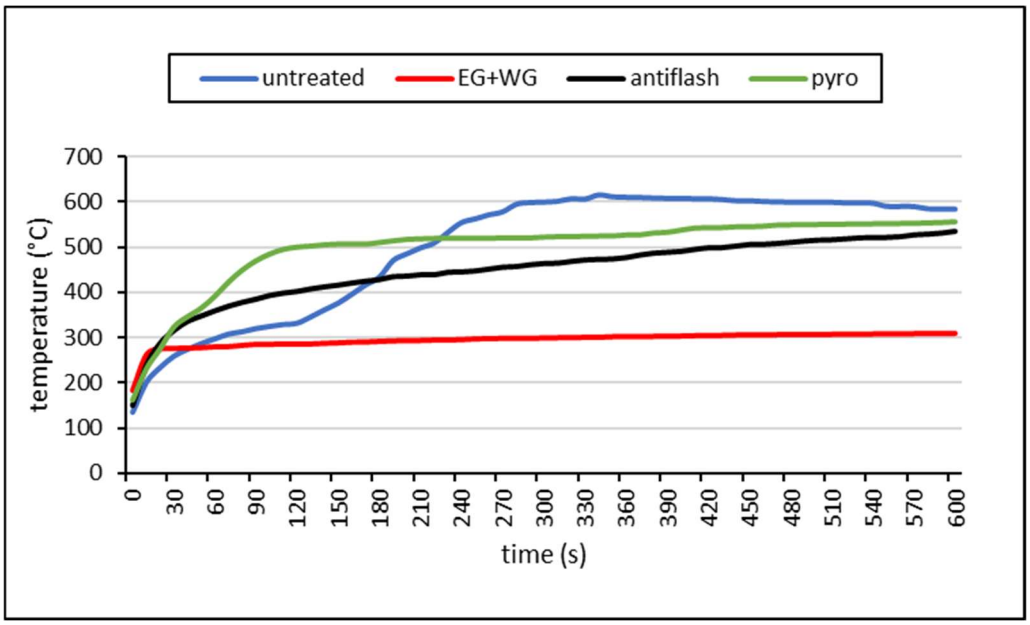

Figure 5. Temperature course of the samples at point A (untreated = reference samples, EG + WG = expandable graphite in combination with water glass, antiflash = Bochemit Antiflash, pyro = Bochemit Pyro).

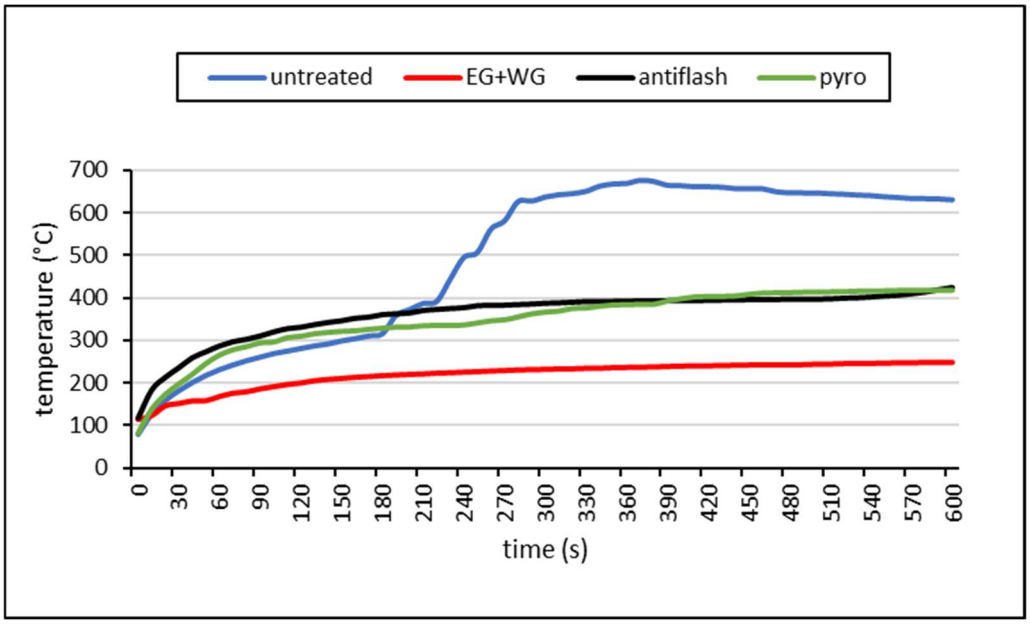

Figure 6. Temperature course of the samples at point B (untreated = reference samples, EG + WG = expandable graphite in combination with water glass, antiflash = Bochemit Antiflash, pyro = Bochemit Pyro).

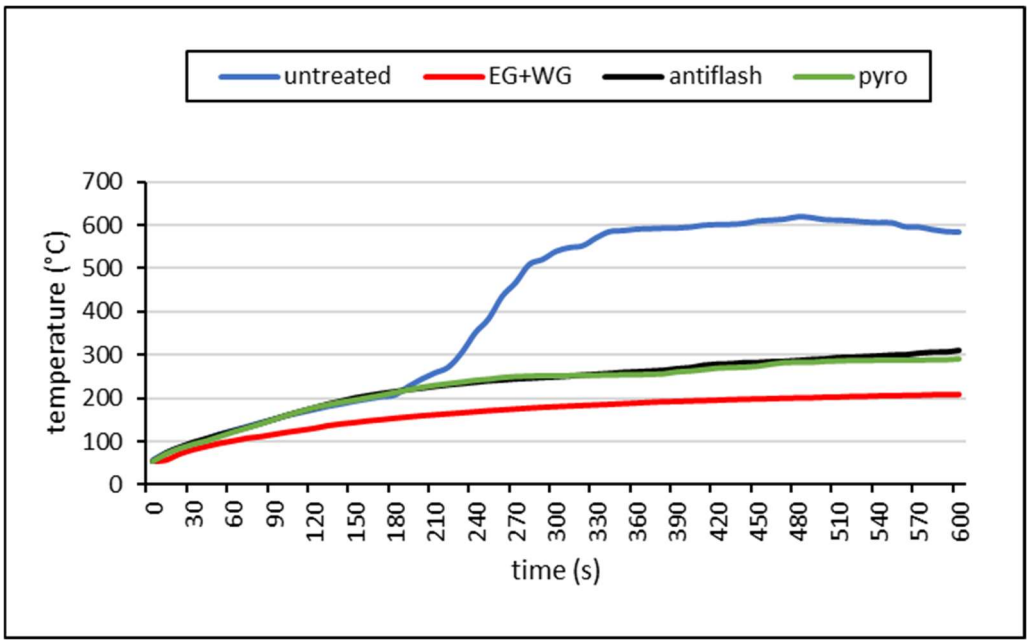

Figure 7. Relative temperature course of the samples at point C (untreated = reference samples, EG + WG = expandable graphite in combination with water glass, antiflash = Bochemit Antiflash, pyro = Bochemit Pyro).

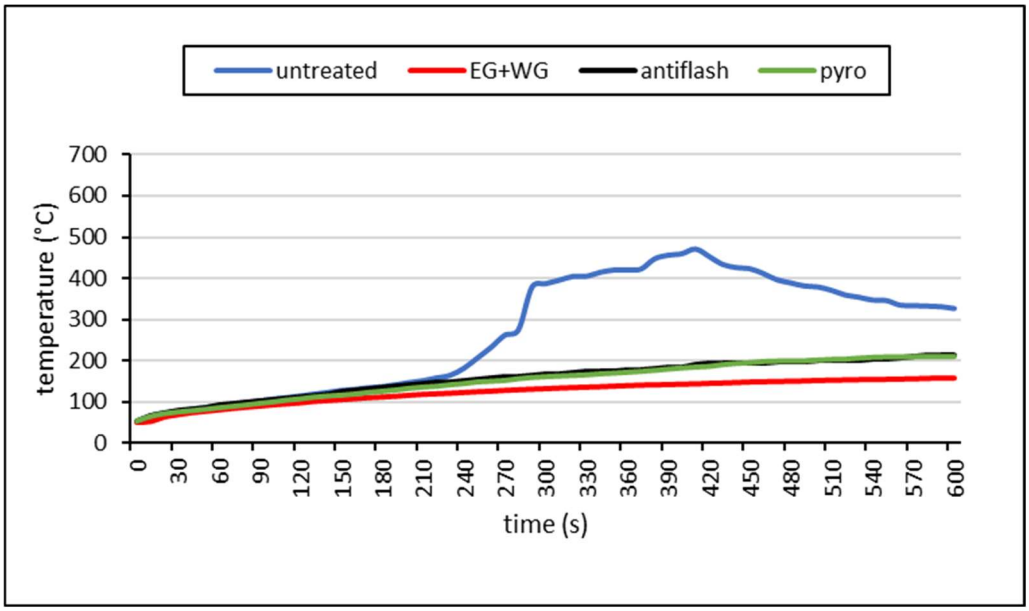

Figure 8. Temperature course of the samples at point D (untreated = reference samples, EG + WG = expandable graphite in combination with water glass, antiflash = Bochemit Antiflash, pyro = Bochemit Pyro).

Kačíková and Makovická [31] also dealt with the speed of sample burning. In this case, the spruce wood samples reached a maximum burning rate of 0.187%·s^{-1} at the 180th s of the test duration.

The temperature course on the sample surface, at point A, is approximately the same for samples treated with Bochemit Antiflash and Bochemit Pyro, where the maximum temperature of 555 °C was reached at the end of the test (at the 600th s) for samples treated with Bochemit Pyro and 537 °C for samples treated with Bochemit Antiflash, also at the 600th s. For samples treated with expandable graphite in combination with water glass, there was a sharp increase in temperature to 280 °C during the first 60 s, and by the end of the test, the temperature increased linearly by only 40 °C. As for the untreated samples, a sharper increase in temperature started to occur at 130 s. The maximum temperature of 617 °C was reached in these samples at the 350th s. And after the end of the flame burning, the temperature started to drop again.

The temperature course at point B is approximately the same for samples treated with Bochemit Pyro, Bochemit Antiflash, and expandable graphite in combination with water glass with different values of measured temperatures. For samples treated with Bochemit Pyro, the maximum temperature reached was 417 °C before the end of the test. Samples treated with Bochemit Antiflash reached a maximum temperature of 420 °C, also before the end of the test. As for the untreated samples, a sharper rise in temperature began at the 220th s, and the maximum temperature of 674 °C was reached at the 380th s.

The temperature course at point C is the same for wood treated with Bochemit Pyro and Antiflash; in both cases, the maximum temperature value was measured at the 600th s of the test. For Bochemit Pyro, it was 290 °C, and for Bochemit Antiflash, it was 310 °C. For samples treated with expandable graphite in combination with water glass, the maximum temperature reached a value of 207 °C at the end of the test. As for the untreated samples, the increase occurred at the 220th s and the maximum temperature was 620 °C at the 480th s of the test duration.

The temperature course at point D was approximately the same for all three types of retardation treatment. In all types of sample treatment, the maximum temperature was measured at the end of the test (600th s). Among the retarded samples, samples treated with Bochemit Antiflash reached the highest maximum temperature value of 215 °C, samples treated with Bochemit Pyro reached 209 °C, and the maximum temperature value of

samples treated with expandable graphite in combination with water glass was 157 °C. For the untreated samples, the increase started to occur at the 240th s and the maximum temperature was 472 °C at the 410th s of the test duration.

If we compare the course of temperatures for individual samples, all samples reached maximum temperatures at point A, except for the untreated wood samples, which overall reached their maximum temperature value at point B. The highest maximum temperature values were reached by untreated samples at all measured points, which we attribute to their ignition. Conversely, the lowest maximum temperature values were achieved by samples with expandable graphite in combination with water glass.

Furthermore, as part of the evaluation of the test results with a radiant heat source, as another evaluation criterion, we present the ignition of the individual tested samples. In the method of thermal loading of the treated test samples with radiant heat, there was no ignition in any case, while untreated spruce wood samples ignited at approximately the 260th s. Even among these samples, however, it could not withstand burning with a flame until the end of the experiment (600 s). We can therefore say that when the samples were loaded with a ceramic infrared emitter, all three types of retardation treatment proved to be satisfactory.

In the test with a radiant heat source, for all evaluation criteria (relative weight loss, relative burning rate, ignition time, and also the temperature course on the surface of the sample), the best results were achieved by the samples treated with expandable graphite in combination with water glass, and on the contrary, the overall worst results, as we expected, were recorded for samples of untreated spruce wood.

In research, many other authors deal with the evaluation of spruce wood in terms of thermal resistance and with the investigation of the effects of flame retardants. Various studies are available dealing with the preservation of wood. Expandable graphite as a fire retardant used to protect wood is still a new area of research, so it is necessary to continue working on these results [3,17,30–33].

4. Conclusions

Fire protection of wood as a building material is a very current issue. In this work, three different treatments to increase the thermal resistance of spruce wood were investigated—samples treated with expandable graphite in combination with water glass and two types of coating substances from Bochemit. In addition, the results were also compared with a sample of untreated wood. The worst results in terms of relative mass loss were achieved by untreated samples, which lost 74 ± 1% more weight than samples treated with EG + VS, which achieved the best results from this point of view. It is important to note that by treating the wood with flame retardants, we managed to significantly reduce the burning rate. Regarding the temperature course, we recorded the highest temperatures on the surface of the samples during the flame burning of the untreated samples. The results showed that the correct treatment of wood has the potential to improve its resistance to fire, but it is essential to choose the right type of flame retardant. However, despite many advantages, expandable graphite (EG) still faces many challenges. First, applying EG to the wood surface requires a suitable bonding material that has the necessary adhesive properties, and is durable, non-toxic, and economically acceptable. Second, the flame-retardant effect depends on several properties of EG, such as particle size, decomposition temperature, etc. It is necessary to find the optimal properties of EG for a specific application. Third, the surface of wood and wood material covered with EG may not be aesthetically suitable for some purposes and it will be necessary to apply an appropriate surface treatment. The results also emphasize the justification, if not the necessity, of using these substances to protect wood, so as not to reduce the possibilities of its use in construction. We realize the even more significant need to treat wood with a retardation treatment when comparing and looking at untreated wood, which is very easily subject to thermal degradation.

Author Contributions: Conceptualization, F.K. and E.K.; methodology, D.K. and E.K.; writing—original draft preparation, E.K.; writing—review and editing, D.K. and F.K.; supervision, D.K.; project admin-

istration, F.K.; funding acquisition, D.K. and F.K. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the Slovak Research and Development Agency under the Contract No. APVV-22-0030. This work was supported by the Slovak Research and Development Agency under the contract No. APVV-16-0326. This work was supported by the project "Progressive research of performance properties of wood-based materials and products" (LignoPro); ITMS 313011T720, supported by the Operational Programme Integrated Infrastructure (OPII) funded by the ERDF.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Additional data will be made available on reasonable request to the authors.

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

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