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**Wojciech Ł. GRZEŚKOWIAK, Krzysztof MOLIŃSKI,  
Marta MOLIŃSKA-GLURA, Grzegorz COFTA**

## **ASSESSMENT OF THE IMPACT OF THE STORAGE TIME OF FIRE RETARDANT AND HEATING OF THE PROTECTED WOOD ON THE EFFECTIVENESS OF FIREPROOFING**

*It was investigated whether and how the storage time of a fireproof salt preparation affects the effectiveness of fire retardance. The impact of long-term heating of the treated wood on the effectiveness was also determined. Statistical methods were used to determine the significance of changes related to the age of the preparation and the effect of heating of treated wood on flame retardant effectiveness. A commercial fire retardant was used for the tests, with three different storage times and in five concentrations. The flammability tests were performed using the Mini Fire Tube method. It was concluded that the storage time of the preparation does not significantly affect the effectiveness values.*

**Keywords:** fire retardants, Mini Fire Tube, wood protection, effectiveness

### **Introduction**

Despite having many advantages, such as strength, low thermal conductivity and ease of processing, wood also has some disadvantages, particularly its susceptibility to biotic factors and fire [Goldstein 1973]. The fire resistance of wooden structures and their elements is directly dependent on the type of wood and its physical properties [Le Van 1989]. To give wood greater durability during a fire, it should be protected with fire retardants (FRs). These preparations can reduce the flammability of wood, but will not lead to a non-combustible material [Oberley 1991]. Research on FRs has a long history, and

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Wojciech Ł. GRZEŚKOWIAK<sup>✉</sup> ([wojciech.grzeskowiak@up.poznan.pl](mailto:wojciech.grzeskowiak@up.poznan.pl)), Marta MOLIŃSKA-GLURA ([mglura3@gmail.com](mailto:mglura3@gmail.com)), Grzegorz COFTA ([grzegorz.cofta@up.poznan.pl](mailto:grzegorz.cofta@up.poznan.pl)), Faculty of Forestry and Wood Technology, Poznań University of Life Sciences, Poznań, Poland; Krzysztof MOLIŃSKI ([krzysztof.molinski@up.poznan.pl](mailto:krzysztof.molinski@up.poznan.pl)), Faculty of Agronomy and Bioengineering, Poznań University of Life Sciences, Poznań, Poland

their economic importance is constantly growing. The introduction of FRs into production and application in practice has enabled a reduction in human and material losses. The quality and effectiveness of FRs are increasingly important for safety, applications, economy and environmental protection [White and Dietenberger 2001].

FRs can significantly improve the reaction to fire of wood and wood-based materials, but this is not always combined with durability in internal and external applications. Requirements for the durability of fire protection are not included in most building regulations [Östman and Tsantaridis 2016]. This is probably due in part to lack of awareness of the problem, but may also be due to a lack of procedures. Currently, the issue of durability of fire protection is gaining more and more attention. Many factors influence the durability of fire protection of structural elements. Many salt fire retardants are leached by water (periodic moisture and precipitation). Another factor associated with the durability of the protection of internal components is the risk of their humidification and, consequently, the migration of chemical compounds through diffusion in the wood product. Swelling salt crystals agglomerate inside the capillaries, leading to mechanical damage, causing a reduction in the strength of elements by up to 50% [Winandy et al. 1988].

There are many reports regarding the durability of the protection of wood and wood-based materials, based on salt and coating preparations subjected to aging in natural and laboratory conditions. Many laboratory methods for accelerated aging use water in liquid or steam form, freezing and heating. In the case of salt fire retardants, the use of water (liquid, steam) in most cases will result in their being washed out of the wood completely, and thus a reduction in FR effectiveness [Holmes and Knispel 1981; Harada et al. 2009; White 2009; Kolbrecki and Sudoł 2014; Sudoł 2014; Nakamura et al. 2016; Östman and Tsantaridis 2016]. A number of publications on FRs and their aging are focused on the strength properties of structural elements [Winandy et al. 1988]. Alongside many positive features characterizing salt FRs for wood, their influence on the strength properties of impregnated wood or wood materials, corrosion of steel elements and biodegradation plays a large role. These properties are dependent on, among others, the type of agent, its pH, the impregnation technology, as well as the use class [EN 335:2013-07] of the treated elements.

In the literature on the subject, there are limited numbers of reports on the impact of the storage of FRs and heating on the quality of the fire protection. In most cases fireproofed wood is used in roof truss construction. In such conditions, the wood is exposed to increased humidity, periodic moisture in cases of roof leaks, as well as higher temperatures resulting from the heating of roof coverings. Similarly, there are no reports regarding the impact of transport conditions and storage time on the properties of FRs and the protected wood. In considering the effectiveness of FRs and the degree of protection of wood, the

durability of the protection as well as the durability of the stored preparations should be taken into account.

In this work, it was investigated whether and how the storage time of a fireproof salt preparation affects the effectiveness of wood protection. It was also determined how the long-term heating of treated wood in constant climatic conditions influences the effectiveness of the protection. Statistical methods were used to determine the significance of changes related to the age of the preparation and the effect of heating on flame retardant effectiveness.

## Materials and methods

A commercial fire retardant, with the components  $\text{NH}_4\text{H}_2\text{PO}_4$ ,  $(\text{NH}_4)\text{HPO}_4$ ,  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{CH}_4\text{N}_2\text{O}$  and the biologically active substances sodium tetraborate, benzyl-C12-18-alkyl dimethyl ammonium chloride and 3-iodo-2-propynyl-N-butylcarbamate, was used for the research. The preparation was selected with regard to the fact that it is the most commonly used fire-retardant preparation in Poland, as well as the possibility of obtaining preparations from the manufacturer with specified years of production. The preparation was tested after three different storage times (Table 1). FR was stored in closed cans, at room temperature ( $20^\circ\text{C}$ ) and relative humidity 60%, and without light. The manufacturer specifies the fireproof stability of the preparation in loose form as 2 years. Wood treated with FR (in 5 concentrations) and control samples were also subjected to a heating process. For each variant, 10 replicas were made.

Samples of Scots pine sapwood (*Pinus sylvestris* L.) were used. The samples taken were free of defects, with a moisture content of  $8 \pm 0.5\%$  and dimensions of  $5 \times 10 \times 100$  mm. Test samples were treated by a full-cell vacuum method with a pressure of 0.085 MPa maintained for 20 min, and then transferred to atmospheric conditions and maintained in solutions for the next 2 hours. After treatment, the samples were conditioned to obtain a wood moisture content of  $8 \pm 2\%$ . Control samples had the same moisture content during the test. The moisture content of the samples was determined by a drying-weight method before the test [Grzeškowiak 2017].

In addition, samples treated with the above variants of the preparation were subjected to aging using a heating and evaporation procedure. The process was carried out in incubators with constant humidity of  $65 \pm 5\%$  at  $40^\circ\text{C}$  for 6 weeks. After the saturation and conditioning period, the samples were subjected to MFT (Mini Fire Tube) flammability tests to determine the effectiveness of the protection. The MFT method is an adopted and modified ASTM E69-02 method. It is more accurate and allows for more repetitions, using smaller samples than the original method. The method is based on the measurement of mass loss and exhaust gas temperature at the outlet of the pipe. A burner with a pre-adjusted flame of approx. 1 cm height was placed inside the pipe. For 6 minutes, every

**Table 1. Test variants with acronyms and average retention values**

Preparation storage time	Concentration (%)	Retention (mean) (kg*m <sup>-3</sup> ) + (SD)	
		unheated	heated – H
K – control	–	–	–
A – 5 years	I – 20	145.06 (19.41)	140.96 (14.48)
	II – 10	71.30 (8.29)	68.51 (4.67)
	III – 5	35.32 (2.38)	33.27 (2.69)
	IV – 2.5	17.11 (1.26)	17.37 (1.23)
	V – 1	6.68 (0.49)	6.97 (0.82)
B – 3 years	I – 20	148.93 (6.94)	147.32 (7.47)
	II – 10	71.57 (1.27)	73.98 (2.98)
	III – 5	35.59 (1.60)	35.03 (1.74)
	IV – 2.5	17.00 (0.29)	17.89 (0.35)
	V – 1	7.16 (0.36)	7.08 (0.35)
C – current production line	I – 20	147.84 (5.95)	145.86 (7.14)
	II – 10	71.86 (2.18)	69.85 (3.37)
	III – 5	34.07 (1.49)	32.69 (3.24)
	IV – 2.5	17.83 (1.04)	17.00 (1.91)
	V – 1	7.04 (0.20)	6.69 (0.99)

1 second the change in mass and temperature of the tested sample was recorded [Grześkowiak 2015; Can et al. 2017; Grześkowiak 2017; Can et al. 2018; Zeinali et al. 2018; Łukawski et al. 2019]. The protective effect was assessed for the final mass loss value (360 seconds) based on the following formula:

$$W = 100\% \cdot \left(1 - \frac{E}{A}\right)$$

where:  $W$  is the conventional effectiveness of the impregnation,

$E$  is the final mass loss of protected samples (%),

$A$  is the final mass loss of control samples (%).

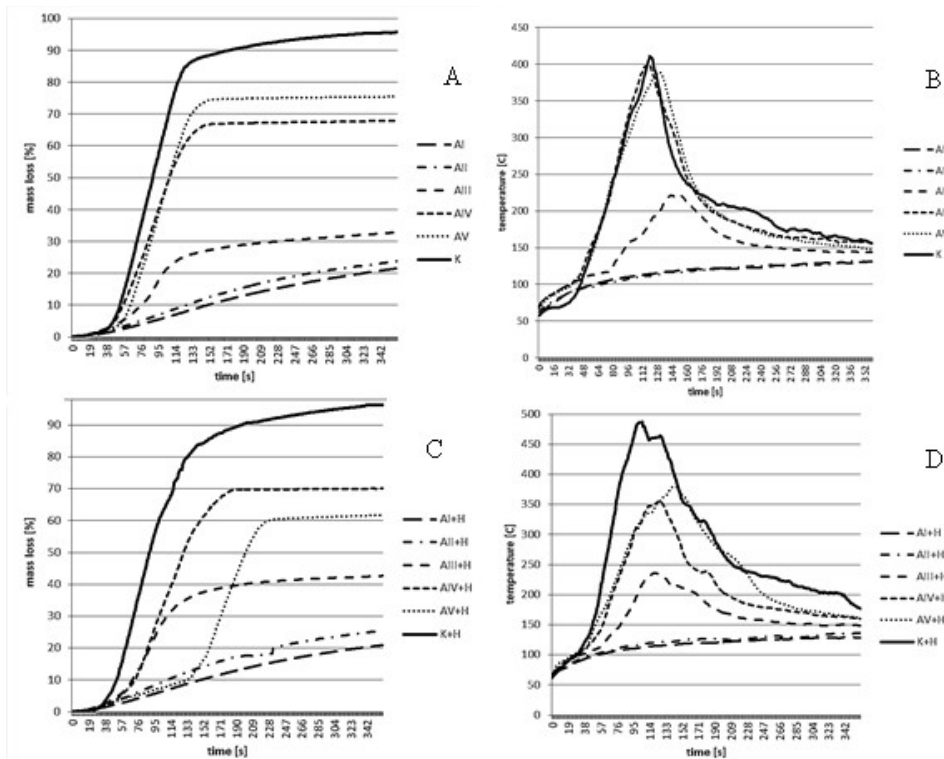
Effectiveness was determined for both heated and unheated variants based on mass losses and temperatures.

For statistical analysis of the flammability characteristics of wood treated with the preparation, the k-means method was used. In this method, objects are divided into a predetermined number of clusters [Grześkowiak and Moliński 2019]. In general, for k-means there will be created k clusters, which are as different as possible. The initial clusters are improved in the course of the agglomeration procedure, transferring objects between them so that the diversity of objects within a cluster is as small as possible and the cluster distances as large as possible [Lloyd 1982; Arthur and Vassilvitskii 2007]. The algorithm operates based on a matrix of Euclidean distances between objects. In this work,

the value  $k = 3$  was adopted, allowing the interpretation of protection variants in terms of mass losses and temperatures. The research used the Statistica 13.1 package with descriptive statistics and tables and General Linear Models.

## Results

The fastest decomposition was exhibited by unheated control samples in the time interval from 0 to 124 seconds, resulting in a final mass loss of 95.64%. The AV variant with the lowest concentration gave the largest mass loss of samples in the period 0–140 seconds, where the final mass loss was 75.42%, yielding 21.11% effectiveness. In the case of the same variant with intermediate solution concentrations of 2.5% and 5%, the respective final mass losses were 67.89% and 32.81%, and the effectiveness values were 28.99% and 65.72%. The best effectiveness was achieved by variants AI and AII: 75.24% and 77.59%, with mass loss 23.72% and 21.50% respectively (Fig. 1A).



**Fig. 1.** Average mass loss for variants A (A) and A+H (C) and temperature curves for variants A (B) and A+H (D)

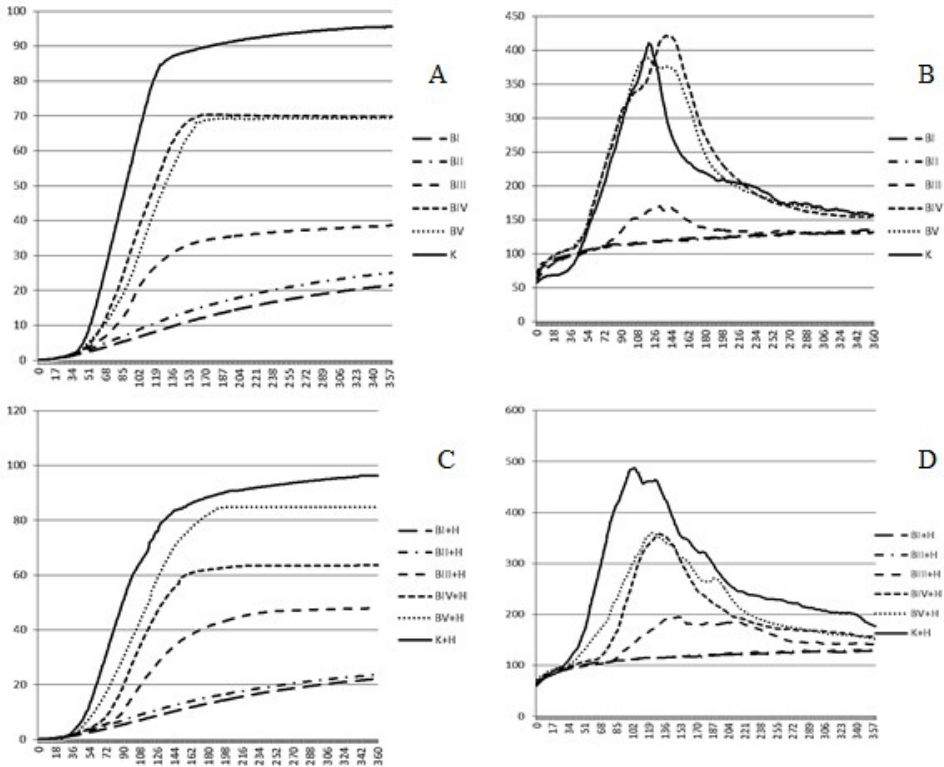
Analyzing the temperature values, it was found that the highest values were obtained by unprotected samples, where the temperature reached about 411°C.

Slightly lower flue gas temperatures were obtained for samples protected with the preparation with the lowest concentrations. For the 1% concentration, the maximum temperature was 389°C, while for a concentration of 2.5% the exhaust temperature was slightly higher, around 400°C. For a higher concentration of 5%, the maximum temperature was 222°C. In the case of higher concentrations, the maximum exhaust temperature was recorded at the last measurement (360 seconds), and was 131.52°C for the AII variant and 131.08°C for AI (Fig. 1B).

Variant A+H, based on heating of the samples, produced a mass loss in the control samples of 96.34%. Significant differences were found between the mass losses for A+H samples protected with lower concentrations. Mass loss was higher than with unheated samples in the case of the 2.5% concentration, where the final loss and effectiveness in 360 seconds were 70.09% and 27.29%, the 5% concentration, with a mass loss of 42.67% and effectiveness 55.74%, and the 10% concentration, with an effectiveness of 73.61% and a mass loss of 25.47%. Lower mass losses compared with unheated samples were obtained for the 1% and 20% concentration series, where the mass loss and effectiveness were respectively 61.71% and 35.95%, and 20.91% and 78.37% (Fig. 1C). In the case of temperature distribution at the outlet of the pipe, a correlation was observed: with an increase in the concentration of the preparation the maximum temperature was lower. In the case of control samples, the maximum flue gas temperature was 487.13°C. The maximum temperatures for concentrations of 1%, 2.5% and 5% were respectively 379.55, 354.87 and 236.17°C. Similarly to the preparation without a heating process, the highest exhaust temperatures for the 10% and 20% variants occurred at the end of the test and amounted to 136.69°C for 10% and 128.72°C for 20% (Fig. 1D).

In the case of the formulation stored for 3 years (B), for the variant without a heating process, higher mass losses were recorded for all concentrations as compared with the formulation stored for 5 years (A), except for the lowest concentration. The final mass losses were as follows: BV 69.43%, BIV 69.83%, BIII 38.65%, BII 25.06%, and BI 21.54% (Fig. 2A). The effectiveness values calculated for the 360-second test were 27.37%, 26.95%, 59.58%, 73.85%, and 77.54% respectively.

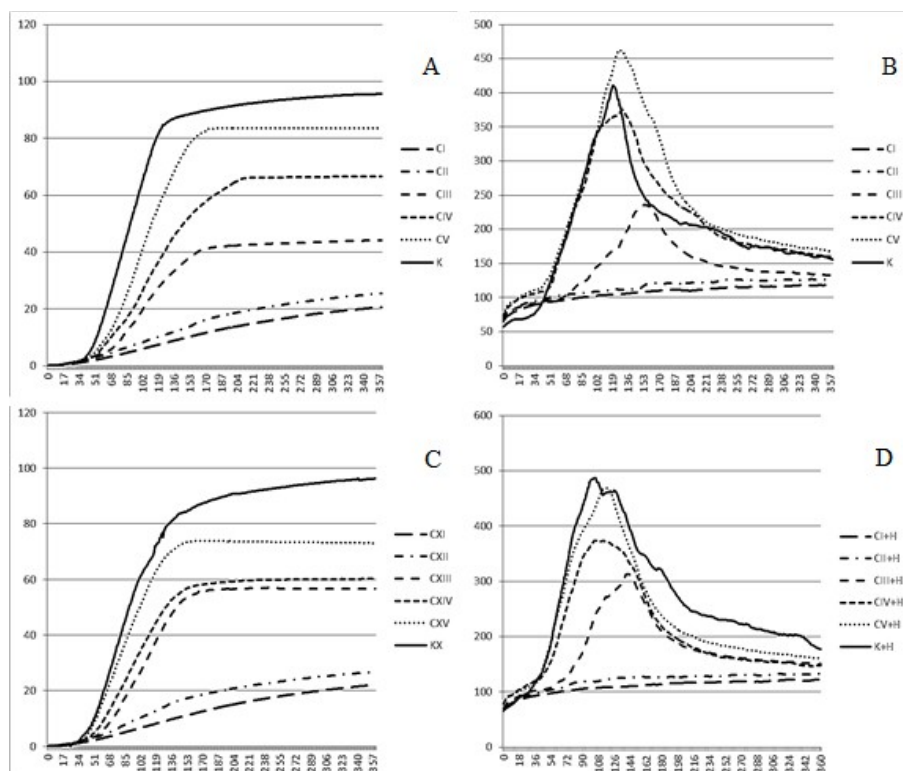
Analyzing the temperature values, it can be observed that samples protected with the BIV variant gave a higher maximum exhaust gas temperature than the control samples. After the initial rapid increase in temperature, it fell off slightly before another rapid increase to a maximum of 421.2°C. The maximum exhaust gas temperature for the BV variant was 389.18°C, while for samples protected with BIII it was 170.39°C. At the highest concentrations, maximum temperatures were obtained at the end of the experiment: 134.21°C for BII and 135.63°C for BI (Fig. 2B).



**Fig. 2.** Average mass loss for variants B (A) and B+H (C) and temperature curves for variants B (B) and B+H (D)

Mass losses in the B variants subjected to heating showed a tendency to decrease with increasing concentration of the preparation (Fig. 2C). Compared with the unheated version, mass losses in the series BV+H, BIII+H and BI+H were respectively 84.88%, 47.95% and 22.17%, yielding 360-second effectiveness values of 11.90%, 50.25% and 77.05%. The remaining concentrations of the preparation gave lower mass losses than the equivalent unheated variants. For the BIV+H variant, the mass loss was 63.54%, yielding an effectiveness of 34.05%, and for BII+H the final mass loss was 23.65% and the effectiveness was 75.51%. The variants with heating produced lower maximum exhaust temperatures for most concentrations used, compared with the unheated version. The maximum temperatures recorded for concentrations of 1% and 2.5% were very similar, at 360.12 and 358.42°C respectively (Fig. 2D). Similarly, small differences in temperature were recorded for variants with the highest concentrations. Samples protected with a 10% solution of the preparation produced a maximum exhaust temperature of 130.48°C, while a 20% solution gave a temperature of 129.29°C. A higher temperature compared with the unheated samples was also obtained for specimens with a concentration of 5%;

this temperature was 196.62°C. In this case, after reaching the maximum, over a long time (from 136 to 221 seconds) the temperature remained at a level in the range 180-186°C, and then until the end of test the temperature decreased. The preparation from current production (C) also showed a decreasing tendency in mass loss with increasing applied concentration (Fig. 3A). In all variants with lower concentrations, the rapid increase in mass loss continued until the temperature approached the maximum exhaust gas temperature. In the 1% series, the maximum flue gas temperature was 466.29°C and was higher than the temperature for the control samples by about 55°C (Fig. 3B). The final mass loss was 83.5%, yielding 12.66% effectiveness. The series protected with a 2.5% solution showed a mass loss of 66.65% (effectiveness 30.29%) and a maximum exhaust temperature of 375.32°C. The 5% variant produced less than half the weight loss compared with the control samples: the value was 44.21%, with an effectiveness of 53.76%, at a maximum temperature of 235.78°C. The variants with the highest concentrations produced series of mass losses and temperatures similar to the previously described A and B variants. The 10% concentration led to a mass loss of 25.51% and a temperature of 127.29°C, while the 20%



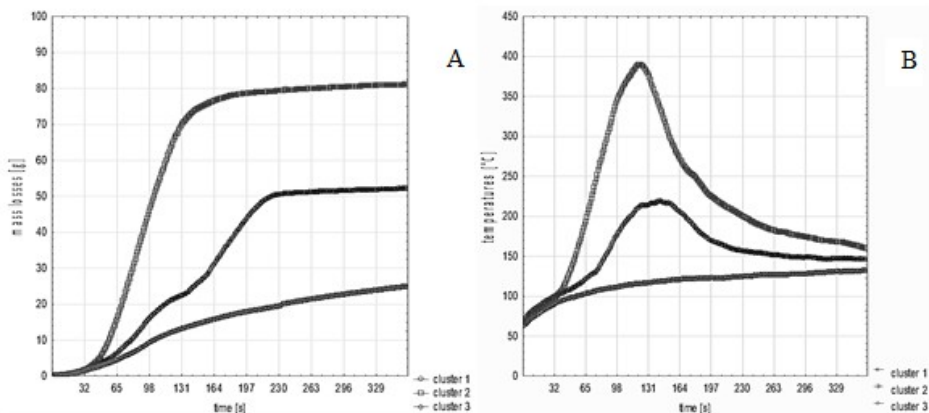
**Fig. 3.** Average mass loss for variants C (A) and C+H (C) and temperature curves for variants C (B) and C+H (D)



concentration gave 20.66% and 119.22°C, yielding respectively 73.37% and 78.45% effectiveness. The variants with the lowest concentrations subjected to heating exhibited lower mass losses (Fig. 3C) than the unheated and approximate maximum exhaust gas temperatures (Fig. 3D). Samples treated with a 1% solution of the formulation achieved a final mass loss of about 10% lower than the unheated samples, and it was 73.84% (23.36% efficiency) at a maximum temperature of 469.16°C. Also, the 2.5% variant showed a mass loss lower by approx. 6% than its unheated equivalent. The value of mass loss was 60.16% (37.57%) and the maximum temperature was 374.35°C. Concentrations of 5, 10 and 20% were characterized by higher mass losses compared to unheated variants. Mass loss, efficacy and maximum temperature values were respectively: 56.72%, 41.13% and 313.39°C; 26.80%, 72.23% and 133.19°C; and 22.14%, 77.11% and 122.04°C.

A mathematical analysis of clusters for particular age variants of the preparation was made, both for the heated and unheated variants, based on mass losses and temperature during the whole experiment (360 seconds), with a division into three clusters.

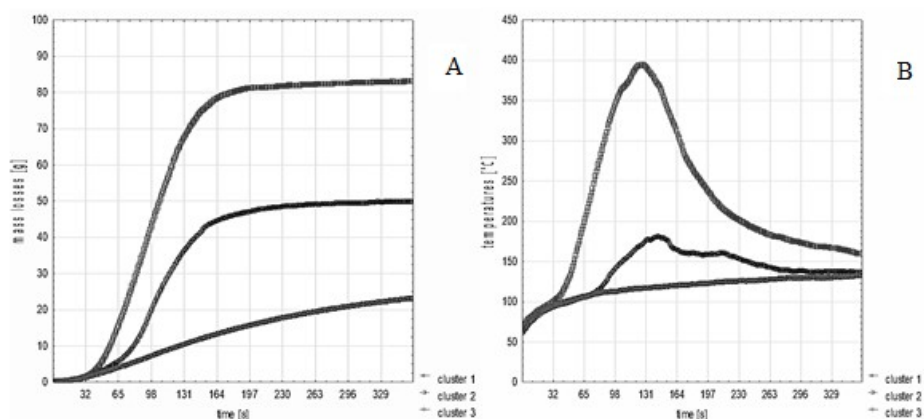
Analyzing formulation A in terms of mass loss, a division into three clusters was obtained, in which cluster 1 contained two cases after heating: AIII+H and AV+H (Fig. 4A). Cluster 2 contained five cases including control samples, namely VA, K, K+H, IVA and IVA+H. These are the variants with the lowest concentrations of the preparation. The highest concentrations of the preparation coincide with cluster 3, also containing five elements: IIIA, IIA, IIA+H, IA and IA+H. Such a cluster system is correlated with the final loss of mass, and thus with the effectiveness of the preparation after 360 seconds. In the case of clusters obtained for temperatures, cluster 1 again contained two cases (AIII and AIII+H), and cluster 2, with six cases, contained variants with the lowest



**Fig. 4. Cluster curves for variant A of the preparation for mass losses (A) and temperatures (B)**

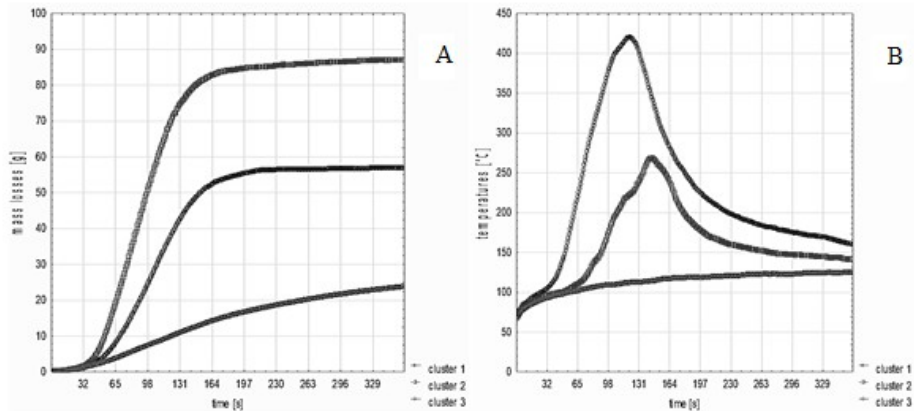
concentrations together with control samples (AIV, AIV+H, AV, AV+H, K, K+H) (Fig. 4B). Cluster 3 contained four cases corresponding to the highest concentrations of the preparation (AI, AI+H, AII, and AII+H). This cluster system corresponds to the temperature efficiency calculated for the critical point.

Taking into account the clusters determined for preparation B based on mass losses, similar relationships were found as for preparation A (Fig. 5A). Cluster 1 contained three cases (BIII, BIII+H and BIV+H) corresponding to the effectiveness calculated for the final values of mass losses. Similarly, cluster 2, containing the lowest concentrations of the preparation and control samples (VB, VB+H, K, K+H, BIV), and cluster 3, with the highest concentrations of the preparation (BI, BII, BI+H, BI+H), correlate with the effectiveness of the preparation at the 360-second point. The clusters based on the temperature profile of exhaust gases for particular variants behave in a similar way (Fig. 5B). In the case of temperature-based clusters for preparation B, the same cases were assigned to the particular clusters as was the case for preparation A.



**Fig. 5. Cluster curves for variant B of the preparation for mass losses (A) and temperatures (B)**

For variant C of the preparation, clusters for mass losses had an even distribution of cases (Fig. 6A). Cluster 1 contained four cases (CIV, CIV+H, CIII, CIII+H), cluster 2 contained samples with the lowest concentration and the controls (CV, CV+H, K, K+H), and as in the other variants, cluster 3 contained cases with the highest concentrations (CI, CI+H, CII, CII+H). This distribution of clusters corresponds to the mass losses for individual concentration variants of the preparation. However, analysis of the clusters for temperatures shows that they do not differ from those obtained for the other age variants of the preparation (Fig. 6B).



**Fig. 6. Cluster curves for variant C of the preparation for mass losses (A) and temperatures (B)**

## Conclusions

1. On the basis of the results for mass losses, no significant differences in the effectiveness of preparations with the higher concentrations of 20% and 10% were found, regardless of the time of storage and whether the samples underwent a heating process. The exhaust temperature ranges obtained for the above variants also do not show significant differences.
2. In the case of lower concentrations of preparations, both without and with the heating process, it is not possible to indicate a clear relationship between storage time and effectiveness changes based on mass loss and exhaust gas temperature.
3. The use of a formulation stored for a longer period of time, in accordance with the recommended saturation parameters, does not affect its fire protection effectiveness, and thus the fire safety of protected elements.
4. The results presented in this study were obtained for pine wood and one type of preparation. Further testing is required for other wood species and preparations.

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### List of standards

- EN 335:2013-07** Durability of wood and wood-based products – Use classes: definitions, application to solid wood and wood-based products. CEN 2013
- ASTM E69-02** Standard test method for combustible properties of treated wood by the fire-tube apparatus

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