1. Introduction

Wood is widely used as a building material in construction and architecture due to its mechanical and operational properties. However, because of its high combustibility, it is a fire-hazardous material. Level of fire safety of objects in which wood building structures are used can be raised by means of fire protection treatment, namely, providing the wood with the ability to withstand flame and its surface propagation, preventing free access of oxygen which promotes wood destruction and accelerates the burning process.

The fires that have occurred in recent years have shown growth of dangerous fire factors, in particular, high temperature since nomenclature of various synthetic materials and hydrocarbons participating in fires and capable of emitting high heat during their combustion has expanded recently. Therefore, establishment of flame depression parameters at elevated temperatures, study of the rate of wood burnout and influence of coating on the burning process is an unsolved component of ensuring fire resistance and, accordingly, determines the need for such studies.

2. Literature review and problem statement

Feature of fire protection of wooden building structures consists in creation of elements of heat-insulating screens on the structure surface that withstand direct fire action and allow structures to maintain their functions for a certain period of time [3, 4]. However, the issues connected with establishment of foam formation temperature remain unresolved there. Also, effect of binders based on vegetative raw materials on properties of flexible insulating materials is considered in [6], however, the flammability related issue remains unresolved. Effect of thermal modification and flame retarding ability was
discovered in [7] by means of burning characteristics such as weight loss, combustion rate and maximum combustion rate but no chemical changes caused by influence of these factors have been reported. The materials presented in [8] are characterized by high fire resistance but the mechanism of coke formation and temperature transitions during thermal action were not shown.

Effectiveness of application of components of a coating based on organic substances is shown in [9], i.e. it is possible to significantly influence formation of a protective foam coke layer due to the action of flame retardants based on polyphosphoric acids and foamers. However, it is necessary to study conditions of formation of a heat barrier and establish an effective action of coating with formation of a coke layer.

Paper [10] presents the most promising flame-retardant compositions of bloater coatings which are complex systems of organic and inorganic components but the issues of manifestation of joint action of the coating components in the foaming process remain unresolved.

Significant increase in stability, density and strength of the protective layer is achieved due to directed formation of certain additives that form high-temperature compounds [11]. However, no relevant physical and chemical calculations were given to confirm this process.

Besides, many coatings have a series of drawbacks, such as application of individual components or loss of functional properties with an increase in environment temperature [12]. This means that it was not found how the process proceeds under thermal conditions in the range of decomposition of the flame-retardant coating.

In addition, studies were conducted on protective materials prepared from organic substances and a solution of colemantine ore [13]. It was shown that due to the established relationship, it becomes possible to adjust contents of components to ensure the heat resistance property.

Synergistic effect of ammonium polyphosphate and aluminum oxide trihydrate playing role of the fire protection components for an epoxy composition reinforced with natural fibers as a flame-retardant material is considered in [14].

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Synergistic effect of ammonium polyphosphate and aluminum oxide trihydrate playing role of the fire protection components for an epoxy composition reinforced with natural fibers as a flame-retardant material is considered in [14]. It was also shown that not always nonreinforced compositions were unable to provide an effective fire resistance when temperature changes. That is why the combustion process with an intense weight loss occurred, so solution of this issue requires development of new approaches.

Therefore, establishment of rate of flame-retarded material burnout and influence of their components on this process is an unsolved part of the problem of ensuring fire resistance of wood building structures. This has necessitated studies in this direction.

3. The aim and objectives of the study

The study objective is to establish patterns of wood specimen burning and parameters of reducing combustion rate when fire protection is applied.

To achieve the objective, the following tasks were solved:
- to establish features of reducing rate of wood burnout under thermal action on the specimen with coating applied;
- to compare experimental dependences of the rate of wood burnout with obtained analytical equations and establish conformity of the protective material structures.

4. Materials and methods used in studying the rate of wood burnout

4.1. Materials used in the experiment

To establish wood combustibility, specimens of straight-grained pine wood with dimensions of 150×60×30 mm, density of 450–470 kg/m³ treated with the following flame retardants (Fig. 1) were used:

- impregnating solutions based on a mixture of ammonium sulphates and phosphates and a polymer (DSA-1 fire protection means);
- flame-retardant bloating organic-mineral coating (Skela-w);
- inorganic coating (Utility Model Patent of Ukraine No. 95440).

The wood specimen surface was coated layer-wise with flame-retardants at the following consumption: 600 g/m² for impregnating composition, 440 g/m² for inorganic coating and 297 g/m² for bloating organo-mineral coating. After drying to a constant weight, treated and untreated wood specimens were tested.

4.2. The procedure of determining indicators of wood burnout rate

A unit for determining material combustibility group was used [1] in the study. It was additionally equipped with a device for recording the specimen weight change during tests. The essence of the tests was to measure loss of the wood specimen weight at certain times points during thermal action and determine burnout rate.

The study of thermal action on wood specimens was conducted at various temperature values since wooden structures are exposed to thermal action of various flame types in a case of fire. Therefore, intensity of combustion of igniting gas mixture was increased by 25 % and 50 %, that is, temperature of the combustion gases at the unit exit was 200 °C, 250 °C and 300 °C, respectively.

Loss of wood specimen weight was measured and the obtained values were used in calculation of burnout rate using the equation:

\[ m = \frac{\Delta m}{\tau \cdot S}, \]  

where \( \Delta m \) is the specimen weight loss in the test, kg; \( \tau \) is the test time, s; \( S \) is the specimen surface area, m².

In this case, the specimen surface area was 0.0306 m², that is, the entire surface of the specimen was subjected to thermal action.

5. Analysis of functional dependences occurring during burning of wood specimens

In general, the wood burning rate is described by equation [2]:

\[ m = m_0 + \frac{\omega_0}{\alpha - \gamma} (e^{-\gamma \tau} - e^{-\alpha \tau}), \]  

where \( \tau \) is the time of burning of the wood specimen, s; \( \alpha \) is the index of flame burning development, s⁻¹; \( \gamma \) is the index of inhibition of combustion reactions due to the action of flame retardants, s⁻¹; \( \omega_0 \) is intensity of combustion at the
time point, kg/(m²·s²); \( m_0 \) is the initial rate of burnout, kg/(m²·s).

Functional dependences of the wood burnout rate in the presence of fire protection and without it are shown in Table 1.

| Wood specimen                  | Functional dependence of wood burnout rate |
|--------------------------------|--------------------------------------------|
| Nontreated (\( \alpha \) \( > \) \( \gamma \) or \( \gamma \) \( \rightarrow \) 0) | \( m_{w-a} = m_0 + \frac{m_0}{\alpha} (1 - e^{-\alpha t}) \). (3) |
| Flame-retarded (\( \alpha \) \( < \) \( \gamma \) or \( \alpha \) \( \rightarrow \) 0)   | \( m_{w-a} = m_0 - \frac{m_0}{\gamma} (e^{-\gamma t} - 1) \). (4) |
| Flame-retarded (\( \alpha \) = \( \gamma \) )                                    | \( m_{w-a} = m_0 + \frac{m_0}{\gamma} \cdot e^{-\gamma t} \). (5) |

Practically, material weight loss during combustion of flame-retarded wood is possible in accordance with the above laws which makes it possible to establish dynamics of material burnout rate depending on properties of the flame-retarding coating. Comparison of theoretical dependences and experimental data allows one to predict flame-retarding qualities of materials depending on physical and chemical properties of the coating.

6. Results obtained in the study of wood burnout rate

6.1. Specimen weight loss during wood burnout

Tests on objects, in particular model specimens of flame-retarded wood (Fig. 1), were performed to determine wood burnout rate.

As can be seen from Table 2, the specimen weight loss occurred at the initial heating stage because of water evaporation and then the stage of intense combustion of untreated wood has taken place. Multiple burnout increase with an increase in the burner flame intensity was observed. Water evaporation is characteristic of flame-retarded wood at the initial stage and then the process of coating bloating takes place with weight loss.

6.2. The wood specimen burnout rate

Fig. 2 shows dynamics of change of untreated wood specimen burnout rate in the course of experimental study (dots). Theoretical dynamics of burning rate was calculated in accordance with this value.

The specimens treated with an impregnating solution based on a mixture of ammonium sulfates and phosphates and a polymer (DSA-1 flame-retardant means) as well as organic-mineral and inorganic coatings were subjected to thermal action during tests. The obtained experimental dependence of specimen weight loss and the accordingly calculated theoretical dynamics of burnout rate for specimens of flame-retarded wood are shown in Fig. 2–5.

The results of processing experimental values obtained in burning wood specimens are shown in Table 2. Experimental studies have confirmed that the untreated wood specimen was ignited under thermal action, flame spread over the specimen surface resulting in its combustion at a burnout rate of 18 g/(m²·s). For the cases of raising intensity of combustion of the igniting gas mixture by 25 % and 50 %, the specimen weight loss rate increased 1.4 and 1.8 times, respectively.

![Fig. 1. Model specimens of flame-retarded wood: untreated (a); treated by flame retardant impregnation (b); treated with a flame-retardant coating on an inorganic base (c); treated with a flame retardantorganic-mineral coating (d)](image)
In the case of wood impregnation, the rate of weight loss has reduced to 4.8 g/(m² s) due to decomposition of fire-retardants under thermal action with release of non-combustible gases inhibiting the processes of material oxidation and forming a coke layer. The obtained experimental data on temperature (200 °C) of flue gases at the unit exit are described by equation (3) and by equation (1) when combustion intensity increases.

Application of organic-mineral bloater coating under thermal action has resulted in formation of a foamed coke layer, suppression of heat transfer from the high-temperature flame to the material and reduction of the burning rate down to 3 g/(m² s). Experimental values are described by equation (3). When wood was treated with an inorganic coating, a heat-resistant ceramic film was formed on the wood surface. It reduced the burnout rate 3.8 times. However, the wood specimen has been ignited with an increase in intensity of combustion of the igniting gas mixture by 50 % which has brought about an increase in the rate of weight loss. Experimental curves of the wood weight loss are described by equations (3) and (4) for an increase in intensity of igniting gas combustion by 50 %.

| Criterion characteristics | Pine wood specimen | Temperature of flue gases, °C | α₀, N/m³ | α × 10⁰⁻⁴, s⁻¹ | γ × 10⁻³, s⁻¹ |
|---------------------------|-------------------|-----------------------------|---------|----------------|----------------|
| Untreated                 | 200               | 0.15                        | 7.01    | 0              |
| -                         | 250               | 0.22                        | 9.35    | 0              |
| -                         | 300               | 0.75                        | 31.89   | 0              |
| Treated with an impregnating solution | 200 | 0.09 | 0 | 18 |
| -                         | 250               | 0.07                        | 2.2     | 0.287          |
| -                         | 300               | 0.10                        | 3.0     | 0.121          |
| Treated with an organic-mineral coating | 200 | 0.09 | 0 | 27.8 |
| -                         | 250               | 0.11                        | 0       | 18.1           |
| -                         | 300               | 0.26                        | 0       | 7.8            |
| Treated with an inorganic coating | 200 | 0.12 | 0 | 24.1 |
| -                         | 250               | 0.20                        | 0       | 7.3            |
| -                         | 300               | 0.16                        | 0.29    | 7.1            |
3.3 times decrease for inorganic coatings and almost 6 times for organic coatings.

The results of comparison of experimental dependences of the wood burnout rates with analytical equations are given in Table 4.

### Table 4

| Time, s | Wood burnout rate, kg/(m²·s) | Wood burnout rate, kg/(m²·s) |
|---|---|---|
|   | Untreated | Treated with a flame-retardant impregnating solution |
|   | 150 | 125 | 100 | 150 | 125 | 100 |
| 50 | Ex(T(3)) | Ex(T(3)) | Ex(T(3)) | Ex(T(2)) | Ex(T(2)) | Ex(T(4)) |
| 50 | 14 | 20.4 | 11.4 | 9.1 | 7.7 | 6.4 | 6 | 4.7 | 4.3 | 2.5 | 2.9 |
| 100 | 21 | 25 | 16.6 | 15 | 12 | 10.8 | 100 | Ex(T(2)) | Ex(T(4)) | Ex(T(4)) | Ex(T(2)) | Ex(T(4)) |
| 150 | 24 | 26.3 | 22 | 20.8 | 17 | 16.1 | 100 | 16 | 14.9 | 12 | 11 | 6.4 |
| 200 | 23 | 26.3 | 20 | 20.2 | 17 | 17.6 | 100 | 16.9 | 17.3 | 13 | 13 | 6.4 |
| 250 | 20 | 26.3 | 20 | 23 | 16.8 | 17.6 | 100 | 17.2 | 19.3 | 14 | 14.6 | 5.5 |

**Note:** Ex: experimental values of the wood burnout rate; T: theoretical values calculated by (2)–(5) and taken in brackets.

The results of comparison of experimental dependences of the wood burnout rate with the derived analytical equations have shown on the whole a conformity between them. However, individual deviations of experimental and theoretical data, in particular, loss of wood weight at burner heat intensity of 150 % can be explained by the period of transition to other dependence which requires other study.

6. Discussion of results obtained in determining the wood burnout rate

As evidenced by the study results (Fig. 3, Table 1), the wood burnout under thermal action of high-temperature flame, proceeds through its decomposition and is characterized by loss of specimen weight. Instead, the processes of ignition and flame propagation for treated specimens (Fig. 4–6, Table 1) considerably slowdown due to the action of flame retardants. Such protective mechanism is determined, first of all, by decomposition of flame retardants under influence of temperature with heat absorption and release of non-combustible gases and a change of direction of material expansion toward formation of a hard-combustible coke residue. When wood is treated with inorganic coating, a heat-resistant ceramic film is formed on the wood surface that reduces burnout rate. However, heat conductivity rises to critical temperatures with growth of thermal action and the wood specimen decomposes which is confirmed by an increase in the rate of burnout. Application of an organic-mineral bloating coating under thermal action leads to formation of a layer of foamed coke, inhibition of heat transfer from high-temperature flame to the material and reduction of burnout rate. Besides, processes of coating decomposition occur with a release of gases and formation of a heat-protecting layer of foamed coke on the wood surface. This agrees with the data known from [5, 8] which associate effectiveness of material thermal protection with the action of protective substances when flame retardants are added.

In contrast to the results of studies [12, 14], the obtained data on the influence of protective means on the process of heat transfer to the material and change of flame-retardant properties have enabled the following assertion:

- not only formation of a flame-retardant coke layer is the main regulator of the process but also decomposition of flame retardants with release of non-combustible gases, in particular, nitrogen and carbon dioxide which effect flame and inhibit the oxidation process in the gas and condensed phases as noted in [1];
- the process of protection of combustible materials with application of flame-retardant coatings is significantly influenced by reactions in a pre-flame zone resulting in formation of products like soot on the surface of the natural combustible material.

However, as can be seen from the study results, when thermal action on the wood specimens treated with flame-retardant impregnation increases, protection is greatly reduced which is indicated by growth of combustion parameters and requires use of a greater amount of flame retardants. The fire protection with inorganic coatings can withstand rising combustion heat but it also must be improved. For the wood specimens treated with bloater coatings, all parameters affecting the burnout rate had lower values and were leveled out with a growth of thermal action. Such conclusions can be considered useful from a practical point of view as they make it possible to reasonably approach to definition of necessary formulations of flame retardants. Treatment with a bloater coating more effectively counteracts high temperatures. This fact should be kept in focus when developing coating formulations for wood. From theoretical point of view, they make it possible to establish mechanism of the fire protection processes which constitute certain advantages of this study. The results of determining the flame-retarded wood burnout rate (Tables 1, 4) indicate an ambiguous effect of nature of protective means on the change of combustion parameters and flame suppression. In particular, this implies availability of data sufficient for obtaining a quality combustion process and determining the moment from which oppression process starts which is a new and unexplored occurrence. Such determination will make it possible to investigate transformation of the wood surface under the action of fire protection with coke formation and flame suppression and identify the variables significantly affecting start of this process.

This study is a continuation of the studies discussed in [1–4] where the mechanism of fire protection of organic natural materials, formation of foamed coke and reduction
of weight loss during wood combustion are fully provided. These studies require development of full-scale test methods and study and description of wood behavior.

Further studies may be aimed at theoretical and experimental elucidation of the wood combustion processes, establishment of a relationship between components and properties of the protection means.

8. Conclusions

1. Experimental studies of burnout rate in a case of ignition of wood specimens were conducted and the effect of flame-retardant coating on this parameter was revealed. Characteristics of parameters of ignition and combustion suppression were determined which makes it possible to know the change of dynamics of wood burnout during decomposition of the flame-retardant coatings. For example, for wood flame-retarded by impregnation, the burning suppression indicator is reduced to zero when thermal action grows and the burning parameter rises from zero to \( \alpha \approx 3.0\) s\(^{-1}\). Instead, the burnout indicator decreased by 3.5 times in the wood protected by coating with growth of thermal action but the burning parameter was absent. At the same time, the rate of burnout of wood specimens protected by impregnation was reduced 3.9 times. Corresponding figures were 3.3 times for inorganic coatings and almost 6 times for organic coatings.

2. The results of comparison of the experimental dependences of the wood burnout rate with the derived analytical equations have shown conformity between them with an admissible deviation. Criterion characteristics of the combustion parameters and the combustion termination parameters were determined. It has been established that the criterion relations changed in an inverse order in presence of fire barrier depending on coating properties, that is, the burning parameter was absent and the burning suppression parameter increased by ten times: 0.0018 s\(^{-1}\) for impregnation, 0.00241 s\(^{-1}\) for inorganic coatings and 0.00278 s\(^{-1}\) for bloater coatings. This indicates formation of a heat-shielding layer on the wood surface which prevents burning and propagation of high temperature to the material which is confirmed by absence of ignition of flame-retarded wood. With an increase in thermal action on flame-retarded wood specimens, the burning parameter increased and the burning suppression indicator decreased and, depending on its value, treatment of wood with the bloater coatings is the most effective protection.

References

1. Tsapko Y., Tsapko A. Establishment of the mechanism and fireproof efficiency of wood treated with an impregnating solution and coatings // Eastern-European Journal of Enterprise Technologies. 2017. Vol. 3, Issue 10 (87). P. 50–55. doi: https://doi.org/10.15587/1729-4061.2017.102393
2. Tsapko Y. Effect of surface treatment of wood on the fire resistance of wooden structures // Eastern-European Journal of Enterprise Technologies. 2013. Vol. 5, Issue 5 (65). P. 11–14. URL: http://journals.uran.ua/ejet/article/view/18104/15850
3. Tsapko Y., Tsapko A., Bondarenko O. Establishment of heat-exchange process regularities at inflammation of reed samples // Eastern-European Journal of Enterprise Technologies. 2019. Vol. 1, Issue 10 (97). P. 36–42. doi: https://doi.org/10.15587/1729-4061.2019.156644
4. Increase of fire resistance of coating wood with adding mineral fillers / Tsapko Y., Kyrycyok V., Tsapko A., Bondarenko O., Guzii S. // MATEC Web of Conferences. 2018. Vol. 230. P. 02034. doi: https://doi.org/10.1051/matecconf/201823002034
5. Neue Wege: Reaktive Brandschutzbeschichtungen für Extrembedingungen / Krüger S., Gühr G. J. G., Watolla M.-B., Morys M., Häbler D., Schartel B. // Bautechnik. 2016. Vol. 93, Issue 8. P. 531–542. doi: https://doi.org/10.1002/bate.201600032
6. Effects of Complex Flame Retardant on the Thermal Decomposition of Natural Fiber / Xiao N., Zheng X., Song S., Pu J. // BioSources. 2014. Vol. 9, Issue 3. P. 4924–4933. doi: https://doi.org/10.15576/biores.9.3.4924-4933
7. The effect of synthetic and natural fire-retardants on burning and chemical characteristics of thermally modified teak (Tectona grandis L. f.) wood / Gaff M., Kačík F., Gáspár M., Todaro L., Jones D., Corleto R. et. al. // Construction and Building Materials. 2019. Vol. 200. P. 551–558. doi: https://doi.org/10.1016/j.conbuildmat.2018.12.106
8. Zhao P., Guo C., Li L. Flame retardancy and thermal degradation properties of polypropylene/wood flour composite modified with aluminum hypophosphate/melamine cyanurate // Journal of Thermal Analysis and Calorimetry. 2019. Vol. 135. Issue 6. P. 3085–3093. doi: https://doi.org/10.1007/s10973-018-7544-9
9. Cirpici B. K., Wang Y. C., Rogers B. Assessment of the thermal conductivity of intumescent coatings in fire // Fire Safety Journal. 2016. Vol. 81. P. 74–84. doi: https://doi.org/10.1016/j.firesaf.2016.01.011
10. Graphene-Borate as an Efficient Fire Retardant for Cellulosic Materials with Multiple and Synergetic Modes of Action / Niem M. J., Tran D. N. H., Tung T. T., Kabiri S., Losic D. // ACS Applied Materials & Interfaces. 2017. Vol. 9, Issue 11. P. 10160–10168. doi: https://doi.org/10.1021/acsami.7b00572
11. Carosio F., Alongi J. Ultra-Fast Layer-by-Layer Approach for Depositing Flame Retardant Coatings on Flexible PU Foams within Seconds // ACS Applied Materials & Interfaces. 2016. Vol. 8, Issue 10. P. 6315–6319. doi: https://doi.org/10.1021/acsami.6b00598
12. An investigation into waterborne intumescent coating with different fillers for steel application / Md Nasir K., Ramli Sulong N. H., Ger J. M., Tran D. N. H., Tung T. T., Kabiri S., Losic D. // ACS Applied Materials & Interfaces. 2017. Vol. 9, Issue 11. P. 10160–10168. doi: https://doi.org/10.1021/acsami.7b00572
13. Erdoğan Y. Production of an insulating material from carpet and boron wastes // Bulletin of the Mineral Research and Exploration. 2016. Issue 152. P. 197–202. doi: https://doi.org/10.19111/bmrc.74700
14. Synergistic of ammonium polyphosphate and alumina trihydrate as fire retardants for natural fiber reinforced epoxy composite / Khalili P., Tshai K. Y., Hui D., Kong I. // Composites Part B: Engineering. 2017. Vol. 114. P. 101–110. doi: https://doi.org/10.1016/j.compositesb.2017.01.049