1. Introduction

Timber, as a building material, is widely used in construction and architecture due to its mechanical and operational properties, but because of increased combustibility, it is a fire hazardous material. Given the fact that this material is sensitive to the effects of high temperature, it is possible to increase the level of fire safety of the sites, where wooden building structures are used, with the help of their fire protective treatment.

The operational reliability and efficiency of fire protection of timber depend on the quality and reactive capability of a fire protection agent, as well as on the class of operating conditions of the site where these materials are used. Sometimes during the operation of fire-protected timber under conditions of fluctuations in temperature and humidity fields in a wide range, water-soluble compositions are washed out, which leads to the ignition of combustible structures under the action of high-temperature flame. An example is a fire that occurred in 2010 in the production structure of the enterprise “VERKON”, Kyiv (Ukraine) during the repair of the roof and the lights, the wooden structures of which lost fire protection ability and caught fire when gas burners were used.

The need for fire protection is also necessary for the sites of mass gathering of people when these sites are made of wooden building structures. The need for developing new types of fire protective materials, was established. That is why there arises a need to determine the conditions for the formation of a barrier for washing out and to establish a mechanism for inhibition of moisture transmission to the material. In this regard, a mathematical model was built of washing out fire retardants using a polymeric shell made of organic material as a coating, which makes it possible to estimate the effectiveness of a polymer shell by the amount of the washed-out fire retardant. According to the experimental data and theoretical dependences, the dynamics of the release of fire retardants from the fire-protective layer of the coating was calculated; it did not exceed 1.0 %, and therefore, ensures fire protection of timber. The results of determining the weight loss of the sample under the influence of water indicate the ambiguous impact of the nature of protection on the washout. In particular, this implies the availability of data sufficient for performing a high-quality process of moisture diffusion inhibition and, based on it, detection of the moment, from which a decrease in efficiency of a coating begins. The experimental studies proved that a sample of fire-protected timber after exposure to water for 30 days withstood the influence of a heat flow. In particular, the loss of timber weight after the temperature exposure was less than 6 %, and the temperature of flue gases did not exceed 185 °C. Thus, there is a reason to assert the possibility of directed control of the processes of fire protection of timber through the use of polymer coatings capable of forming a protective layer on the surface of fire-protected material, which inhibits the rate of washing out the fire retardants.

Keywords: protective equipment, weight loss, treatment of timber surface, washing out a fire retardant, polymer shell...
of wooden structures and operated without heating, periodically, since fire protection agents are washed out of timber, and crumble under the action of gravitational forces. An example is a fire in the children’s camp “Victoria”, Odessa (Ukraine).

That is why the studies aimed at determining the regularities of inhibition of the process of washing out water-soluble fire retardants from fire-protected timber through the shell of waterproof polyurethane coating are relevant.

2. Literature review and problem statement

In paper [1], it is shown that reactive fire-protective coatings very effectively increase the fire resistance of wooden structures. However, coatings and fire resistance tests must be adapted and refined for increasingly complex applications, including changes in temperature and humidity. Current research focuses on the development of new materials (for example, geopolymers, ceramic coatings, silicone-based coatings) for extreme ignition scenarios (extreme temperatures, long exposure time) and the implementation of complex functions (complex geometry, changes in operating conditions). However, it is not said what classes of operation they belong to.

The value of the effect of hygroscopicity on washing-out resistance affects the indicators of fire-protective substances [2]. To increase fire protection, nano-SiO$_2$ zol was added to various nitrogen-phosphorus-boron fire-retardants to produce four new impregnation compounds, specifically: (1) ammonium dihydrophosphate+tetrahydrate dinatrum octaborate (AD), (2) ammonium dihydrophosphate+tetrahydrate dinatrum octaborate+nano-SiO$_2$ sol (ADS), (3) ammonium dihydro phosphate phosphate+boric acid (AB) and (4) ammonium dihydrogen phosphate+boric acid+nano-SiO$_2$ sol (ABS). After that, hygroscopicity, washing out resistance, and heat resistance were studied with scanning electron microscopy (SEM), energy-dispersion X-ray spectroscopy (EDX), thermogravimetric (TG) and Fourier transform infrared spectrometer (FTIR), and optimal complex fire-retardant – ABS. The results show that the addition of nano-SiO$_2$ sol cannot only effectively reduce the hygroscopicity of fire-protective materials by more than 20 % but also improve its resistance to washing out. However, the relevant physicochemical data on the washout during the operation were not provided to substantiate this process.

Description of behavior of timber [3], impregnated with urea guanil phosphate, boric acid, and melamine-formaldehyde resin, showed that resistance to leaching increased significantly while maintaining excellent fire resistance, as tested by the method of limit oxygen index. It was found that urea guanil phosphate is well retained in the structure of timber, as evidenced by the FTIR spectroscopy. The loss of effectiveness of fire protection substance by 7.4 % was achieved after excessive washing out of the treated timber. However, the issues that are associated with the mechanism of formation of coke foam remain unresolved. The reason for this may be the subtleties regarding the formation of a protective layer, which accordingly makes such studies difficult.

Study [4] was aimed at discussing the method for evaluating the indicators of the fire reaction of timber, treated with flame based on phosphorus organic substances, which was used as facades of buildings, taking into consideration the deterioration due to aging. The characteristics of timber resistance to the fire were studied and it was found that the initial indicators deteriorated during fire reaction testing by more than 12 %.

Effectiveness of the application of coating components is shown in research [5], dealing with the effects of combined timber treatment with nitrogen-phosphorus fire protection and of the heat treatment, which was subjected to hygroscopicity, washing out, heat resistance, research using Fourier transform infrared spectra (FTIR), and X-ray diffraction properties. Timber samples were impregnated with 10 % fire retardant solution and subjected to thermal action at 140–160 °C for 30 minutes. Heat-treated timber reduced moisture absorption by 5 % and increased the resistance to fire-retardant washing-out up to 10 % and improved thermal stability by more than 3 %. It was established that heat treatment decomposes a fire retardant, and the acidity of these decomposition products has a dehydrating effect on timber polysaccharides, and the number of hydroxyl groups in timber decreased significantly. Low-temperature heat treatment increased the fixation of a fire retardant and hydrophobicity of timber treated with a fire retardant. However, the execution of these works requires special equipment at the stage of manufacturing materials.

The aim of research [6] was to explore the effects of boron-based fire retardants and fiber load on mechanical, fire, and thermal indicators of impregnated timber filled with high-density polyethylene composites. To do this, timber was impregnated with a solution of boric acid, boron, and their mixtures and dried before making composites. The effect of wash-out on the limit levels of the oxygen index (LOI) of fire-protected samples was also explored, and it was revealed that boron samples ensured better mechanical properties compared to boric acid samples and showed a 19 % improvement in the stretching module at fiber load by 40 % compared to the control samples. In addition, fire-protected samples with boron increased the levels of LOI and charring, as well as reduced combustion rate by 50 %. The presence of boric acid in the composition after washing out somewhat decreased the protection effectiveness up to 5 %.

The studies were conducted to study the functionality of timber treated with fire retardants [7]. Sodium silicate penetrated the timber, the following agents, such as boric acid, ammonium borate, diammonium phosphate, were used to fix a fire retardant. As a result, the washing-out resistance was improved by 10 %. A fire-protective combination such as sodium silicate, boric acid, and diammonium phosphate showed high hygroscopic properties and corrosion effectiveness to metals. However, it was not said about the impact of environmental changes on the coating, its destruction in time.

In paper [8], aluminum hypophosphate and melamine cyanide that were used as a fire retardant in a composite of polypropylene and timber meal were explored. The flammability of the composites was tested using a limit oxygen index (LOI), a vertical combustion test, and a conical calorimeter test. When 20 % of fire retardant were loaded into the composite, the LOI increased up to 29.5 %. In addition, bending strength increased by about 11.0 %. The results of the calorimeter test showed that the heat release rate and the peak heat release rate of the composite were significantly reduced. The mechanism of thermal destruction of composites was explored by the methods for thermogravimetric analysis, and Fourier transform infrared spectrometry, and it was found that fire retardants have the effect of slowing down combustion in the gas phase and the condensed phase under
the thermal destruction of the composite, but it is not said about water-resistance of this composition.

The effect of inorganic fillers on water-based fire protective coating was studied in paper [9]. The results show that the combination of coating with the CaCO$_3$ filler significantly improved the fire-protecting characteristics due to the resulting layer of coke foam and equilibrium temperature equal to 264 °C. The coal layer showed a homogeneous dense foam structure in the microphotograph, and this composition had an adhesion strength of 2.13 MPa, indicating the effectiveness of adhesion to the substrate. On the contrary, the coating combination with Al(OH)$_3$ showed the highest oxygen index, equal to 35 %, which led to resistance to ignition. However, the mechanism of coating swelling, in this case, is not specified and the operating conditions of the coating were not detected.

The mathematical model and the procedure of the numerical study of the kinetics of the state of heat and humidity of the capillary-porous body, constructed on the simultaneous solution of the equation of thermal conductivity and moisture transmission, were proposed in paper [10]. However, these studies are characteristic of inorganic material, and it is impossible to apply them to timber.

Thus, it was found from literary sources that fire protection coatings are able to be washed out from the timber surface during operation and the parameters that provide resistance to loss of fire protection by timber, as well as what affects this process, were not identified. The meagerness of mathematical models for explanation and description of the process of washing out fire retardants, the negligence of the use of organic substances to inhibit their output lead to ignition of wooden structures under the influence of a flame. That is why the establishment of counteraction to washing out fire retardants from fire-protected timber and the impact of the components on this process caused the need for research in this direction.

3. The aim and objectives of the study

The aim of this research is to identify the regularities of washing out water-soluble salts through a polyurethane shell from fire-protected timber. This makes it possible to substantiate the use of fire protection coating at sites with high humidity.

To reach the aim, the following tasks were to be solved:
– to model the process of washing out water-soluble salts through a polyurethane shell from fire-protected timber;
– to establish the effectiveness of fire protection of timber after washing out water-soluble salts through a polyurethane shell under the influence of water.

4. Materials and methods of research

4. 1. Studied materials used in the experiment

The studies of counteraction to washing out a protective agent from timber, treated with the fire protection composition, were carried out on the samples of straight-grained timber [11] with dimensions of 150×60×30 mm (Fig. 1).

The samples were treated with covering impregnation solution “Skela-i” (mixture of carbamide 28…30 % and phosphoric acids 23…24 % and starch 20 %) in the amount of 270.2 g/m², while the thickness of the fire-protecting coating was about 100 μm [12]. To increase resistance to water, the samples were covered with polymer mixtures based on polyurethane organic coatings produced in Ukraine in the amount of 100 g/m² [13]. The thickness, respectively, was about 40 μm.

4. 2. Procedure for determining the indicators of washing out water-soluble phosphorus-ammonium salts from a fire-protecting coating of timber through a polyurethane shell

To obtain the values of mass transfer of a fire retardant from timber, special equipment was developed and manufactured (Fig. 2).

Fig. 1. Model samples of tested timber:

a – pine; b – spruce; c – beech

Fig. 2. Device to test washing out of fire-protective agents

The samples treated with the fire-protecting composition and the applied protective layer with the ends treated with wax (2 mm thick) were placed and fixed in a vessel with distilled water of the volume of 100 cm$^3$ (Fig. 2). After a certain period, the solution was stirred with a glass stick, 5 cm$^3$ of water were taken with the help of a pipette, and the concentration of the fire retardant was determined [14].

The studies on determining the timber combustibility after exposure to water were carried out by thermal exposure.

The essence of the test method for experimental determining of the combustibility group of fire-protected timber is to influence the sample located in the setup, the flame of a burner, or the radiation panel with the specified parameters.

During the experimental studies on determining the combustibility group, the maximum temperature increase in gaseous combustion products (ΔT) and the weight loss of the sample (Δm) were recorded.

According to the test results, the materials are classified as:
– difficultly combustible – ΔT<60 °C and Δm<60 %;
– combustible – ΔT≥60 °C or Δm≥60 %.
5. Results of the study of the process of washing out water-soluble phosphorus-ammonium salts from a fire-protective coating of timber through a polyurethane shell

5.1. Modeling the process of washing out water-soluble phosphorus-ammonium salts from a fire-protective coating of timber through a polyurethane shell

The mechanism of washing out the protective coating from timber during operation is as follows: when moistening timber, fire retardants of protective agents dissolve and gradually move to the timber surface, where their concentration decreases over time (Fig. 3). The existence of a polymeric shell on the sample surface slows down the washing out process.

Fig. 3. Calculation and analytical scheme of the process of the coating washing out from timber

Four areas are considered:
1 – external environment, \( x < 0 \);
2 – a polymer shell of thickness \( \delta \), mm, \( 0 \leq x \leq \delta \);
3 – a part of the coating with dissolved substance, \( \delta < x \leq Z(\tau) \) (\( Z \) is the dissolution coordinate, m);
4 – material of the coating with hard substance, \( \delta \leq x \leq \delta + R \) (\( R \) is the thickness of the fire-protecting coating, m).

The differential equation of diffusion describing the washing-out process has the form of [15]:

\[
\frac{\partial^2 C}{\partial x^2} = \frac{1}{\phi_i \tau} \frac{\partial C}{\partial \tau},
\]

where \( C \) is the substance concentration in the \( i \)-th area, \( \text{kg/m}^3 \); \( \phi_i \) is the coefficient of substance diffusion in the \( i \)-th area, \( \text{m}^2/\text{s} \); \( \tau \) is the time of sample being in a humid environment, s.

Initial and limit conditions:
- at \( x < \delta \), \( \tau = 0 \):
  \[ C_i(x,0) = C_{\text{min}}, \ Z(0) = 0; \]  \hspace{1cm} (2)
- at \( x > \delta \):
  \[ C_i(x,0) = C_{\text{max}}. \]  \hspace{1cm} (3)

At the boundary of a polymer film and the environment, the boundary condition of the third order is assigned as:

\[
\phi_i \left. \frac{\partial C}{\partial x} \right|_{x=0} = \beta \left( C_2 - C_1 \right)_{x=0},
\]

where \( \beta \) is the coefficient of mass exchange between the surface of the coating surface and the environment, m/s.

At the dissolution of the coating and diffusion of the protective coating components, the boundary conditions of the fourth order are assigned:
- at \( x > \delta \), \( \tau > 0 \) at the separation boundary:
  \[ C_2(\delta, \tau) = C_1(\delta, \tau); \]  \hspace{1cm} (5)
- at the front of coating dissolution
  \[ C_i[Z(\tau), \tau] = C_i[Z(\tau), \tau] = C_f. \]  \hspace{1cm} (6)

The solution to equation (1) is represented in the integral form or in infinite series, which requires a significant amount of calculation, that is why numerical methods of analysis were applied. To do this, dimensionless variables were introduced:

\[
\frac{\tau}{R} = \frac{x}{R}; \quad F_0 = \frac{\phi_i \cdot \tau}{\phi_i \cdot \tau}; \quad Z = \frac{Z}{R}; \quad C = \frac{C}{C_{\text{max}}},
\]

where \( F_0 \) is the Fourier diffusion number; \( R \) is the coating thickness, mm.

For a one-dimensional problem we have the following calculation dependence [16]:

\[
C_{i+1,n} = (1 - 2F_0)C_{i,n} + \Delta F_0(C_{i-1,n} + C_{i+1,n}),
\]

where \( C_{i,n} \) is the concentration at the front of phase transformations; \( Z(\tau) \) and \( \tau \) are the dissolution coordinate and the time of sample being in a humid environment, respectively.

The calculation grid is represented by nodes in space \( i = 0 \ldots 100 \), obtained by conditional dividing the sample by cross-sections with pitch \( \Delta x \) and into temporary layers \( n \) with pitch \( \Delta F_0 \) on condition that

\[
\Delta F_0 = \frac{1}{6},
\]

that ensures the stability of the explicit scheme in the calculation, then from the equation (8) we obtain:

\[
C_{i,n+1} = \frac{1}{6} \left( C_{i-1,n} + 4C_{i,n} + C_{i+1,n} \right).
\]

The calculation scheme for dependence (10) is represented in Fig. 4.
and at the boundary of a polymer film and the environment – of Nusselt numbers:

\[ N = \frac{\phi^2}{\beta \Delta \lambda^2} \]  

(12)

In the case of using a polymer coating, the fire-retardant content on the surface of the polymer shell was taken equal to 1% of its original content in timber at \( F = 0.0001 \) and \( N = 0.0001 \). This value of a fire retardant was pre-assigned for calculation since it is associated with the procedure of manufacturing samples, namely the introduction of a fire retardant into the polyurethane shell when treating timber. The results of the calculation of the dynamics of fire retardant washing out from equation (10) are given in Table 1.

Fig. 5 shows the dependences of the value of fire-retardant concentration in the sample volume with a change in the thickness of the polymer shell.

As Fig. 5 shows, the value of concentration, when exposed to water, decreases over time, since criterion \( Fo \) is the dimensionless time of the non-stationary process of washing out fire retardants. In this case, criterion \( Nu \) increased, since it characterizes the degree of the ratio of mass transfer rates due to concentration transfer to the interphase surface, that is, to the shell. That is why it can be predicted that the polyurethane shell, according to the calculated parameters of Table 1, was not destroyed. When the protective shell is destroyed, the wash-out flows (\( Fo \)) and the inhibition flows (\( Nu \)) converge [16].

5.2. Results of the experimental study of the process of washing out water-soluble phosphorus-ammonium salts through a polyurethane shell during exposure of the surface of a fire-protected timber sample to water

The influence on the inhibition of the process of fire retardants washing out from fire-protected timber in the presence of a polymeric shell was determined in the course of research (Fig. 6).

![Fig. 6. The process of fire retardants washing out from the timber sample in water](image)

The results of the studies on determining the duration of the induction time of the release of a fire retardant from fire-protected timber through a layer of the polymeric shell are given in Table 2.

| Table 2 Results of the studies on determining the amount of washed-out fire retardant in water |
| Exposition time, days | Amount of washed-out fire retardant in water, mg |
| --- | --- | --- |
| Timber | Pine | Spruce | Beech |
| 0.5 | Not found | Not found | Not found |
| 1 | Not found | Not found | Not found |
| 6 | Not found | Not found | Not found |
| 12 | Not found | Not found | Not found |
| 18 | Traces | Traces | Traces |
| 24 | 0.01 | 0.03 | 0.02 |
| 30 | 0.18 | 0.16 | 0.15 |
Table 2 shows, when timber samples were exposed to water, the polymer shell withstood the effects of moisture, and a small amount of washed-out fire retardant was recorded after 30 days of timber exposure to water that did not exceed 3.0 %.

Thus, the results of the experimental determining the amount of washed-out salts in water (Table 2) coincide with the results of theoretical studies of changes in the fire retardant concentration (Table 1) and are characterized by a decrease up to 3.0 %. For more complete proof of the resistance to washing out water-soluble salts of this composition, the studies were carried out to determine the combustibility group of the fire-protected timber after exposure to water.

After exposure to water, the samples were dried and subjected to thermal action to determine the combustibility group. Fig. 7 shows the results of the action of a high-temperature flame, Fig. 8 shows the weight loss of the samples after tests, Fig. 9 shows the temperature of flue gases.

The studies revealed that the samples that were exposed to water within 30 days after the thermal action lost the weight of not more than 6.2 %, and the temperature of flue gases during combustion was not more than 220 °C. According to these results, fire-protected samples belong to difficultly combustible materials.

Thus, the obtained results on determining the combustibility group of fire-protected timber correspond to the properties of the formation of a heat-resistant layer of coke foam under the action of a high-temperature flame, so it indicates the resistance of the polymer shell to washing out water-soluble fire retardants under the influence of water, which proves the results of the theoretical studies.

In the study of the process of washing out water-soluble phosphorus-ammonium salts from the fire-protective coating of timber through a polyurethane shell, as it follows from the obtained results (Tables 1, 2), it is natural to extend the time of the destruction of the polymer shell and the release of fire retardants. This is due to the resistance of the polyurethane coating and the formation of a barrier on the surface of the fire-protected timber, which slows down the processes of diffusion of water-soluble fire retardants.

It should be noted that the existence of a polyurethane shell leads to the blockage of the timber surface from moisture penetration. Obviously, such a mechanism of influence of the polyurethane shell is the factor in regulating the process due to which the fire resistance of timber is preserved. In this sense, it is interesting to interpret the results of determining the difficult combustibility of timber after exposure to water, namely the weight loss of the samples during the thermal exposure and the temperature of flue gases during combustion. The weight loss did not exceed 6.2 %, and the temperature was no more than 220 °C. This indicates the formation of a barrier for the release of fire retardants, which can be identified by the method of thermal effect on the studies samples.

This means that consideration of this fact opens the possibility for effective regulation of the properties of fire-protected timber directly under conditions of serial industrial production.

Comparison of experimental studies of washing out water-soluble fire retardants and theoretical studies of mass transfer of the fire retardant through the polyurethane shell indicates inhibition of processes of washing out fire retardants since no more than 3 % of them were washed out.

This does not differ from the practical data well known from papers [5, 6], the authors of which, by the way, also associate the resistance to washing out with the formation of compounds that are resistant to washing out in the composition. However, unlike the results of the studies published
in [5, 6], the obtained data on the influence of a polyurethane shell and complex fire retardant on the process of inhibition of washing out water-soluble salts, make it possible to state the following:

- the main regulator of the process is not so much the formation of a significant number of water-insoluble complexes, since separate fire-protective coatings are destroyed under the influence of moisture;
- a significant impact on the process of protection of natural combustible material, when applying a fire-protective coating, is made in the direction of waterproofing the surface of timber with a polymer shell resistant to the destruction under the action of moisture.

Such conclusions may be considered appropriate from the practical point of view because they allow a reasonable approach to determining the required amount of a polymer shell. From the theoretical point of view, they make it possible to state about determining the mechanism of processes of inhibition of fire retardants release, which is a certain advantage of this study. However, it is impossible not to note that the results of determining (Fig. 2) indicate the ambiguous effect of a polymer shell on a change in fire-protective effectiveness. This is manifested, first of all, in the temperature of flue gases during testing fire-protected timber. Such uncertainty imposes certain restrictions on the use of the obtained results, which may be interpreted as the disadvantages of this study. The impossibility to remove these restrictions within the framework of this study gives rise to a potentially interesting direction for further studies. They, in particular, can be focused on detecting the moment when the fire-protective properties of timber ignition under the influence of high temperature begin to decrease. Such detection will make it possible to explore the structural transformations of the shell that begin to occur at this time and to determine the input variables of the process that significantly affect the beginning of such a transformation.

1. According to the calculation scheme, the dynamics of the release of water-soluble fire retardants from the fire-protected layer of timber at the use of a polyurethane shell were studied. The influence of the shell on the resistance to water action was found and the criterial ratios that make it possible to obtain a change in the fire retardants on the surface of timber were obtained. They are the values of the concentration of washed-out fire retardants due to their exposure to water over time that are characterized by criterion $Fo$. At the same time, criterion $Na$, which characterizes mass transfer due to the concentration transfer to the interphase surface, that is, the shell retaining the fire retardant, was substantiated. We calculated the dynamics of fire retardants release from the fire-protecting layer of the coating that does not exceed 3.0 % and is proved experimental and, therefore, provides fire protection of timber.

2. The specific features of the inhibition of the process of fire retardants release from timber when exposed to water include the insulation of the surface of fire-protected timber with a polymer shell. Thus, the sample of fire-protected timber after exposure to water for 30 days withstood the temperature influence of the heat flow. In particular, the weight loss of timber after the temperature exposure did not exceed 6.2 %, and the temperature of flue gases did not exceed 220 °C.

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7. Conclusions
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