

Проведено аналіз способів визначення вогнезахисної ефективності очерету і встановлено необхідність розробки надійних методів дослідження процесу займання та розповсюдження полум'я по поверхні будівельної конструкції, необхідних для створення нових типів вогнезахисних матеріалів. Оцінка часу займання і проходження фронтом полум'я ділянок поверхні виявило недостовірність реальних значень індексу горючості матеріалу. Був обґрунтований спосіб визначення процесу займання та розповсюдження полум'я вогнезахисних матеріалів, а з урахуванням постійних умов тепломасообміну в ході випробувань, розроблено пристрій. Визначення індексу горючості полягає у впливі на зразок теплового потоку електричної радіаційної панелі і запалюванні зразка паликом. А також визначення теплового коефіцієнта установки, вимірювання максимальної температури продуктів горіння і час її досягнення, часу займання і проходження фронтом полум'я ділянок поверхні, довжину згорілої частини зразка і розрахунку індексу горючості.

Проведені дослідження процесу займання та розповсюдження полум'я по поверхні очерету за даною методикою показали, що необроблений зразок під термічним впливом зайнявся на 52 с, полум'я поширилося по всьому зразку протягом 100 с. Вогнезахисний зразок, оброблений просочувальним розчином на основі суміші неорганічних та органічних речовин, а саме суміш карбаміду і фосфорних кислот та природного полімеру у кількості 47,1 г/м², зайнявся на 595 с, поширення полум'я по поверхні відбулося тільки на першу ділянку, максимальна температура димових газів становила 114 °С, а індекс горючості знизився до 0,42.

Результати визначення індексу горючості показали, що при впливі високотемпературного потоку на очерет вогнезахисний покриттям у кількості 46,2 г/м², загорання і поширення полум'я не відбулося, індекс горючості склав 0. За рахунок інтенсивного спучування відбулося незначне підвищення температури у вентиляційній трубі. Зниження антипірену у композиції у двічі за тих же витрат призвело до підвищення індексу горючості для покрівельного просочувального розчину до 5,8, а спучуючого покриття до 0,96 відповідно. Вищенаведені результати дозволяють встановити співвідношення антипіренів та полімерів у даних композиціях та необхідну кількість їх кількість

Ключові слова: вогнезахист очерету, просочувальні розчини, покриття, оброблення поверхні, час займання, поширення полум'я

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ESTABLISHMENT OF FIRE PROTECTIVE EFFECTIVENESS OF REED TREATED WITH AN IMPREGNATING SOLUTION AND COATINGS

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1. Introduction

Reed has a number of unique properties, such as the small specific weight, low thermal conductivity, rather high atmospheric resistance, and high chemical resistance. In addition, it is also possible to manufacture details on a construction site, it is cost effective, etc., despite a high rate of development of new technologies, products made of reed are widely used in construction.

At the same time, despite its light flammability and combustibility, reed is now widely used in Ukraine as a decorative material. It is used for making a variety of products and decoration of premises.

Reed stems have a large-pore structure and the walls of stems are quite dense, a reed mat, plate or a roof have high heat- and sound insulation properties.

Elasticity, strength, shape stability are the basic mechanical properties of reed, due to the location of its cells. The layers of cells with a high content of lignin, as if reinforced with cellulose fibers, are located in a circle and determine a high bending strength of reed while retaining its hardness and strength.

The fact that fire protection issues of such products have not been resolved yet decreases the possibility of their application in construction. Resonance facts of fires on the sites of mass gathering of people (restaurants, cafes, etc.) indicate the relevance of fire protection of building structures made of reed.

It is known that cellulose-containing construction materials are not capable of flame combustion by themselves, only the products of their decomposition burn under the influence of temperature [1, 2]. Introduction of flame retardants into

the material decreases the formation of flammable volatile products, inhibits gas-phase reactions of flames and excludes non-flame combustion of carbonized residue [3, 4].

The means that are capable to form an insulating layer, which greatly inhibits the processes of heat transfer to the material, on the surface of the building structure, became increasingly common [5].

Given the peculiarities of chemical construction and reed structure, when using reed products during fire protection, there is some difficulty related with the application of the impregnating fire-retardant agents. This is primarily due to the fact that the structure and composition of reed differ from them and the process of treatment is not implemented. That is why development of effective fire protection means is aimed at the application in the construction of facilities, where the usage of existing compositions is ineffective. Applying the new ones needs reliable ways of studying the properties of the coating. This caused the need for research in this direction.

2. Literature review and problem statement

In order to protect reed from fire, the impregnating agents are used that cover a building structure, which leads to the decomposition of flame retardants under the influence of temperature with heat absorption and release of noncombustible gases. However, such substances are ineffective and are required in significant amounts for ignition elimination; moreover, an ignition can occur in places that are unreachable for extinguishing [6].

The specific feature of fire protection of wooden building structures is creating the elements of thermal insulation shields on the surface, which can withstand the direct effect of fire and maintaining their functions within a specified period of time; however, it is not always advisable for reed [7]. Description of the behavior of flame retardant coating is a separate and challenging task and covers both swelling of the coating and subsequent heat transfer [8]. The kinetics of forming a foam coke layer that is formed at swelling has its own peculiarities and depends on the properties of substances [9]. This necessitates the study into conditions of formation of a barrier for thermal conductivity and establishment of the effective action of the coating with the formation of a coke layer.

Effectiveness of the application of components of the coating based on organic substances was shown in paper [10], where it is possible to significantly influence the formation of the protective layer of a foam coke due to the action of flame retardants based on polyphosphoric acids and foam agents. A significant increase in stability, density and strength of a protective layer is achieved due to direct formation of certain additives that produce high-temperature compounds [11].

In most cases, they are modified by polymer complexes and flame retardants, however, such coatings belong to the materials that are easily washed out and are suitable for inner premises [12]. To decrease water solubility and washing out fire retardants from impregnating solutions and coatings, the surface of fire protected material is treated with hydrophobic agents [13]. A fire-retardant composition is prepared based on organic binders, which are not soluble in water [14]. All these requirements are inherent to the wood, but are not always feasible for reed and require development of new approaches [15].

The effectiveness of flame retardants for a particular material is determined by the level of the fire protection ability and is caused by: flame retardants' decomposition under the action of temperature with heat absorption and release of noncombustible gases. In addition, the direction of decomposition of the material changes toward the formation of a heavy coke residue and formation of a heat protective coke layer on the surface of the material.

In the general form, the boundary of fire propagation over building structures is determined by the influence of the thermal action of a fire chamber furnace on a building structure at the expense of fuel combustion. After that the extent of thermal damage in the control zone is measured and this damage is compared with the damage of a reference sample of the structure [14]. The disadvantage of this method is that it works only for classification comparison of substances and materials between themselves and does not give any information about the actual capacity of the sample to the ignition and flame propagation.

There is also a known technique for determining the flammability index, in which a sample of the material is exposed to the influence of heat flow from the radiation plane over certain time [1]. The criterion of flammability index evaluation is the behavior of a sample under the influence of heat flow, such as ignition time of a sample, the length of the combusted part of a sample. The dimensionless coefficient – flammability index – is calculated based on these results. The disadvantage of this method is that it does not make it possible to estimate reliably the parameters of ignition and flame propagation, in particular, to take into account the effect on temperature of the flue gases and the flame propagation rate of samples during the creation of temperature conditions from infrared radiation of gas burners and because of the unstable heat flow and the influence of heat exchange with the surrounding environment.

Given the above, it was established that for such material as reed, there are no data as for the improvement of its fire resistance and evaluation of its treatment at the thermal action and, accordingly, the establishment of fire protection effectiveness for a specific means, particularly reed, that has specific properties and forms of application.

That is why a promising issue is the establishment of the thermal resistance of roofing means for reed in the long-term influence of temperatures and the influence of a mixture of inorganic and organic substances, which are included in the composition of a coating and are provided with thermal resistance to a flame. To evaluate the flame-retardant effectiveness of protective means for reed, there arises the need to create both appropriate methods and equipment. All the above issues must be investigated.

3. The aim and objectives of the study

The aim of this study is to explore the effectiveness of fire protection of reed with roofing impregnating compositions and coatings of a certain concentration of flame retardants that are based on the protection of materials by means of formation of a coke layer under the thermal influence.

To achieve the set aim, the following tasks were set:

- to develop the procedure and equipment for the evaluation of effectiveness of fire protection for reed;
- to establish dependences of flammability index under thermal influence, using a sample of reed, on the nature of a fire protective roofing substance.

4. Materials and methods of research

4. 1. Examined materials used in the experiment

To establish fire protective effectiveness for reed, we used the reed samples both raw and treated with two fundamentally different compositions, specifically, the water solution that is able to impregnate the material and to leave dry flame retardants in its structure after evaporation of water, as well as the coating that forms a colorless film on the surface and is able under the influence of high temperature to create a foam coke protective layer on the surface, specifically:

- by the roofing impregnating solution based of the mixture of organic and inorganic substances (a mixture of urea and phosphoric acids and natural polymer at the ratio of 2:1);
- a swelling coating that under the thermal influence forms a foam coke layer on the surface of the material (composition based on the salts of polyphosphoric acid and the polymer at the ratio of 2:1).

The resulting mass was stirred, adding 100 % water and applied to the reed sample (Fig. 1), and to study the influence of the composition of the mixtures of flame retardants, their concentration was decreased to the ratio of 1:1.



Fig. 1. Model samples of reed for testing

To study the effectiveness of the fire protective material, we used the reed samples of medium dimensions with a diameter of up to 10 mm and a height of 310 mm, which were bound in mats with dimensions 310×140 mm and treated with the roofing impregnating solution with a flow rate of 47.1 g/m², a swelling coating, at a flow rate of 46.2 g/m².

4. 2. Development of the method and equipment for determining the effectiveness of fire protection for reed; results of the study

Combustion of the mat made of reed as an example occurs as a result of heating to the temperature of the release of volatile combustion products, which in the case of existence of an ignition source are able to ignite and to support combustion independently. Application of fire protection changes decomposition of components under the action of temperature, inhibition of oxidation processes in the gas and condensed phase.

That is why using the method for determining the parameters of ignition and flame propagation of heat-insulating building materials, which includes the impact on the sample of the heat flow of the electric radiation plane and ignition of the sample by the burner, we additionally, determined thermal coefficient of the plant, measured maximum temperature of combustion products and the time it takes to achieve it, ignition time and time of passing the surface areas by the flame front, the length of the burnt part of the sample. The magnitude of the dimensionless flammability index by coefficient *I* is calculated based on the obtained data:

$$I = \sqrt{\frac{q \cdot Q}{W} \cdot \frac{\Delta T_{\max}}{\Delta T_{\text{HO}}} \cdot \frac{\tau_{\max} - \tau_0}{\tau_0} \cdot \left[1 + \frac{60 \cdot l_r}{l} \cdot \sum_{i=1}^n \frac{1}{\tau_i} \right]}, \tag{1}$$

where *q* is the specific heat of combustion of propane gas (23,630), kJ×l⁻¹; *Q* is the consumption of gas of ignition burner (0.001), l×s⁻¹; *W* is the power of the electric radiation plate, 0.5 kW; Δ*T*_{max} is the maximum increase in temperature of flue gases:

$$\Delta T_{\max} = T_{\max} - T_0,$$

where *T*₀ is the ambient temperature, °C; *T*_{max} is the maximum temperature of flue gases, °C; Δ*T*_{he} is the maximum increase in temperature of the heating equipment:

$$\Delta T_{\text{he}} = T_1 - T_0,$$

where *T*₀ is the ambient temperature, °C; *T*₁ is the temperature of output air during the operation of the heating equipment, °C; τ₀ is the time of sample ignition, s; τ_{max} is the time of reaching the maximum temperature of flue gases, s; τ_i is the time of passing the control sections by the flame front, s; *l* is the length of the sample, mm; *l*_g is the length of passing the sample, mm.

5. Experimental research into fire protection effectiveness of reed; results

Fig. 2 shows the test chamber for conducting research.

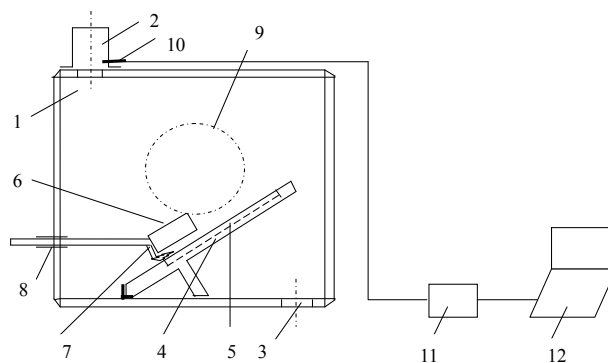


Fig. 2. Test chamber for determining parameters of ignition and flame propagation: 1 – test chamber, 2 – air pipe, 3 – ventilation opening, 4 – sample holder, 5 – sample, 6 – radiation plane, 7 – ignition device, 8 – regulation pipe, 9 – viewing glass, 10 – thermocouple, 11 – analog-to-digital converter, 12 – computer

As Fig. 2 shows, the test chamber for determining the ignition and flame propagation consists of the radiation plane with the ignition device and the sample holder mounted opposite it. The thermocouple, which measures the temperature of flue gases is mounted in the air pipe. The radiation plane of low power ensures the density of heat flow in the stationary mode for the first reference point (32±3) kW/m², which allows obtaining the temperature of 250±10 °C on the surface of the test sample at the distance of 50±5 mm and imitates low-calorie ignition source that can ignite organic material. Radiation plane along with ignition device is located opposite the middle of the bottom part of the sample in the chamber, which is made

of high-temperature insulating material to reduce heat exchange with the environment.

Research with the use of the presented method is performed in the following way. The analog-to-digital converter 11, temperature recording device 12 are turned on, gas burner 7 is lit and gas consumption is regulated so that the height of the flame is (10±2) mm. Electrical radiation plate 6 is switched on and initial temperature of the outlet pipe of the chamber is determined using thermocouple 10. The sample of material, prepared for testing, is set in holder 4, fixed with a clip and brought to radiation plane 6. During testing, the temperature in the air pipe is measured by thermocouple 10 and the maximum temperature of flue gases and the time of its achievement are measured using the markup of the sample holder through viewing glass 9. In the course of testing, we determined time of the sample ignition, time from the beginning until the passage of the zero mark by the flame front, time of passing the *i*-th section of the sample surface by the flame front and the distance, at which the flame front propagated.

The test proceeds until the flame propagation around the sample surface stops or at reaching 600 s in the absence of sample ignition.

Fig. 3, 4 show the ignition process and reed flame propagation.

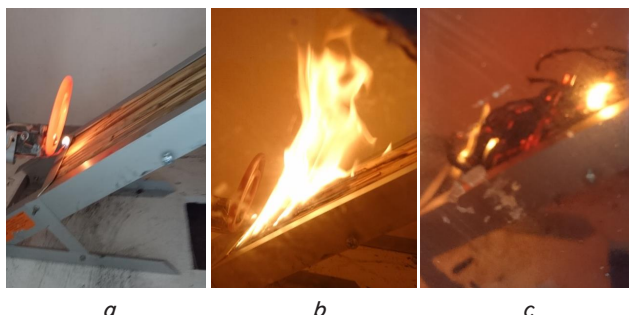


Fig. 3. Test results of the ignition process and flame propagation of raw reed: *a* – influence of flame on the sample, *b* – sample ignition, *c* – reed combustion

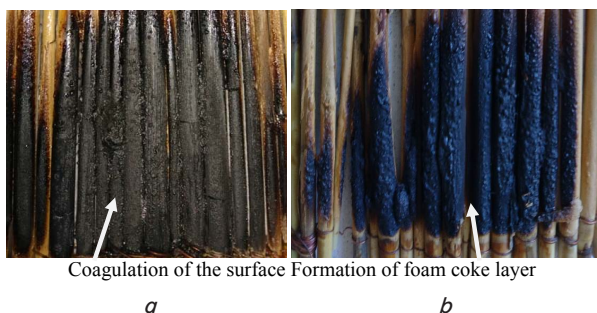


Fig. 4. Test results of the process of ignition and flame propagation of the fire protected reed: *a* – treated with roofing impregnating solution, *b* – treated with swelling coating

The results of the research into determining an increase in the maximum temperature of the gas-like combustion products (*t*, OS) and weight loss of the reed samples (*m*, %) under laboratory conditions, are shown in Fig. 5, 6.

We obtained regression dependences of the temperature of flue gases in the test chamber on time of the fire activities, which are described by dependences of type:

$$v(t) = a_0 + a_1 \cdot t + a_2 \cdot t^2, \tag{2}$$

where *t* is the time of the thermal influence on the sample, *s*; *a*₀, *a*₁, *a*₂ are the regression coefficient.

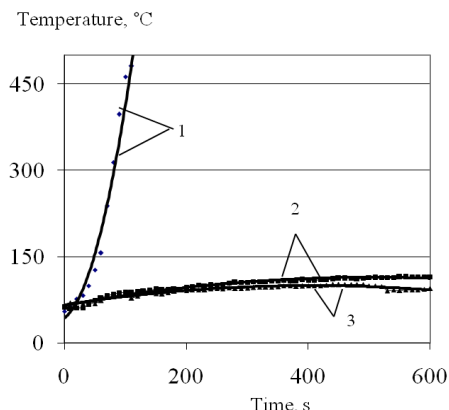


Fig. 5. Dynamics of increase in temperature of flue gases when testing the fire protected reed: 1 – raw; 2 – treated with roofing impregnating solution; 3 – treated with swelling coating

The experimental data were processed by the least square method. The variance was minimized

$$D = [v(t_i) - T_i]^2, \tag{3}$$

where *v*(*t*_{*i*}) are the theoretical values of temperature, derived from formula (2); *T*_{*i*} is the experimental value of temperature.

Table 1 shows a sample of experimental data for reed combustion.

Table 1

Sample of experimental data for reed combustion

Time, s	Temperature of flue gases, °C at combustion of protected sample			Time, s	Temperature of flue gases, °C at combustion of protected sample	
	Raw	Impregnation	Coating		Impregnation	Coating
0	54.6	61.3	64	130	90.2	85.4
10	66	59.8	68.6	140	91.4	86.7
20	76.7	60.9	72.6	150	92.5	85.1
30	82.3	60.6	68	160	93.1	84.8
40	99	66.6	70.3	180	94.2	89.5
50	127	74.2	68	200	96.3	90.9
60	156	78.5	73.3	240	99.6	93.5
70	237.5	82.2	78.7	300	105.3	94.8
80	314	84.4	80.6	380	107.6	97.9
90	397	86.7	82.3	450	111.5	100.8
100	462	86.4	82	510	112.8	98.6
110	481	86.3	77.4	560	114.1	91.5
120	502	87.5	81.2	600	115	93.8

After minimizing *D*, root mean square quadratic deviation σ was calculated from formula

$$\sigma = \sqrt{D / (n - n_0)}, \tag{4}$$

where n is the number of measurements; n_0 is the number of unknown parameters.

The results of processing the experimental data for wood samples combustion are shown in Table 2.

Table 2

Results of treatment of experimental data for reed samples combustion

Sample of fire protected reed	Values of parameter			
	a_0	a_1	a_2	σ
Raw	43.85	0.7468	0.0294	4.97
Roofing impregnating solution	62.33	0.2254	-0.0002	3.54
Swelling coating	65.64	0.1638	-0.0002	3.35

Discrepancy between the experimental and calculated values does not exceed 5 %, which is accepted to be reliable during engineering calculations in the region of modeling of fire resistance [17].

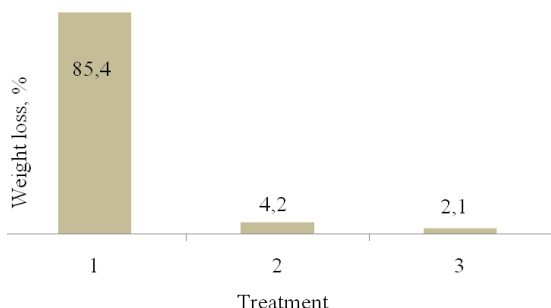


Fig. 6. Results of weight loss of samples Δm (%) of fire protected reed: 1 – raw; 2 – treated with a roofing impregnating solution; 3 – treated with a swelling coating

The study showed (Fig. 5, 6) that reed belongs to combustible materials, the impregnated reed sample withstood

the temperature influence and belongs to hard-to-fire combustible materials by the indicator of the weight loss. At the initial temperature of gaseous combustion products $T=76\text{ }^\circ\text{C}$, under the influence of the radiation plane on the protected sample (curve 2, Fig. 5), the temperature of gaseous combustion products was $T\leq 120\text{ s}$ and the weight loss did not exceed 4.2 % (Fig. 6), and for the sample, protected by coating, it amounted to 2.1 %.

During testing the reed samples, it was found that the raw sample ignited at second 52, the flame propagated throughout the sample over 100 s, while the sample that was protected by the roofing impregnating solution, specifically, a mixture of urea and phosphoric acids and natural polymer of 47.1 g/m^2 , ignited at second 595, the flame propagation around the surface occurred only in the first section, the maximum temperature of flue gases amounted to $114\text{ }^\circ\text{C}$ within the time that is 5 times longer, and flammability index decreased by 0.42. An even greater effect was achieved during the treatment of the reed sample with the swelling coating (Table 3). A decrease in the flame retardant in the composition by 2 times at the same consumption led to an increase in flammability index for roofing impregnating solution by 14 times, and for the swelling coating to 0.96, respectively. These results allow us to establish the ratio of flame retardants and polymers in these compositions and their required quantity.

Thus, it was established that the sample of reed at high temperatures is able to ignite and propagate the flame around the surface. Treatment of reed with the roofing impregnating solution to a certain extent prevents combustion and flame propagation, but at the end of the sixth minute, we observed the ignition of the material on the first section, which is not enough for protection and requires an increase in the amount of the flame-retardant agent. Ignition and flame propagation were not recorded for the reed, protected by the swelling coating presented above.

Table 3

Time of passing control points by the flame front

Fire protected reed sample	Temperature of flue gases, $^\circ\text{C}$		Ignition time, s	Time of passing sections of sample by flame front, s									Time of achievement of the maximum temperature of flue gases, s	Length of combustion of sample, mm	Flammability index
	T_1	T_{\max}		1	2	3	4	5	6	7	8	9			
Raw	61	323	52	2	8	7	10	6	8	7	6	7	101	294	177.5
Treated with a roofing impregnating solution in ratio of flame retardants to polymer of 2:1	62	114	595	-	-	-	-	-	-	-	-	-	596	22	0.42
Treated with a roofing impregnating solution in ratio of flame retardants to polymer of 1:1	62	173	501	44	36	18	22	57	-	-	-	-	598	158	14.7
Treated with a swelling coating with ratio of flame retardants to polymer of 2:1	64	101	-	-	-	-	-	-	-	-	-	-	600	0	0
Treated with a swelling coating with ratio of flame retardants to polymer of 1:1	63	119	594	-	-	-	-	-	-	-	-	-	599	18	0.96

6. Discussion of results of determining the effectiveness of fire protection of reed

Reed flammability, as indicated by the results of research (Fig. 5, 6), is determined by means of its ignition and rapid flame propagation around the surface and the weight loss of the sample. Instead, for the protected sample, due to the action of flame retardants, the processes of ignition and flame propagation slow down significantly. The mechanism of the protective agent is determined, first of all, by the decomposition of flame retardants under the influence of temperature with heat absorption and release of noncombustible gases, a change in the direction of decomposition of material towards formation of difficult-to-fire coke residue. In addition, there occur the oxidation inhibition processes in the gas and condensed phase and a heat protective coke layer is formed on the surface of wood. This is in line with the data, contained in papers [6, 7], the authors of which also attribute the effectiveness of thermal protection of the material to the effects of protective substances at adding flame retardants.

In contrast to the findings by authors of papers [13, 15], the obtained data on the influence of the protective agents on the process of heat transfer to the material and a change in heat-insulating properties make it possible to state the following:

- the main regulator of the process is not only the formation of the thermal protecting coke layer, but also decomposition of flame retardants with the release of noncombustible gases, including nitrogen, carbon dioxide, which interact with the flame with retardation of the oxidation process in the gas and condensed phase, which is noted in paper [1];

- the process of the protection of combustible material at the application of flame retardant coating is significantly influenced by reactions in the pre-flame area towards the formation of soot-like products on the surface of the natural combustible material.

Such conclusions can be considered relevant from the practical point of view, because they allow us to approach reasonably determining the required formulation of a flame-retardant agent. Application of a mixture of salts of phosphoric acids and the polymer included in the roofing impregnating solution is able to protect reed from thermal effects, but requires an increase in the amount of such agent. The treatment with the swelling coating more effectively counteracts high temperature, which should be paid special attention to, while developing the formulation of coatings for reed. From the theoretical point of view, they can make it possible to argue about determining the mechanism of the fire protection process, which are certain merits of this research. The results from determining the process of ignition and flame propagation of the protected reed (Table 1) indicate the ambiguous impact of the nature of the protective agent on a change in temperature. In particular, this implies the availability of the data, sufficient for high-quality conduction of the temperature inhibition process and detection of the moment of the beginning of ignition based on it. Such detection will allow studying the conversion of the surface and the structure of reed under the influence of fire protection towards the coke formation and flame inhibition, and

determining the variables that significantly influence the beginning of this process.

This work is continuation of research reported in [1–5], which describe in detail the mechanism of fire protection, of organic natural materials, foam coke formation, displacement and implementation of thermal insulation of high temperature.

The presented studies are limited to fire protection of reed, require the development of the field methods of testing and research and description of the behavior of material.

Future research can be directed to the theoretical and experimental studying of the processes of combustion of thermal insulating materials, establishment of the relationship between the components and properties of the protective agents and their impact on the processes of thermal stability of building structures.

7. Conclusions

1. An analytical study into determining the fire protective effectiveness of reed was carried out, the equipment and the method for determining the flammability index of the reed samples during the thermal influence were developed. The essence of this method lies in the effect similar to heat flow of electric radiation plate that imitates a low-calorie ignition source and ignition of the pattern with a burner. In this case, thermal coefficient of the plant is determined, the maximum temperature of combustion products and the time of its achievement, ignition time, the time of passing the surface areas by the flame front, and the length of the burnt part of the sample are measured.

2. Specific features of the ignition processes and propagation of the flame of reed, treated by the roofing impregnating solution include several aspects. They are decomposition of fire retardants under the action of temperature with heat absorption and the release of noncombustible gases (nitrogen, carbon dioxide), which causes inhibition of oxidation in the gas and condensed phase, as well as a change in the direction of wood decomposition towards formation of the noncombustible gases and difficult-to-fire coke residue, a decrease in the combustion of material and accordingly an increase in flammability index.

Instead, the coating under the influence of high temperature forms a significant swelling coefficient, promotes the formation of the heat isolated coke layer, which prevents burning out and passage of high temperature to the material, which is proved by the absence of the ignition process of the protected reed. This indicates the possibility of directed regulation of processes of high temperature transfer to the organic material through the use of special swelling coatings for products made of reed.

A decrease in the flame retardant in the protective composition by two times at the same consumption, led to increased flammability index for roofing impregnating solution by 14 times, and for the swelling coating by 0.96, respectively, which allows us to establish the ratio of fire retardants and polymers in these compositions and, accordingly, their required quantity.

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