

Наведено результати досліджень вогнезахисних властивостей засобів захисту для деревини та визначено оптимальні умови проведення процесу піролізу. Методом хроматографії здійснено якісний і кількісний аналіз одержаних летких продуктів піролізу та встановлено зниження горючих газів. Встановлено, що вогнезахист переводить деревину у матеріал важкогогорючий, який не поширює полум'я поверхнею, з помірною дымоутворювальною здатністю

Ключові слова: захисні засоби, вогнестійкість, леткі продукти, втрата маси, оброблення поверхні, ефективність захисту

Приведены результаты исследований огнезащитных свойств средств защиты древесины и определены оптимальные условия проведения процесса пиролиза. Методом хроматографии выполнены качественный и количественный анализ полученных летучих продуктов пиролиза и установлено снижение горючих газов. Установлено, что огнезащита переводит древесину в материал трудногорючий, не распространяющий пламени по поверхности, с умеренной дымообразующей способностью

Ключевые слова: защитные вещества, огнестойкость, летучие продукты, потеря массы, обработка поверхности, эффективность защиты

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ESTABLISHMENT OF THE MECHANISM AND FIREPROOF EFFICIENCY OF WOOD TREATED WITH AN IMPREGNATING SOLUTION AND COATINGS

Y. Tsapko

Doctor of Technical Sciences*

V. D. Glukhovskiy Scientific Research Institute for Binders and Materials

Kyiv National University of Construction and Architecture

Povitroflotskyi ave., 31, Kyiv, Ukraine, 03680

E-mail: juriyts@ukr.net

A. Tsapko

Posgraduate student*

E-mail: alekseystsapko@gmail.com

*National University of Life and Environmental Sciences of Ukraine

Heroiv Oborony str., 15, Kyiv, Ukraine, 03041

1. Introduction

Given the fact that cellulose-containing materials and products made of them (lumber, plywood, paper and fibrous materials and others) are the main conductors of the propagation of flame, they need appropriate protection. That is why fire safety puts forward high requirements both to the effectiveness of fire-protective means and to the quality of fire-protected materials.

Main requirements to the fire protection of cellulose-containing materials imply providing them with the capability to resist the action of fire and not to spread flame over surface. A change in the arrangement of material at fire protection is directed toward the formation of noncombustible gases and coke residue that is difficult to combust, as well as the inhibition of oxidation in the gas and condensed phase.

The fires that have occurred in recent years led to the death of people from hazardous factors, such as smoke generation and toxicity of combustion products. This prompted testing on smoke generation but only in relation to textile materials used inside the premises.

The research conducted established [1] that a non-treated wood sample is able to ignite and spread the flame over surface after ignition, which leads to significant smoke generation. Application of coatings makes it possible to slow the heating of the material and to maintain their functions during fire over a specified period of time. But relating wood to the materials that are difficult to combust does not always

affect other indicators, in particular smoke generation and the toxicity of products of the combustion of materials [2, 3].

Therefore, examining the mechanism of fire protection, and the impacts of components that make up their composition on this process is an unresolved constituent in the provision of fire resistance to building structures and it necessitates multifactor determining of flammable properties. This predetermined the need to conduct research in this direction.

2. Literature review and problem statement

In recent years, there have been studies into fire protection that focused on the synthesis of coatings using inorganic substances modified by polymer complexes, capable of forming a coke layer on the surface [4, 5]. The indicated articles presented component compositions, their efficiency and thermal characteristics of the means, however, the specified means are characterized by weak atmosphere resistance. The most common are the enamel and glass crystalline coatings [6], they, however, cannot provide reliable protection of structures at long-lasting temperatures because they fail to form a necessary coke layer, instead they are heated and destroyed.

A promising area of research is to improve the fire resistance of building structures using the fire retardant agents. In most cases, they are modified with polymer complexes and flame retardants, but such coatings relate to the mate-

rials that are easily washed out and are suitable for interior premises [7, 8].

Modern methods of fire protection include the use of coatings that swell, which represent complex systems of organic and inorganic components. Materials are characterized by high intumescent ability, but the studies [9] did not report the mechanism of forming the coke, phase and temperature transitions of the coating to the coked cellular material [9].

In recent years, among the studies in the proposed area of research are the papers that report creating fire protective means, which in the process of heating form a coke insulating layer on the surface of wood [10]. The most common among them are the impregnating means for wood, however, they are easily washed out, corrode with metals and cannot provide reliable protection of structures.

Authors from European countries report very high efficiency of fire protection of cellulose-containing materials. The treated materials are transferred to the group of those difficult to combust, with low smoke-forming ability, which do not spread the flame over surface; however, the process is not described in detail [11]. Influence of inorganic fillers on the fireproof water-based coating demonstrated its effectiveness, however, the mechanism of coating swelling is not specified [2].

Therefore, the establishment of the mechanism of a fireproof coating, effects of components that make up their composition on this process and comprehensive determining the effectiveness of fire protection is the unsolved constituent in the provision of fire resistance of building structures. This necessitated research in this area.

3. Research goal and objectives

The goal of present work is to study the mechanism of fireproof efficiency of wood treated with impregnating means and coatings.

To accomplish the goal, the following tasks have been set:

- to establish a mechanism for the protection of wood with impregnating solutions and coating under thermal action;
- to explore fireproof efficiency of wood treated with impregnating solutions and coatings using the multifactor determining of flammable properties.

4. Materials and methods for examining a front of phase transitions during swelling of a fireproof coating

4.1. Examined materials that were used in the experiment

In order to establish flammability, spread of flame and smoke generation of wood, we used wood samples, non-treated and treated (Fig. 1) with:

- impregnating solutions based on ammonium phosphate and sulfate;
- a fireproof swelling coating;
- a non-norganic coating.

For the pyrolysis of a fireproof material, we used samples of wood of average size 10×10 mm and height 10 mm, treated with fireproof coatings given above.

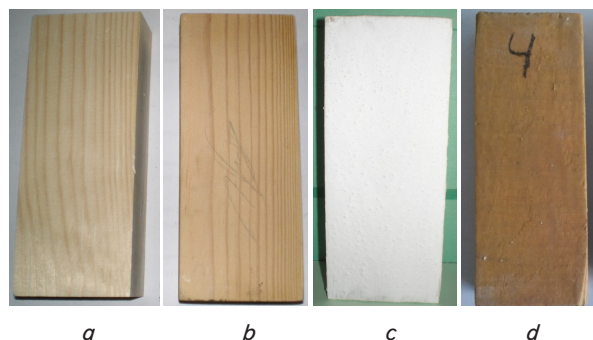


Fig. 1. Model samples of fireproof wood: *a* – non-treated, *b* – treated with a flame-retardant impregnation; *c* – treated with a fireproof swelling coating; *d* – treated with a flame-retardant coating on non-organic base (patent of Ukraine for useful model No. 95440)

4.2. Procedure for determining the indicators of properties of the samples of the flame retardant coating of wood

Research into determining a group of flammability of wood, the Flame Spread Index and smoke generation, treated with impregnating solutions and the coating was carried out in accordance with [8].

The essence of a method for testing experimental determining of the group of wood that is difficult to combust is the impact of the flame of a burner or a radiation panel with the assigned parameters on the sample placed in the installation.

In the course of experimental research into determining a group of flammability, we register a maximal gain in the temperature of the gaseous products of combustion (Δt) and the loss of mass of the sample (Δm).

Based on the results of testing, the materials are categorized as follows:

- difficult to combust – $\Delta t < 60$ °C and $\Delta m < 60$ %;
- combustible – $\Delta t \geq 60$ °C or $\Delta m \geq 60$ %.

When determining the Flame Spread Index, we register a distance of thermal destruction of the sample and the temperature of smoke gases.

Based on the values of Flame Spread Index, the materials are categorized as follows:

- those that do not spread flame on the surface – spread index 0;
- those that spread slowly – spread index 0÷20;
- those that spread quickly – spread index >20.

When determining a coefficient of smoke generation, we determine optical conductivity of the formed smoke gases.

Depending on the obtained coefficient of smoke generation, the materials are categorized as follows:

- with a reduced smoke-generating capacity – larger than 50 m²/kg;
- with an average smoke-generating capacity – larger than 50 m²/kg to 500 m²/kg inclusive;
- with a high smoke-generating capacity – smoke generation coefficient exceeds 500 m²/kg.

4.2.1. Development of the method and equipment for determining kinetics of the swelling of a fireproof coating and research results

One of the methods that provides for the opportunity to explore the formation of volatile products of fireproof wood is a gas chromatographic analysis. A composition of products of thermal degradation depends on many conditions: a kind

of plant material, moisture content, temperature, dynamics of air intake, etc. The highest content of combustible components is demonstrated by gaseous products formed as a result of the pyrolysis (thermal decomposition without air supply).

To obtain gaseous products of thermal degradation, we designed and manufactured specialized equipment based on a tubular electric furnace with a thermoregulator. The installation provides the possibility to maintain the temperature to 800 °C with an accuracy of ±10 °C. Thermal destruction of the samples of materials was performed in a cylindrical steel reactor with an inner diameter of 40 mm, whose working volume is 0.5 dm³ (Fig. 2). A sample of the examined material is placed in housing 4 between pistons 3 and 5 supported by spring 6. Reactor from both sides is shut by lids 2 and 7, one of which has plunger 8 for the release of gases, while the second has an in-built thermocouple 1 whose junction is located inside the reactor.

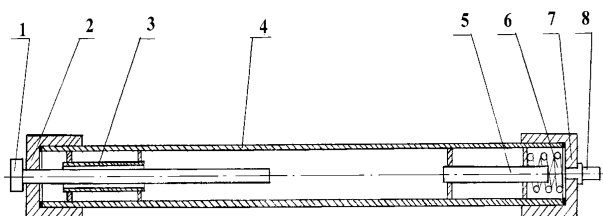


Fig. 2. Schematic of reactor for thermal destruction of the samples of materials: 1 – thermocouple; 2, 7 – lids; 3, 5 – pistons; 4 – housing; 6 – spring; 8 – plunger

Schematic of the installation for separating the gaseous products of thermal destruction of materials is shown in Fig. 3.

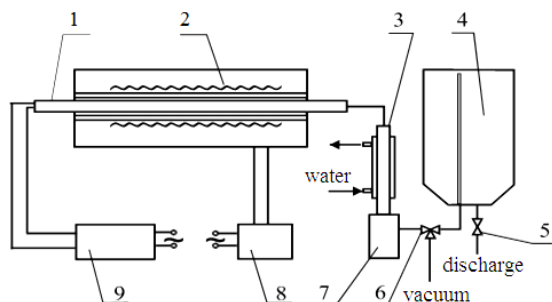


Fig. 3. Schematic of equipment for separating the gaseous products of thermal destruction of materials: 1 – reactor; 2 – tubular furnace; 3 – heat exchanger; 4 – gas collector; 5, 6 – taps; 7 – vessel to collect condensate; 8 – thermostat; 9 – millivoltmeter

In tubular furnace 2 there is reactor 1 connected through a heat exchanger 3 to vessel 7, to which, through three-way tap 6, glass gas collector 4 is attached, filled with a saturated sodium chloride solution. The use of such a solution is due to the low solubility of gases in it compared to their solubility in water. Given this, the composition of the received gas mixture when it enters gas collector 4 undergoes minimal changes. It should be noted that the use of elastic gas collectors made of polymeric materials is not feasible for this purpose because of their permeability for hydrogen and other gases.

To conduct thermal destruction of the examined material and separate the gas mixtures that form, the following operations are carried out:

- place a sample of the examined material in reactor 1;
- vacuum the system to a residual pressure not exceeding 1kPa, controlling the pressure in the system by the readings of vacuum gauge;
- feed water to the casing of heat exchanger 3;
- heat furnace 2 to the set temperature;
- symmetrically place reactor in furnace 2 and connect the contacts of the thermocouple located inside it to millivoltmeter 9;
- after leveling the pressure in the reactor to the atmospheric, turn tap 6 in position "reactor – collector" and open tap 5.

Gaseous products enter collector 4, and a sodium chloride solution through tap 5 flows into a specialized vessel. Products of destruction, which are liquid under normal climatic conditions, are condensed in heat exchanger 3 and collected in vessel 7.

Upon completion of collecting the obtained products, collector 4 via tap 5 is filled with a saturated solution of sodium chloride, which pushes the gas mixture to a sample collector.

Qualitative and quantitative composition of these mixtures were determined by a gas-chromatographic method using the gas chromatograph LHM-7A, made by research enterprise "Chromatograph", Moscow.

Results of a gas-chromatographic analysis of the received combustible gas mixtures are given in Table 1.

Table 1

Qualitative and quantitative composition of the gaseous products of the thermal destruction of wood

Component	Content of components in the volatile products of destruction of pine wood, % by volume			
	non-treated	treated with impregnating mixture	treated with swelling coating	treated with non-organic coating
CO	39.08	18.37	12.80	16.35
CO ₂	51.93	67.36	76.20	82.48
CH ₄	6.05	2.74	0.54	0.46
C ₂ H ₆ +C ₂ H ₄	0.45	0.37	not detected	0.15
C ₃ H ₈	0.19	not detected	not detected	not detected
C ₃ H ₆	0.32	not detected	not detected	not detected
H ₂	0.73	0.63	not detected	0.56
O ₂	0.26	not detected	not detected	not detected
N ₂	0.99	10.53	10.46	not detected

Table 1 shows that after the pyrolysis of non-treated wood and the wood treated with fire retardant means, the mixtures of products of destruction significantly differ in the content of nitrogen, carbon dioxide and the amount of combustible gases. Thus, in the wood treated with a mixture of phosphates and sulfates of ammonium, the amount of nitrogen increased by more than 10 times. The wood treated with a swelling coating revealed an increase in carbon dioxide by 1.5 times and in nitrogen by 10 times, and a reduction in combustible gases. The wood treated with a non-organic coating demonstrated an increase in the amount of carbon

dioxide by 1.6 times and a decrease in the combustible gases by more than two times.

However, chromatographic research we conducted makes it difficult to identify one or another group of fireproof efficiency, which matches the impregnating means and coatings, which is why we conducted studies into fireproof efficiency that would conform to the multifactor determining of fire dangerous properties.

5. Experimental studies into fireproof efficiency of wood treated with coating and their results

In order to establish fireproof efficiency when examining an impregnating solution and coatings, we conducted studies to determine a group of flammability of wood treated with the specified compositions. Results of research to determine the weight loss of the samples ($\Delta m, \%$) and the gain of maximum temperature of the gaseous products of combustion ($\Delta t, ^\circ\text{C}$) of the samples of fireproof wood, conducted under laboratory conditions, are shown in Fig. 4, 5.

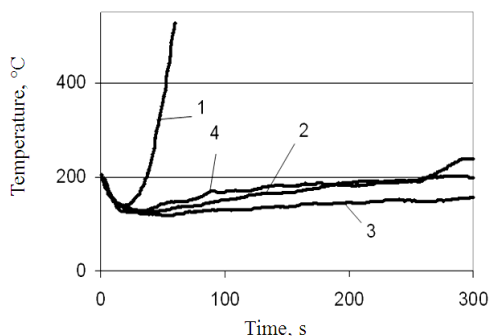


Fig. 4. Dynamics of an increase in the temperature of smoke gases in the tests of fireproof wood: 1 – non-treated; 2 – impregnation; 3 – swelling coating; 4 – coating on non-organic base

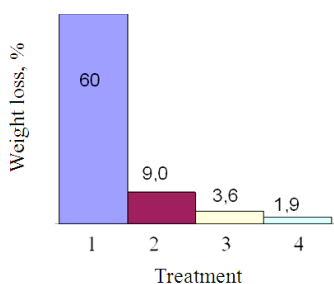


Fig. 5. Results of weight loss of the samples $\Delta m, \%$ of fireproof wood: 1 – non-treated; 2 – impregnation; 3 – coating on non-organic base; 4 – swelling coating

Studies have shown (Fig. 1, 2) that wood relates to combustible materials, the impregnated wood survived the temperature impact and belongs to the materials difficult to combust by the indicator of weight loss. At initial temperature of the gaseous products of combustion $T=200\text{ }^\circ\text{C}$, under the action of flame of the burner on the sample protected by a non-organic coating (Curve 2), temperature of the gaseous products of combustion was $T\leq 260\text{ }^\circ\text{C}$; while weight loss did not exceed 9% (Fig. 2).

An even higher efficiency was demonstrated by the samples treated with the non-organic coating Curve 4 at weight loss of 3.6% and with the swelling coating (sample 3) (Fig. 4, 5).

The magnitude of dimensionless Flame Spread Index was computed by coefficient I:

$$I = \sqrt{\frac{q \cdot Q}{W} \cdot \frac{T_{\max} - T_0}{T_1 - T_0} \cdot \frac{\tau_{\max} - \tau_0}{\tau_0} \cdot \left[1 + \frac{60 \cdot l_d}{l} \cdot \sum_{i=1}^n \frac{1}{\tau_i} \right]}, \quad (1)$$

where q is the specific heat of combustion of the gas propane (23630), $\text{kJ}\cdot\text{l}^{-1}$; Q is the flow rate of the gas burner (0.003), $\text{l}\cdot\text{s}^{-1}$; W is the power of electric radiation panel, 0.75 kW; T_0 is the ambient temperature $^\circ\text{C}$; T_1 is the initial temperature of smoke gases, $^\circ\text{C}$; T_{\max} is the maximum temperature of smoke gases, $^\circ\text{C}$; τ_0 is the time of ignition of the sample, s; τ_{\max} is the time of reaching the maximum temperature of smoke gases, s; τ_i is the time for the flame front to pass control sections, s; l is the length of the sample, mm; l_d is the length of damage to the sample, mm.

We initially tested the non-treated samples of wood. When exposed to the action of flame for a short period of time, the non-treated samples started burning and intense spread of flame over the surface, Flame Spread Index was 56.7. Next, we tested the samples treated with the impregnating substance and the coating. During tests of the samples of fireproof wood, it was found that the sample ignited at second 113, spreading of flame over the surface occurred only in the first and second sections, maximum temperature of smoke gases amounted to $134\text{ }^\circ\text{C}$ over the time longer than 2.0 times, while the Flame Spread Index decreased by 8 times (Table 2).

Table 2 shows that the Flame Spread Index of the untreated sample is 56.7, while in the treated samples this value is reduced by 8 times, and when applying the swelling coating, flame spread did not occur.

Coefficient of smoke generation (D_m) in m^2/kg is determined by formula:

$$D_m = \frac{V}{L \cdot m} \ln \frac{T_0}{T_{\min}}, \quad (2)$$

where V is the volume of the measuring chamber, $V=(0,664\pm 0,004)\text{ m}^3$; L is the path that a light beam passes in smoke, $L=(0,800\pm 0,002)\text{ m}$; m is the mass of the sample, kg; T_0, T_{\min} are the values of the initial and resulting light transmittance, %, respectively.

For each of the test modes, we determined a coefficient of smoke generation for the non-treated and fireproof wood: impregnation, by a swelling coating and on non-organic base (Table 3).

The mean value of coefficient of smoke generation for wood under mode of smoldering is $756.11\text{ m}^2/\text{kg}$ and it relates wood to the materials with high smoke-generating capacity. Fireproof treatment reduces the smoke generation of wood by two times; this material is categorized as the one with a mild smoke-generating capacity.

Thus, we developed a comprehensive approach to the evaluation of fire-resistant properties. Results of determining the fire-resistant indicators of fireproof wood demonstrated that the goods belong to the materials that are difficult to combust, which spread the flame slowly at low smoke-generating capacity.

Table 2

Time of passing control points by the flame front

Wood sample	Temperaturte of smoke gases, °C		Time to travel over sample's sections by the flame front, s										Time to reaching T_{max} of smoke gases, s	Length of damage to sample, mm	Flame Spread Index
	T_1	T_{max}	0	1	2	3	4	5	6	7	8	9			
Non-treated	88	367	21	25	5	22	48	47	32	45	99	32	121	298	56.7
Impregnated	90	134	113	87	-	-	-	-	-	-	-	-	256	19	7.01
Protected with non-organic coating	91	106	180	-	-	-	-	-	-	-	-	-	288	15	4.2
Protected with swelling coating	91	98	-	-	-	-	-	-	-	-	-	-	394	0	0

Table 3

Results of testing a coefficient of smoke generation of wood

Test mode and density of heat flow	Number of sample for testing	Sample weight (m), $kg \times 10^{-3}$	Light transmittance, %		Coefficient of smoke generation for each sample (D_m), m^2/kg
			(T_0) initial	(T_{min}) resulting	
Flame burning (35 kW/m ²)	1	0.75	100	51	674.45
	2	0.79	100	80	224.20
	3	0.83	100	87	140.26
	4	0.84	100	86	151.81
Smoldering (35 kW/m ²)	1	0.76	100	47	756.11
	2	0.80	100	76	275.47
	3	0.85	100	74	302.08
	4	0.86	100	75	288.65

6. Discussion of results of examining the process of advancing of the phase transformation front

When establishing the mechanism of fire protection of wood under the thermal action of high-temperature flame, as evidenced by research results (Table 1), there is a natural process of reducing flammable volatile products of the pyrolysis. Under the action of temperature, volatile decomposition products are characterized by the formation of inert components. This is due to the mechanism of work of the coating with the formation of a layer of coked cellular material, which slows down the processes of heat transfer. It should be noted that the protection by coatings, for example swelling, leads to the formation of coke layer on the surface of wood and reduces flammable components. Obviously, such a mechanism of influence is the factor regulating the degree of protection and effectiveness of the thermal insulation of a material. However, at the same time, this mechanism adversely affects smoke-generating capacity of the coating. It agrees with data known from articles [7, 10], whose also attributed a change in the process of smoke generation when treated with swelling coatings. In contrast to research results [8, 11], obtained data on the influence of a coating on the process of the formation of a foamed layer of coke and a change in the thermal insulation properties allow us to argue that:

– the main regulator of the process of thermal insulation is not only the formation of a layer of coked cellular material but thermal resistance of a fireproof coating;

– a significant impact on the process of transition of a combustible material at application of a fireproof coating is exerted by a transition to the group of materials that are difficult to ignite, which spread the flame over surface slowly at low smoke-generating capacity.

Results of determining a transition of wood under the action of impregnation and a flame retardant coating and the formation of thermal insulation layer of coke (Fig. 4, 5) point to the ambiguous impact of protection on the change in fire efficiency of a coating. This uncertainty cannot be solved within the framework of the present study, because to do so would require conducting additional experiments in order to obtain more reliable data. In particular, this implies the existence of data that are sufficient for conducting the process of fire protection properly and for the detection of moment when thermal resistance falls. Detecting it will make it possible to explore the transformation of the coating surface, which shifts in the direction of elevated temperature with the formation of coke, and to define those variables that significantly influence the start of transformation of this process.

Present work is continuation of the studies given in [1–3] that report in detail the mechanism of fire protection

of wood, the formation of coked cellular material, shift and thermal insulation of high temperature.

7. Conclusions

Result of the conducted studies is the establishment of wood fireproof efficiency by the impregnation and by coatings of the swelling type and non-organic origin, in particular:

– we conducted research into mechanism of wood fire protection with impregnating means and coatings during formation of a thermal insulating layer of coke, we defined volatile combustion products and received a change in the components during fire protection that allow us to obtain the volatile combustion products of a fireproof coating when exposed to temperature. Based on data received, it was found that at thermal decomposition of fireproof wood there occurs a reduction of combustible gases by over 50 % and an increase in the inert gases by more than 8 times;

– tests on the model samples of fireproof wood demonstrated that the impregnation is characterized by a decomposition of fire retardants under the influence of temperature with the heat absorption and release of noncombustible gases, inhibition of oxidation in the gas and condensed phase and the formation of thermal protective layer of coke at the surface of wood. Instead, the coating under the influence of high temperature forms a significant coefficient of swelling, contributes to the formation of thermal insulating layer of coke, which prevents wood from burning as well as the passage of high temperature to the material. In this case, smoke generation and material combustion decrease. In general, wood fireproof efficiency, obtained by multifactor determining of flammable properties, demonstrated that the goods refer to the materials that are difficult to combust, which spread the flames slowly and at low smoke-generating capacity.

Research in the future can be directed towards studying the processes of establishing a relationship between the components and properties of coatings and their impact on the processes of thermal resistance of building structures.

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