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ESTABLISHING THE THERMAL CHANGES IN THE FOAM LAYER OF A BIOCOMPOSITE COATING UPON THE ADDITION OF POTASSIUM NITRATE

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The object of research is the fire-retardant properties of biocomposite coating for wood when potassium nitrate is added during thermal action. The problem of fire resistance of biocomposites, which is aimed at increasing the efficiency of fire protection of wooden structures, consists in the formation of fire-resistant coatings by adding inorganic compounds. Such compounds are characterized by thermal stability, reduced smoke-forming ability and toxicity of combustion products and are mostly used as synergistic additives. Therefore, the work proves that during thermal action on wood treated with biocomposite coating, intensive formation of a foam coke layer began after 62 s to a height of 16 mm, which thermally insulated the wood. Instead, after the radiation panel was exposed to a sample of wood treated with a biocomposite coating with the addition of potassium nitrate, the formation of a foam coke layer began after 48 s of thermal exposure, the height of which was 15 mm, and the temperature of the flue gases did not exceed 100°C. At the same time, chemical reactions of potassium nitrate begin in the coating with the release of carbon monoxide, with the subsequent destruction of ammonium polyphosphate, which interacts with the biopolymer. The latter, in turn, affects the dehydration of pentaerythritol with the formation of coke, and the simultaneous decomposition of melamine causes the coke to foam. The composition of the gaseous products of thermal destruction of the biocomposite coating showed that when potassium nitrate was added, the amount of combustible gases decreased by more than 19%, and the amount of nitrogen increased by more than 10%. The practical significance is that the results obtained were taken into account when developing a fire-retardant coating for construction. Thus, there are grounds to argue about the possibility of effective protection of wood with a coating containing biopolymers

***Keywords:** biocomposite coating, wood processing, coating swelling, foam coke layer, combustion products*

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1. Introduction

Wood and products based on it are widely used for the manufacture of building structures and structures, but they are combustible materials and are prone to the spread of flame. Since fire safety is of great importance in the operation of structures, the use of wood materials increases the risk of ignition and its spread, which prompts the implementation of fire protection of building structures. The simplest preparations used for fire protection of wood and plant fibers contain flame retardants based on inorganic salts. These include mixtures such as ammonium sulfate and diammonium phosphate, sodium orthoborate and boric acid, and a mixture of sodium carbonate with boric acid. However, they are capable of leaching from the surface of the building structure.

Fire-resistant coatings based on inorganic binders, where sodium liquid glass, Portland cement, alumina cement, aluminosilicate binders are used as a binder and contain bound water. Which evaporates during heating and blocks the transfer of heat to the surface being protected. However, such coatings are short-lived and ineffective, since they do not provide sufficient adhesive strength and have a large temperature coefficient of linear expansion. For fire protection of building structures, special organic-based coatings have been widely used, namely, additives to polyolefin, which contain halogens and are quite effective. However, they can form efflorescence, and when exposed to high temperatures, they emit toxic combustion products.

The effectiveness of fire protection of building structures made of wood consists in creating heat-insulating screens on the surface that can withstand high temperatures and direct fire. Their presence allows to slow down the heating of the material and maintain the structure's functions during a fire for a given period of time. The disadvantage of intumescent coatings is that the temperature of formation of the thermal insulation layer of foam coke is over 300°C, which makes it impossible to use them to protect wood, the intensive decomposition of which begins at a temperature of 215°C. And the question arises of reducing the temperature of formation of the protective layer for these materials.

Therefore, the formation of fire-resistant coatings by adding inorganic compounds (hydroxides, nitrates, carbonates and sulfates), which are characterized by thermal stability and reduced smoke-forming ability and toxicity of volatile combustion products. They are mostly used as synergistic additives that can increase the efficiency of the coating and act by cooling the combustion source and diluting combustible gases with inert diluents. And are also characterized by the formation of non-combustible carbon layers that inhibit heat and mass transfer.

Thus, studies aimed at establishing the efficiency of the formation of a fire-resistant coating and changes in its structure during operation, which are necessary to determine the parameters of fire resistance, are relevant.

2. Literature review and problem statement

The paper [1] highlighted the fire resistance, physical, chemical, mechanical and thermal properties of intumescent fire-retardant coatings (IFRC). Five IFRC formulations were mixed with vermiculite and perlite to produce prototype fireproof wooden doors in this research project. In addition, the prototype fireproof door with the best fire resistance performance was selected and compared with the commercial prototype in a fire resistance test. The inventive prototype fireproof door (P2) with a low density of 636.45 kg/m^3 demonstrated outstanding fire resistance performance, resulting in a temperature reduction of up to 54.9°C compared to the commercial prototype. Importantly, the new prototype fireproof wooden door with the addition of a specially designed intumescent coating proved to be effective in preventing fires and maintaining its integrity, maintaining fire resistance for 2 hours. However, the optimal solutions for their use have not been established.

In [2], the fire extinguishing mechanism of intumescent fire-retardant coatings, the fire protection system, the effect of functional fillers on the fire resistance of intumescent fire-retardant coatings, and the application of intumescent fire-retardant coatings in tunnels are summarized. In addition, the synergistic effect of the combined use of different functional fillers is presented. Finally, some key issues regarding the use of intumescent fire-retardant coatings in the tunnel environment are raised, as well as prospects and opportunities. However, their impact on the environment is not established. In [3], it is proven that coatings require a thin layer on the surface for fire protection. Which in turn expands many times its initial thickness under the influence of fire, forming an insulating coal that acts as a barrier between the fire and the surface. But some research papers discuss the types of intumescent coatings, application methods, manufacturing processes, cost-effectiveness, adhesion properties, toxicity and different uses, still need to conduct a comprehensive study covering all these topics. This review article explores the different types of intumescent coatings, their formulation and production methods, application processes and use on different surfaces. It also discusses the key materials of intumescent coatings and their interaction during fire. However, little attention is paid to the issue of char formation temperature and toxicity.

In [4], it is said that three types of shell-shaped biofillers, including eggshell (CES), conch shell (CHS) and clam shell (CMS), were obtained by purification. After ultrasonic treatment and grinding of biowaste, they were applied to the intumescent fire-retardant coatings. The results show that the three types of biofillers have excellent synergistic effects in improving the fire resistance and char formation properties of intumescent fireproof coatings, while the shell has the best synergistic effect among the biofillers. In particular, the IFRC-CMS coating containing 3 wt.% of the decomposition exhibits the best fire protection performance and the lowest smoke and heat generation, providing an equilibrium backside temperature of 134.6°C at 900 s, a flame spread coefficient of 14.4, and a smoke density value of 22.8%. The synergistic effect of biofillers in intumescent coatings depends on the polymorphs of $CaCO_3$ in the biofillers, and aragonite $CaCO_3$ exhibits higher synergistic effect compared to calcite $CaCO_3$ and a mixture of aragonite and calcite $CaCO_3$. CMS, consisting of aragonite, exhibits the best synergistic effect, CHS, consisting of aragonite and calcite, ranks second, and CES, consisting of calcite, has the weakest synergistic effect. However, it has not been established how they affected the flame in the gas-condensate phase.

In the study [5], it is noted that the study presents a systematic method for the synthesis of a transparent intumescent flame retardant coating based on bisphenol-A epoxy resin. Compared with transparent epoxy resin-based flame retardant coatings reported in the literature, the proposed coating exhibits improved performance with excellent flame retardancy. These improvements are achieved by removing urea and adjusting the ratio of phosphoric acid, which leads to simplified synthesis procedures and a significant increase in transparency. It is noteworthy that when the ratio of epoxy resin, diethanolamine, and phosphoric acid is optimized to 1:2:4, a 1.2 mm thick coating achieves optimal flame retardancy. In a large panel simulation method for 45 minutes, the backside temperature stabilized at approximately 148°C. However, the impact of flame retardants on the ecosystem was not reported. In the study [6], thermoplastic polyurethane (TPU) was used as the matrix; antimony trioxide (primary flame retardant) and montmorillonite organoclay (secondary flame retardant) as well as benzoflex (plasticizer) were used as fillers to study their synergistic effect. Nanocomposites of different compositions (from TPU-1 to TPU-6) were obtained by melt mixing and compressed to form sheets of desired sizes using a hydraulic compression molding press. The samples were characterized by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Tensile testing was performed using a universal testing machine (UTM), which showed that the Young's modulus improved from 147.348 MPa for the pure sample (TPU-1) to 244.568 MPa for TPU-6. UL-94 testing was performed to observe the fire resistance. The sample of interest (TPU-6) received a V-0 classification. All these results confirmed the synergistic effect of primary and secondary flame retardants, however, nothing was said about the operating conditions of the coating.

In the study [7], to develop an organic/inorganic synergistic flame retardant and reduce the dosage and cost of flame retardants, organic/inorganic synergistic flame retardants, hexakis(4-boronic acid-phenoxy)-cyclophosphazene (CP-6B) and magnesium hydroxide (MH), were selected. The flame retardant properties of CP-6B/MH in epoxy resin (EP) were discussed. EP/CP-6B/MH had better flame retardancy and heat resistance compared with EP/CP-6B and EP/MH. The limiting oxygen index of EP/3.0%CP-6B/0.5%MH was achieved at 31.9%, and the vertical combustion rating of V-0 was also achieved. Compared with EP, the cone calorimetry data of EP/CP-6B/MH decreased. CP-6B/MH suppressed combustion and had little damage to mechanical properties. In addition, the mechanism of fire-retardant action was studied using scanning electron microscopy, energy-dispersive X-ray spectroscopy, X-ray diffraction, Fourier transform infrared spectroscopy, and pyrolysis gas chromatography with mass spectrometry. CP-6B/MH demonstrated good synergistic effects. But it is not said how their properties change during operation.

In the study [8], halogen-free flame-retardant expanded polystyrene (PS) foams were obtained by supercritical carbon dioxide (SC-CO_2) foaming. The flame retardants included expandable graphite (EG) and melamine phosphate (MP), and their effects on the foaming, decomposition behavior, flame resistance and mechanical properties of PS foams were investigated. It was shown that the flame retardants can generate inert gases and catalyze the carbon formation from PS, and the formed thick carbon layer with a noticeable barrier property can significantly reduce the heat release of PS foams. The addition of triphenyl phosphate (TPP) or hexaphenoxycyclotriphosphazene (HPCTP), which act as a flame-retardant plasticizer, can obviously improve the foaming and flame resistance of the foams. TPP or HPCTP can generate active phosphorus compounds and phenoxy radicals to enhance the gas-phase flame retardant effect. Numerical evaluation of the synergistic effect of TPP and HPCTP on further improving the fire resistance of PS foam was carried out using microcalorimetric (MCC) testing. Further X-ray photoelectron spectroscopy (XPS) study of the carbon residues of PS foam demonstrates the formation of P–O–C and other stable structures. However, whether their stability under severe fire exposure plays a key role. In [9], in order to improve the fire resistance of high impact polystyrene (HIPS), HIPS compounds were synthesized with the addition of a swelling flame retardant (IFR: APP and PER mass ratio was 3:1) and diatoms to the HIPS matrix by melt blending. It was found that the IFR/diatom system PHRR values for HIPS-2 compound are about 460.58 kW/m^2 compared to 937.22 kW/m^2 of pure HIPS. And the THR value of the HIPS-2 compound is about 32.9 MJ/m^2 compared to 62.7 MJ/m^2 of pure HIPS. This indicates that the addition of the IFR/diatom system can reduce the PHRR and THR values, demonstrating the synergistic effect between IFR and diatoms in reducing heat release. The thermogravimetry results showed that the IFR/diatom flame retardant system functioned in the gas phase to suppress the flame. SEM images showed that the formed char residue was more compact and continuous, indicating that the IFR/diatom flame retardant system exhibits barrier and catalytic effects. At the same time, blocking heat transfer and promoting char formation, but it is not said why the char layer is well expanded.

In [10], a strategy based on γ-aminopropyltriethoxysilane (KH550) and graphene oxide (GO) functionalized 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) was presented. To fabricate an integrated P–N–Si flame retardant material [KDOPO modified GO (DGO)] via mild Mannich reactions and silanization to overcome the single-phase fire resistance problem of DOPO. DGO-based phenolic epoxy resin (DGO/PER) is prepared and applied to the surface of expandable polystyrene (EPS) boards to achieve fire safety, which are used as thermal insulation exterior wall in buildings and structures. The DGO/PER paint provides high fire safety for EPS foam board, showing a high oxygen index limit of 29%, and the UL-94 V-0 classification is achieved with only a layer thickness of 300 μm compared to the DOPO/PER paint. Meanwhile, all combustion parameters such as peak heat release rate, heat release rate, total heat release, smoke release rate, total smoke release rate and ignition time provide excellent advantages for EPS@DGO compared to EPS@DOPO. However, no mention is made of the environmental friendliness of these products. Piperazine pyrophosphate (PAPP) combined with melamine polyphosphate (MPP) was used to prepare a water-based fire-retardant intumescent coating (IC) for structural steel [11]. A silicone acrylic emulsion was used as the binder. During the 2-hour burner test, the PAPP/MPP-IC coating showed excellent fire resistance. The equilibrium temperature on the reverse side of the steel sheet decreased to 170°C after MPP/PAPP-IC protection compared to 326°C for APP/PER/MEL-IC. After 72 hours of immersion in water, MPP/PAPP-IC could still provide sufficient thermal insulation, but APP/PER/MEL-IC failed the test. The water absorption of the MPP/PAPP coating also decreased. Thermogravimetric analysis showed that PAPP/MPP-IC had a unique initial decomposition temperature of 296°C and a higher residue of 33.8 wt. %, which demonstrated better thermal stability and fire resistance in the condensed phase. In addition, the images obtained by scanning electron microscope (SEM) demonstrated that the structure of the carbon layer formed by MPP/PAPP-IC was dense, complete and uniform, indicating the improvement of the mechanical strength and thermal insulation of the carbon. The synergistic effect between the piperazine and phosphoric acid groups in MPP/PAPP contributed to the improved fire resistance. Therefore, MPP/PAPP-IC was much more effective than the traditional APP/PER/MEL-IC. However, the values of these parameters do not cover a wide range of applications.

In [12], cerium oxide (CeO_2) was introduced into ethylene vinyl acetate (EVA)/magnesium hydroxide (MDH) composites to improve the flame retardancy, mechanical properties and UV aging resistance. The target EVA/MDH/CeO_2 composites were obtained by extrusion and injection molding. The effect of CeO$_2$ addition was investigated by thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), X-ray diffraction (XRD), limiting oxygen index (LOI), UL-94, cone calorimetry and UV aging resistance. As a rule, the addition of CeO$_2$ allows to significantly increase the elongation at break and Young's modulus compared to EVA/MDH by 52.25% and 6.85%, respectively. The pHRR index decreased significantly from 490.6 kW/m^2 for EVA/MDH to 354.4 kW/m^2 for the EVA/MDH/CeO^2 composite. It was found that the combination of CeO^2 with MDH is a new and simple method for improving the interaction and dispersion of the filler and the polymer, which led to improved mechanical properties, fire resistance and resistance of the composites to ultraviolet aging. However, the optimal solutions for their concentration have not been established.

Thus, from the literature on the creation of fire-retardant coatings [1–5, 7, 8, 10, 11] and biocomposites [6, 9, 12] it was established that fire-retardant coatings with the addition of inorganic compounds and biopolymers exhibit protective properties for building structures against fire, but the parameters that ensure their resistance to thermal effects have not been determined. Therefore, establishing protection indicators and the influence of flame retardant additives on this process necessitated the need to conduct research in this direction.

3. The aim and objectives of the study

The aim of the study is to identify the patterns of fire protection of wood materials under thermal influence by a coating with the addition of potassium nitrate. This makes it possible to increase the fire protection efficiency of the coating in construction.

To achieve this aim, the following objectives were accomplished:

– to substantiate and optimize a biocomposite coating with the addition of potassium nitrate;

– to determine the mechanism of formation of a thermal insulation layer of foam coke on the surface of wood when treated with a coating with the addition of potassium nitrate.

4. Materials and methods

4. 1. The object and hypothesis of the study

The object of the study is the fire-retardant properties of a biocomposite coating for wood when potassium nitrate is added during thermal action. The scientific hypothesis is to establish the effectiveness of fire protection of a biocomposite coating on the surface of wood when a heat-resistant layer of foam coke is formed under the influence of high temperature.

It was assumed that the temperature, humidity, and pressure of the wood processing process do not change.

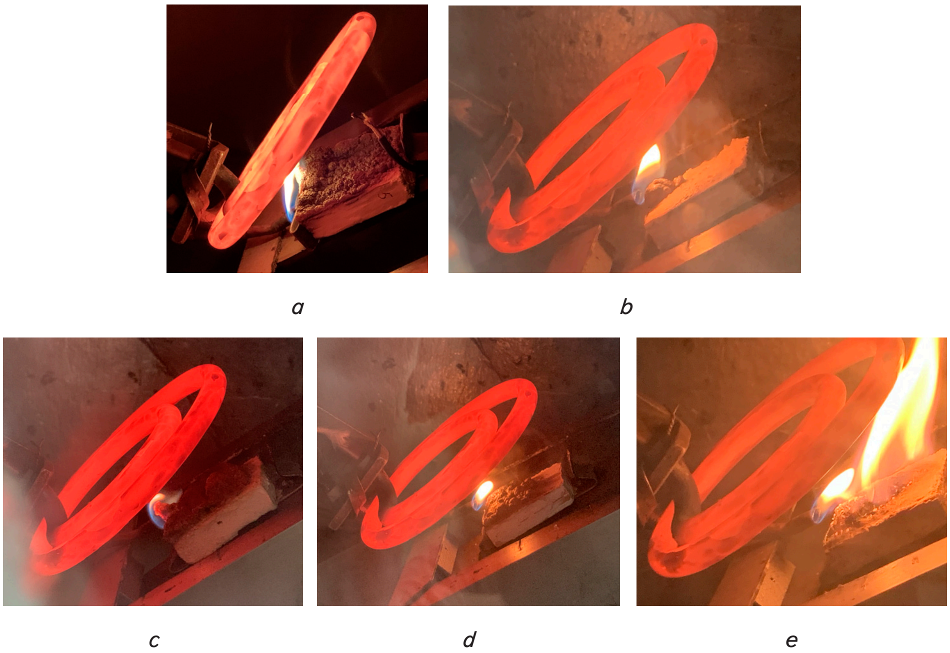
It was simplified that the process of processing wood with a protective coating is constant under the influence of external conditions.

4. 2. The materials under study that were used in the experiment.

To establish the effectiveness of the use of a biocomposite coating when potassium nitrite is added, pine wood samples were used. The coating includes the following substances: ammonium polyphosphate, pentaerythritol, melamine, chloroparaffin, titanium dioxide and starch in an amount of 9% (wt.), which were placed in a binder (acrylic resin) and diluted with a solvent (biocomposite coating) [13]. Potassium nitrite was added to the coating in an amount of 1%, 4%, 16%, 28%. The resulting coating was applied to wood after intensive mixing. To determine the foam coke layer, wood samples 40 × 40 × 10 mm were used, which were covered with the resulting coating with a consumption of about 300 ÷ 310 g/m^2 (Fig. 1). The film thickness was within 0.6 ÷ 0.7 mm. After the samples were aged for 14 days, tests were carried out on the swelling of the coating and its effectiveness.

Fig. 2 shows the results of research into the process of foam coke formation.

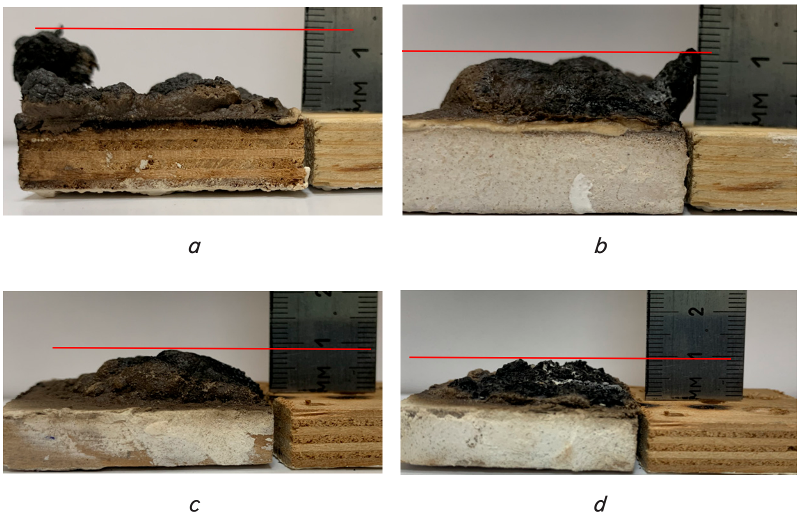
Fig. 1. Model sample of wood treated with a biocomposite coating with the addition of potassium nitrite



a *b* *c* *d* *e*

Fig. 2. Results of research on the swelling process of biocomposite coating: *a* – without additive and with the addition of potassium nitrite; *b* – 1%; *c* – 4%; *d* – 16%; *e* – 28%

As can be seen from Fig. 2, under the action of the radiation panel, the swelling of the coating occurred, which is associated with the decomposition of flame retardants under the influence of temperature with the absorption of heat and the release of non-combustible gases (nitrogen, carbon dioxin). In particular, the beginning of swelling of the biocomposite coating occurred at 62 s, and the height of the swelling was 12.5 mm over an area of 75%. When adding 1% potassium nitrite to the coating, the beginning of swelling of the biocomposite coating decreased to 48 s, when adding 4% potassium nitrite to the coating, it decreased to 45 s. And with a further increase, the beginning of swelling did not decrease, but the coating was ignited. Fig. 3 shows the change in the height of the coating swelling depending on the amount of potassium nitride.



a *b* *c* *d*

Fig. 3. Swelling height of biocomposite coating with potassium nitrite addition: *a* – 1%; *b* – 4%; *c* – 16%; *d* – 28%

From Fig. 3, for biocomposite coating with 1% potassium nitrite addition, the swelling was 14 mm, with 4% potassium nitrite addition, it decreased to 12 mm, and with an increase in the amount of potassium nitrite, the swelling was less than 10 mm for a concentration of more than 16%.

4. 3. Methods for studying the properties of the coating and fire-retardant wood

The study of the process of forming a foam coke layer under thermal action on the coating was carried out according to the method given in [14]. The essence of the method for determining the swelling coefficient is the effect on the fire-retardant coating applied to the wooden substrate of the heat flux of the radiation panel and measuring the height of the foam coke layer after cooling.

Studies to determine the thermal stability of a sample of wood treated with the above coating were carried out using a method that involved exposing the sample to a radiation panel and igniting it. As well as measuring the temperature of combustion products and the time it reached, the time of ignition and passage of the flame front through the surface areas, and the length of the burned part of the sample. The flammability index was calculated using the data obtained [15].

The study of the mechanism of the fire-retardant coating was determined by the method of thermogravimetric analysis and the formation of volatile products of wood pyrolysis.

Thermogravimetric analysis was carried out according to [16]. Thermogravimetric studies were carried out on a Linseis STA 1400 derivatograph (Germany). Samples weighing 10 mg were heated in an air atmosphere from 20 to 700°C at a rate of 10°C/min.

The gaseous products of thermal destruction of fire-retardant wood were obtained using the method [17]. The analysis of samples was performed on a gas chromatograph 6890 N from Agilent. Analysis conditions: detector – catharometer, detector temperature 250°C; carrier gas – helium, flow rate 60 ml/min.; column – PLOT HP-MOLSIV, length 15 m, column temperature 50°C/min. from 50°C to 200°C. Dose value 0.25 ml. Calculation of component concentrations was performed by the peak area normalization method. Gas samples were introduced directly into the chromatograph dispenser, squeezing out of sealed vessels with saline solution.

5. Results of determining the thermal stability of a biocomposite coating with the addition of potassium nitrate

5. 1. Results of optimizing a biocomposite coating with the addition of potassium nitrate

Taking into account the results of determining the swelling height of a biocomposite coating (Fig. 3), using the three-factor simplex-central method of experimental planning in the mathematical environment Statistica 12, statistical processing of the results was carried out.

The following variation factors were selected as the following: the amount of potassium nitrite, % (factor X_1); coating consumption when processing wood, g/m^2, (factor X_2), the values of which are given in Table 1.

Table 1

Variation factors during optimization

Factors	Code	Variation levels			Variation interval
		–1	0	+1	
Potassium nitrite content, %	X_1	0	14	28	14
Coating consumption for wood treatment, g/m^2	X_2	290	300	310	10

The swelling height of the biocomposite coating was chosen as the initial parameter (response function), the value of which was recorded on samples subjected to thermal stress. The experimental planning matrix and its mathematical implementation are given in Table 2.

As a result of the modeling, regression equations were obtained and ternary surfaces of changes in the output parameter were constructed depending on changes in the variation factors (Fig. 4).

Regression equation

$$Y_{calc} = 10.444 - 4.167X_1 + 1.167X_2 + \\ + 1.833X_1 \cdot X_1 - 0.167X_2 \cdot X_2 - 0.250X_1 \cdot X_2. \quad (1)$$

Based on the computer modeling, the best value of the indicator that ensures the fulfillment of the task was determined. Namely, the largest value of the swelling height of the coating, which was obtained for a coating consumption of 310 g/m^2 and a potassium nitride content in the coating of about 1%.

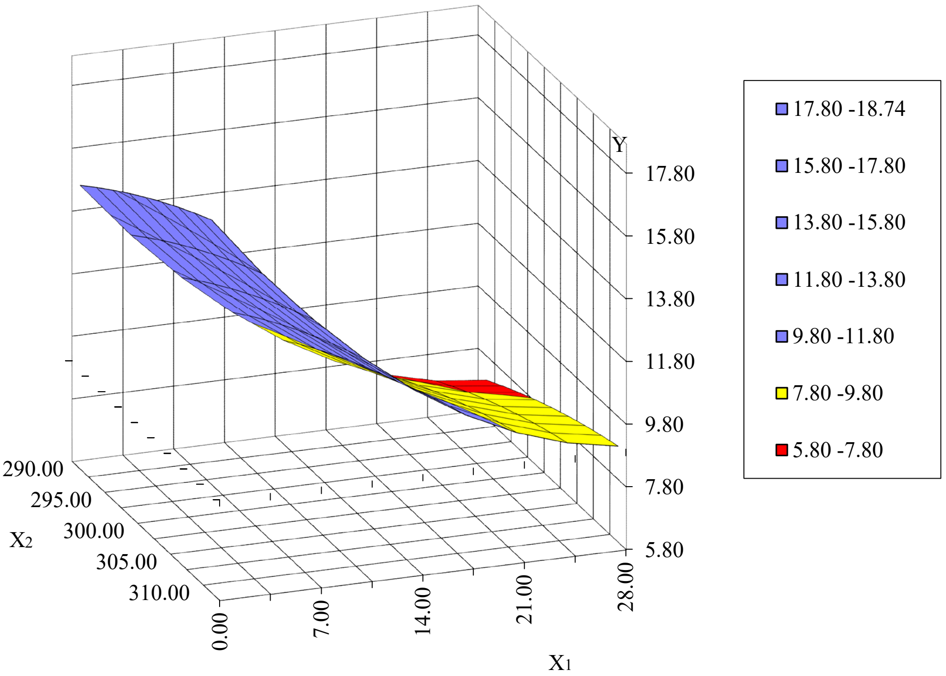


Fig. 4. Ternary surfaces of changes in the output parameter depending on changes in the factors of variation of the mixture flow rate during processing

Table 2

The experimental matrix and its implementation

No.	Factors, type		Planning matrix		Feedback function	
	X_1	X_2	Amount of potassium nitrite, %	Coating consumption, g/m^2	Y_{act}	Y_{calc}
1	1	1	28	310	9	8.86
2	1	–1	28	290	7	7.03
3	–1	1	0	310	18	17.69
4	–1	–1	0	290	15	14.86
5	1	0	28	300	8	8.11
6	–1	0	0	300	16	16.44
7	0	1	14	310	11	11.44
8	0	–1	14	290	9	9.11
9	0	0	14	300	12	10.44
10	0	0	14	300	10	10.44
11	0	0	14	300	11	10.44

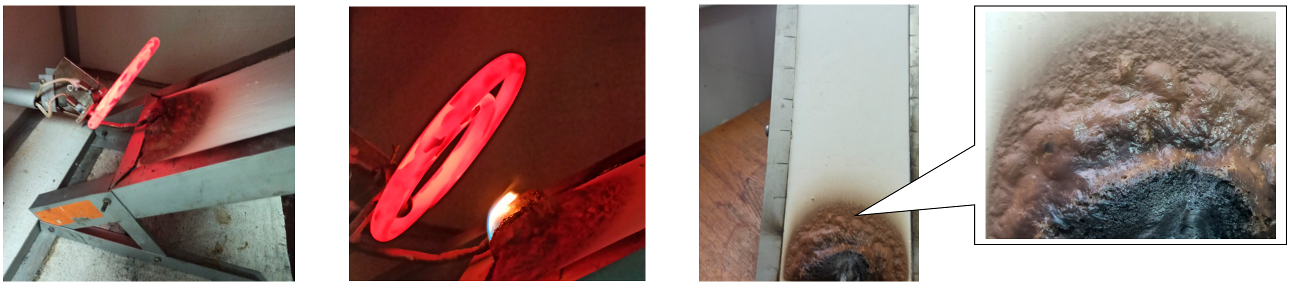
To establish the resistance of wood to thermal effects, studies of the biocomposite coating were conducted to determine the flammability index and maximum temperature of the output gases from the installation. The results of the studies are shown in Fig. 5, 6, Table 3.

Studies have shown that at the initial temperature of gaseous combustion products $T = 65 \div 66°C$, when a radiation panel is applied to a wood sample treated with a biocomposite coating. Intensive formation of a foam coke layer began after 62 s of thermal exposure with a height of 16 mm on a 75% sample area, which thermally insulated the wood. In contrast, after exposure to a radiation panel on a wood sample treated with a biocomposite coating with the addition of potassium nitride, the formation of a foam coke layer began after 47 s of thermal exposure, the height of which was 15 mm. The maximum temperature of flue gases did not exceed 100°C. The flammability index of the samples was zero.

Table 3

Time of passage of the flame front of the control points

Sample coated	Flue gas temperature, °C		Ignition time, s	Time of passage of the flame front through the sample sections, s									Time of reaching T_{max} of flue gases, s	Sample fracture length, mm	Flammability index
	T_1	T_{max}		1	2	3	4	5	6	7	8	9			
–	65.7	98	–	–	–	–	–	–	–	–	–	–	600	0	0
Potassium nitrite	66.1	95	–	–	–	–	–	–	–	–	–	–	600	0	0



a b c

Fig. 5. Results of studies of the ignition process and flame spread on a wood sample treated with a biocomposite coating: *a* – flame effect on the sample; *b* – swelling of the coating; *c* – protective layer of foam coke

a b c

Fig. 6. Results of studies of the ignition process and flame spread on a wood sample treated with a biocomposite coating with the addition of 1% potassium nitride: *a* – flame effect on the sample; *b* – the beginning of the coating swelling; *c* – the result of the formation of protective foam coke

5. 2. Results of studies of the process of foam coke layer formation under thermal exposure to a biocomposite coating

To establish the original properties of the biocomposite coating samples, thermogravimetry was performed. The analysis curves are shown in Fig. 7, 8.

In all coating samples at temperatures of about 100°C, endothermic processes occur, showing mass loss due to evaporation of water without decomposition of coating materials.

Endothermic effects within the temperature range of 170°C are associated with the fact that the coating components lose water and with the subsequent destruction of potassium nitrate and dehydration of melamine. Within the temperature range of 200–300°C, the destruction of biopolymers begins, which is superimposed on the effect of the destruction of pentaerythritol with the formation of ketones and aldehydes, as ammonium polyphosphate melts and a foam coke cell is formed.

The interaction of melamine degradation products with pentaerythritol aldehydes forms methylol, and then polymer-oligomeric structures of amino resin. At temperatures of about 300–370°C, intensive decomposition of ammonium polyphosphates into ammonia and polyphosphoric acid begins, which is confirmed by endothermic peaks. The beginning of intensive decomposition of the fireproof coating is characterized by a temperature of 320–340°C, when the peak of melamine decomposition is superimposed, which begins at a temperature of about 330°C and ends at more than 420°C. This is shown by the intensive growth of foam coke, when mass loss reaches about 60%. At the first stage, pyrolysis occurs with the cleavage of volatile biopolymer products, the rate of which does not depend on the subsequent chemical transformations of these products. At the second stage, the rate of mass loss is determined by the kinetics of the interaction of the carbonized residue with the oxidant.

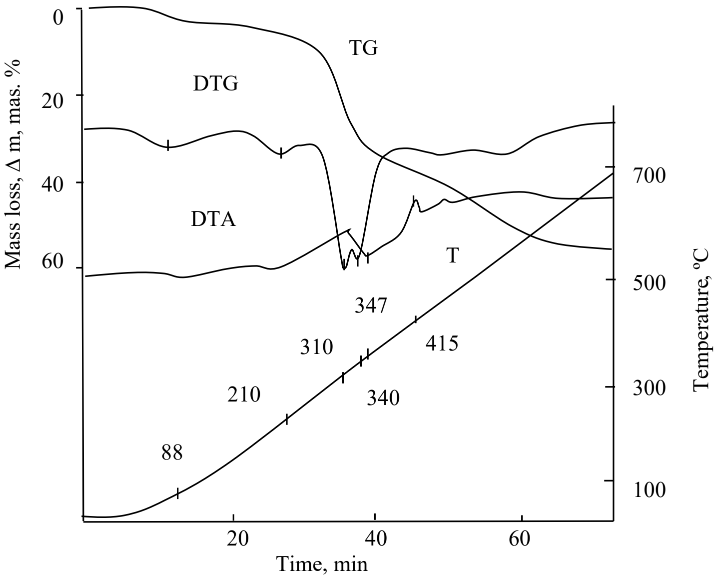


Fig. 7. Thermogravimetric analysis curves for a starch-based biocomposite coating sample

Thus, thermogravimetric studies have revealed that under thermal action, chemical reactions of potassium nitrate begin with the release of inert gases, with the subsequent destruction of ammonium polyphosphate, which decomposes and releases phosphoric acid. Which, in turn, interacts with the biopolymer and affects the dehydration of pentaerythritol with the formation of coke, and the simultaneous decomposition of melamine leads to the release of non-combustible gases that cause coke to foam.

From the thermograms, the temperatures at which intensive pyrolysis of the coating is achieved were determined and the stages of decomposition processes were estimated when heated to 700°C. Taking into account the results of thermogravimetric studies, the process of thermal destruction of coating samples was carried out under conditions under which the formation of gaseous products occurs at the highest speed.

The results of gas chromatographic analysis of the obtained combustible gas mixtures are given in Table 4.

Table 4

Qualitative and quantitative composition of gaseous decomposition products of pine wood

Component	Content of components in volatile decomposition products of pine wood, % vol.	
	Treated with biocomposite coating	Treated with biocomposite coating with the addition of potassium nitrate
CO	22.34	16.88
CO_2	37.42	38.64
CH_4	2.55	2.67
$C_2H_6 + C_2H_4$	0.56	0.75
C_3H_8	0.18	0.23
C_3H_6	not detected	not detected
H_2	0.44	0.36
O_2	not detected	not detected
N_2	36.51	40.47

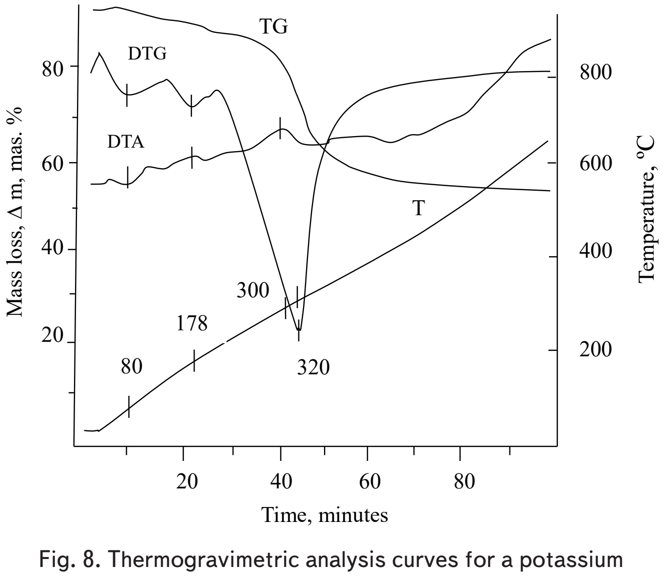


Fig. 8. Thermogravimetric analysis curves for a potassium nitrate-added biocomposite coating sample

As can be seen from Table 4, after pyrolysis of wood treated with a coating, the mixtures of degradation products differ in the content of inert gases (carbon dioxide, nitrogen) and the amount of combustible gases. Thus, when potassium nitrate was added to the coating filled with starch, the amount of combustible gases decreased by more than 19%, and the amount of nitrogen increased by more than 10%.

6. Discussion of the results of the study of the fire protection patterns of wood by biocomposite coating with the addition of potassium nitride

The results of the studies of the coating degradation process showed that the onset of swelling of the biocomposite coating occurred at 62 s, and the swelling height was 12.5 mm over an area of 75%. When adding 1% potassium nitrite to the coating, the onset of swelling of the biocomposite coating decreased to 48 s, when adding 4% potassium nitrite to the coating, to 45 s. And with a further increase in concentration, the onset of swelling did not decrease, but the coating flared up (Fig. 2). Under the influence of thermal action on the biocomposite coating, chemical reactions begin (Fig. 3), leading to swelling. Thus, for a coating with the addition of 1% potassium nitrite, the swelling was 14 mm, with the addition of 4% potassium nitrite it decreased to 12 mm, and with an increase in the amount of potassium nitrite, the swelling was less than 10 mm for a concentration of more than 16%.

According to the results of computer modeling studies (Fig. 4), the best value of the indicator that ensures the fulfillment of the task was determined, namely the largest value of the swelling height of the coating, which was obtained at a coating consumption of 310 g/m^2 and a potassium nitride amount in the coating of about 1%. Studies (Fig. 5, 6 and Table 3) showed that when a radiation panel was applied to wood treated with a biocomposite coating, after 62 s of thermal exposure, an intensive formation of a foam coke layer with a height of 16 mm began on the sample area of 75%. In contrast, after exposure to a radiation panel on a wood sample treated with a biocomposite coating with the addition of potassium nitride, a foam coke layer began to form after 48 s of thermal exposure, the height of which was 15 mm. The maximum temperature of the flue gases did not exceed 100°C. The flammability index of the samples was zero. This confirms that the biocomposite coating will effectively create a thermal insulation layer of foam coke on the surface of the wood [18, 19]. As can be seen from Fig. 8, thermogravimetric studies have revealed that under the influence of thermal exposure, chemical reactions of potassium nitrate with the release of an oxidant begin. Which interacts with the biopolymer to form carbon monoxide, and then ammonium polyphosphate, which, when decomposed, releases phosphoric acid. Which, in turn, affects the decomposition of the biopolymer and pentaerythritol with the formation of soot, and the simultaneous decomposition of melamine induces the release of non-combustible gases, which dries the soot to foam and form foam coke. This reflects the participation of potassium nitride in the formation of the foam coke layer, which can be identified directly by high-temperature exposure to samples of the fire-retardant composition [20, 21].

However, as an alternative to the studies cited in [4, 9], where interest is paid to coatings based on biofillers, coatings that are known on the market are considered here.

However, their difference from the results shown in [10] regarding the protection mechanism allows to state the following:

– the main means of penetration of thermal action into wood is not only the evaporation of water, which affects the suppression of temperature with the formation of ceramic compounds, which is characteristic of geocement coating;

– the mechanism of operation of the intumescent coating is reduced to the fact that under thermal action, the decomposition of flame retardants begins with the absorption of heat and the release of non-combustible gases, which induce soot to foam and form foam coke.

Understanding the obtained results, since the coating is confirmed by the reaction to the action of high-temperature flame, gives a vision of the specific aspects of the operation of the coating. The results of experimental data show that the obtained coating has the ability to form a heat-insulating layer of coke, and the establishment of the swelling height shows how potassium nitrate acts on the beginning of foam coke swelling.

The obtained results on the mixtures of wood degradation products treated with biocomposite coating have certain limitations in determining the components in volatile degradation products, in particular, those associated with the conditions of the experiments, under which the formation of gaseous products occurs at the highest speed.

In addition, the resistance of the coating to high temperature gives little information on the formation of the thermal insulation layer of foam coke and narrows the results obtained, since the disadvantages of the experimental approach to obtaining data include the fact that it extends to the photographic form of reflection. However, as a result of the implementation of this method, a path was obtained to the results that allow establishing the influence of potassium nitrate on the swelling of the biocomposite coating. Possible directions for further development of research may be the development of a coating production technology.

7. Conclusions

1. According to analytical data, the largest value of the coating swelling height was determined by the experimental design method, which was obtained for a coating consumption of 310 g/m^2 and a potassium nitride content in the coating of about 1%. The experimental value of the optimal amount was substantiated by thermal action on the coating sample and it was established that at the initial temperature of gaseous combustion products the temperature was 65÷66°C. When a radiation panel was applied to a wood sample treated with a biocomposite coating, after 62 s of thermal action, an intensive formation of a foam coke layer with a height of 16 mm began on the sample area of 75%. In contrast, after the radiation panel was applied to a wood sample treated with a biocomposite coating with the addition of potassium nitride, the formation of a foam coke layer began after 47 s of thermal action, the height of which was 15 mm. At the same time, the temperature of the flue gases did not exceed 100°C, and the flammability index of the samples was zero.

2. Thermogravimetric studies have established that under the influence of thermal action, chemical reactions of potassium nitrate begin with the release of carbon monoxide, with the subsequent destruction of ammonium polyphosphate, which, when decomposed, releases phosphoric acid. Which, in turn, interacts with the biopolymer and affects the dehydration of pentaerythritol with the formation of coke, and the simultaneous decomposition of melamine leads to the release of non-combustible gases, which cause the coke to foam. The qualitative and quantitative composition of the gaseous products of thermal destruction of wood treated with a biocomposite coating showed that volatile mixtures differ in the content of inert gases and the amount of combustible gases. Adding potassium nitrate to the biocomposite coating reduced the amount of combustible gases by more than 19%, and increased the amount of nitrogen by more than 10%.

Conflict of interest

The authors declare that they have no conflict of interest regarding this study, including financial, personal, authorship or other nature, which could influence the study and its results presented in this article.

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Data availability

The manuscript has no linked data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies in the process of creating the presented work.

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