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SETTING THE PARAMETERS OF THERMAL DESTRUCTION OF FIRE-RESISTANT WOOD

The problem of using wood is to ensure resistance to high-temperature flame and application technology. Therefore, the object of research was to change the parameters of thermal destruction of wood during fire protection by impregnation and intumescent coating. It has been proven that for wood treated by impregnation, the destruction processes slow down, so the mass loss is reduced by 3–5 times, the process increases in the region of higher temperatures with a significant coke residue. As for wood treated with intumescent coating, in the temperature range of 200–300 °C, pentaerythritol begins to decompose with the formation of aldehydes and a foam coke center is formed. The beginning of intensive mass loss coincides with the temperature of 320–330 °C, on which the sublimation peak of melamine is superimposed, starting at a temperature of 330 °C, which ends at a temperature of more than 420 °C. The obtained activation energy of wood is 30.03 kJ/mol, treatment of wood with impregnating agents increases the activation energy during its thermal decomposition by more than two times, and treatment with an intumescent coating by more than 4.4 times. After pyrolysis of wood treated with flame retardants, the mixtures of destruction products differ significantly in the content of carbon dioxide, nitrogen and the amount of combustible gases. Thus, for wood treated with the composition DSA-1, the amount of nitrogen increased by more than 46 times, and the amount of combustible gases decreased by more than 3 times. An even greater difference was recorded during treatment of wood with an intumescent coating. In particular, it was found that the amount of combustible gases decreased by more than 4 times, and the amount of nitrogen increased by more than 56 times. The practical significance lies in the fact that the results obtained were taken into account when developing a reactive coating. Thus, there are grounds to argue about the possibility of directed regulation of the wood protection process through the use of coatings capable of forming a protective layer on the surface.

Keywords: *protective agents, fire resistance, volatile products, mass loss, surface treatment, protection efficiency.*

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1. Introduction

The use of wood and products made of it is gaining a very wide range of applications every year. But it is these materials and products that are the main conductors of flame spread, since they are combustible materials and require fireproofing. For fire protection of wood, fireproofing impregnation is mainly used, as well as coatings, plasters, and slab heat-insulating materials are also used. However, impregnating compositions are washed out of wood, and finishing with plaster and slab materials increases the material consumption of the structure. And, therefore, fire safety puts forward increasingly high requirements for the effectiveness of fireproofing agents, as well as the quality of fireproofing materials.

In this regard, representatives of the construction industry are increasingly intensively searching for new highly effective fireproofing agents for wood. Since fire protection today must not only ensure the standardized fire resistance of wood, but also maintain its operational parameters, solve environmental safety and durability. Therefore, an important problem of today is ensuring the life and safe operation of construction objects from an economic, technological and environmental point of view, and the development of intumescent fire-retardant coatings for building structures is of paramount importance. Such coatings can be used not only on a par with existing analogues, but also be highly effective in special construction industries, which makes it possible to prevent the occurrence of man-made accidents.

Thus, there is a need to develop work towards the development of effective fire-retardant coatings for their use during the construction of both general construction and special-purpose objects, where the use of flame retardant mixtures is ineffective, are relevant.

In the work [1], the material for the protective coating of building structures made of wood is considered. The possibility of chemical processes occurring in the material, leading to its expansion, has been investigated. The coefficient of expansion of the material upon heating has been practically established. It has been established that the material can swell, both under the influence of flame and at a low rate of temperature increase. The swelling coefficient in this case reaches 8. The temperature range of swelling is 150–250 °C, which is confirmed by thermodynamic calculations and experimentally. The temperature at which the material begins to swell is below the temperature of thermal destruction of wood, but questions regarding the manifestation of the joint action of the components remain unresolved. Wood, as noted in [2], has remained an integral part of building structures around the world. Nevertheless, concerns have arisen about wood, which has high flammability and threatens the fire safety of buildings and residents. This leads to the need to increase fire resistance. The use of flame retardants, which are characterized by ease of application and cost-effectiveness to mitigate the threat of wood flammability, is convincing in current developments. Among the variety of methods, fire retardant coating applied to wood provides improved protection against fire.

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This review aims to provide a substantial insight into the assessment methods and regulations followed worldwide, serving as a guide for assessing the fire resistance of wood structures in construction. A comprehensive discussion of the mechanisms of the various elements for imparting fire resistance to polymer coatings is provided. In addition to these traditional flame retardants, the review highlights the growing interest in biomaterials as sustainable alternatives. However, it is not stated how bio-based flame retardants offer multiple environmental benefits and demonstrate promising fire protection properties through a variety of mechanisms.

In the study [3], the development of a new type of wood sawdust (WS) composite based on modified $CuSiF_6$, epoxy-amine polymers (EAP) with reduced flammability is reported. The thermo-oxidative behavior of the obtained WS/EAP–$CuSiF_6$ and WS/EAP samples was investigated using a comprehensive thermal analysis. The thermal analysis data show that the introduction of $CuSiF_6$ (a flame retardant) into WS/EAP increases the resistance of WS/EAP–$CuSiF_6$ to thermal oxidation. It is shown that for WS/EAP–$CuSiF_6$ and WS/EAP, the thermo-oxidative destruction ends at 571 °C and 625 °C, and the maximum temperature of this exothermic process is 435 °C and 499 °C, respectively. The flammability of WS/EAP–$CuSiF_6$ and WS/EAP was investigated using the "ceramic tube" (CT) method. The obtained CT results demonstrate striking differences in the flammability of these two composites; the maximum temperature of gaseous combustion products for WS/EAP–$CuSiF_6$ is 265 °C, for WS/EAP – 910 °C. Flammability tests for WS/EAP–$CuSiF_6$ and WS/EAP were carried out in accordance with ASTM D635-18 and ASTM D3801-19a. According to the results of vertical combustion tests, the WS/EAP–$CuSiF_6$ composite material was classified as V-0. However, the mechanism of coke formation and temperature transitions under thermal action are not shown. In [4] it is noted that flammability limits the application of cellulose functional materials in certain specific environments. To overcome this problem, an environmentally friendly and effective flame retardant modification is required. Using porous wood with oriented pores as raw material, a highly efficient halogen-free cellulose-based nanocomposite was fabricated by incorporating hydroxyapatite (HAP) nanosheets synthesized by chemical co-deposition. The structure and flame retardant properties of the cellulose-based nanocomposite were investigated. The results showed that the larger pore size promotes the growth of HAP and that the HAP nanosheets have a sheet-like structure with a thickness of 10 nm and fill the pore channels. The nanocomposite had an impressive limiting oxygen index (LOI) (44.45 %) and total heat release (THR) (3.2 kJ/g). The vertical burning test showed that the material did not burn after 60 s of flame exposure. The flame retardant mechanism involves volatiles generated by the decomposition of HAP and the oxygen barrier effect of the carbon layer formed by the carbonization of oriented pores composed of cellulose. However, it is unknown how the prepared flame retardant cellulose-based nanocomposites have potential applications.

Flame retardant composite hydrogels, as reported in [5], have many advantages over conventional flame retardants, such as high water retention capacity, enhanced fire resistance and mechanical strength. In this paper, flame retardant dynamic covalent hydrogels are developed using wood-derived cellulose nanocrystals (CNCs) cross-linked by boronate ether linkages. Environmental and health concerns associated with the presence of non-biodegradable synthetic polymer components and/or inorganic nanoparticles in existing systems are addressed. This rheological study demonstrates the liquid-to-soft solid transition of CNC dispersions with tunable network elasticity in the range of ≈ 0.2 kPa to 3.5 kPa and immediate self-healing ability. Pine wood coating with these hydrogels delayed ignition by approximately 30 s compared to native wood, and achieved an excellent limiting oxygen index of 64.5 %. In addition, it was found that the increased borax content in the gels reduced and delayed the first peak of the heat release rate by up to 40 s, resulting in a 277 % increase in the fire resistance index. The microstructure and rheological behavior were correlated with fire-fighting mechanisms. However, for the rational design of sustainable fire-retardant materials, the results of using plant-based dynamic gels to prevent wood ignition are not presented.

In [6], phytic acid, a natural source of phosphorus extracted from rice bran, was used to synthesize phytic acid-based flame retardants (PFRs) by reflux esterification with powdered chicken eggshell (CES) using calcium carbonate ($CaCO_3$) as a biofiller. These components were incorporated into melamine formaldehyde resin to produce a transparent IFR coating. It was found that the developed intumescent flame retardant (IFR) coatings achieved the highest fire rating based on UL94 flammability standards compared to the control. The coatings also gave increased LOI values, indicating self-extinguishing properties. An increase in the melting point of the IFR coating by 17 °C and a significant increase in the enthalpy change of ~172 % compared to the control were observed, indicating increased fire resistance. The thermal stability of the coatings was improved, as indicated by a decrease in mass loss and an increase in residual mass after thermal degradation. As confirmed by microscopy and spectroscopy, the large number of phosphorus and carbon groups in the condensed phase of the coatings after combustion indicates enhanced char formation. In the gas phase, TG-FTIR showed the release of non-flammable CO_2 and flame retardants PO and P–O–C. Mechanical property testing confirmed the absence of a decrease in the adhesion strength of the IFR coating. Due to these results, the developed IFR coating demonstrated increased fire resistance while remaining optically transparent, which indicates a two-phase protective mechanism of IFR, which includes the release of gaseous combustion diluents and the formation of a thermally insulating char layer. However, it has not been determined how the decomposition process of the flame retardant coating proceeds.

As reported in [7], to reduce flammability, a polyelectrolyte complex (PEC) coating consisting of sodium polyborate (SPB) and polyethyleneimine (PEI) was applied to OSB using a simple two-dip process. This PEC treatment imparts self-extinguishing properties to the OSB and reduces the total heat release by 21 % and the total smoke release by 79 %, while increasing the time to ignition by 18 % compared to untreated OSB. In addition, the PEI/SPB coating adds a small additional weight (5.8 wt. %) to the oriented strand board, while maintaining visual aesthetics and mechanical properties. The main fire protection effect is due to the action of the condensed phase through a combination of swelling and thermal barrier mechanisms. Improving the fire protection of OSB and other engineered wood materials through simple and safe processing will increase their potential as a largely renewable building material, contributing to a sustainable bioeconomy. However, they cannot provide reliable protection for structures. In [8], polyurethane (PU) was used to obtain simultaneously improved fire-retardant and mechanically strengthened wood-polyurethane composites (WPUC). It was made from ammonium polyphosphate (APP) and PU in a simple way. The results showed that a reduction in smoke production was measured during the cone calorimeter test. At an APP weight of 18 % of PU, the limiting oxygen index can reach 31.2 %. During the combustion test, the peak heat release rate and total smoke production of WPUC were reduced by 42.1 % and 89.7 %, respectively, with the above APP to PU ratio. In addition, the results of the functional group test show that the polyurethane contains highly active –NCO, which is bound to –OH and moisture in the wood fiber. This leads to the improvement of physical and mechanical properties. The mechanism of fire resistance of WPUC showed that the polyphosphoric acid formed by the pyrolysis of APP catalyzed the PU into char, and the PU was distorted by gases such as NH_3 to form a tiny spherical structure that blocked heat and metabolism. It is shown that the WPUC with APP obtained by this method has improved performance, however, it is expected to provide a new strategy for the preparation of fire-retardant WPCs.

In [9] describes the synergistic evolution of a fire-resistant and biodegradable composite structure of polyvinyl alcohol, starch and kaolin for wood-based coatings. The resulting composite and its components were characterized for chemical structure, morphology, viscosity, flame resistance, biodegradability and physical properties using infrared spectrometer (FT-IR), X-ray diffractometer (XRD), nuclear magnetic resonance (NMR), scanning electron microscope (SEM), laboratory tests (UL-94), soil burial tests and relevant standard methods. Thus, the obtained analysis results indicate the formation of an unsaturated hybrid structure with a viscosity of 79 cP, a pencil hardness of 5 N and strong adhesion to the wood surface. The coating of the developed paint with a thickness of 0.15 micrometers on 0.5 mm thick wooden strips demonstrated extraordinary fire safety in terms of 4 times increase in ignition time, 80 % reduction in burning rate and effective charring. In addition, based on the chemical interaction, gas permeability and synergistic interaction between the components, a mechanism for understanding the fire resistance, adhesion and physical protection of wood was presented.

In the study [10], it was noted that in order to recycle waste, waste bagasse (sugar cane) was used as a fire-retardant filler to produce a water-based intumescent fireproof coating. The effect of bagasse on the properties of the refractory coating was investigated by flame retardant test, thermogravimetric analysis, smoke suppression test, scanning electron microscope, energy dispersive spectrometer, X-ray diffraction, Fourier transform infrared spectroscopy, water resistance test and mechanical property test. The results showed that the coating with 1.5 % bagasse showed the best performance in flame retardant, thermal stability and smoke suppression tests. In addition, the char layer of the sample after fire protection was dense and continuous. However, the refractory coating containing 2 % bagasse showed excellent performance in water resistance test and mechanical properties due to the fiber properties. In addition, the components and chemical structure of the coal layers were not characterized to study the flame retardant mechanism of bagasse in the refractory coating.

In [11], the thermal and rheological behavior of several varnishes and paints used for wood protection available on the market was analyzed. Thermogravimetric analysis combined with mass spectrometry and Fourier transform infrared spectrometry showed that the degradation mechanism is complex and involves two to five decomposition steps, depending on the composition. Analysis of the gases formed during the decomposition processes of the water-based samples showed that carbon dioxide was the main potentially harmful product, while for those containing solvents, the harmful substances were: benzene, xylene and carbon dioxide. The decomposition of the sample containing urea-alkyd resins also leads to the formation of hydrocyanic acid. Rheological tests showed a viscoelastic solid behavior in the case of water-based film-forming products, and a viscoelastic liquid behavior in the case of solvent-based products. Microscale combustion calorimetry confirmed that this sample has the lowest thermal stability and can contribute the most to fires.

The mechanisms of nanomaterials, as stated in [12], not only protect wood from biological degradation agents, but also provide adequate protection against weathering and fire. Also reduce the effects of abrasion and chemicals. Various methods of nanomaterial-based wood protection have been investigated, including biocide delivery systems, metal-based nanoparticles, green compounds and nanominerals. The biocide delivery system is used for the controlled release of termiticides, insecticides and fungicides. Some metal-based nanoparticles, such as zinc oxide nanoparticle and copper oxide nanoparticle, were resistant to leaching, inhibited decay fungi harmful to termites, and increased the photostability of wood against UV radiation. However, the potential of nanomaterials for wood preservation is not used in a wider scale of commercial wood protection practice due to limited available information and high cost.

Thus, analytical studies of the literature have established that fire protection agents protect the surface of wood from the effects of fire, but the parameters that provide fire protection ability have not been determined. A small number of studies and descriptions of the process of fire protection of wood leads to the burning of wooden structures. Therefore, establishing the parameters of wood protection and the role of fire protection in wood decomposition necessitates research.

The aim of research is to identify the patterns of inhibition of thermal destruction when exposed to high temperature on fire-protected wood with an impregnating composition and an intumescent coating. This makes it possible to substantiate the requirements for the use of fire protection agents and expand the scope of fire protection.

To achieve the aim, the following objectives were solved:

– to investigate the activation energy of fire-protected wood during thermal destruction;
– to determine the effect of fire retardants on the formation of volatile components of thermal destruction of wood.

2. Materials and Methods

2.1. Object and hypothesis of research

The object of research is the change in the parameters of thermal destruction of wood during fire protection by impregnation and intumescent coating.

The scientific hypothesis is to inhibit the indicators of thermal destruction of wood when treated with fire retardants.

The following simplifications were adopted in the study, which relate to the process of thermal destruction of wood as a model. These include the features that determine the effect of changing conditions on the object of research and the course of thermal destruction of wood: external and internal influences, temperature, humidity, atmospheric pressure.

2.2. Materials for experimental studies

The flammability of wood was carried out in samples of pine wood treated with fire retardants:

– impregnating solution (fire retardant composition DSA-1);
– fire retardant intumescent coating "FIREWALL-WOOD".

The surface of the wood samples was treated with fire retardants at a consumption of: DSA-1 in the amount of 600 g/m^2 and coating in the amount of 241 g/m^2. After drying the samples to a constant mass, the necessary studies were carried out.

2.3. Methodology of studies of thermal destruction of wood

Thermogravimetric analysis was carried out according to [13]. In order to determine the temperature range at which thermal destruction of wood occurs most intensively, thermogravimetric study of destruction processes in dynamic mode was carried out. Thermogravimetric studies were carried out on a Linseis STA 1400 derivatograph (Germany). Wood samples weighing 10–11 mg were heated in a derivatograph from 20 to 700 °C at a rate of about 8–10 °C/min.

Obtaining volatile mixtures of thermal destruction of wood was carried out according to [14]. Analysis of gaseous mixtures was introduced into a gas chromatograph 6890 N from Agilent. Conditions under which the analysis was carried out: detector – catharometer with a temperature of 250 °C; carrier gas – helium with a flow rate of 60 ml/min; column – PLOT HP-MOLSIV, 15 m long with a temperature change rate of 50 °C/min from 50 °C to 200 °C. Analytical dose of the mixture 0.25 ml. Calculation of the value of the components was carried out by the area of the peaks. Samples of volatile products were fed into the chromatograph dispenser, displacing saline solution from sealed vessels.

Calculations of kinetic parameters from the TG curve, which sufficiently characterizes the thermal destruction of wood, are based on the equation [15]:

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$$-\frac{dm}{dt}=k\cdot m^{n}, \quad (1)$$

where m – sample mass that entered the decomposition reaction, mg; n – the order of the reaction; k – the specific reaction rate of the material decomposition.

The calculation of the activation energy (E) is based on numerical modeling of the TG curve taking into account the dependence [13]:

$$\ln\left(\ln\frac{100}{100-\Delta m}\right)=-\frac{E}{R}\cdot\frac{1}{T}, \quad (2)$$

where E – activation energy, kJ/mol; R – the universal gas resistance, kJ/(mol·K).

In this inequality, Δm is the mass of the destroyed material (%) for each temperature value in the interval of its decomposition, which is described by the 1st order process ($n=1$) and the linearization of the ratio:

$$\ln\left(\ln 100/\left(100-\Delta m\right)\right) \text{ from the temperature T, K.} \quad (3)$$

In this case, the value of the activation energy (E) is calculated according to the equation:

$$E=\mathrm{tg}\varphi\cdot R. \quad (4)$$

From the derivatograms, the temperature at which the loss of wood mass is obtained was determined, and the stage of the destruction process and mass loss upon heating were also estimated.

3. Results and Discussion

3.1. Results of studies of the activation energy of wood treated with fire retardants

The results of thermogravimetric studies show the direct processes that occur in wood samples when heated in a dynamic mode. The decomposition of wood samples, as well as those treated with protective agents, was carried out in an air atmosphere with an oxygen content of 21 % vol., which are shown in Fig. 1–3.

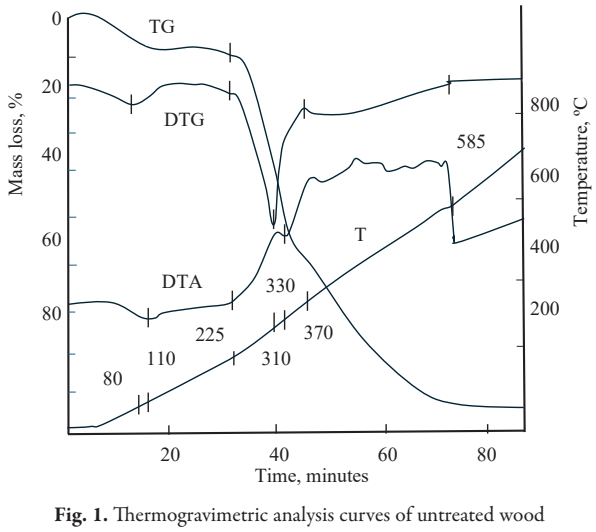

Fig. 1. Thermogravimetric analysis curves of untreated wood

Studies have shown that in the samples of the studied wood at temperatures up to 100 °C, endothermic processes occur, which are characterized by the loss of unbound water, and at 190 °C – the organic components of wood lose water.

In the untreated wood sample, along with endothermic decomposition processes, exothermic processes occur at 225 °C, which is characterized by a stage of intensive mass loss at a temperature within 370–390 °C, which is due to the flame combustion of volatile substances. As well as a sluggish stage (at elevated temperatures – after 60–70 % burnout), which is caused by the burnout of wood residues. For wood treated with DSA-1, the temperature of the onset of thermo-oxidative destruction at 190–200 °C and the temperature at which the maximum rate of decomposition occurs (210–325 °C) were noted. The pyrolysis processes at the second stage depend significantly on the properties of the flame retardant: as it acts, the mass loss slows down by 3–5 times, the thermal destruction process shifts to the region of higher temperatures with a significant coke residue. For wood treated with an intumescent coating, an endothermic reaction was recorded within the temperature range of 180–200 °C, which correlates with the decomposition of ammonium polyphosphate, the transformation and dehydration of pentaerythritol. Within the temperature range of 200–300 °C, it begins due to the decomposition of pentaerythritol with the transformation of aldehydes and the nucleation of foam coke cells. The beginning of intensive mass loss corresponds to a temperature of 320–330 °C, at which the sublimation peak of melamine approaches, which begins at 330 °C.

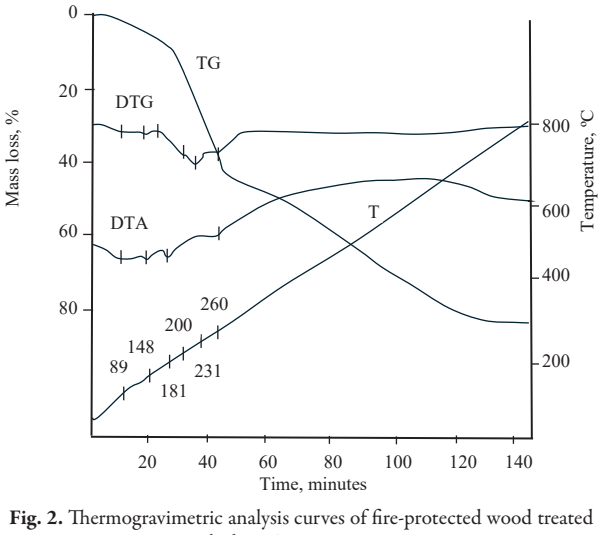

Fig. 2. Thermogravimetric analysis curves of fire-protected wood treated with the DSA-1 composition

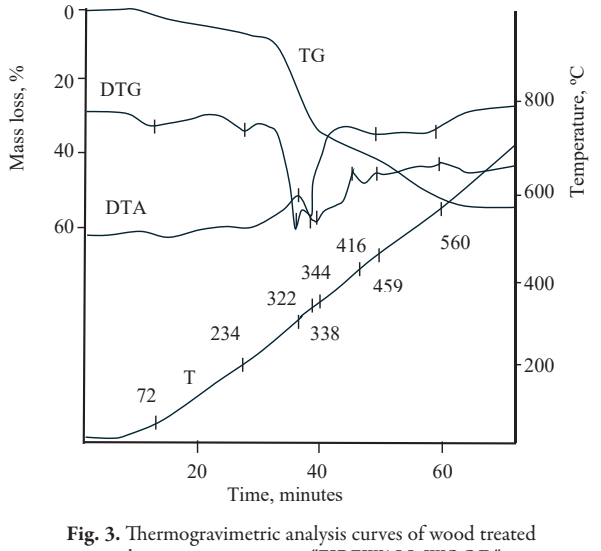

Fig. 3. Thermogravimetric analysis curves of wood treated with intumescent coating "FIREWALL-WOOD"

And, which ends at a temperature above 420 °C (when the relative mass loss reached 60–70 %).

Thus, the obtained thermogravimetric data make it possible to establish the rate of thermal destruction of the sample at a given temperature

and show the value of thermal effects. However, it is important to establish the activation energy during thermal oxidative destruction.

Table 1 shows the results of calculating the parameters necessary to determine the activation energy for wood.

Table 1

Results of treatment with derivatizers

T, K	Δm, %	$Ln(Ln(100/100-\Delta m))$	T, K	Δm, %	$Ln(Ln(100/100-\Delta m))$
Wood in normal air atmosphere					
473	9.1	–2.34957	603	45.9	–0.48721
498	10.3	–2.21917	693	66.0	0.075858
523	25.1	–1.24127	733	77.2	0.390967
573	33.0	–0.9151	773	85.2	0.647387
Wood treated with DSA-1 impregnating composition					
453	12.1	–2.04817	593	42.6	–0.58856
503	23.2	–1.33194	763	71.2	0.218971
533	27.1	–1.15176	863	75.8	0.349824
Wood treated with intumescent coating "FIREWALL-WOOD"					
473	5.5	–2.87227	793	42.2	–0.60115
573	15.0	–1.81696	873	45.1	–0.5114
723	40.0	–0.67173	1013	55.4	–0.21389

Fig. 4 shows the dependence of the rate of wood destruction on the inverse temperature.

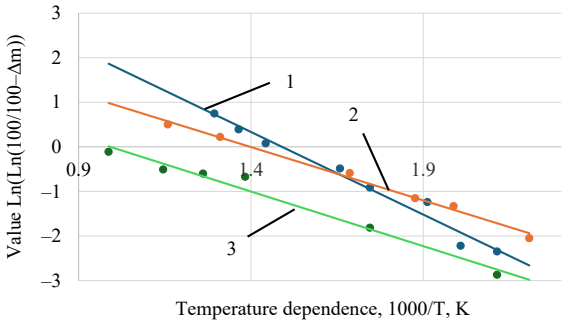

Fig. 4. Graphical dependence of the rate of thermal destruction of wood on the inverse temperature: 1 – untreated; 2 – treated with the impregnating composition DSA-1; 3 – treated with the intumescent coating "FIREWALL-WOOD"

Table 2 shows the values of the activation energy during thermal decomposition of wood.

Table 2

Activation energies during thermal destruction of wood

Pine wood	Activation energy E (kJ/mol)
Untreated	30.03
Treated with impregnating composition DSA-1	60.20
Treated with intumescent coating "FIREWALL-WOOD"	133.98

As can be seen from Table 2, the activation energy of wood is 30.03 kJ/mol, treatment of wood with impregnating agents increases the activation energy of wood during its thermal decomposition by more than two times, and treatment with an intumescent coating by more than 4.4 times.

3.2. Results of studies on the formation of volatile substances during thermal action on fire-protected wood

The results of the analysis of the content of the obtained combustible gas mixtures of wood decomposition are given in Table 3.

Table 3

Composition of volatile products of thermal decomposition of wood

Component	Content of components in volatile products of pine wood destruction, % vol		
	untreated	treated with DSA-1	treated with "FIREWALL-WOOD"
CO	39.09	12.46	10.25
CO_2	51.92	41.16	32.26
CH_4	6.06	0.32	0.42
$C_2H_6+C_2H_4$	0.44	not detected	0.32
C_3H_8	0.19	0.8	0.18
C_3H_6	0.33	not detected	not detected
H_2	0.74	0.14	0.12
O_2	0.27	0.04	not detected
N_2	0.98	46.99	56.45

As can be seen from Table 3, after pyrolysis of wood treated with fire retardants, the mixtures of degradation products differ significantly in the content of carbon dioxide, nitrogen and the amount of combustible gases. For wood treated with DSA-1, the amount of nitrogen increased by more than 46 times, and the amount of combustible gases decreased by more than 3 times. An even greater difference was recorded when the wood was treated with a coating, in particular, it was found that the amount of combustible gases decreased by more than 4 times, and the amount of nitrogen increased by more than 56 times.

3.3. Discussion of the results of the study of the process of fire protection of wood with a reactive coating

Wood samples at temperatures up to 100 °C lose chemically unbound water, water, and at 190 °C organic substances lose constitutional water. In a sample of untreated wood, along with endothermic processes of pyrolysis, exothermic processes occur at 225 °C, which is characterized by intensive mass loss at a temperature of 370–390 °C, which occurred during the flame combustion of volatile products. For wood, fire-protected by DSA-1, the temperature of the onset of thermo-oxidative destruction was noted at 190–200 °C and the temperature at which the maximum rate of decomposition occurs (210–325 °C). Pyrolysis processes at the second stage significantly depend on the properties of the fire retardant: as it acts, mass loss slows down by 3–5 times, the process of thermal destruction shifts to the region of higher temperatures with a significant coke residue. For wood treated with an intumescent coating, an endothermic reaction was recorded within the temperature range of 180–200 °C, which is correlated with the decomposition of ammonium polyphosphate, the transformation and dehydration of pentaerythritol. Within the temperature range of 200–300 °C, this reaction begins due to the decomposition of pentaerythritol with the transformation of aldehydes and the nucleation of foam coke cells. The beginning of intensive mass loss corresponds to a temperature of 320–330 °C, at which the sublimation peak of melamine approaches, which begins at 330 °C and ends at a temperature above 420 °C (when the relative mass loss has reached 60–70 %). The obtained activation energy of wood is 30.03 kJ/mol, treatment of wood with impregnating agents increases the activation energy during its thermal decomposition by more than two times, and treatment with an intumescent coating by more than 4.4 times.

Gas chromatographic studies (Table 3) have established that after pyrolysis of wood treated with fire retardants, the mixtures of destruction

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products differ significantly in the content of carbon dioxide, nitrogen and the amount of combustible gases. Thus, for wood treated with DSA-1, the amount of nitrogen increased by more than 46 times, and the amount of combustible gases decreased by more than 3 times. An even greater difference was recorded when wood was treated with a coating, in particular, it was found that the amount of combustible gases decreased by more than 4 times, and the amount of nitrogen increased by more than 56 times.

This indicates the inhibition of the thermal destruction process [16, 17], which can be determined by the gas chromatography method of analyzing wood decomposition products.

Unlike the studies presented in [4, 6, 8], where the main attention is paid to the development of flame retardants for wood-polymer composites, this study considers substances that are widely available on the market.

However, unlike the results obtained in [18, 19] regarding the inhibition of the destruction process of fire-protected wood and in [20] regarding the effectiveness of its protection, the results of this study allow to state the following:

– the regulator of the inhibition of thermal destruction process is not only the inhibition of oxidation in the gas and condensed phases, but also a change in the direction of wood decomposition towards the formation of non-combustible gases and a difficult-to-burn coke residue;
– a significant impact on the process of inhibiting thermal destruction when using fire protection is carried out in the direction of chemical reactions that inhibit the flame, the coating foams and forms a thermal insulation layer of foam coke.

However, as established from research, with thermal action on fire-protected wood, thermal destruction increases significantly, as indicated by a decrease in activation energy, and requires an increase in the amount of fire retardant. For wood samples treated with an intumescent coating, when exposed to high temperatures, all parameters that affect thermal decomposition have much lower values.

The results obtained have certain limitations when determining the activation energy due to the unpredictability of the process of inhibiting heat transfer to wood. Taking into account the process of inhibiting thermal destruction by an intumescent coating is possible provided that thermal insulation is guaranteed by a layer of foam coke formed during the decomposition of the coating.

This uncertainty is difficult to resolve within the framework of this study, since it is necessary to conduct additional experiments to obtain more data. For example, this requires the availability of data necessary for a sound study of the thermal destruction process and establishing the time point at which the drop in thermal resistance begins. Such a manifestation will allow to establish the transformation of the coating itself, which moves towards increased temperature with the formation of a thermally insulating layer of foam coke. And also, to establish those differences that change the transformation of the thermal destruction process of wood.

4. Conclusions

Wood samples at temperatures up to 100 °C lose chemically unbound water, water, and at 190 °C organic substances lose constitutional water. In a sample of untreated wood, along with endothermic processes of pyrolysis, exothermic processes occur at 225 °C. A characteristic stage is the stage of intensive mass loss up to temperatures of 370–390 °C, which is due to the formation and flame combustion of gaseous products. For wood treated with DSA-1, the temperature of the onset of thermo-oxidative destruction was noted at 190–205 °C, and the temperature at which the maximum rate of destruction occurs (210–325 °C). The destruction processes at the second stage largely depend on the properties of the flame retardant: as it acts, the mass loss slows down by 3–5 times, the thermal destruction process shifts to the region of higher temperatures with a significant coke residue. Regarding wood treated with an intumescent coating, endothermic effects were recorded in the temperature range of 180–200 °C, associated with the melting of ammonium polyphosphate, rearrangement and dehydration of pentaerythritol. In the temperature range of 200–300 °C, pentaerythritol begins to decompose with the formation of aldehydes and a foam coke center is formed. The beginning of intensive mass loss coincides with the temperature of 320–330 °C, which is superimposed by the sublimation peak of melamine, which begins at 330 °C. And, which ends at a temperature above 420 °C.

The obtained activation energy of wood is 30.03 kJ/mol, treatment of wood with impregnating agents increases the activation energy during its thermal decomposition by more than two times, and treatment with an intumescent coating by more than 4.4 times.

Gas chromatographic studies have established that after pyrolysis of wood treated with fire retardants, mixtures of destruction products differ significantly in the content of carbon dioxide, nitrogen and the amount of combustible gases. Thus, for wood treated with the composition DSA-1, the amount of nitrogen increased by more than 46 times, the amount of combustible gases decreased by more than 3 times. An even greater difference was recorded when wood was treated with an intumescent coating, in particular, it was found that the amount of combustible gases decreased by more than 4 times, and the amount of nitrogen increased by more than 56 times.

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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 research and its results presented in this article.

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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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