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FIRE PROTECTION OF WOOD BASED ON MULTICOMPONENT MIXTURES OF SUBSTANCES

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ABSTRACT: The article presents the results of scientific research aimed at determining and improving the fire-protective properties of powdered substances and their impregnating solutions for the fire protection of wooden products, building materials and structures. A research methodology is proposed for the development of effective fire-extinguishing and fire-retardant agents for wood, based on three-component mixtures in which one of the components is a nitrogen-containing compound. Experimental findings show that a three-component mixture consisting of monoammonium phosphate, ammonium sulphate and carbamide, in appropriate proportions, provides highly effective fire protection for wood. In addition, a composition in the form of an epoxy polymer was developed, containing a mixture of ammonium phosphate and urea-formaldehyde resin, intended for use as a surface fire-retardant coating for wood.

Keywords: *firefighting powder, fire efficiency, gas chromatography, epoxy polymer, non-additivity.*

INTRODUCTION

Recent decades have seen a rise in fire hazards due to the widespread use of wood, synthetic, and polymeric materials in industry and daily life. This has increased the likelihood of fires and, consequently, led to a greater number of deaths and injuries from dangerous fire factors like high temperatures and toxic decomposition products released during combustion.

It is difficult to encounter a fire involving a single class; most incidents typically involve class A, B, and C fires. Among the fire loads represented by solid combustibles (Class A fires), wood is the most commonly encountered material.

An analysis of scientific, technical, and patent literature (Shkarabura et. al. 2002, Shkarabura et. al. 2003) indicates that most developments of fire-extinguishing powder compositions are based on improving formulations using already known and well-tested components. Intensive research is currently underway to determine the optimal composition of these powders to achieve maximum fire-extinguishing efficiency and versatility, which is reflected in the large number of existing formulations.

New formulations of fire-retardant agents are being developed based on two-component flame retardants. Research is underway to create new formulations aimed at both enhancing efficacy and reducing the cost of fire-extinguishing and fire-protective agents—objectives that cannot be fully achieved with the existing single- or two-component mixtures. This highlights the relevance of further scientific research aimed at developing new, more effective, and economically viable multicomponent mixtures of substances as fire-extinguishing and fire-retardant agents for wood.

1. MAIN RESULTS

To facilitate the development of efficient fire-extinguishing and fire-retardant agents, it is essential to investigate the theoretical framework underlying the preparation of three-component compound mixtures. To represent the composition (active basis) of three-component systems, it is appropriate to use an equilateral triangle (Figure 1). The vertices of the triangle ABC correspond to the pure components A, B, and C. The sides of the triangle represent the binary mixtures AB, BC, and AC. The points on the sides of the triangle ABC indicate the percentage content of components in the binary systems AB, BC, and AC. Any point inside the triangle corresponds to a defined composition of a three-component mixture. For example, point G represents a ternary composition abc (where a is the concentration of component A, b is the concentration of component B, and c is the concentration of component C). This method of constructing the composition of three-component systems is known as the Gibbs–Rosenbaum method.

Using the above-described method, we constructed an equilateral triangle whose sides are divided into percentages at 20% intervals (Figure 2).

Three-component systems are studied by first obtaining a composition diagram. The corresponding efficiency values are then plotted on perpendiculars drawn to the plane of the diagram. By connecting these points, an efficiency surface is generated.

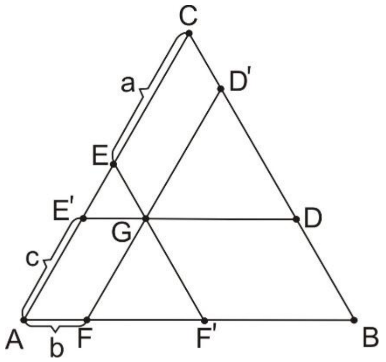


Figure 1 Three-component system

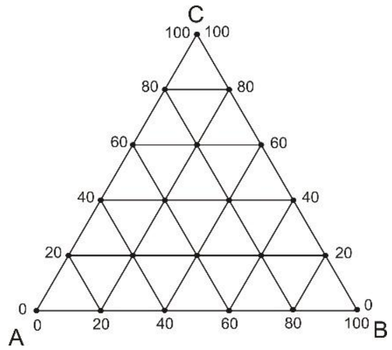


Figure 2 Gridded triangle for constructing three-component compositions

After determining the efficiency for each point, the data were entered into the Statsoft Statistica software matrix. This program was then used to construct the additive surface (Figure 3), the efficiency surface of the three-component mixture (Figure 4), a zone map (Figure 5), and a contour plot (Figure 6). Furthermore, the equation describing the dependence of efficiency on the mixture's composition was derived.

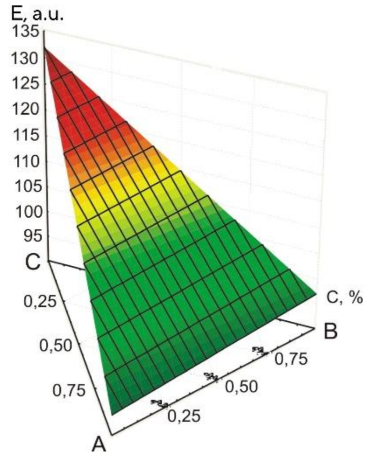


Figure 3 Additive efficiency surface of the three-component mixture

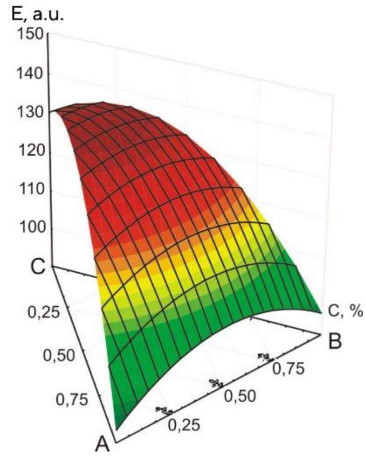


Figure 4 Efficiency surface of the three-component mixture

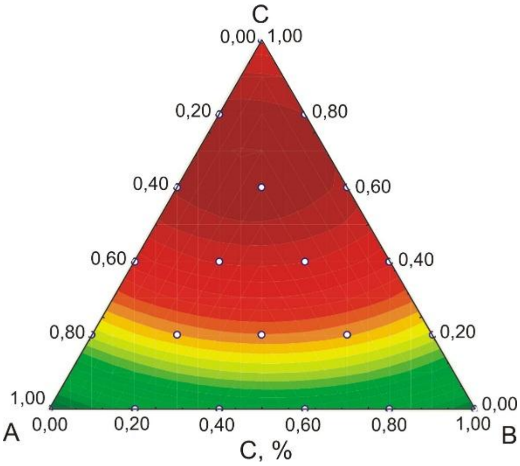


Figure 5 Zonal map of the efficiency surface of the three-component mixture

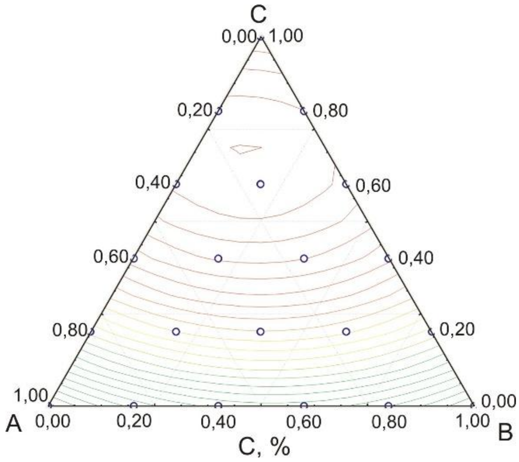


Figure 6 Contour plot of the efficiency surface of the three-component mixture

The darkest color on the zonal map indicates the maximum efficiency of the given mixture. By selecting the mixture concentrations within this zone, further studies can be conducted to determine the most optimal concentrations of the components in the mixture.

Experimental studies of the fire-extinguishing powders were carried out using a laboratory setup based on a Bunsen burner.
A mathematical calculation was performed to determine the dependence of the fire-extinguishing efficiency of powders on the initial temperature. The results showed a linear relationship:

$$E(t) = a_0 + a_1 \cdot t, \tag{1}$$

where t is the temperature in °C, E is the fire-extinguishing efficiency, a_0 is the free term, a_1 is the angular coefficient.

During the analysis of the experimental graphs, as described in the work (Maladyka, 2005), to determine the nature of the dependence of the slope coefficients on the melting temperature of the powders and to approximate this dependence using the corresponding function $f(t_m)$ the MathCAD program was applied. This program allowed the determination of the unknown coefficients of the function. As a result, the following function was obtained:

$$f(t_m) = 22 \cdot t_m{}^{-0,75} + 0.45, \tag{2}$$

where t_m is melting temperature of the powder in °C.

This function allows determining the slope coefficient a_1 for a fire-extinguishing powder if its melting temperature is known. Using the function $f(t_m)$, it is possible to determine the predicted increase in fire-extinguishing efficiency E_2 of a powder when preheated to a certain temperature t_2, if its fire-extinguishing efficiency E_1 at a lower temperature t_1 (ambient temperature) is known.

$$E_2 = E_1 + (22 \cdot t_m{}^{-0,75} + 0.45) \cdot (t_2 - t_1) \tag{3}$$

Calculations using this formula allow drawing conclusions regarding the feasibility of preheating the fire-extinguishing mixture.

Research was conducted on the effect of nitrogen-containing compounds (carbamide, urotropine, and diphenylamine) on the effectiveness of fire-extinguishing powders. The results showed a maximum increase in efficiency at a nitrogen compound content of about 1%. Similar results were obtained by adding 1% carbamide to two-component salt mixtures of monoammonium phosphate and ammonium sulfate in a 1:1 ratio, and diammonium phosphate and ammonium sulfate in a 1:1 ratio. These mixtures are used for wood impregnation. The obtained results indicate the possibility of increasing the effectiveness of existing fire-extinguishing powders and creating advanced fire-extinguishing and fire-protective compositions for wood.

A study was conducted to investigate three-component mixtures of inorganic salts that had previously shown a synergistic effect as two-component mixtures. The experiments demonstrated (Figure 7) that the effectiveness of the three-component mixtures was enhanced compared to their two-component counterparts.

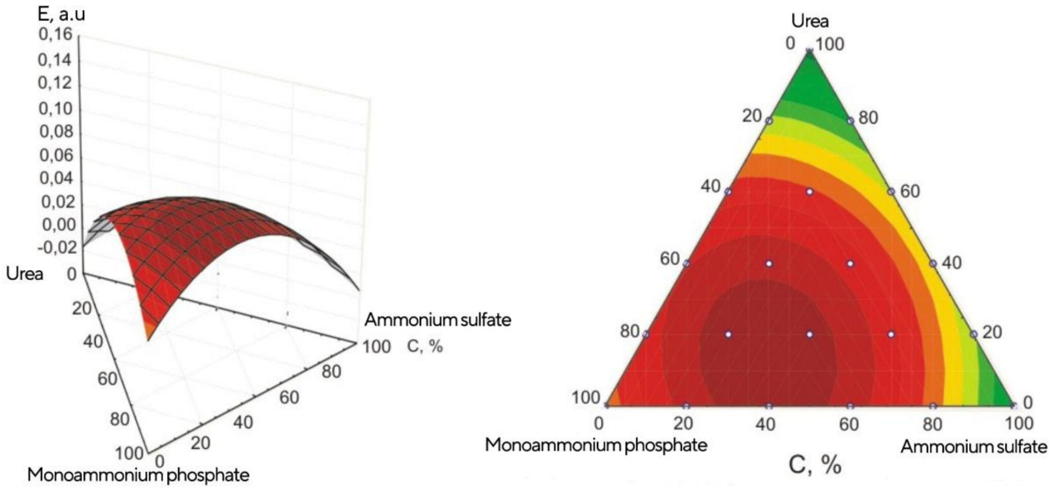


Figure 7 Results of determining the zone of maximum efficiency for a three-component mixture (monoammonium phosphate, ammonium sulfate, carbamide)

As can be seen from Figure 7, the zone of maximum efficiency is observed between points (80; 20; 0), (40; 60; 0), and (40; 20; 40), which form an equilateral triangle, and further research was continued inside it.

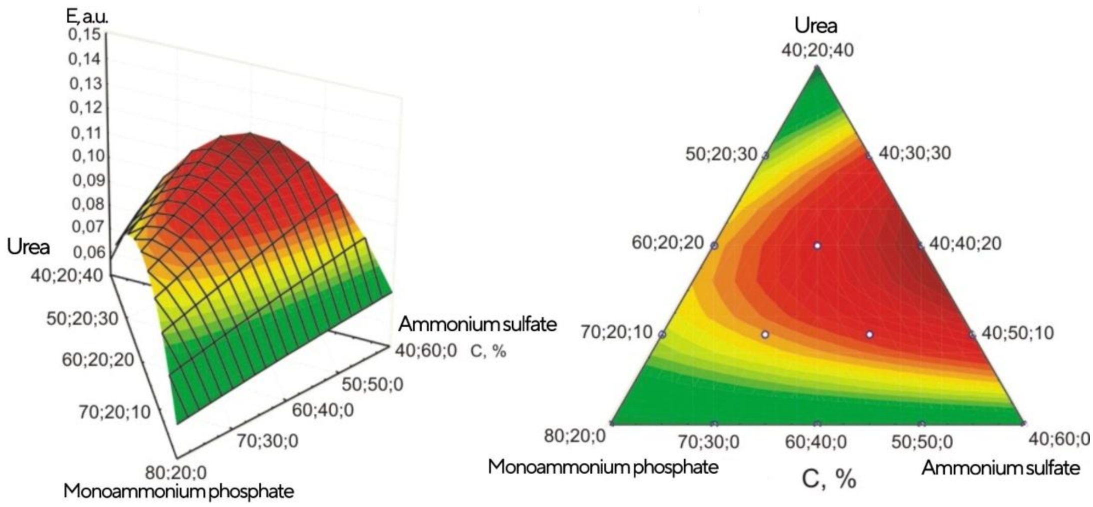
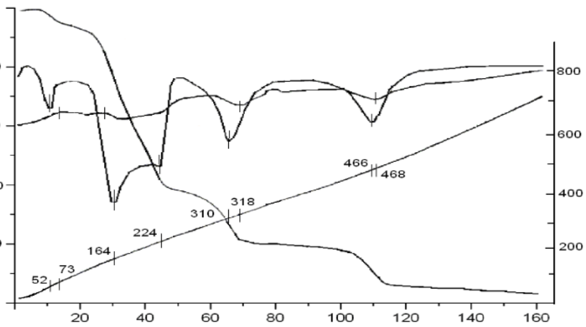


Figure 8 Results of determining the zone of maximum efficiency for a three-component mixture (monoammonium phosphate, ammonium sulfate, carbamide)

As the studies have shown, a mixture of the aforementioned salts in a ratio of monoammonium phosphate (40%), ammonium sulfate (40%), and carbamide (20%) yielded the best results for fire extinguishment.

To assess the feasibility of preparing impregnation solutions, a study was conducted using optical spectroscopy to investigate the inhibitory effect of aqueous solutions of ammonium sulfates, phosphates, and carbamide on an n-heptane flame. It was established that these solutions have a significant inhibitory effect on combustion processes and can be effectively used for the fire protection of cellulose-containing materials. Furthermore, it was found that at a carbamide mass concentration in the range of 15–25%, a synergistic effect is enhanced, which reduces the relative intensity of OH• radical emission by 24%. These properties are also preserved in a composition based on monoammonium phosphate, ammonium sulfate, and carbamide.

For the purpose of determining the temperature ranges at which the thermal decomposition of salts occurs most intensively, thermogravimetric studies of the thermal decomposition processes were carried out in a dynamic mode using a Q-1500 D derivatograph. The following mixtures were tested: mixture of diammonium phosphate (50%), ammonium sulfate (49%), and carbamide (1%); mixture of monoammonium phosphate (50%), ammonium sulfate (49%), and carbamide (1%); mixture of carbamide (50%) and monoammonium phosphate (50%); mixture of monoammonium phosphate (40%), ammonium sulfate (40%), and carbamide (20%). The results demonstrated that incorporating urea into the mixture increases the wood's heat absorption capacity. The most significant effect was observed in mixture 4, corresponding to the endothermic events at 164 °C (Figure 9).



Time, minutes

Figure 9 Thermogravimetric analysis curves of a mixture of monoammonium phosphate (40%), ammonium sulfate (40%), and urea (20%)

Thermogravimetric studies of the thermal degradation processes of pine sawdust samples were carried out, as well as of wood treated with fire-retardant agents based on ammonium phosphates and sulfates with the addition of carbamide, in an atmosphere of normal air composition (oxygen content – 21%)

The results of gas chromatographic studies on the pyrolysis of untreated and treated wood, using fire retardants based on monoammonium phosphate, ammonium sulfate, and carbamide (Table 1), show that the degradation products differ significantly in their nitrogen content and the amount of combustible gases.

Table 1 Qualitative and quantitative composition of gaseous products of wood thermal decomposition.

Component	Content of components in volatile decomposition products, %			
	Pine wood, untreated	Pine wood treated with diammonium phosphate (20 %) + ammonium sulfate (10 %)	Pine wood treated with monoammonium phosphate (15 %) + carbamide (15 %)	Pine wood treated with monoammonium phosphate (15 %) + ammonium sulfate (10 %) + carbamide (10 %)
CO	39.08	15.9	11.91	11.04
CO_2	51.93	Not detected	40.51	38.76
CH_4	6.05	0.58	0.19	0.16
C_2H_6 + C_2H_4	0.45	Not detected	Not detected	Not detected
C_3H_8	0.19	Not detected	0.07	Not detected
C_3H_6	0.32	Not detected	Not detected	Not detected
H_2	0.73	0.44	0.14	0.12
O_2	0.26	Not detected	0.44	Not detected
N_2	0.99	83.27	46.99	49.92

The average mass loss of samples treated with a mixture of monoammonium phosphate and carbamide was 8.9%, with a fire-retardant salt absorption rate of 54.0 kg/m³ (on a dry basis). The maximum temperature of the flue gases was 237 °C.

In order to evaluate the fire-retardant properties of wood treated with mixtures based on monoammonium phosphate and urea, as well as mixtures of monoammonium phosphate, ammonium sulfate, and urea, experimental investigations were carried out to determine the fire-protective effectiveness of the impregnation agents, following the methodology prescribed by GOST 16363.
The average mass loss for samples treated with a mixture of monoammonium phosphate, ammonium sulfate, and carbamide was 7.4%, with a fire-retardant salt absorption rate of 54.0 kg/m³ (on a dry basis). The maximum temperature of the flue gases was 213 °C.

Based on the mass loss values of the samples after fire tests, wood treated with the impregnating mixtures of monoammonium phosphate and carbamide, as well as monoammonium phosphate, ammonium sulfate, and carbamide, demonstrated high fire-retardant effectiveness.

To develop fire-retardant polymer coatings for wooden structures, it is necessary to investigate the flammability of filled epoxy polymers with the addition of flame retardants. The influence of a two-component fire retardant and intumescent additive system (monoammonium phosphate: carbamide and monoammonium phosphate: urea-formaldehyde resin) on the flammability of epoxy polymers was investigated using the "fire tube" method. The results showed that with the combined use of these fire retardants and additives, the materials transitioned into the group of non-combustible materials. The flammability of the filled epoxy polymers was assessed based on the oxygen index (OI) and resistance to a heated rod (heat resistance), which was used to calculate the mass loss of the polymer coating samples.

The results showed that the highest Oxygen Index (OI) value is achieved in compositions with a higher content of monoammonium phosphate. Regarding heat resistance, compositions containing a mixture of monoammonium phosphate with urea-formaldehyde resin are more resistant to a heated rod than those with a mixture of monoammonium phosphate and carbamide. Based on these findings, optimal compositions of epoxy composites were identified. These materials belong to the group of non-combustible materials and have an OI of 33-35%.

To determine the effect of selected fire retardants and additives on the thermo-oxidative degradation of an epoxy coating, thermogravimetric analysis was conducted on epoxy polymer samples containing monoammonium phosphate, urea, and urea-formaldehyde resin. The results indicate the optimal conditions for forming a fire-retardant coating for wood based on epoxy-polymeric binders (as reported in the work (Maladyka, 2006)).

To test the fire-retardant coating for wood based on epoxy-polymeric binders (Maladyka, 2006), experimental studies were conducted to determine its fire-retardant effectiveness in accordance with GOST 16363. The results of these studies are presented in Table 2.

Table 2 Results of the studies on determining the fire-retardant effectiveness of coatings

Composition of the fire-retardant coating	Protective layer thickness					
	0.5 mm		1 mm		1.5 mm	
	Average sample mass loss, g	Average sample mass loss, %	Average sample mass loss, g	Average sample mass loss, %	Average sample mass loss, g	Average sample mass loss, %
Carbamide	9.36	6.84	8.89	5.93	11.61	6.38
Urea-formaldehyde resin	19.51	13.82	17.05	11.96	19	11
Monoammonium phosphate, urea-formaldehyde resin	4.05	2.4	3.99	2.65	4.73	2.63
Monoammonium phosphate, carbamide	4.15	2.86	3.88	2.51	5.05	3.09

As shown by the research results, the coatings with mixtures of monoammonium phosphate and carbamide, and monoammonium phosphate and urea-formaldehyde resin, demonstrated the highest fire-retardant effectiveness.

CONCLUSIONS

The study confirms that two-component powder mixtures of inorganic salts can produce a synergistic or antagonistic effect, depending on their proportions. A synergistic effect was also found in two- and three-component fire-extinguishing powder mixtures when one of the components is an amine. Thermal analysis of multicomponent mixtures showed that a two-component mixture of monoammonium phosphate and ammonium sulfate is thermally stable up to 200 °C. In contrast, a three-component mixture of monoammonium phosphate, ammonium sulfate, and carbamide exhibits significant endothermic effects in the 120–165 °C temperature range. Furthermore, thermogravimetric analysis demonstrated that adding a mixture of monoammonium phosphate and urea-formaldehyde resin to epoxy polymers reduces the oxidation rate of the carbonized residue at 500–600 °C. Consequently, this blend is recommended as a more effective fire-protective coating for wood.

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