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SILICOPHOSPHATE FIREPROOF COATINGS FOR BUILDING MATERIALS

The composition of silicophosphate fire-resistant coatings for wooden building structures was developed and their properties were investigated. Fireproof compositions were obtained by mixing aqueous solutions of liquid glass and acetic acid. As a phosphate-containing additive, phosphate buffer solutions were used, which were added to the silicic acid sol in different amounts and with different ratios of the components of the buffer pair. Adjusting the ratio of the components of the buffer solution led to a change in the pH of the buffer solutions, but adding them to the sol did not change its acidity, which was in the pH range of 5, 5–6. The effect of the content and ratio of the components of the buffer pair on the change in the optical density of the obtained sols over time was studied. The highest durability of the flame retardant composition was recorded when using a buffer solution with a pH of 7 at a content of 20 %. The embedding of phosphate ions into the siloxane framework of experimental gels has been chemically proven, which increases their fire resistance. It is shown that the amount of free phosphate anion in the intermicellar liquid of the experimental gels is less than 5 %. The mechanism of the strengthening effect of the acetate buffer solution, which is formed during the mixing of the liquid glass solution with acetic acid, on the phosphate buffer solution is proposed. Fire-retardant compositions were applied to wood samples by the bath method and dried at temperatures of 80–100 °C in a drying cabinet. The fire protection effect of coatings was determined during fire tests in a ceramic pipe. The effect of the content of phosphate buffer solution on the fire-retardant properties of experimental coatings was studied. It is shown that increasing the content of the phosphate buffer solution reduces mass loss during fire tests, allows to increase the fire resistance of wood and transfer it to the group of «highly flammable».

Keywords: fire-resistant coatings, building materials, liquid glass, SiO₂ sol, phosphate buffer solution, fire resistance

1. Introduction

According to the new DBN B.1.2-7:2021, the problem of «fire safety engineering», which consists in the design and development of systems and measures aimed at preventing and limiting the spread of fires in buildings, becomes of key importance. Such a complex of actions necessarily includes the selection of fire-resistant building materials and their processing with special compositions for the purpose of forming fire-resistant coatings.

Almost any type of construction involves the use of wooden building structures. The wide use of wood is explained by its environmental friendliness, strength, ease of processing and good thermal insulation properties. However, according to flammability indicators, this material belongs to group 4, that is, under the action of an open fire, it ignites, supports combustion and burns almost completely, which can lead to the loss of material values and the death of people.

To reduce the flammability class, compositions based on flame retardants are used to impregnate and treat wood. Intumescent type fireproof coatings are quite promising today. As a rule, these are multi-component systems, the composition of which must necessarily include porophores – substances capable of decomposing when heated and

burning with the release of gaseous products that provide swelling (swelling). The main problem in the application of such coatings is ensuring the durability and preservation of the integrity of the intumescent layer on the surface of the protected substrate. After all, flame retardant components can be washed out of environmentally safe water-based coatings over time under the influence of adverse climatic factors. Increasing the adhesive strength and uniform distribution of the composition on the surface is usually achieved by introducing modifying additives, which often have quite high prices.

In view of the above, the search and development of relatively cheap and environmentally friendly fire retardant materials for wood are urgent.

2. Analysis of literary data and formulation of the problem

For effective fire protection of wood, as a rule, silicate and sol-gel coating methods are used. The silicate method consists in impregnating samples directly with a solution of a silicon-containing compound, most often liquid glass. In particular, in works [1–3], it was established that the treatment of wooden samples with a solution of sodium silicate contributes to an increase in fire resistance, a reduction in the time of burning and ignition was recorded, and an improvement in the mechanical and thermal insulation properties of wood was also observed. Just as the formation of a coating exclusively on a pure inorganic silicate base is often accompanied by uneven distribution on the surface of the sample, unsatisfactory adhesion characteristics, lack of resistance to climatic conditions, and as a result – cracking and delamination, in works [4, 5] organic modifying additives were used: isocyanates [1, 2] and acrylates [3–5]. It was experimentally established that the addition of these substances to the compositions makes it possible to provide more effective fire resistance indicators and improve the surface interaction between the coating and the base. The above-mentioned works are of great practical and theoretical importance, but one should take into account the fact that the proposed organic modifying additives can pose a threat to human life and the environment.

Today, the sol-gel method is a promising method for obtaining silicate coatings, as it allows for improved resistance to atmospheric influences, adhesion, and increased strength compared to silicate coatings [6, 7]. In most of the works devoted to fire-resistant coatings of wood, silica sols are obtained on the basis of organosilanes with simultaneous modification with inorganic components. For example, in work [8] solutions of tetraethoxysilane and hydrochloric acid were used as precursors, and the synergistic effect of potassium carbonate was also investigated. K_2CO_3 contributed to the ignition of the wood sample at lower temperatures, and the sol formed a compact and molten barrier on the surface, which prevented the transfer of heat and combustible gases in the condensed phase. The authors of the paper [9] conducted a study of fire-retardant properties and kinetics of thermal decomposition of wood treated with silicic acid sol modified with boric acid. The sol was also obtained on the basis of tetraethoxysilane. It was found that the addition of H_3BO_3 contributed to a significant improvement in fire resistance compared to wood treated with unmodified sol. Undoubtedly, the results of works [8, 9] deserve attention, but it should be taken into account that organosilanes are expensive and often less environmentally safe than inorganic analogues.

Silicic acid sols are usually stable in strongly acidic and strongly alkaline environments, but the treatment of samples with such compositions can cause a weakening of the physical and mechanical properties of wood [10]. It was established that in order to obtain sols with high fluidity in an environment close to neutral, it is advisable to use

buffer systems. In particular, it was previously investigated [11] that acetic acid, which is added to the liquid glass solution for the formation of silica sol, contributes to the formation of the buffer pair $\text{CH}_3\text{COOH} / \text{CH}_3\text{COO}^-$, ensures the maintenance of pH in the range of 5–6. This value of the hydrogen index is satisfactory for obtaining more durable SiO_2 sols of low concentration.

It is known that phosphorus- and nitrogen-containing compounds demonstrate good flame retardant properties. The mutual positive influence of nano- SiO_2 airtel and ammonium phosphate on the fire resistance of wood was evaluated by the authors of the paper [12], and the ability of phosphorous flame retardant to be embedded in the structure of the siloxane frame, which prevented excessive leaching, was noted.

Taking into account the rationality of using buffer solutions in the studied systems and improving the fire resistance of coatings in the presence of Phosphorus, it would be expedient to consider the effect of phosphate buffer systems on the properties of silica sols and analyze the problem of developing the composition of a flame retardant composition based on them.

3. The purpose and tasks of the research

The purpose of the work is to determine and justify the optimal composition of silicophosphate composition as a basis for fire-resistant coating of wooden building structures.

To achieve the goal, it was necessary to complete the following tasks:

- to investigate the effect of the content and composition of the phosphate buffer solution on the physical and chemical properties of silicic acid sols;
- to determine the influence of silicon phosphate coating on the fire resistance of wood samples.

4. Research materials and methods

The object of research is fire protection of wooden building structures.

The subject of research are the processes of transformation of fire-resistant coatings during the action of fire.

The main hypothesis of the research is the possibility of incorporating phosphate ions into the siloxane framework of the flame retardant composition, which leads to an increase in the fire resistance of siliceous coatings.

Silicic acid sol was obtained using solutions of liquid glass and acetic acid. Phosphate buffer solutions with pH 6, 7 and 8 prepared by mixing NaH_2PO_4 and Na_2HPO_4 solutions were used as modifying additives, while the volume fraction of the buffer solution was varied.

To determine the content of phosphates embedded in the gel structure, a photometric method was used using a molybdenum mixture (solutions of ammonium molybdate, stibium (III) chloride, sulfanilic and tartaric acids) in the presence of ascorbic acid. Changes in the optical density of sols over time were determined using a KFC-2 photocolormeter. A solution of molybdenum mixture and ascorbic acid was used as a comparison solution.

Scots pine wood samples with dimensions of 9x6x3 cm were pre-dried in a drying cabinet at 100 °C to a constant mass. Silica sol, modified with phosphate buffer solutions, was applied to the wood surface using a bath method and dried in an oven at 80 °C. The coated samples were additionally treated with a solution of ammonium hydrogen phosphate by spraying and dried again.

5. Study of the influence of the content of phosphate buffer solution on the properties of experimental sols

The dependence of the change in the optical density of the experimental silicic acid sols (obtained with the help of acetic acid) over time on pH was studied for compositions with a phosphate buffer solution content of 15, 20 and 25 % by volume. All three dependencies had an identical character, and the curves practically overlapped each other. Fig. 1 shows changes in the optical density of sols over time depending on the pH of the buffer solution with a volume fraction of 20 %.

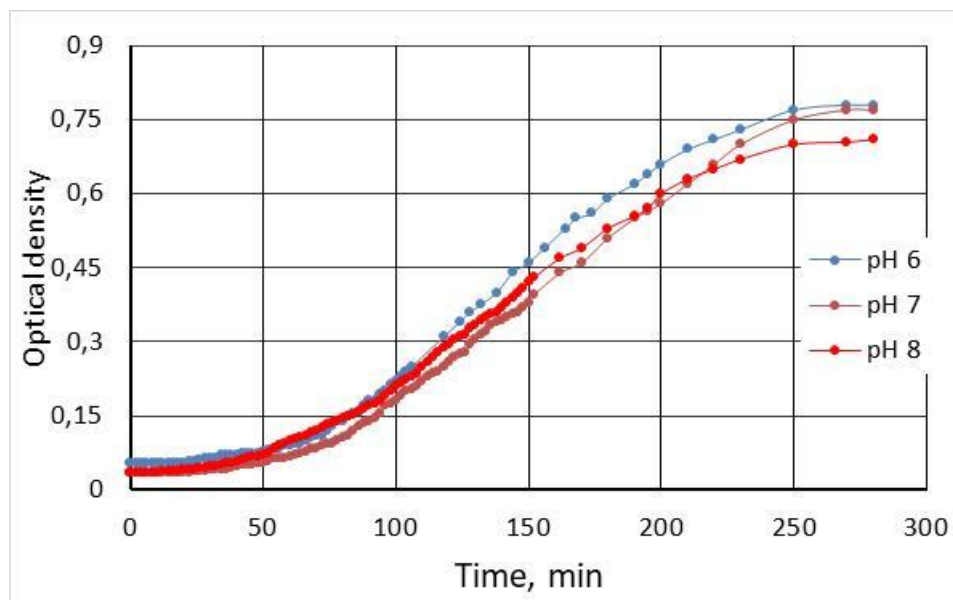


Fig. 1. The dependence of the change in the optical density of the experimental silicic acid sols over time on the pH of the phosphate buffer solution

Analysis of the obtained dependencies indicates an increase in the survival time of sols compared to systems with orthophosphate acid additives, which were studied earlier [13]. The value of the optical density at which the sols lost their fluidity remained practically unchanged regardless of the pH and the content of the buffer solution.

Fig. 2 presents the dependences of latent coagulation time and sol viability on the buffer solution content.

Fig. 2 shows that the latent coagulation time was the lowest for sols with buffer solution additives with a pH of 6, while it is possible to note the growth of the parameter with an increase in the buffer solution content. For compositions with pH 7 and 8, this characteristic was practically the same.

In order to establish how completely phosphate ions are incorporated into the siloxane framework, the difference between the content of phosphate added with the buffer solution and the mass fraction of PO_4^{3-} ions in the intermicellar liquid, which is released during the aging of the sol and transition to a gel, was determined (Tabl. 1).

Tabl. 1. Results of determining the mass fraction of phosphate ions that were incorporated into the siloxane framework of gels

№	pH of the buffer solution added to the composition	pH of the intermicellar fluid	w(PO_4^{3-}), which is embedded in the siloxane framework, %
1	6	5	97,3
2	7	5	96,6
3	8	5	98

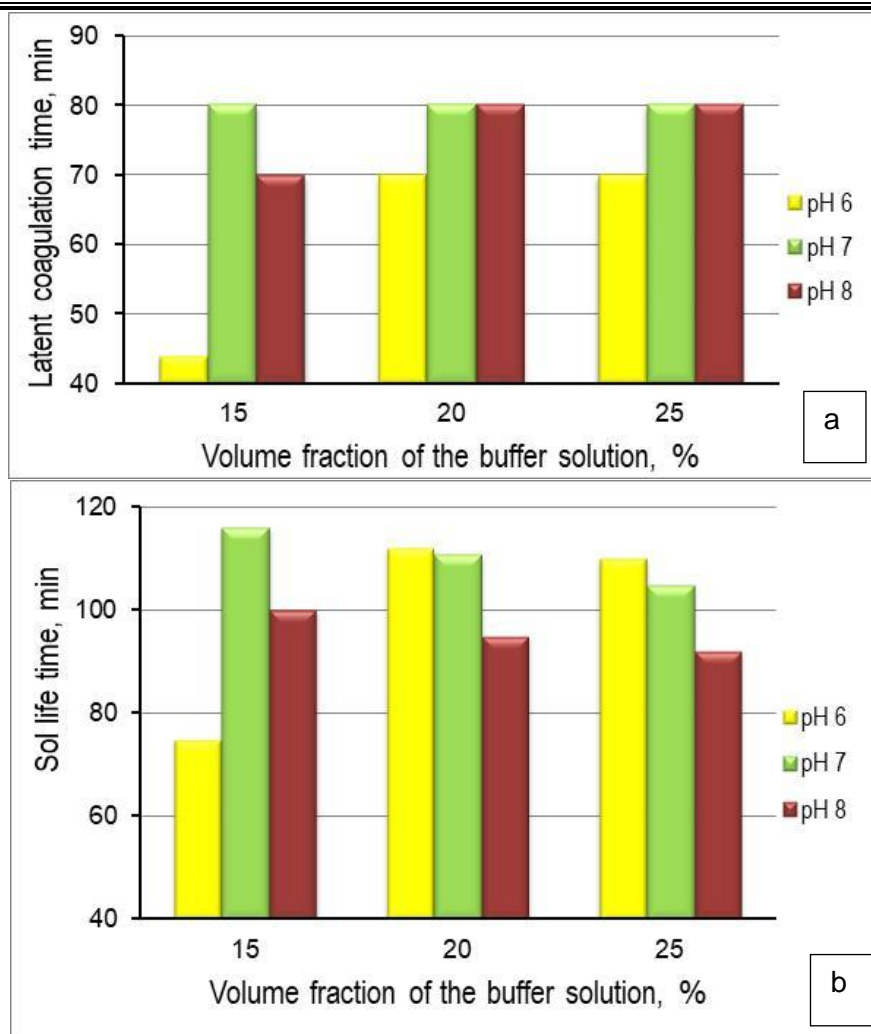
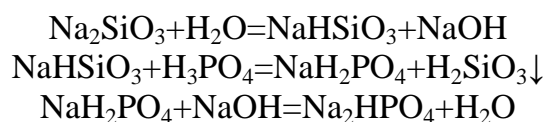


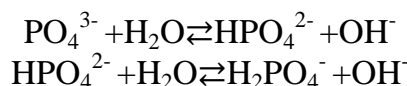
Fig. 2. Dependence of latent coagulation time (a) and sol viability (b) on the volume fraction of the buffer solution

The results of the experiment indicate that phosphate ions are almost completely embedded in the siloxane framework. On the other hand, clathrates are formed in the structure of the gel coating, where both water molecules and phosphate ions can be retained. The pH of the intermicellar fluid remained constant regardless of the pH value of the added buffer solution. This can be explained by the higher buffer capacity of the acetate buffer solution (pH 5 is in the interval of its buffering effect), which is formed when adding acetic acid to the composition, compared to phosphate.

It is known that orthophosphate acid can also be used to obtain SiO_2 sol. At the same time, processes with the formation of a phosphate buffer solution can occur in the «liquid glass – H_3PO_4 » system, which confirms the following reactions:



In addition, it can be assumed that the orthophosphate ion undergoes hydrolysis, also forming a buffer pair of hydrogen phosphate-dihydrophosphate:



On the other hand, if orthophosphate acid is added to the acetate buffer solution as a modifying additive, the formation of two buffer solutions can be considered, the joint action of which is shown in Fig. 3.

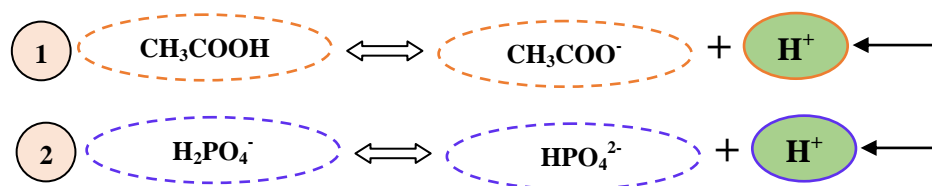


Fig. 3. Scheme of joint action of acetate and phosphate buffer systems

Equilibrium processes 1 and 2 are closely related: when the concentration of acetic acid increases, the equilibrium shifts in process (1) to the right, and in reaction (2) to the left. That is, it can be stated that when the concentration of CH₃COO⁻ ions increases, the concentration of HPO₄²⁻ decreases. But the dissociation constants of these processes should also be taken into account:

$$K_a(1) = 1,8 \cdot 10^{-5} > K_a(2) = 6,2 \cdot 10^{-8}$$

The dissociation constant of acetic acid is almost 300 times higher compared to the dissociation constant of the dihydrophosphate ion, so the CH₃COO⁻/CH₃COOH system exerts a greater buffering effect and at the same time strengthens the effect of the phosphate buffer solution.

6. Study of the effect of silicon phosphate coating on the fire resistance of wood samples

The results of fire tests of wood samples with a coating applied in three layers are presented in Fig. 4.

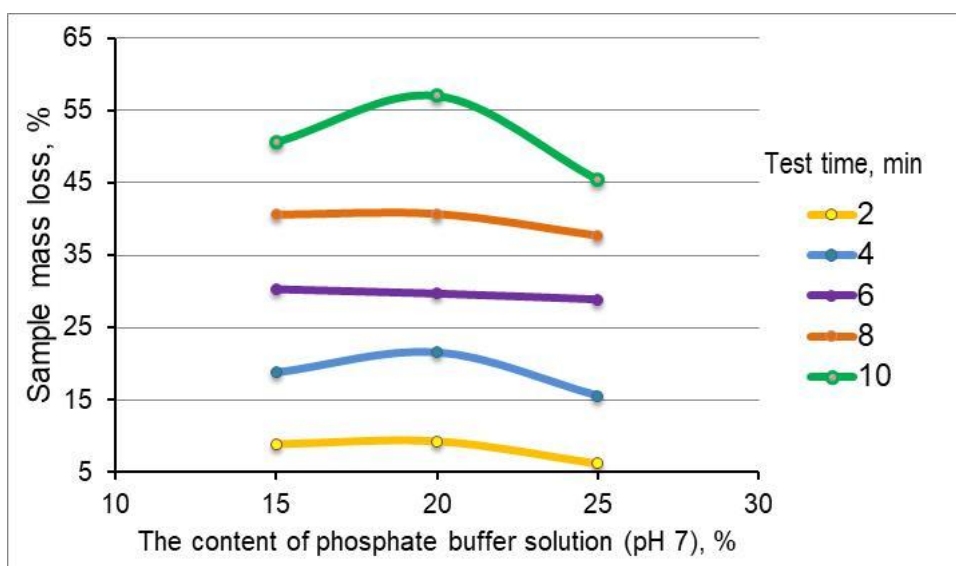


Fig. 4. Influence of the content of the phosphate buffer solution with pH 7 and the duration of the fire test on the weight loss of samples with a three-layer coating

Fig. 4 shows a decrease in the weight loss of the samples with an increase in the phosphate buffer solution additive content. Regardless of the duration of the fire test, there is a slight decrease in the mass loss of the samples in the case of adding 25 % of

the buffer solution.

Fig. 5 shows the change in mass and temperature of a sample with a three-layer coating based on a composition with a 25 % phosphate buffer solution with a pH of 7. The graph clearly shows a periodic decrease in temperature, which corresponds to the gradual decomposition of the gel coating under the action of fire.

Tabl. 2 shows the results of studies on the determination of the group of fire-resistant effectiveness of the coating.

The results of studies on the determination of the flammability group of wood samples with a fire-resistant coating with the addition of 25 % phosphate buffer solution are shown in the Tabl. 3.

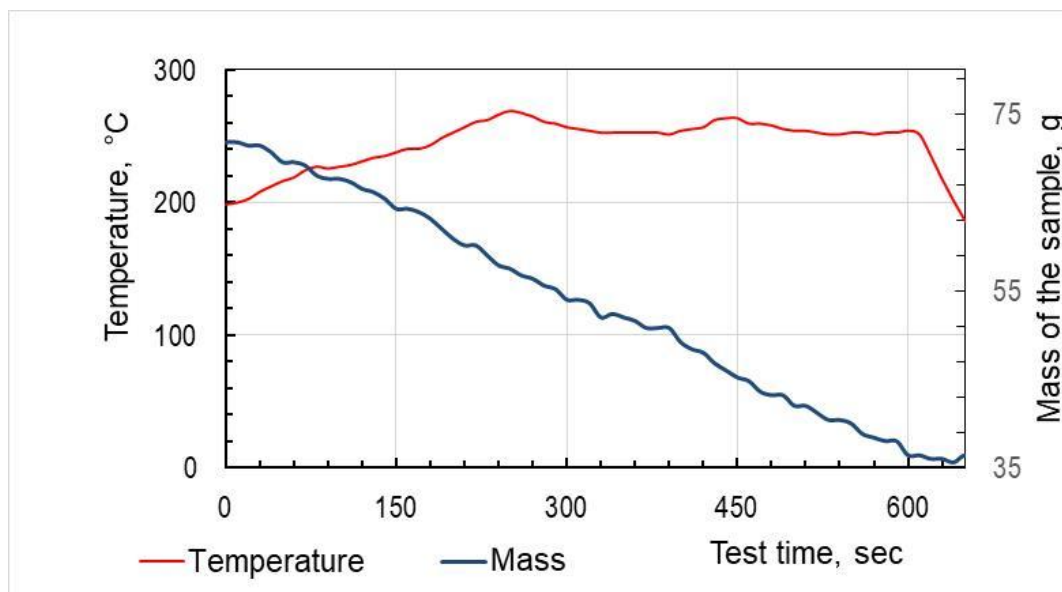


Fig. 5. The nature of the change in mass and temperature of the sample with a coating based on a composition with a 25 % phosphate buffer solution with a pH of 7

Tabl. 2. Results of determination of the group of fire-resistant efficiency of experimental coatings on wood

Number of coating layers	Mass of the sample		Sample mass loss, %	Group of fire protection efficiency of the coating
	to the tests,	after 2 min of testing, g		
1	71,83	66,17	7,9	I
3	72,58	68,06	6,2	I

The mass loss values of samples with one- and three-layer coating do not exceed 9 %, i.e., the first group of fire-resistant coating efficiency is established according to [15].

According to [16], wood covered with one layer of flame retardant composition belongs to non-flammable materials, since the maximum increase in the temperature of gaseous combustion products is $<60\text{ }^{\circ}\text{C}$ and the loss of sample weight is less than 60 %. The sample covered with three layers of sol is combustible because $\Delta t_{\text{max}} > 60\text{ }^{\circ}\text{C}$ and belongs to nonflammable materials because the time to reach $\Delta t_{\text{max}} > 240\text{ s}$. Therefore, based on the calculations, better fire resistance is provided by a single-layer coating.

7. Discussion of the results of the study of the influence of phosphate buffer systems on the properties of sols

The addition of orthophosphate acid, even in small quantities and at any degree of dilution, leads to instant coagulation in the silicic acid sol. But in the case of adding

H₃PO₄ to a mixture of liquid glass and acetic acid, coagulation does not occur for the reasons given in the hydrolysis reactions of the orthophosphate ion. The same reactions accompany the processes of silicophosphate coating formation in the case of adding a phosphate buffer solution. Despite the fact that the pH of the buffer solutions was changed from 6 to 8 by changing the ratio of the components of these solutions, after their addition to the mixture of liquid glass and acetic acid, the pH of the composition was 5. A weakly acidic environment significantly reduces the viability of sols, but due to the fact that phosphate buffer solutions are diluted, when their content is increased, some dilution of the composition also occurs, which increases its survivability, which is illustrated in Fig. 1 and Fig. 2. Analysis of the curves in Fig. 1 allowed us to establish that the highest fluidity and the longest latent coagulation time were characteristic of the sol with the additive buffer solution with pH 7.

Tabl. 3. Results of studies on the determination of the flammability group of wood samples with a fire-resistant coating

Number of coating layers	Temperature of gaseous combustion products, °C		Δt_{\max} , °C	The time interval until the maximum temperature is reached	Mass of the sample		Sample mass loss, %	Flammability
	primary	maximum			to the tests, g	after testing, g		
1	200	255	55	300	71,83	54,00	24,8	heavy-flammable
3	196	269	73	300	72,58	57,88	20,3	flammable, highly flammable

In this case, it is possible to consider the joint action of the acetate and phosphate buffer systems in the system (Fig. 3). At the same time, the acetate buffer solution will enhance the effect of phosphate.

The controlled content of H⁺ ions by the action of a complex acetate-phosphate buffer solution leads to the initiation of net polycondensation in the silicic acid sol and, as a result, to the formation of clathrates in the structure of the gel coating. Such a coating is able to decompose in a wide temperature range. It should also be taken into account that the water content in the compositions increases, which, evaporating, also provides local cooling of the surface of the gel coating. Thus, the greater the content of the phosphate buffer solution, the smaller will be the mass loss of samples during fire tests (Fig. 4). If several layers of coating are applied to the surface of wood samples, under the action of fire, graded thermal destruction of each layer of coating will occur, which is illustrated in Fig. 5: the temperature of the sample periodically decreased during the fire tests. The gradual removal of water vapor from the surface of the coating at temperatures of 200–300 °C leads to an increase in the porosity of the coating, which additionally thermally insulates the surface of the wood. The assumptions made are confirmed by the results presented in Tabl. 2 and Tabl. 3. The presence of clathrates in the structure of the gel coating, formed due to the presence of a phosphate buffer solution, increases the fire resistance of wood samples with an experimental coating and brings it to the 1st group of fire-retardant efficiency. Depending on the degree of homogeneity of the gel coating and the number of applied layers, the samples can be either heavy-flammable or highly flammable.

Further research involves studying the structure of a sol-based coating with a defined composition on the surface of building materials, in particular wood and extruded polystyrene.

8. Conclusions

1. The effect of the content and composition of phosphate buffer solutions on the physical and chemical properties of silicic acid sols was studied. The highest durability of the flame retardant composition was recorded when using a buffer solution with a pH of 7 at a content of 20 %. Using a chemical method, it was established that phosphate ions are almost completely incorporated into the siloxane framework under the conditions of adding a phosphate buffer pair. In the case of adding orthophosphoric acid as a phosphate-containing additive, a phosphate buffer solution is also formed, the effect of which is enhanced by the presence of an acetic buffer solution, which is formed during the production of SiO₂ sol from liquid glass. The mechanism of enhancing effect of acetate buffer solution on phosphate buffer pair is proposed.

2. Theoretically justified and experimentally proven fire protection effect of the developed coatings on wood. It is shown that the complex action of two buffer solutions – acetate and phosphate – manifests itself in the initiation of net polycondensation in silicic acid sol and, as a result, in the formation of clathrates in the structure of the gel coating, which gradually decompose under the influence of fire with an endothermic effect. Coating with compositions based on silicic acid sol, modified with a phosphate buffer solution allows you to transfer the wood to the "high-burning" group. The set composition of the composition provides the I group of fire-resistant coating efficiency.

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СИЛІКОФОСФАТНІ ВОГНЕЗАХИСНІ ПОКРИТТЯ ДЛЯ БУДІВЕЛЬНИХ МАТЕРІАЛІВ

Розроблено склад силікофосфатних вогнезахисних покриттів для дерев'яних будівельних конструкцій та досліджено їх властивості. Вогнезахисні композиції отримували змішуванням водних розчинів рідкого скла та оцтової кислоти. Як фосфатовмісну добавку використовували фосфатні буферні розчини, які додавали до золю кремнієвої кислоти в різній кількості та з різним співвідношенням компонентів буферної пари. Коригування співвідношення компонентів буферного розчину призводило до зміни рН буферних розчинів, але додавання їх до золю не змінювало його кислотність що знаходилася в інтервалі рН 5,5–6. Досліджували вплив кількості та співвідношення компонентів буферної пари на зміну оптичної густини отриманих золів в часі. Найбільша живучість вогнезахисної композиції зафіксована при використанні буферного розчину з рН 7 при вмісті 20 %. Хімічним шляхом доведено вбудовування фосфат-іонів в силоксановий каркас експериментальних гелів, що забезпечує підвищення їх вогнестійкості. Показано, що в інтерміцелярній рідині експериментальних гелів заходиться вільний фосфат-аніон у кількості менше 5 %. Запропоновано механізм підсилювальної дії ацетатного буферного розчину, який утворюється під час змішування розчину рідкого скла з оцтовою кислотою, на фосфатний буферний розчин. Вогнезахисні композиції наносили на зразки деревини ванним методом та сушили за температур 80–100 °С в сушильній шафі. Вогнезахисну дію покриттів визначали під час проведення вогневих досліджень в керамічній трубі. Досліджено вплив вмісту фосфатного буферного розчину на вогнезахисні властивості експериментальних покриттів. Показано, що збільшення вмісту фосфатного буферного розчину знижує втрати маси під час вогневих досліджень, дозволяє підвищити вогнестійкість деревини та перевести її до групи «важкогорючі».

Ключові слова: вогнезахисні покриття, будівельні матеріали, рідке скло, золь SiO₂, фосфатний буферний розчин, вогнестійкість

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