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TECHNOLOGICAL FEATURES OF OBTAINING COMPLEX SILICA SOLS FOR FIRE PROTECTION OF BUILDING FINISHING MATERIALS

The influence of the technology for obtaining fire-retardant compositions based on liquid glass for fire protection of building finishing materials was studied. The influence of the silicate module of liquid glass on the rheological properties of silicic acid sols modified with phosphate-containing compounds was studied. According to the results of spectrophotometric measurements, it was established that the ratio $n(SiO_2)/n(Na_2O)$ in liquid glass within 2.5–3 does not significantly affect the survivability of the compositions. The influence of the ratio of the initial components on the duration of solidification of the sols was determined. It was established that the preliminary introduction of 0.1 wt. % Trilon B into tap water allows obtaining stable silicic acid sols over time, which is a prerequisite for the formation of a homogeneous fire-retardant coating. Fire tests were carried out on samples of wood and extruded polystyrene foam coated with compositions of the studied composition. It was found that the content of 2 % orthophosphoric acid and 0.1 % sodium hexametaphosphate provides mass loss of wood samples less than 7.5 %, which corresponds to the I group of fire-retardant efficiency of coatings, and the protected material belongs to the group of low-flammability. For extruded polystyrene foam, the best fire-retardant effect was demonstrated by compositions with a sodium hexametaphosphate content of 1 %: mass loss of samples varied within 1–3 %, burning drops were not formed, and the samples did not support combustion. It is assumed that the increased fire resistance of coatings with a higher content of phosphorus-containing additive is associated with the fusibility of sodium compounds and their ability to transfer the fire-retardant coating to a visco-plastic state, which contributes to the dissipation of deformation stresses and prevents the formation of cracks in the coating.

Keywords: fire-retardant silica-containing coatings, silicophosphate coatings, modifying phosphorus-containing additives, building materials, heat resistance, fire resistance, extruded polystyrene foam

1. Introduction

Modern approaches to building design are increasingly focused on ensuring high energy efficiency. In order to reduce heat loss through the external enclosing structures of buildings, thermal insulation materials are now widely used. They are most often used in facade systems of buildings – both during new construction and during the reconstruction of existing facilities. However, energy-efficient optimization often occurs without taking into account fire safety aspects, which creates potential threats to the safe operation of buildings.

Inadequate level of fire safety control of facade systems made of combustible materials has repeatedly become the cause of large-scale incidents. One of the most striking evidence that design solutions without taking into account the requirements for fire resistance of building materials can have catastrophic consequences was the fire in the 24-story building "Grenfell Tower" (London, 2017), which led to the death of at least 70 people and the hospitalization of at least 74 victims. The data of researchers [1, 2], who performed the analysis of this emergency situation, indicate the critical role of the facade system in the dynamics of fire development. The presence of flammable thermal insulation materials, in particular polyisocyanurate and a polyethylene layer in the cladding, created conditions for rapid vertical and horizontal flame spread along the outer surface of the building. This event highlighted the need for a comprehensive approach to the selection of facade building materials, with a special emphasis on their behavior under the influence of high temperatures. Among the materials widely used for the purpose of exterior decoration of buildings, the leading positions are currently occupied by extruded polystyrene foam (XPS) and wood. One of their key advantages is good thermal insulation properties. XPS is also valued for its lightness and economy, wood attracts with its environmental friendliness and aesthetic expressiveness. At the same time, both materials are characterized by a high level of flammability, which requires the implementation of effective fire-fighting solutions, especially when used in facade systems.

One of the promising approaches to ensuring the fire resistance of such materials is the use of silica-containing compounds capable of forming heat-resistant barrier layers that reduce the intensity of combustion and prevent the spread of flame. It should be noted that when developing such systems, it is especially important to take into account technological aspects, in particular the choice of raw materials, the features of the composition preparation, the conditions for its application, etc. The developed technology must be not only effective and efficient in terms of fire protection, but also suitable for use in real production conditions – with predictable properties and the ability to scale without loss of quality.

Therefore, within the framework of solving the important scientific problem of increasing the fire resistance of finishing building materials, the issue of finding and developing a simple, economically feasible and technologically suitable fire protection system for building materials based on silicophosphate compositions is relevant.

2. Analysis of literary data and problem statement

In recent decades, there has been an active development of research in the field of silicon-containing flame retardant systems, which is associated with their ability, due to their unique physicochemical properties, to significantly surpass traditional flame retardants. Such compounds have high thermal and thermo-oxidative stability, do not emit toxic or corrosive gases during combustion. The main mechanism of action of silicon-containing flame retardants is the formation of a protective layer in the condensed phase, which prevents oxygen from reaching the material being protected and slows down heat transfer [3].

Quite often, the object of study in the context of the problem is organic silicon-containing compounds. For example, in [4], a silicone-modified thermoplastic polyurethane (TPU) was synthesized. The addition of polydimethylsiloxane (PDMS) contributed to the formation of a fine three-phase microstructure, a decrease in the rate of thermal decomposition and an increase in elongation at break by 173.8 %. The modified TPU also demonstrated improved fire resistance (LOI=27), a decrease in heat release by more than half and suppression of smoke formation were observed. In [5], polydimethylsiloxane was also used as part of a fire protection system for expanded polystyrene. The addition of 4 wt. % of the flame retardant provided UL-94 V-0, LOI 26 %, and also made it possible to maintain the fire resistance of the material after exposure to water due to the hydrophobicity of siloxane. The object of the study [6] was siloxane-silsesquioxane resins used to increase the fire resistance of polypropylene.

The thermal stability of the composites was evaluated using thermogravimetric analysis and cone calorimetry. The results showed that even a small amount of functional resins contributes to the formation of a protective ceramic layer during combustion, which improves the thermal stability of polypropylene composites. Also noteworthy are the results of work [7], in which the authors synthesized a flame retardant by imidization of aminopolysiloxane using maleopimaric acid, which inhibits the conversion of polysiloxane to cyclic siloxane, thereby increasing thermal stability. The synthesized flame retardant material was used as part of the soft segment to obtain rigid polyurethane foam. The peak heat release rate was reduced by more than 50 %, and the oxygen index was increased by 39.7 %, compared to pure foam. The compressive strength was also improved.

In some studies, in order to increase the efficiency of fire protection and ensure better compatibility with the polymer matrix, combined systems obtained by modifying silicate compositions with phosphorus, nitrogen or boron compounds were studied. In particular, in [8] a new phosphonate flame retardant based on γ-aminopropyltriethoxysilane was proposed for thermoplastic polyurethane foam, which simultaneously increased fire resistance and preserved the mechanical properties of the material. The modified sample passed the UL-94 V-0 test and had a limiting oxygen index of 28.5 %, which corresponded to the non-flammable level. The studies in [8] were conducted only for one flame retardant concentration value – 5 wt. %, which does not allow to fully assess the relationship between the amount of the additive and its fire-retardant properties, as well as to determine the efficiency limit. Despite the proven efficiency, most of the considered organic silicon-containing flame retardants [4–8] have limited potential for practical application due to the high cost of synthesis. In addition, given the potential environmental burden, it is advisable to conduct additional studies on the biodegradability and toxicity of thermal degradation products of the proposed flame retardants.

Taking into account the above, the attention of researchers is increasingly attracted to inorganic silicon-containing compounds. In a number of scientific works, silica is considered as a key component of hybrid fire-retardant systems. For example, in [9], a nanocomposite paper based on silica aerogel and hydroxyapatite was developed, which was characterized by exceptional thermal stability (sample mass loss was <10 wt. % even at 800 °C) and good mechanical characteristics.

One of the most studied approaches to the synthesis of silica is the sol-gel method, as it allows the creation of materials with controlled structural characteristics, which is critically important for the formation of effective fire-retardant systems. In particular, in [10] a method of hybrid treatment of wood with silica sol modified with a surfactant was proposed. The treated samples demonstrated increased fire resistance, a decrease in the rate of heat release, smoke release, and a decrease in ignition and burnout times were recorded. The authors of [11] developed a fire-retardant coating for cotton fabrics based on silica sol, phytic acid, and sodium alginate, which increased the thermal stability of the material, reduced smoke and heat release, and also contributed to the formation of a carbon layer. Despite the scientific significance of the results obtained, work [10] lacks research results on the behavior of treated wood in real operating conditions, in particular, the impact of ultraviolet radiation, moisture, and mechanical loads is not assessed. In study [11], consumer properties of the fabric, such as softness and breathability, are insufficiently studied.

It is also worth noting that despite the excellent functional properties of such fire-retardant systems, the high cost of the components [9] and the large number of components [11] may cast doubt on the economic feasibility of their implementation in large-scale production.

In previous studies, silica-containing fire-retardant coatings were obtained on the basis of liquid glass [12–14]. Taking into account the synergism of silicon and phosphorus compounds, orthophosphoric acid, sodium hexametaphosphate and phosphate buffer solutions were used as modifying additives. The obtained coatings demonstrated high efficiency in increasing the fire resistance of wood and extruded polystyrene foam. At the same time, it should be taken into account that the implementation in the production process requires a deeper understanding of the technological features of the process of synthesis of the fire-retardant coating, in particular the influence of the quality and degree of purity of the starting components. In this context, it is important to consider the influence of the silicate module of liquid glass on the quality of fire-retardant coatings. A number of works [15–17] note that such a parameter can affect the rheological properties of the silicic acid sol as well as the morphological and structural characteristics of the resulting gel.

It is also expedient and rational from a practical point of view to use tap water, but under conditions of controlling its hardness, because the presence of Ca^{2+} and Mg^{2+} ions can cause coagulation and premature gelation. It can be assumed that this effect can be neutralized by using a complexing agent that can bind metal ions [18], but at the same time significantly affect the kinetics of gelation.

In view of this, it would be advisable to investigate the influence of the silicate module of liquid glass and the complexon – disodium salt of ethylenediaminetetraacetic acid (Trilon B) on the properties of silica-containing fire-retardant compositions for wood and extruded polystyrene foam.

3. Purpose and objectives of the study

The purpose of the work is to study the influence of technological features of obtaining silicic acid sols on the formation of the structure and fire-retardant properties of coatings based on liquid glass.

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

– to investigate the rheological properties of silicic acid sols with different contents of disodium salt of ethylenediaminetetraacetic acid (Trilon B), obtained on the basis of liquid glass with different values of the silicate modulus, and the gelation processes in them;

– to conduct fire tests of samples of wood and extruded polystyrene foam to assess the effectiveness of the fire-retardant silicophosphate coating.

4. Materials and research methods

The object of research is the technology of increasing the fire resistance of finishing building materials made of wood and extruded polystyrene foam.

The subject of research is the physicochemical processes that occur in fire-retardant coatings during fire exposure.

The main hypothesis of the research is the possibility of using technical water for the preparation of fire-retardant compositions by adding a complexing agent to prevent coagulation of the sol-gel composition and ensure its stable fluidity.

To prepare the experimental compositions, solutions of liquid glass (silicate module 2.5 and 2.88 – manufacturer "Nova-Khim") and acetic acid were used. The silicate module of liquid glass was determined according to DSTU-N B V.2.7-304:2015 [19]. Solutions of orthophosphoric acid (1–4 wt. %) and sodium hexametaphosphate (0.1 and 1 %) were added to the obtained silicic acid sols. Tap water was used as a solvent for the initial components. The concentration of the Trilon B additive was varied within the range of 0.001–0.1 wt. %, in order to simultaneously assess its effect on the water softening efficiency and fire-retardant properties of the obtained coatings. Potassium phosphate buffer solution with pH 7 (20 vol. %) was added to the silicic acid sols.

To assess the survivability of the compositions, the change in optical density over time was measured using a KFK-2 photocolorimeter (λ=490 nm, l=5 cm, reference solution – distilled water).

Heat treatment of silica gels was carried out at 800 °C in an oxidizing environment (atmospheric air), maintaining at the maximum temperature for 2 h (temperature rise rate 4–5 °C/min).

To study the structural changes in raw and heat-treated gels, infrared absorption spectra were recorded on an FTIR-8400S spectrometer equipped with a QATR 10 (Shimadzu) attachment in the range of 400–4000 cm–1. The spectra were analyzed using IRsolution and ACD/Labs software.

Modified silicic acid sols were applied to the surface of experimental samples by immersion. Extruded polystyrene foam manufactured by Technoplex and Penoboard was used. 2–3 layers of coating were applied to wood samples measuring 9x6x3 cm, which were previously dried to constant mass in a drying cabinet at 100 °C, as well as samples of extruded polystyrene foam of the XPS brand measuring 5x5x3 cm. Each layer was dried at 80 °C before applying the previous one. Additionally, a 20 % aqueous solution of ammonium dihydrogen phosphate was applied to the surface of the dried samples by spraying, after which they were dried again.

The fire-retardant efficiency group of coatings on the surface of wood was determined according to DSTU 4479:2005 [20], the flammability group according to DSTU 8829:2019 [21].

The mass of the extruded polystyrene foam samples was recorded before and after exposure to fire for 10 s, the mass loss was expressed in percent.

Since XPS is mainly used for thermal insulation in facade systems, the conditions for conducting fire tests were close to those of the field. Two layers of a fire-retardant composition of the specified composition were applied to samples of extruded polystyrene foam measuring 30x20x3 cm, each of which was dried at 80 °C, after which they were finished with a plaster layer. The samples were mounted on a brick substrate that simulated the base of a real facade system. The fastening was carried out in a vertical position with continuous pressure on the brick using the same plaster solution that was used for the external decoration of polystyrene boards. The fixation was carried out over the entire contact area of the samples to prevent access of oxygen and delamination during heating.

5. Study of rheological properties of modified sols and gelation processes in them

Fig. 1 shows the dependence of the survival time of sols on the content of orthophosphoric acid, the value of the silicate modulus, and the volume ratio of acetic acid and liquid glass solutions.

Analysis of the diagram allows us to establish a regularity of increasing the survival time of sols with increasing orthophosphoric acid content. Regardless of the value of the silicate modulus, the sol survival time increases with increasing orthophosphoric acid content. In addition, a longer solidification time is noted for compositions with a higher volumetric ratio of acetic acid and liquid glass solutions (i.e., with a higher acidity of the medium). An increase in the silicate modulus of liquid glass, which corresponds to an increase in the content of silicon dioxide in the system, also contributes to an increase in the survival time of sols.

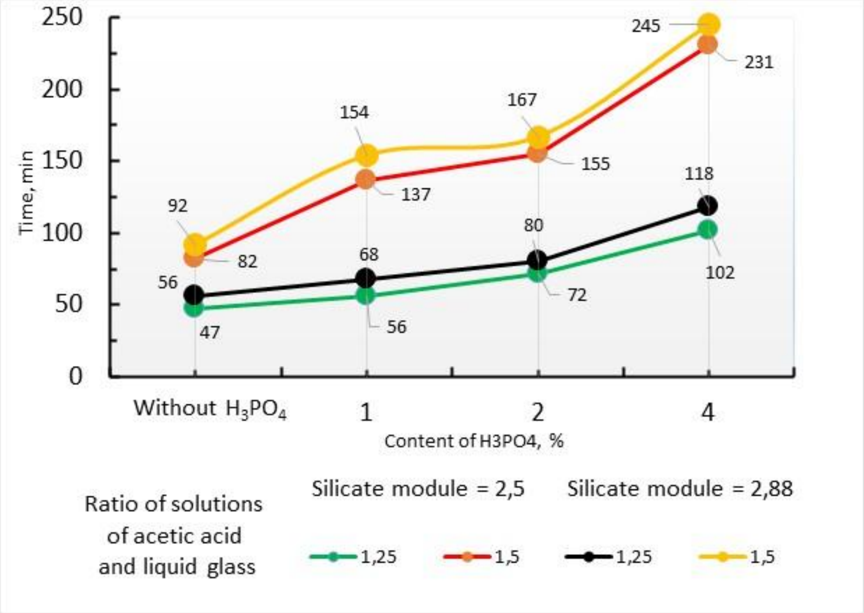

Fig. 1. Dependence of the survival time of sols on the content of orthophosphoric acid, the value of the silicate module and the volume ratio of acetic acid and liquid glass solutions

In order to assess the synergistic effect of phosphorus-containing components, further studies were focused not only on compositions with orthophosphoric acid, but also on compositions that simultaneously contained orthophosphoric acid and sodium hexametaphosphate. Fig. 2 shows the dependences of the change in optical density over time for modified sols.

For unmodified sols with an increase in the silicate modulus, a sharp decrease in the slope angle for the optical density curves of sols with the addition of 2 % orthophosphoric acid and 0.1 % sodium hexametaphosphate is recorded (especially for compositions obtained on the basis of liquid glass with a silicate modulus of 2.88). With an increase in the GMP concentration, the increase in optical density becomes less intense and smoother – the latent coagulation time becomes somewhat longer, which increases the survival time of the compositions.

The infrared spectra of raw unmodified silica gels obtained from compositions with different silicate modulus of liquid glass (Fig. 3) are characterized by identical sets of characteristic bands. Both spectra show a broad absorption band in the range of 2700-3600 cm^{-1}, which contains individual peaks at 3160, 3275 and 3405 cm^{-1}. Such absorption bands correspond to stretching vibrations of hydroxyl groups, which indicates the presence of adsorbed water and Si–OH groups on the gel surface.

The spectra also contain quite intense absorption bands at 1405, 1540 and 1650 cm^{-1}, corresponding to the deformation vibrations of the O-H groups, and present in the spectra of other silica gels studied in previous studies.

In both spectra, the presence of an absorption band at 1050 cm^{-1} is noted, which is due to the stretching asymmetric vibrations of the Si-O-Si bonds. The presence of siloxane bonds is also confirmed by the absorption bands at 790 cm^{-1}.

In the low-frequency region (400–600 cm^{-1}) there are also absorption bands corresponding to deformation vibrations of siloxane fragments [22, 23].

To analyze the gelation processes, the relative intensity of the bands corresponding to vibrations of the Si–OH (970 cm^{-1}) and Si–O–Si (790 and 1050 cm^{-1}) bonds was estimated (Fig. 4). With the same volume ratio of acetic acid and liquid glass solutions (1.5), but different values of the silicate modulus, the relative intensities of the characteristic bands remain practically unchanged, which indicates a slight influence of this parameter on the gel structure formation processes. The maximum value of the relative intensity of the band at 1050 cm^{-1} is recorded for a sample with a silicate modulus of 2.5 and a volume ratio of components of 1.25, but it is comparable with other obtained results.

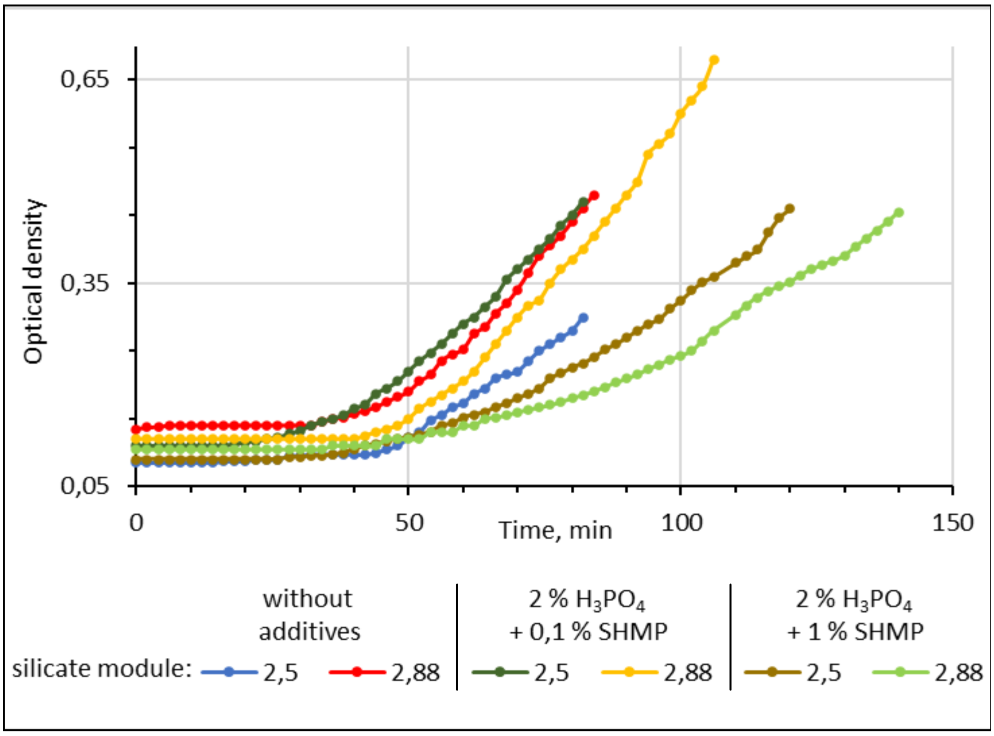

Fig. 2. Graph of the dependence of the change in the optical density of sols over time on the value of the silicate module and the content of phosphorus-containing additives (V(HAc)/V(liquid glass) = 1.5)

The nature of polycondensation in silicic acid sols was evaluated by the value of the I/d index – the ratio of the intensity of the band at 1050 cm^{-1} to its half-width.

This indicator indirectly indicates the nature of polycondensation of polysilicic acid: the lower its value, the greater the probability of linear polycondensation, the increase in this parameter indicates the development of the network structure of the siloxane framework [14].

Calculations allowed us to determine that predominantly linear type of polycondensation will occur for the conditions of use of compositions with a lower value of the silicate module (2.5) and a lower content of liquid glass in the initial composition (V(HAc)/V$_{\text{(liquid glass)}}$ = 1.5).

After high-temperature treatment of silica gels, the absorption band in the range of 2800–3600 cm^{-1} completely disappears, which may indicate the removal of water. In the interval of 800–1200 cm^{-1}, a broad absorption band appears, which is the result of the merging of peaks that were clearly expressed in the spectra of raw SiO_2 gels. For the IR spectrum of the heat-treated silica gel obtained on the basis of a composition with a liquid glass silicate modulus of 2.88, within the above-described band, separate peaks at 870, 930 and 1025 cm^{-1} are noticeable, while for the case with a silicate modulus of 2.5 the band has a more smoothed character.

Fig. 5 presents the results of a study of the influence of the Trilon B additive on the rheological properties of experimental compositions.

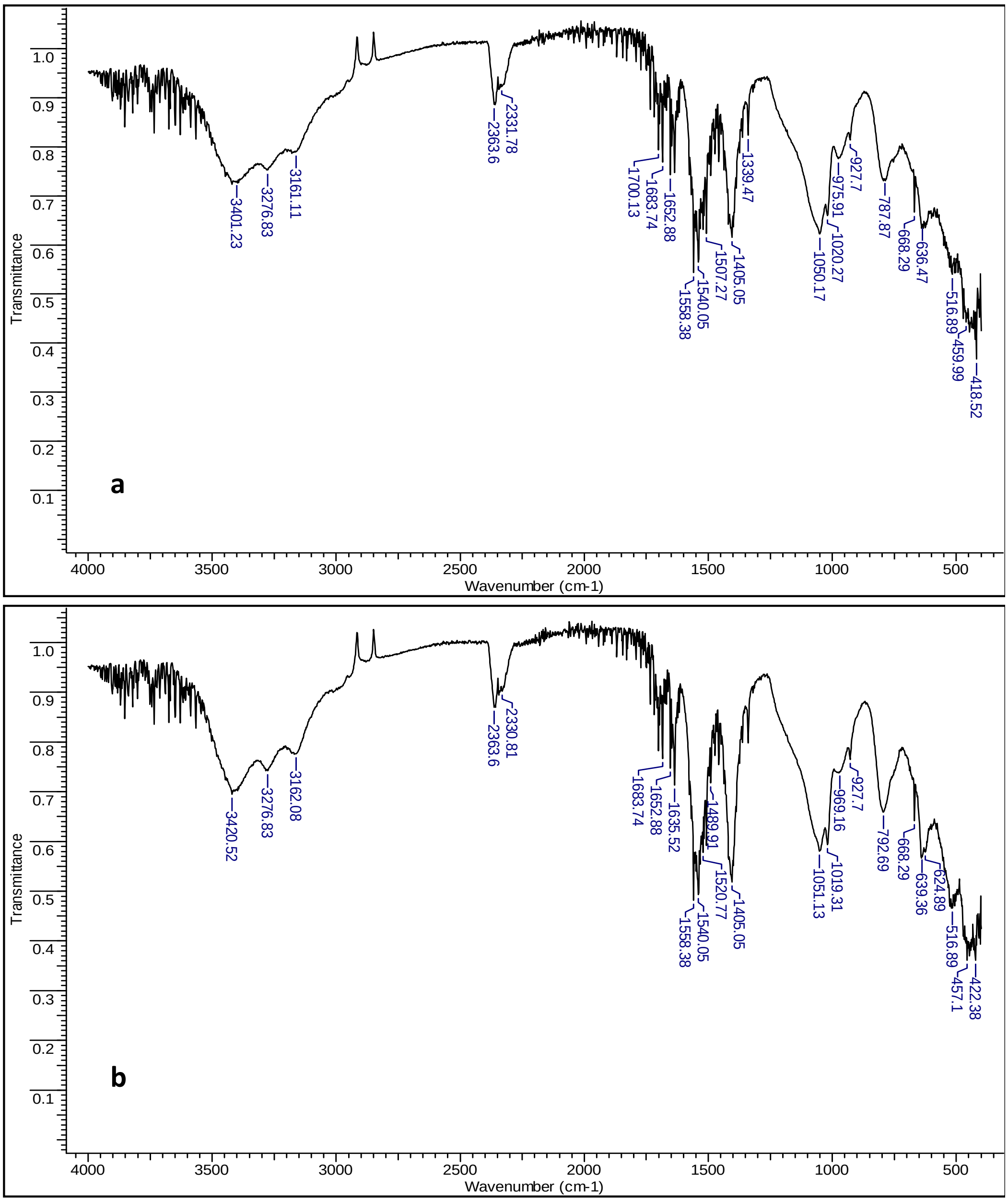

Fig. 3. IR spectra of raw SiO_2 gels obtained on the basis of compositions with a silicate modulus of liquid glass: 2.5 (a) and 2.88 (b) (V(HAc)/V(liquid glass) = 1.5)

Analysis of the results indicates a direct relationship: with an increase in the Trilon B content, a gradual increase in the duration of the viability of the sol is observed. For compositions with an additive content of 0.35 and 0.45 wt.%, the solidification time is the same. In the entire range of the studied Trilon B concentrations, the latent coagulation time varies within 7–15 minutes, which is insufficient for the formation of a structurally homogeneous silicic acid gel.

The gelation processes were evaluated by the relative intensities of the Si–OH (970 cm^{-1}) and Si–O–Si (790 and 1050 cm^{-1}) vibration bands (Fig. 6).

The relative intensities of the bands at 790 and 970 cm^{-1} for the studied compositions, regardless of the Trilon B content, are almost the same. The main differences are observed for the absorption band at 1050 cm^{-1}: with an increase in the additive content from 0.09 to 0.21 %, its relative intensity increases by more than 1.5 times. With a further increase in the Trilon B content (up to 0.27 %), a significant increase in the relative intensity is not observed, and the I/d indicators are almost the same.

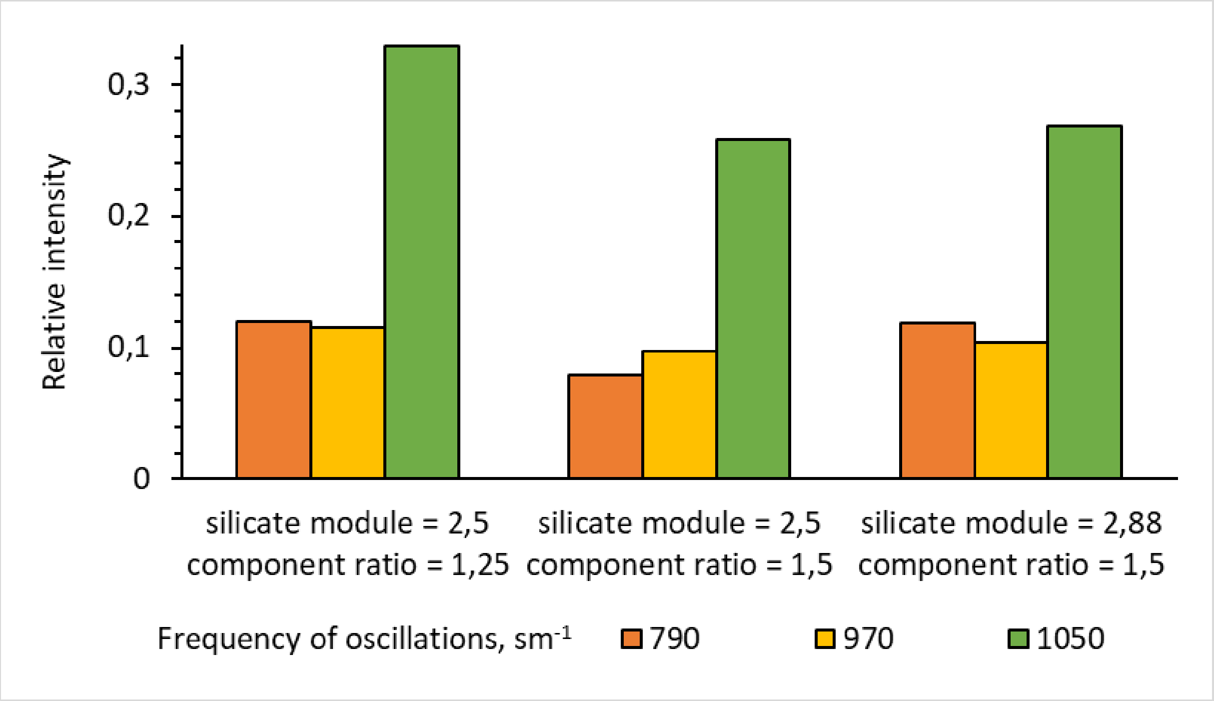

Fig. 4. Change in the relative intensity of the characteristic bands of raw SiO_2 gels obtained on the basis of compositions with different silicate modules and different ratios of the initial components

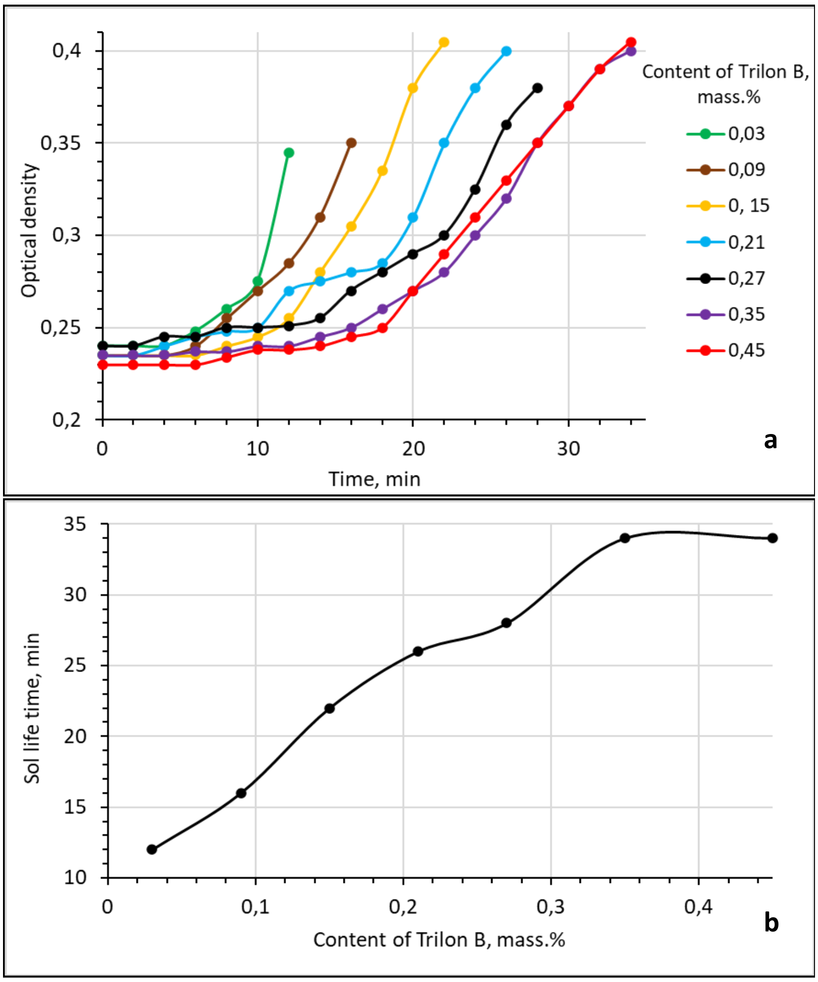

Fig. 5. Dependence of the change in optical density over time (a) and the survival time of sols on the content of Trilon B (b)

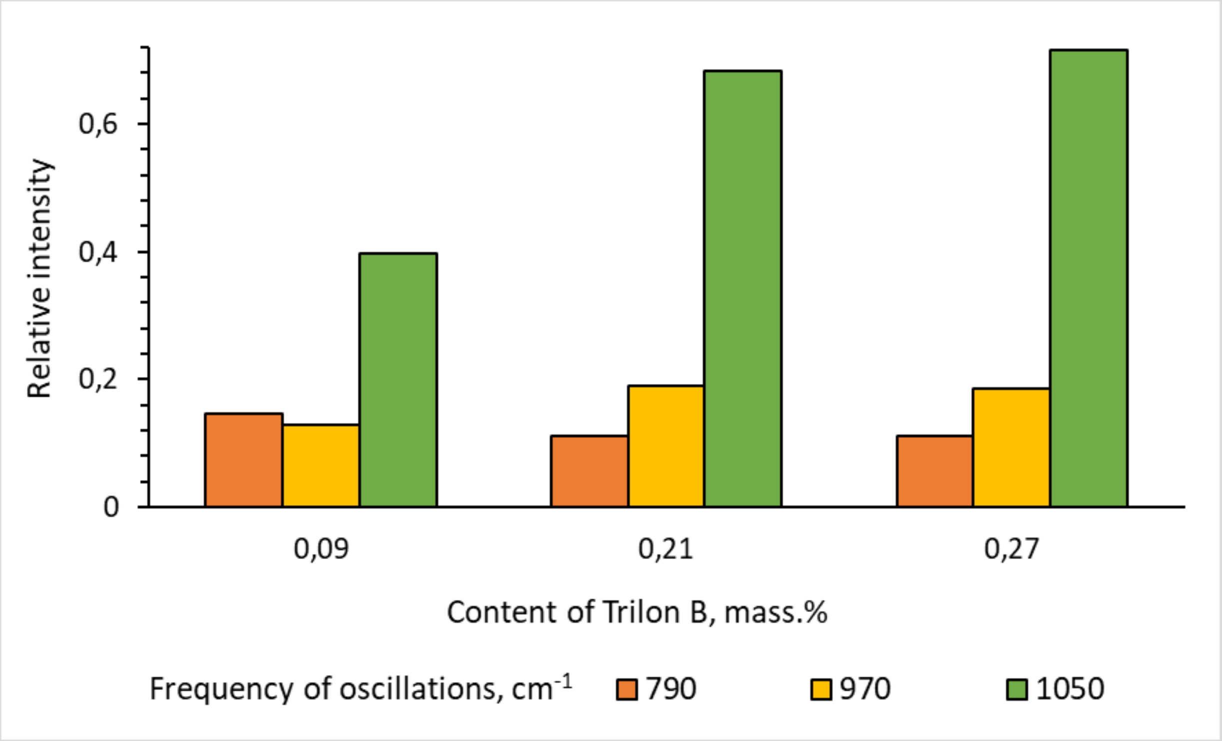

Fig. 6. Change in the relative intensity of characteristic bands of raw SiO_2 gels obtained on the basis of compositions with different Trilon B contents

High-temperature treatment of silica gels promotes the removal of water, which is confirmed by the disappearance of the absorption band at 2800–3600 cm^{-1} in the spectra of heat-treated samples. In the region of 800–1200 cm^{-1}, an extended band is formed, caused by the merger of peaks that were clearly separated in the spectra of raw silica gels.

6. Results of fire tests of silicophosphate coatings on wood and polystyrene foam samples

Tabl. 1 shows the results of determining the fire-retardant efficiency group of the coating and the flammability class for wood samples.

The smallest mass losses (5.8–7.5 %) were recorded for samples coated with compositions with the addition of 2 % orthophosphoric acid and 0.1 % sodium hexametaphosphate at a volume ratio of acetic acid and liquid glass of 1.5. At the same time, coatings with such an additive content at different values of the silicate module gave similar heat resistance indicators, in both cases, regardless of the number of coating layers, the I group of fire-retardant efficiency was established.

A decrease in the fire-retardant properties of the coating was observed in cases of increasing the sodium hexametaphosphate content to 1 % – according to the results of the conducted studies, higher mass losses of the samples were obtained, as well as higher values of the maximum temperature increase. It should be noted that negative trends were recorded regardless of the value of the silicate module.

In most cases, with an increase in the number of coating layers, the fire-retardant efficiency of the coating increased: mass losses of the samples became smaller.

It is also worth noting that the key factor in creating an effective fire-retardant coating is the optimal volumetric ratio of the starting components in the composition. Comparatively lower mass losses were recorded for samples coated with compositions where V(HAc) / V(liquid glass) was 1.5.

Tabl. 1. Results of studies on determining the fire protection efficiency group and flammability class for wood samples

№	Silicate module	V(HAc) / V(liquid glass)	GMF content, %	Number of coating layers	Sample weight loss (120 s), %	Coating fire protection efficiency group	ΔT_{max}, °C	Time to reach maximum temperature, c	Sample mass loss, %	Combustibility
1	Untreated sample				26,4	-	118	30	5,1	flammable, combustible
2	2,5	1,25	0,1	1	11,14	II	107	280	30,94	flammable, highly flammable
3				2	10,95	II	78	500	43,03	
4				3	9,17	II	85	590	50,86	
5		1,5	0,1	1	5,75	I	63	320	21,71	flammable, highly flammable
6				2	7,38	I	74	300	23,86	
7				3	6,79	I	75	320	20,66	
8			1	1	10,88	II	90	220	23,52	flammable, medium flammability
9				2	9,86	I	80	520	38,42	flammable, highly flammable
10				3	9,65	II	96	500	35,03	
11	2,88		0,1	1	7,45	I	65	320	23,56	flammable, highly flammable
12				2	7,31	I	74	340	27,78	
13				3	6,46	I	45	300	23,37	hardly flammable
14			1	1	10,2	II	102	550	56,68	flammable, highly flammable
15				2	8,26	I	78	570	39,21	
16				3	9,83	II	56	560	37,02	

* all modified compositions contained 2 % H_3PO_4

Tabl. 2 shows the results of fire tests for samples of extruded polystyrene foam treated with compositions obtained on the basis of liquid glass with different values of the silicate modulus.

The use of all silicophosphate compositions prevented the formation of burning drops – one of the main factors of fire hazard of expanded polystyrene.

The silicate module did not demonstrate a decisive influence on fire resistance, which is consistent with the results of fire tests of wood samples (Tabl. 1). Samples coated with compositions with the same content of phosphorus-containing additives (2 % orthophosphoric acid and 1 % sodium hexametaphosphate), but with a different silicate module for liquid glass, did not support combustion. Mass losses ranged from 1.5 to 3 %, the ignition time was increased by 2 to 2.5 times, compared to the untreated sample.

The optimal content of GMF required to create an effective fire-retardant coating on the surface of expanded polystyrene turned out to be higher than for wood samples, which is obviously due to different physicochemical properties of the materials.

Tabl. 3 shows the results of fire tests for samples of extruded polystyrene foam treated with compositions with different Trilon B contents.

The survival time of silicophosphate compositions increased with increasing Trilon B content in tap water, which was a solvent for the initial components.

Tabl. 2. Fire test results for extruded polystyrene foam samples

№	Silicate module	V(HAc) / V(liquid glass)	GMF content, %	Number of coating layers	Sample weight loss (10 s), %	Ignition time, s	Note
1	Untreated sample				-	3	actively burned with the formation of burning drops
2	2,5	1,25	0,1	1	-	5	burned without the formation of burning droplets
3				2	-	6	
4				3	3,23	-	did not burn
5		1,5	0,1	1	-	6	burned without the formation of burning droplets
6				2	1,13	7	
7				3	-	5	
8			1	1	1,86	7	did not support combustion
9				2	2,94	7	
10			1	3	-	4	burned without the formation of burning droplets
11	2,88		0,1	1	-	4	
12				2	-	4	
13				3	-	5	the fire started after a crack formed
14			1	1	1,92	6	did not support combustion
15				2	1,54	4	
16				3	2,53	8	

* усі модифіковані композиції містили 2 % H_3PO_4

It is worth noting that for none of the studied samples the formation of burning drops, which is one of the determining factors of fire hazard of expanded polystyrene, was recorded. The most stably reproducible positive results were obtained for fire-retardant compositions prepared using tap water with a Trilon B content of 0.1 wt.%. Mass losses were less than 1 % for samples of the “Technoplex” brand and about 2 % for “Penoboard”. The ignition time was increased 3 times.

Fig. 7 shows the appearance of extruded polystyrene foam samples treated with a fire-retardant coating of the studied composition (the composition was prepared using tap water pre-conditioned with 0.1 wt.% Trilon B) and a plaster layer during and after fire tests.

For XPS without a fireproof coating and a plaster layer, intensive flame spread was recorded over the entire height of the samples. The formation of burning drops was observed, which is a common marker of the fire hazard of thermoplastic insulation materials. After fire tests, the surface of the samples was significantly deformed and melted.

The Technoplex samples retained their integrity both when applying a fireproof coating and a plaster layer, and in the version with only a plaster layer. Only uniform darkening of the samples was recorded.

For polystyrene foam of the Penoboard brand, which was covered only with a plaster layer, cracks appeared and the protective coating was subsequently peeled off, which led to melting and ignition of the sample and further spread of fire at the site of damage. In the case of comprehensive protection, only local end peeling of the plaster was recorded. At the same time, burning drops were not formed, and the flame did not spread.

7. Discussion of the results of the study of the influence of silicate module and Trilon B on the fire-retardant efficiency of coatings

Analysis of the rheological properties of silica sols showed that changing the silicate modulus in the range of 2.5–3 for unmodified compositions has an insignificant effect (Fig. 1). The slight increase in the survival time is apparently due to the fact that with an increase in the ratio n(SiO_2)/n(Na_2O) the number of alkali metal cations in the diffusion layer of micelles decreases, so particle aggregation occurs more slowly.

For compositions with orthophosphoric acid additives (Fig. 1), the variation of the silicate module also does not cause significant changes. The increase in the survivability of compositions with an increase in the orthophosphoric acid content is due to the initiation of the network mechanism of polycondensation. The effect of changing the silicate module is more noticeable in the case of the complex use of phosphorus-containing additives (Fig. 2). Compositions with 2 % H_3PO_4 and 1 % sodium hexametaphosphate are more stable over time than similar ones with a GMP content of 0.1 %. Obviously, with an increase in the concentration of hexametaphosphate, its stabilizing effect increases, which prevents premature aggregation of particles.

Tabl. 3. Effect of Trilon B on the fire-retardant efficiency of coatings for extruded polystyrene foam samples

№	Content of Trilon B in tap water, wt.%	Composition viability, min	Foam brand	Sample mass loss, %	Ignition time, s	Note
1	Необроблені зразки		«Technoplex»	-	3	actively burned with the formation of burning drops
2			«Penoboard»	-	3	
3	0,1	35	«Technoplex»	-	9	burned without the formation of burning droplets
4				0,79	9	did not support combustion
5				0	-	did not burn
6			«Penoboard»	-	9	burned without the formation of burning droplets
7				2,26	9	did not support combustion
8				1,48	-	did not burn
9	0,01	25	«Technoplex»	1,08	-	did not burn
10				-	6	burned without the formation of burning droplets
11				-	5	
12			«Penoboard»	-	6	
13				-	4	
14				-	4	
15	0,001	17	«Technoplex»	-	5	
16				0,55	5	
17				-	4	
18			«Penoboard»	-	4	
19				-	4	
20				-	4	

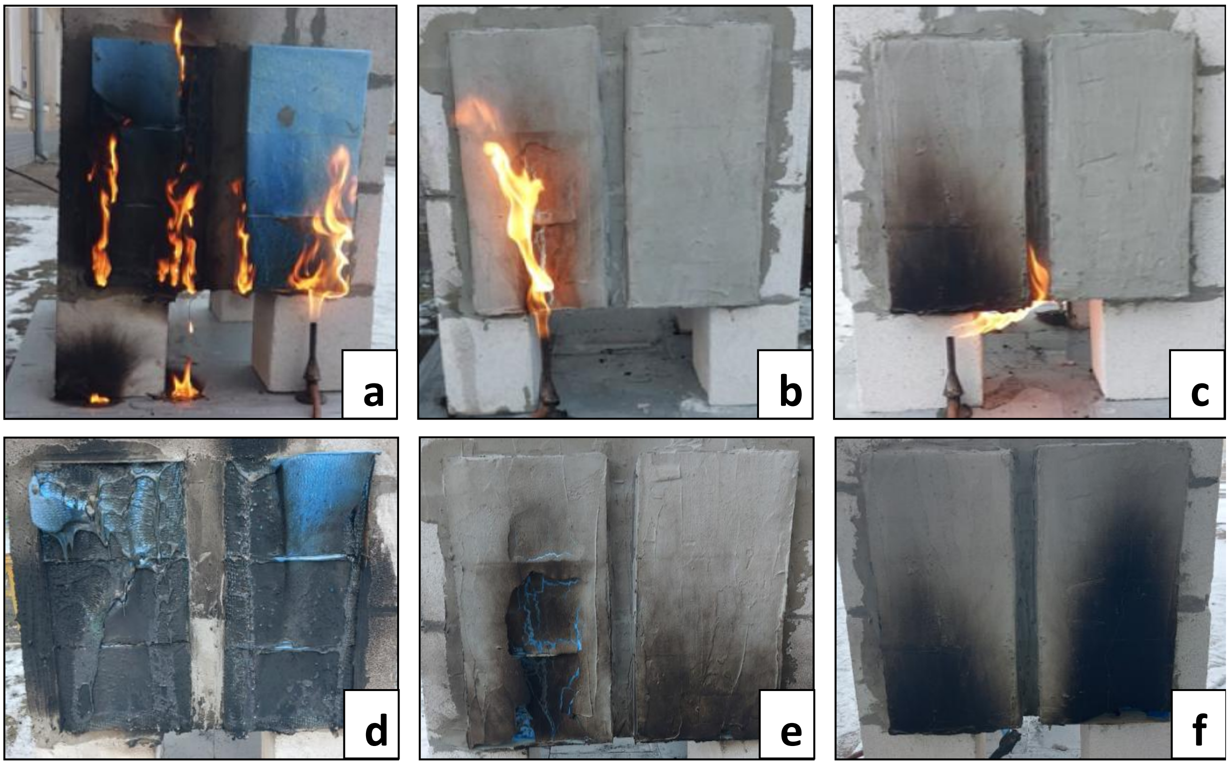

Fig. 7. Appearance of XPS samples during (a-c) and after fire tests (d-f): a,d – without fireproof coating and plaster layer; b,e – without fireproof coating, with plaster layer; c,f – with fireproof coating and plaster layer (in each photo on the left – samples of expanded polystyrene "Penoboard", on the right – "Technoplex")

An important factor is also the volumetric ratio of the initial components. In all cases (Fig. 1, 2), higher values of the duration of solidification of sols were recorded at $V_{(HAc)/}V_{(liquid\ glass)}$=1.5. A decrease in the liquid glass content leads to a general decrease in the amount of SiO_2 in the composition, corresponding to a decrease in the frequency of particle collisions, and therefore to a slowdown in coagulation processes.

These assumptions were confirmed by the results of the analysis of infrared spectra. The maximum values of the ratio of the intensity of the absorption band of the vibration of the bonds –Si–O–Si– at 1050 cm–1 to its half-width were obtained for samples with the ratio $V_{(HAc)}/V_{(liquid\ glass)}$=1.25. With an increase in this parameter, the ratio I/d decreases, which indicates the occurrence of predominantly linear polycondensation: the SiO_2 content decreases, and accordingly, the crosslinking points necessary for building a spatial network are formed less frequently. When using a composition with the same liquid glass content but a higher silicate modulus, the I/d value increases insignificantly, which does not indicate the decisive role of the silicate modulus in the formation of the structure of the silica-containing coating.

Rheological tests of silica sols also showed that the introduction of Trilon B and an increase in its content increases the survivability of the compositions (Fig. 5). Ca_2+ and Mg_2+ ions contained in tap water significantly accelerate the aggregation of negatively charged particles in the sol, weakening the repulsion between them and literally "stitching" them together. On the other hand, an increase in the number of cations in the diffusion layer leads to a compression of the double electric layer and a decrease in the ζ-potential, which also reduces the stability of the system. Trilon B chelates ions that cause water hardness, forming strong water-soluble complexes with them. In the range of 0.35–0.45 wt.% EDTA, the dependence of the change in optical density over time has a plateau (Fig. 5, a), which may indicate a “saturation” effect, when most of the multiply charged ions are already bound, and the electrostatic parameters of the system almost do not change.

The results of the infrared spectra analysis are consistent with the assumptions made above. With increasing Trilon B content, the intensity of the band at 1050 cm^{-1} increases, which corresponds to asymmetric stretching vibrations of siloxane bonds (Fig. 6). This indicates a more controlled polycondensation process: at a higher EDTA content, the sol remains stable longer, “random” coagulation decreases, and the reactions of conversion of –Si–OH to –Si–O–Si– occur more evenly.

The differences in the I/d parameter for silica gels with different chelating additive contents are insignificant. However, with increasing Trilon B concentration, the probability of network polycondensation increases. EDTA complexes with Ca_2+ and Mg_2+ cations are quite bulky and can create certain steric barriers that mask the reactive –Si–OH centers and prevent linear chain growth. On the other hand, the probability of cross-linking and the formation of a network structure increases. The transition from 0.21 to 0.27 wt.% obviously gives only a minimal increase in the effect, so the relative intensity of the band at 1050 cm^{-1} (Fig. 6) and the I/d parameter remain almost at the same level.

When Trilon B is added directly to the silicic acid sol, the latent coagulation time (Fig. 5,a) is insufficient to form a morphologically uniform fireproof coating. The Trilon B content, which provides approximately optimal from a technological point of view the solidification time of the sol, promotes the occurrence of predominantly network polycondensation. Given the above considerations, it was determined to be advisable to introduce Trilon B at the stage of solvent preparation.

As is known, in the case of linear polycondensation, elongated polymer chains based on siloxane bonds are formed, which is a prerequisite for the formation of coatings with a more flexible, elastic structure. The results of fire tests confirm their fire-fighting efficiency. For samples of both wood and extruded polystyrene foam, the smallest mass losses of the samples were recorded for the ratio $V_{(HAc)/}V_{(liquid\ glass)}=1.5$, i.e. with a predominant linear polycondensation mechanism (Table 1, 2). Changing the value of the silicate modulus did not have a significant impact on the fire-retardant properties of the coatings. In particular, for cases with a silicate modulus of both 2.5 and 2.88, the wood samples were transferred to the “difficult to ignite” group and the I group of fire-retardant efficiency was established. XPS samples also did not support combustion.

Special attention is paid to the influence of sodium hexametaphosphate content on the ability of coatings to resist fire. For wood, the best results were obtained in the case of using compositions with a GMP content of 0.1 % (Tabl. 1). Presumably, this content of phosphorus-containing additive was sufficient to ensure optimal fire-fighting characteristics. On the other hand, with a 10-fold increase in the GMP content, the mass loss of the samples increased, which may indicate oversaturation of the surface of the material, as well as its porous structure with the phosphorus-containing component. Wood, as is known, has high hygroscopicity and the ability to absorb moisture and aqueous solutions well, but an excessive amount of GMP can reduce the adhesion of the protective layer and worsen the mechanical properties of the building material, thereby weakening its resistance to fire.

For polystyrene foam, the best fire protection indicators were given by compositions with a higher GMP content (Tabl. 2). Sodium compounds are fusible and are capable of converting the fire-retardant coating into a visco-plastic state under the influence of high temperatures, which prevents the access of oxygen and slows down the combustion process, but 0.1 % of the additive was apparently not enough to form an effective thermal protective layer.

The study of the effect of Trilon B on the fire-retardant properties of coatings (Tabl. 3) showed that increasing the additive content (from 0.001 to 0.1 wt. %) in the water used to prepare the sol contributed to a monotonous increase in the survivability of the compositions (from 17 to 35 minutes), as well as the ignition time (from 4 to 9 s). A stable positive effect for both brands of the studied XPS was observed at a Trilon B content of 0.1 wt. %, which indicates the formation of an effective fire-retardant coating on the surface of the foam. Under the influence of fire, such a layer is able to transition to a visco-plastic state, which limits the access of oxygen and inhibits combustion processes. The variability of individual results (“did not burn” / “burned without the formation of burning drops”) for coatings of the same composition may be due to technological fluctuations (the condition of the surface of the samples, the drying regime between layers, etc.) and does not contradict general trends.

The role of the plaster layer also deserves special attention, since it is used to finish extruded polystyrene foam in real facade systems. Based on the results of fire tests (Fig. 7), it can be stated that the plaster layer alone can provide only a limited thermal protection effect and is capable of cracking and peeling off under the influence of flame. In combination with a fire-retardant coating, a synergistic effect is likely to be realized. The coating, which has passed into a visco-plastic state, is able to "heal" microcracks and create a continuous thermal protection barrier. It is this configuration that guarantees the absence of burning drops and prevents the spread of fire even in the presence of edge defects.

For wood, a series of fire tests was not conducted for the variant with pretreatment of water with Trilon B. However, given the stable correlation for other compositions, as well as the better mechanical adhesion of the silicate coating due to the formation of chemical bonds with wood cellulose, positive fire test results can be expected for this material with a high probability.

Therefore, the effectiveness of fire-retardant coatings based on silica sols significantly depends on the content of modifying additives. Optimal results are achieved in the case of a volumetric ratio of acetic acid and liquid glass of 1.5, which promotes the occurrence of predominantly linear polycondensation and ensures the formation of a more uniform coating. Changing the silicate modulus in the range of 2.5–3, which is most common among liquid glass manufacturers, does not have a significant impact on the rheological properties of sols and the fire-retardant efficiency of coatings, which indicates the technological flexibility of using the proposed composition and simplifies its practical implementation, since it does not require precise control of this parameter. The use of Trilon B can be considered as a tool for adapting the developed technology for fire protection of building materials to work with technical (non-distilled) water. The EDTA content is 0.1 wt. % is optimal for softening hard water, but can be adjusted depending on the degree of purity of tap water to ensure sufficient time for the composition to solidify and form a structurally intact and effective fire-retardant coating. This approach is economically feasible and facilitates the integration of the technology into production processes.

9. Conclusions

1. The rheological properties and gelation processes in silicic acid sols obtained on

the basis of liquid glass modified with Trilon B were investigated.

It was established that the silicate modulus in the range of 2.5–3 does not significantly affect the rheological properties of silicic acid sols, as well as the gelation processes. It was found that at a volumetric ratio of acetic acid and liquid glass solutions of 1.5, the highest stability of the compositions over time is ensured, and the linear mechanism of polycondensation prevails, which is a prerequisite for the formation of elastic fire-retardant coatings. It was established that Trilon B can be used to remove iron, calcium and magnesium cations from the technical water used to dissolve the main components of the composition, which are capable of compensating the charges on the surface of silicic acid micelles and leading to premature coagulation of the sol. It was found that the introduction of a chelating agent directly into the sol-gel composition initiates predominantly network polycondensation, therefore, a scheme of preliminary preparation of technical water by conditioning with Trilon B was chosen.

2. Fire tests of wood and extruded polystyrene foam samples were performed to assess the effectiveness of the fire-retardant silicophosphate coating.

The fire-retardant properties of silicophosphate coatings on the surface of wood and extruded polystyrene foam were evaluated. It was established that the optimal content of GMF for wood is 0.1 %, for extruded polystyrene foam – 1 %. It was determined that the variation of the silicate module in the range of 2.5–3 practically does not affect the fire-retardant efficiency of the coatings, which makes the technology of their formation more flexible and universal. It was established that the additive content of 0.1 wt. % is optimal for softening water, ensuring a technologically acceptable setting time of the composition and forming an effective fire-retardant coating. It was found that the plaster layer on the XPS surface alone demonstrates limited thermal protection and is prone to cracking and delamination, while in combination with the studied fire-retardant coating it implements a synergistic barrier effect, significantly increasing the fire resistance of the building material.

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ТЕХНОЛОГІЧНІ ОСОБЛИВОСТІ ОТРИМАННЯ СИЛІКОФОСФАТНИХ ВОГНЕЗАХИСНИХ ПОКРИТТІВ ДЛЯ БУДІВЕЛЬНИХ ОЗДОБЛЮВАЛЬНИХ МАТЕРІАЛІВ

Досліджено вплив технології отримання вогнезахисних композицій на основі рідкого скла для вогнезахисту будівельних оздоблювальних матеріалів. Вивчено вплив силікатного модуля рідкого скла на реологічні властивості золів кремнекислоти, модифікованих фосфатовмісними сполуками. За результатами спектрофотометричних вимірювань встановлено, що співвідношення $n(SiO_2)/n(Na_2O)$ в рідкому склі в межах 2,5–3 суттєво не впливає на живучість композицій. Визначено вплив складу композиції на тривалість застигання золів. Методом інфрачервоної спектроскопії підтверджено відсутність істотного впливу силікатного модуля в досліджуваному діапазоні на процеси структуроутворення в гелях. Проведено дослідження можливості використання водопровідної води для отримання композицій, що є необхідним у разі впровадження розробки. Встановлено, що попереднє введення до водопровідної води 0,1 мас. % Трилону Б дозволяє отримати стабільні в часі золі кремнекислоти, що є передумовою для утворення однорідного вогнезахисного покриття. Проведено вогневі випробування зразків деревини та екструдованого пінополістиролу, вкритих композиціями досліджуваного складу. Встановлено, що вміст 2 % ортофосфатної кислоти та 0,1 % натрій гексаметафосфату забезпечує втрати маси зразків деревини менше 7,5 %, що відповідає I групі вогнезахисної ефективності покриттів, а захищений матеріал відноситься до групи важкозаймистих. Композиції з умістом 1 % натрій гексаметафосфату показали найкращий вогнезахист екструдованого пінополістиролу: втрати маси зразків варіювалися в межах 1–3 %, палаючі краплі не утворювалися, зразки не підтримували горіння. Припущено, що підвищена вогнестійкість покриттів з більшим вмістом фосфоровмісної добавки пов’язана з легкоплавкістю натрієвих сполук та їх здатністю переводити вогнезахисне покриття у в’язкопластичний стан, що сприяє дисипації деформаційних напружень та запобігає утворенню тріщин в покритті.

Ключові слова: кремнеземвмісні покриття, силікофосфатні покриття, фосфоровмісні добавки, термостійкість, вогнестійкість, екструдований пінополістирол

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