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STUDY OF WATER RESISTANCE OF SILICA PROTECTIVE COATINGS BASED ON LIQUID GLASS

The water resistance of cotton textile materials impregnated with SiO_2 sols obtained on the basis of liquid glass was studied. Experimental coatings on fabrics were prepared by the bath method. After applying each coating layer and removing excess ash, the experimental samples were dried at (60–80) ºC. Fabric samples impregnated with sol SiO_2 were immersed in containers with distilled water maintaining the same sample/water ratio. The fire-retardant properties of the coatings were determined after standing in water for 2–72 hours. The degree of destruction of coatings during hydrolysis was studied by determining the optical density of the hydrolyzate above the surface of the samples using the spectrophotometric (KFK-2) method. Fire-resistant properties were determined at a laboratory installation for fire tests. Under the influence of water, partial hydration of the surface of the silica coating occurs, which does not lead to its destruction. The presence of a layer of adsorbed water molecules on the surface of the coating is the reason for an additional increase in the fire-retardant properties of the samples. It is shown that the degree of homogeneity of the SiO_2 sol affects the resistance to hydrolysis of the gel coatings. Low-concentration SiO_2 sols (8 %), which are characterized by high fluidity and have a long service life, have a predominant effect. The long-term effect of water provides an increase in the fire-retardant properties of impregnated samples in comparison with non-impregnated fabric samples. The concentration and degree of homogeneity of the SiO_2 sol have a predominant effect on the flame retardant properties. The surface layer of flame-retardants prevents the final burning and smoldering of the samples after removing the fire source, but does not significantly affect values of flame-retardant properties.

Keywords: liquid glass, fire-retardant coatings, textile materials, water resistance, lay-by-layer assembly, fire resistance

1. Introduction

The problem of preserving human life and health is one of the most important in the field of civil defense and fire safety. Solving this problem consists of solving a large number of humanitarian, social and technical tasks, including issues of reducing the level of fire danger for people in places of large concentration, preventing the occurrence of fire, as well as developing ways of its localization [1].

One of the important tasks in this sense is the development of technological principles for increasing the fire-resistant properties of textile materials used as upholstery and decoration materials in residential buildings, offices and places of large crowds [2–4]. In addition, it is important to reduce smoke emission during a fire by suppressing the ignition of textile finishing materials [5].

From this point of view, research related to the development of flame-retardant coatings on textile materials, which are able to be reliably fixed on the surface of the fabric, reduce the overall thermal effect of ignition or prevent the ignition of the fabric under the influence of a fire source, are relevant.

2. Analysis of literary data and formulation of the problem

Recently, silica coatings, which do not oxidize, do not burn, and do not undergo reconstruction under the influence of flame, are used for fire protection of textile mate-

rials. A class of organosilicon compounds of different spatial structure, which contain phosphorus, bromine, nitrogen and other atoms, are usually used as precursors for the production of silica coatings. In an attempt to increase the fire resistance of siliceous coatings, various methods of applying these substances to the fabric are used: impregnation, spraying, layer-by-layer assembly, etc. [6–9].

It is known that the nature of the siliceous precursor and the history of its preparation affect the degree of homogeneity of the gel coating and, accordingly, the quality of the fire-retardant effect of the coating [10, 11].

The introduction of titanium, zirconium and aluminum oxides into the compositions significantly increases the fire resistance of treated fabrics, but their effectiveness is lower than that of silica [11].

The use of phosphorus-containing compounds as components of a protective coating helps to increase the fire-resistant properties of impregnated fabrics, but increases the emission of smoke. A small content of phosphorus compounds (up to 15 wt.% in relation to the organosilicon precursor) significantly improves the fire resistance of the fabric due to a synergistic effect [12]. But the complexity of the synthesis of phosphorus-containing compounds and the toxic effect on the human body and the environment limit the use of such compounds.

The method of forming a fire-resistant coating by impregnating the fabric with aqueous suspensions in which SiO_2 nanoparticles are the dispersed phase is quite widely used [13, 14]. But the quality of the application of the protective layer is affected by the reaction conditions of the deposition of SiO_2 nanoparticles on the surface of the fabric fibers, the modification time and the content of tetraethylorthosilicate, the distribution of nanoparticles by diameter. It should be noted that the layer-by-layer application of a siliceous coating is a very long process, and requires additional fixing of each layer by heat treatment, which increases the cost and energy consumption of the process.

SiO_2 sols of inorganic origin, for example, obtained as a result of the exchange reaction of sodium silicate with mineral acid, can be considered as low-concentration suspensions, in which the dispersed phase is silicic acid micelles capable of polycondensation with the formation of nanoparticles. From the point of view of applying SiO_2 nanolayers, silicic acid salts obtained by the action of mineral acids on sodium silicate (liquid glass) are interesting. However, in the publications cited in the technical literature, ways of obtaining gel powders of the desired structure, given porosity or pore morphology were considered, but not the issue of obtaining a stable sol.

In previous studies [15], we proposed a method for obtaining a stable SiO_2 sol based on liquid glass in the concentration range (4–16%), studied the rheological properties of the sols and showed the positive effect of protective coatings based on them on increasing the fire protection of textile materials. But, taking into account the inorganic origin of such sols, that is, the absence of hydrocarbon groups on the surface, it is possible to assume a partial decrease in the adhesion of the coating to the cellulose fiber.

Fire-resistant coating on textile materials works reliably only in case of sufficient adhesion to the fabric fiber. It is possible to evaluate the adhesion of the coating to the fiber of the fabric threads during the study of the water resistance of impregnated samples. This is explained by the fact that water has a wedging effect at the points of connection of the coating with the threads of the fabric.

Thus, an unsolved part of the problem is determining the reliability of applying siliceous coatings based on stable concentrated SiO_2 sols based on liquid glass.

3. The purpose and objectives of the research

The purpose of the work is to study the processes occurring in the protective coating based on liquid glass during water resistance tests.

To achieve the goal, the following research tasks needed to be solved:

– determine the effect of long-term action of water (hydrolysis process) on the structure of the coating based on liquid glass;

– determine the effect of long-term hydrolysis on the fire-retardant properties of coatings.

4. Research materials and methods

Experimental SiO_2 sols were obtained by mixing aqueous solutions of liquid glass and acetic acid. Cotton samples measuring 9 x 13 cm were impregnated by the "bath" method, excess sol was removed on wringing rollers and dried at temperatures of 80-100 ºC. The coating was applied in three layers. Fire retardant solutions (diammonium hydrogen phosphate and urea) were applied to the dried samples by spraying and dried again. The marking of the samples consists of the concentration of SiO_2 sol (8, 11, 14) and the "a" mark in the case of using flame retardants.

Fabric samples impregnated with sol (SiO_2 concentration 8, 11, and 14%) with and without flame retardants were immersed in containers with distilled water with a volume of 3 liters, maintaining the same sample/water ratio. For comparison, samples of non-impregnated fabric were placed in a separate container. After standing in water for some time (2, 4, 6, 24, 48 and 72 hours), one sample was removed from each container and dried in a drying cabinet. At the same time, a portion of pre-scalated water was taken from each container to determine the optical density of the liquid above the samples subject to hydrolysis (hydrolyzate).

The optical density was determined using a KFK-2 photocolorimeter, using distilled water as a standard. Every day, the contaminated water was drained and the containers were filled with clean water.

After the samples were dried, they were subjected to fire tests using a laboratory setup consisting of a protective metal horizontal screen with a hole in the middle with a diameter of 30 mm. The burner was fed from the bottom of the hole in such a way that the hole was in the middle of the flame from burning gas supplied under a pressure of 0.2 MPa.

A fabric sample was fixed on top of the screen. The process of the fire test was recorded on a video camera, which was used to determine the time of the beginning of carbonization of the fabric under the action of fire, the time of the beginning of its destruction, the time of final burning and smoldering after the removal of the fire source. The area of damage to fabric samples from the action of fire for 6 seconds was determined separately.

5. Results of studies of the effect of long-term action of water on the structure of the coating

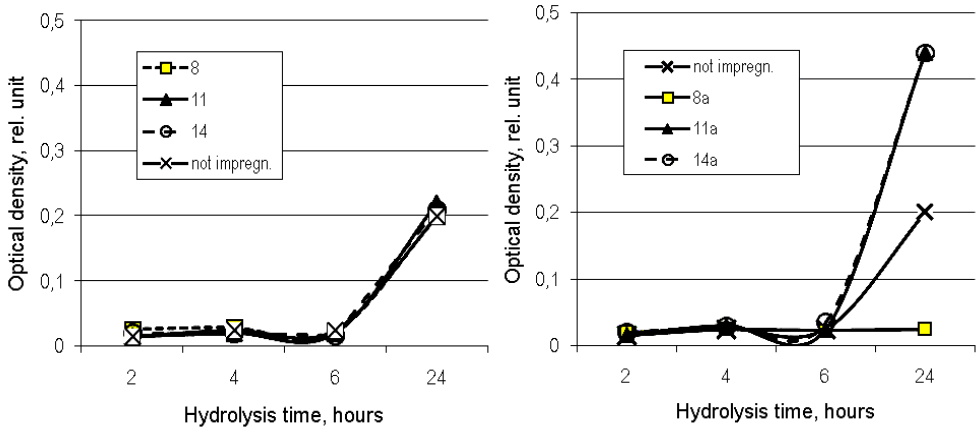
Holding the samples in water for 2–6 hours does not lead to significant changes in the optical density of the hydrolyzate (fig. 1a).

Practically the same situation is observed on the graph of the change in the optical density of the hydrolyzate over the tissue samples impregnated, in addition to the SiO_2 sol, with flame retardant solutions (fig. 1b). The compositions show sufficient stability in the first 6 hours of hydrolysis.

With increasing hydrolysis time, there is a sharp increase in the optical density of the non-impregnated sample, which indicates a partial loss of their coloring. The optical density curves of the impregnated samples are superimposed on the curve of the non-impregnated sample (fig. 1a).

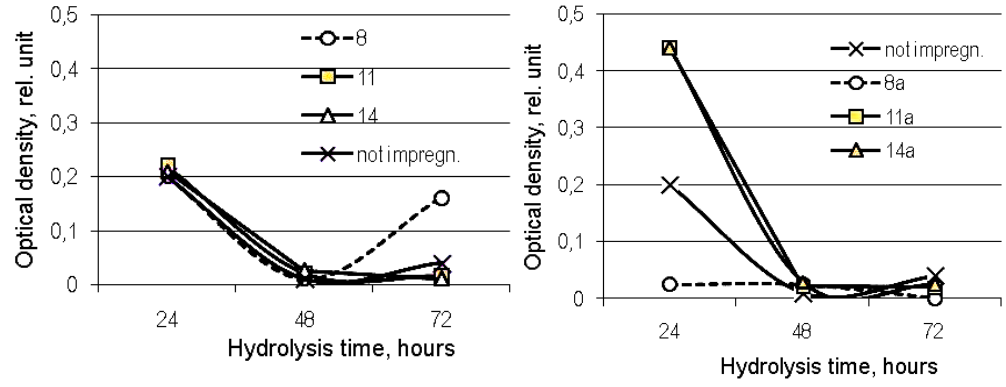
In the case of additional impregnation of the samples with flame retardants, a sharp increase in the optical density of the hydrolyzates is observed in comparison with the curve for the non-impregnated sample (fig. 1b).

The shape of the curves in graph 2(a) shows that the main damage to the coating occurs after 24 hours of hydrolysis. Changing the water in the containers and waiting for another 24 hours does not lead to noticeable changes in the coatings. Only on the 3rd day of hydrolysis, there is a slight increase in the optical density of the hydrolyzate of samples impregnated with sol of 8% concentration.



a) **b)**

Fig. 1. Change in the optical density of the hydrolyzate depending on the time of hydrolysis of the samples impregnated with sols of 8, 11 and 14% concentration: a – samples impregnated with SiO_2 sol; b – samples additionally impregnated with flame retardant solutions



a) **b)**

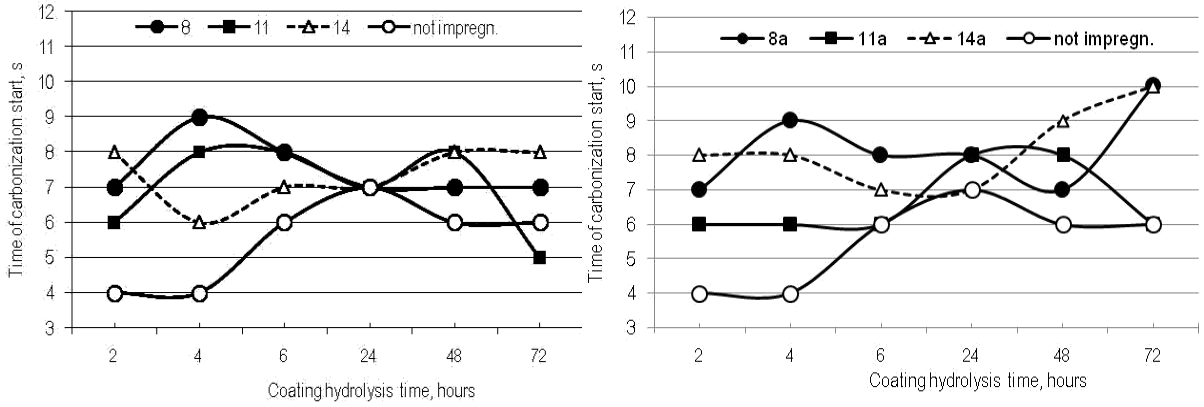
Fig. 2. The influence of the hydrolysis time of impregnated samples on the optical density of the hydrolyzate: a – samples impregnated with SiO_2 sol; b – samples additionally impregnated with flame retardant solutions

A similar situation is observed in graph 2(b): the maximum damage to the coating occurs in the first 24 hours of hydrolysis, and the subsequent two days of testing practically did not change the optical density values.

6. Results of studies of the effect of long-term hydrolysis on the fire-retardant properties of coatings

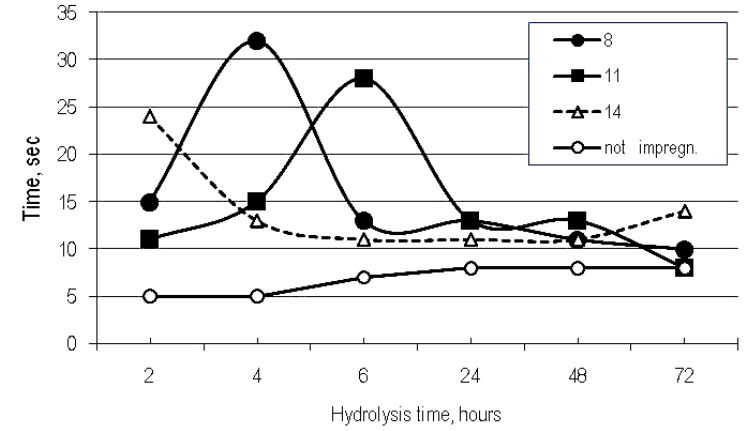
The results of fire tests of experimental samples are shown in fig. 3–4. Curves of

changes in the time of the beginning of charring and the time of the beginning of tissue destruction from the time of hydrolysis of coatings. It should be noted that the non-impregnated fabric significantly reduces its flame-retardant properties after hydrolysis: the time of the beginning of charring decreases from 6 to 4 s, the time of the beginning of destruction ranges from 5 to 8 s.

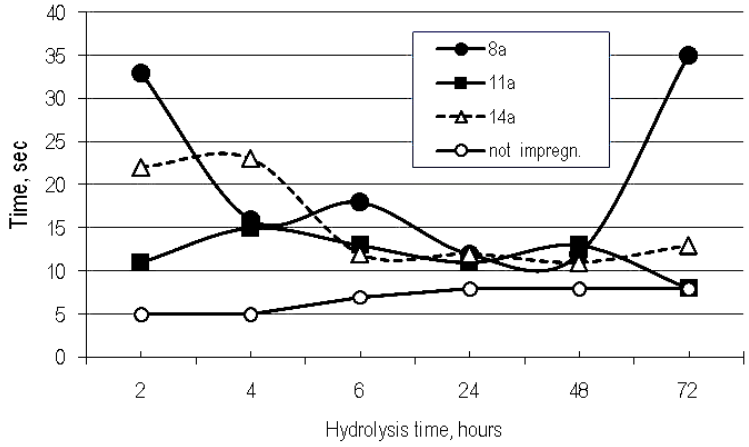


a) **b)**

Fig. 3. The dependence of the time of the beginning of charring during the action of fire on the samples impregnated with SiO_2 sol (a) and additionally impregnated with flame retardants (b) on the hydrolysis time of the coatings



a)



b)

Fig. 4. The influence of the time of hydrolysis of coatings on the time of the beginning of destruction of samples during fire tests: a – samples impregnated with SiO_2 sol; b – samples additionally impregnated with flame retardant solutions

All non-impregnated samples after removal of the fire source showed final burning in the interval 11–130 s, final smoldering was observed only in the sample after hydrolysis for 2 hours and in the sample that was not subjected to hydrolysis.

The time of the beginning of charring of non-impregnated samples (fig. 3a) increases with the increase of the hydrolysis term, which indicates the hydration of cellulose fibers. The time of the beginning of charring of samples impregnated with sols of 8 and 11 % concentration after hydrolysis for 2–4 hours varies from 6 to 9 s (fig. 3a), probably due to partial hydration of the surface of the protective coating. Adsorption of water molecules on hydrophilic parts of the surface during hydrolysis for 2–4 hours increases not only the time of the beginning of charring, but also the time of the beginning of tissue destruction (fig. 4).

Further hydrolysis reduces the fire-retardant properties of the coatings, but in comparison with non-impregnated samples, a significant improvement in fire-retardant properties remains.

7. Discussion of the results of the study of water resistance of protective coatings

Hydrolysis of the samples within 24 hours is probably the cause of the partial loss of staining by the samples, as evidenced by a sharp increase in the optical density of the hydrolyzate in a container with non-impregnated tissue samples (fig. 1a).

The curves of changes in the optical density of the hydrolyzate in the containers with samples practically overlap each other, so it can be assumed that the coatings have some porosity, due to which the dyeing of the fabric partially reaches the hydrolyzate, increasing its optical density.

Samples additionally impregnated with flame retardants more easily give off a certain amount of not only fabric coloring, but also partially flame retardants (fig. 1b). The reason for this is probably the weak fixation of flame retardants on the surface of the silica coating, which lacks hydrocarbon groups. Previous studies have already proven that the concentration of SiO_2-sol affects its rheological properties [7] (flowability, viscosity), which, in turn, affect the quality (degree of uniformity) of coating application. Applying flame retardants to a thin coating protects the fabric to a greater extent: the optical density of the hydrolyzate practically does not change. Taking into account that clean water was poured into the container every 24 hours, the change in the optical density of the hydrolyzate can show the interval of stability of the coating (fig. 2). All samples marked "a" were impregnated, except for SiO_2 sols, with solutions of diammonium hydrogen phosphate and urea of the same concentration under the same conditions of impregnation, wringing and drying, so it can be concluded that the durability of the coating depends, first of all, on the concentration of SiO_2 sol.

Conclusions regarding water resistance of experimental coatings based on processing only optical density curves, of course, are not justified without the results of fire tests after hydrolysis of the coatings.

It is known that fabric impregnation with flame retardant solutions has a negative effect on physical and mechanical characteristics. This is explained by the loosening of cellulose fibers during impregnation. If, at the time of impregnation, the threads of the fabric are loosened into individual fibers, the protective coating should also be destroyed, so it was expected to receive a significant decrease in fire-retardant indicators.

Long-term hydrolysis (within 2–3 days) reduces the time of the beginning of charring of the fabric (fig. 3), but, taking into account the similarity of the shape of the curves for samples without flame retardants and samples additionally impregnated with

flame retardants, we can also conclude about the predominant role of concentration and, accordingly, the degree of homogeneity SiO_2 sol in increasing flame retardant properties. The fire retardant effect of flame retardants is mainly manifested during the determination of the time of the beginning of the destruction of the fabric, the time of final burning and smoldering, because after removing the source of fire, the samples did not burn and did not smolder.

The curves shown in Figures 1–4 agree with each other. In the first 5 hours of hydrolysis, the flame-retardant properties of the samples are significantly higher than those of non-impregnated samples, and the use of SiO_2 sols of low concentration (8–11 %) has a greater effect. The layer of adsorbed water molecules formed during hydrolysis on the surface of the coating cannot be desorbed in the process of drying the samples, so the time for the beginning of charring and destruction is significantly increased. The long-term effect of water neutralizes the effect of the concentration of silica sol, but under the conditions of the participation of flame retardants, it highlights the effect of the degree of homogeneity of the gel coating on the fire-retardant properties.

8. Conclusions

1. The water resistance of the developed compositions of fire-resistant siliceous coatings on textile materials was studied. It was established that partial hydration of the surface of the siliceous coating occurs under the action of water for 2–6 hours, which does not lead to its destruction. The main damage to the coating occurs after 24 hours of hydrolysis. It is shown that the degree of homogeneity of the SiO_2 sol affects the resistance to hydrolysis of the gel coatings. SiO_2 sols of low concentration (8 %), which are characterized by high fluidity and have a long life, demonstrate greater resistance to prolonged action of water.

2. The influence of the hydrolysis term on the fire-retardant properties of impregnated fabric samples was studied. It is shown that even the long-term effect of water provides an increase in the fire-retardant properties of impregnated samples in comparison with non-impregnated fabric samples by 2–2.5 times. It is shown that the concentration and degree of homogeneity of the SiO_2 sol have a predominant effect on the flame retardant properties. A thin layer of coating based on sols of 8–11 % concentration increases the time of the beginning of fabric destruction by 2 times compared to a coating based on SiO_2 sol of 14 % concentration and by 6 times compared to non-impregnated fabric. The surface layer of flame retardants prevents the final burning and smoldering of the samples after removing the fire source, but does not significantly affect numerous indicators of flame-retardant properties.

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ВОДОСТІЙКІСТЬ КРЕМНЕЗЕМИСТИХ ЗАХИСНИХ ПОКРИТТІВ НА ОСНОВІ РІДКОГО СКЛА

Досліджено водостійкість бавовняних текстильних матеріалів, просочених золями SiO_2 які одержані на основі рідкого скла. Експериментальні покриття по тканинах готували ванним методом. Після нанесення кожного шару покриття і видалення зайвого золю експериментальні зразки сушили в сушильній шафі при (60–80) ºС. Просочені золем (концентрацією SiO_2 8, 11 та 14 %) зразки тканини з антипіренами та без них, заглиблювали в ємності з дистильованою водою, дотримуючись однакового співвідношення зразки/вода. Вогнезахисні властивості покриттів визначали після витримування в воді протягом 2–72 годин. Ступінь руйнування покриттів підчас гідролізу досліджували, визначаючи оптичну густину гідролізату над поверхнею зразків за допомогою спектрофотометричного (КФК-2) методу. Вогнезахисні властивості визначали на лабораторній установці проведення вогневих випробувань. Встановлено, що під дією води відбувається часткова гідратація поверхні кремнеземистого покриття, яка не призводе до його руйнування. Наявність шару адсорбованих молекул води на поверхні покриття є причиною додаткового підвищення вогнезахисних властивостей зразків. Показано, що ступінь однорідності золю SiO_2 впливає на стійкість до гідролізу гелевих покриттів. Переважний вплив оказують золі SiO_2 низької концентрації (8 %), які характеризуються високою текучістю та мають тривалий термін життя. Показано, що навіть довготривала дія води зберігає підвищені вогнезахисні властивості просочених зразків у порівнянні з не просоченими зразками тканини. Показано, що переважний вплив на вогнезахисні властивості оказує концентрація та ступінь однорідності золю SiO_2. Поверхневий шар антипіренів запобігає остаточному горінню та тлінню зразків після видалення джерела вогню але не значно впливає на численні показники вогнезахисних властивостей.

Ключові слова: рідке скло, вогнезахисні покриття, текстильні матеріали, водостійкість, пошарова збірка, вогнестійкість

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