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O. Skorodumova, *DSc, Professor, Professor of the Department (ORCID0000-0002-8962-0155)*
O. Tarakhno, *DSc, Professor, Professor of the Department (ORCID0000-0001-9385-9874)*
O. Chebotareva, *Adjunct (ORCID0000-0002-7321-8700)*
K. Bajanova, *Cadet (ORCID 0000-0002-5719-6759)*
National University of Civil Defence of Ukraine, Kharkiv, Ukraine

SILICON PROTECTIVE COATINGS FOR TEXTILE MATERIALS BASED ON LIQUID GLASS

The problem of development of technological principles of obtaining silica coatings on the basis of stable concentrated SiO_2 sols on the basis of liquid glass is solved in the work. The composition was developed and SiO_2 sols based on liquid glass of technical purity were obtained by mixing a solution of liquid glass and acetic acid. Experimental coatings on tissues were prepared by the bath method. After applying each coating layer and removing excess sol, the experimental samples were dried by heating in an oven at (60–80) ºC. The resulting silicic acid solution is characterized by pH5-6, has sufficient resistance to coagulation for about an hour. To improve the quality of impregnation of fabric threads with the composition, ethanol was added in the amount of 5 to 15 vol. %. The obtained compositions were examined by spectrophotometric (CPK-2) and microscopic (Digital Microscope S10) 1000x methods of analysis. The behavior of experimental sols in the induction period of maturation was studied and it was found that the viability of sols increases with increasing alcohol content. It is shown that small amounts of alcohol lead to a decrease in the buffer capacity of the composition and, accordingly, to a decrease in the viability of sols. The alcohol content of 15 vol. % significantly increases the survivability of the sol. The influence of alcohol content on fire-retardant properties of impregnated tissue samples was studied. It has been shown that regardless of the concentration of the SiO_2 sol, 10 % ethanol must be added to the composition to improve the fire-retardant properties of the impregnated tissues. After the fire tests, the fabrics have a fairly dense structure, but all the threads have become much thinner. All samples did not lose their elasticity, the coating did not crumble. Given that the stability of impregnated fabric samples compared to non-impregnated samples increased 5–7 times, it can be concluded that the use of SiO_2 sols based on liquid glass for fire protection of textile materials is promising.

Keywords: liquid glass, siliceous coatings, fire protection of textile materials, fire resistance, sol-gel method

1. Introduction

Recently, the scientific direction on the development of fire-resistant coatings on textile materials using the sol-gel method has been actively developing. This method has been known for a long time, it was developed in the 50 s of the last century. Initially, it was used to obtain self-hardening binders for ceramic products, molding soils for metal casting, hydrophobic substances. Later, sol-gel technology developed in the direction of creating especially pure ceramic materials of a given composition, optical and quartz glass, products for fiber optics, protective coatings for automobile and window glass, and other applications. This technology makes it possible to create new materials with a high degree of homogeneity at the molecular level and with exceptional physical and chemical properties that are significantly different from the properties of materials obtained by traditional methods.

The sol-gel method is based on conducting hydrolysis and condensation reactions of metal alkoxides, such as tetraethoxysilane, tetramethoxysilane, titanium tetraisopropoxide, etc., which leads to the formation of completely inorganic, organic compounds or organic-inorganic hybrids, which are widely used for fire protection of various textile materials purpose [1–3]. These coatings are able to protect the surface of the fabric, creating a physical barrier, improving the fire-resistant properties and combustion characteristics of the treated materials.

The use of sol-gel processes for fixing silicon dioxide nanoparticles on the surface of tissues is well described in the technical literature [4–6]. Usually, the sol-gel approach is used to create new functional properties, such as protection against UV radiation, superhydrophobicity, antimicrobial protection, etc., which additionally opens up new areas for the application of protective coatings [7–9].

However, in some works, conflicting data on the influence of the sol-gel process on increasing the fire resistance of fabrics of various nature have been published. For example, in work [10] it was found that sol-gel treatment acts selectively on different fibers in fabrics: the fire resistance of polyester does not improve, and in mixed fabrics containing 35 % cotton, a noticeable increase in the time to flash was established. In addition, there have been many publications on the negative impact of components of flame retardant compositions, as well as modified fabrics on human health and the environment [11, 12]. The described situation makes it necessary to solve the problem of ensuring safe fire protection of textile materials without losing their functional properties for wide application. Taking into account the importance and complexity of the processes of ensuring the safety of people during a fire, the problem of creating effective elastic fire-resistant siliceous coatings on textile materials is an urgent one.

2. Analysis of literary data and formulation of the problem

Silicon dioxide applied to the fabric by sol-gel technology plays a protective role during the pyrolysis of fabrics, but the degree of influence of the silica coating depends on the nature of the silica precursor, that is, on the conditions of preparation of the original SiO_2 sols, as well as the chemical composition and spatial structure of flame retardant and modifying additives [13].

As an alternative to coatings based on silicon dioxide (derived from tetraethylorthosilicate), oxide phases synthesized from other precursors are used, as well as alkoxides (aluminum tetraethylorthotitanate, zirconate, and isopropylate) that can be applied to cotton fabrics to increase fire resistance**.** Titanium, zirconium, and aluminum oxides significantly increase the fire resistance of treated fabrics, but their effectiveness is lower than that of silica [14].

The use of phosphorus-containing compounds as components of organosilicon sol helps to increase the fire-retardant properties of impregnated fabrics. A high content of phosphorus compounds (30 and 50 wt. %) increases the emission of smoke. On the contrary, with a low content of phosphorus compounds (5 and 15 wt. % in relation to tetramethoxysilane), the fire resistance of the fabric is significantly improved due to the synergistic effect [15]. But the complexity of the synthesis of phosphorus-containing compounds and the toxic effect of flame retardants on the human body and the environment limit the use of such compositions.

There is a known method of obtaining nanoparticles for applying them in the form of aqueous suspensions to fabric fibers, which were used as polyester, cotton, and polyester-cotton mixtures [16]. Similar to the sol-gel process used to create a silicon dioxide screen on the surface of fibers, nanoparticles can also be applied to fabric as a protective layer. 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 tetraethyllorthosilicate, that is, those factors directly affect the amount of silica applied to the surface of the fiber, the distribution of nanoparticles by diameter, as well as thermal stability. However, 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 of the process.

From the point of view of applying SiO_2 nanolayers, silicic acid salts obtained by the

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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 stable sol.

Thus, the problem of developing technological principles for obtaining siliceous coatings on the basis of stable concentrated SiO_2 sols on the basis of liquid glass is not solved.

3. The purpose and objectives of the research

The purpose of the work is the development of a stable concentrated SiO_2 sol based on liquid glass in the pH range of 5–6 for fire protection of textile materials against the action of open fire.

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

– to investigate the influence of the organic solvent content on the rheological properties of the impregnation composition

– to investigate the effect of alcohol content on the fire-resistant properties of siliceous coatings (the time of the beginning of charring, the time of the beginning of the destruction of the fabric and the area of damage to the fabric under the action of fire).

4. Results of studies of the effect of organic solvent on the rheological properties of the impregnation composition and their durability

Liquid glass of technical purity, acetic acid and ethanol were used for research.

Sols based on liquid glass were prepared by mixing aqueous solutions of liquid glass and acetic acid. Experimental coatings on fabrics were prepared by the bath method. After applying each coating layer and removing excess sol, the experimental samples were dried by heating in a drying oven at a temperature of (60–80) ºC (Fig. 1).

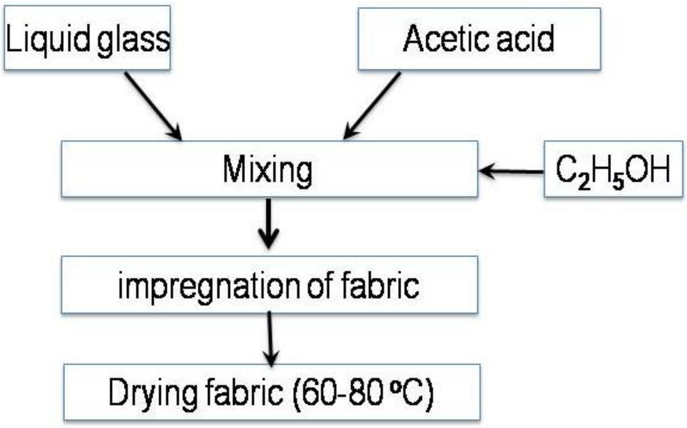

Fig. 1. Scheme of obtaining experimental coatings based on liquid glass

The optical density of sols was studied using a KFK-2 photo colorimeter. The fluidity of the sols was determined by the time it took for the sols to flow out of a burette with a capacity of 10 ml through a standard hole with a diameter of 3 mm. The microstructure of fabric coatings was studied using a Digital Microscope S10 1000x optical microscope.

Fire tests were carried out on a modernized laboratory unit, holding the samples in the upper part of the flame of a gas burner and determining the time of the beginning of charring (τ_{bc}) and the time of the beginning of tissue destruction (τ_{td}). The installation consists of a burner, a gas cylinder with a reducer and a metal horizontal screen. With the help of a manometer installed on the reducer, a constant value of gas pressure was maintained. A hole with a diameter of 35 mm was cut in the center of the protective screen, with the help of which a sample of fabric fixed on top of the screen is brought into contact with the fire. The test process was recorded on a video camera. During the tests, the fabric on the reverse side changed its color from pink to dark yellow, and in places of deep damage – to dark brown.

The damage area of impregnated tissue samples (from the wrong side) was determined at the value of the ignition time of non-impregnated fabric τ_i (8 s), at the value of the time of the beginning of charring (τ_{bc}) and the time of the beginning of destruction (τ_{td}) of the impregnated samples as a percentage of the total area of the sample that was in the fire zone.

In previous studies, we managed to obtain only a time-stable diluted SiO_2 sol of 4 % concentration. The fire-retardant effect of a thin coating based on such sol was recorded, but due to the low concentration of SiO_2, the impregnated fabrics smouldered after the removal of the fire source during fire tests.

In these studies, an attempt was made to obtain SiO_2 salts based on liquid glass of higher concentration (8–10 %). But, an increase in the concentration of sol leads to a decrease in its stability over time, therefore, at the first stage of research, the influence of SiO_2 concentration on the fluidity of sols was determined.

An increase in the concentration of SiO_2 in the sol increases its density, which complicates the uniform impregnation of the fabric. From previous studies, it was established that the impregnation of fabric samples with SiO_2 sols improves when small amounts of ethanol are used, so the influence of the alcohol content in SiO_2 sols of 10 % concentration on changes in fluidity over time was determined (Fig. 2).

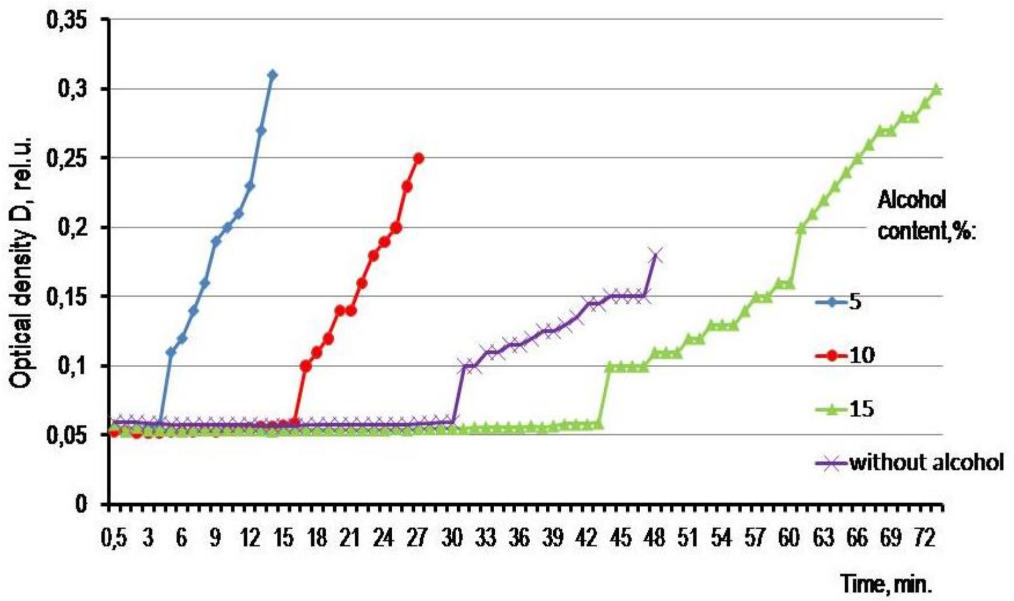

Fig. 2. Changes in the optical density of 8 % SiO_2 sol over time depending on the ethanol content

The effect of the content of the organic solvent on the change in the optical density of the SiO_2 sol over time was studied (Fig. 2), when ethanol in the amount of 5, 10, and 15 vol. % was added to the freshly prepared sol with a 10% concentration of SiO_2 with constant stirring.

The shapes of the curves are similar. All curves have an inflection point at which the process of increasing the optical density of the sols is activated. With an increase in the amount of alcohol, the inflection point shifts to a longer time: in the case of using 5 % alcohol, an increase in optical density is observed after 5 minutes, and an increase in the alcohol

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content to 15 % increases this time to 44 minutes. According to the results of determining the fluidity of sols with different alcohol content, the time during which the sols completely lost their fluidity was determined (Fig. 3).

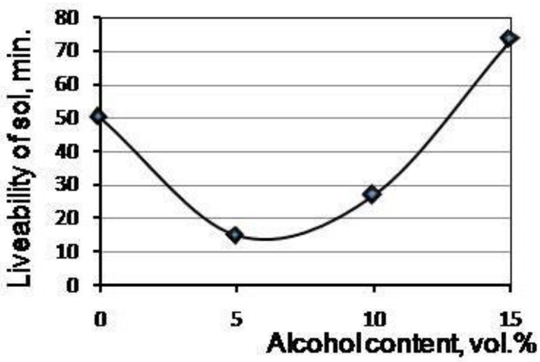

Fig. 3. Effect of alcohol content on the survivability of 8% SiO_2 sol

It can be seen from the graph that the introduction of 5–10 % alcohol reduces the survivability of the sol. Increasing the alcohol content to 15 % leads to an increase in the life of the sol.

5. Study of the effect of alcohol content on the fire-retardant properties of silica coatings

All samples impregnated with SiO_2 sol, with different alcohol content, were subjected to fire tests. Table 1 shows the results of studies of the influence of alcohol content on the time of the start of charring of coatings (τ_{bc}), the time of the start of tissue destruction (τ_{td}), as well as the area of tissue destruction at τ_{td} ($S(\tau_{td})$). To prevent smoldering, a solution of diammonium hydrogen phosphate was applied to the dried silica coating by spraying.

After the fire tests, the fabrics have a fairly dense structure (Fig. 4), but all the threads have become much thinner.

a) b)

c)

Fig. 4. Microstructure of samples impregnated with 8 % SiO2 sol with the addition of 5 % alcohol depending on the number of coating layers: (a) 1; (b) 2; (c) 3

Tabl. 1. Composition and fire-retardant properties of protective coatings based on liquid glass

Sample No	SiO_2 concentration, %	Alcohol content in sol, %	The number of coating layers	Carbonization start time, sec	Time of onset of destruction τ_{td}, min	Area of destruction $S(\tau_{td})$, mm^2
1	8	5	1	8	6,04	38
2			2	7	6,07	72
3			3	7	6,42	6
4		10	1	8	2,53	16
5			2	7	5,15	36
6			3	7	5,08	48
7		15	1	7	6,44	31
8			2	6	5,47	29
9			3	8	6,10	36
10	10	5	1	7	6,58	43
11			2	8	7,18	29
12			3	7	1,19	32
13		10	1	8	5,32	8
14			2	7	6,13	38
15			3	7	4,21	50
16		15	1	7	6,45	51
17			2	7	5,39	12
18			3	8	2,04	without flame retardant smoldering 3 min
19		without alcohol	1	7	5,19	50
20	non-impregnated fabric sample		-	7	1,02	completely burnt

An increase in the number of fabric impregnations, that is, the thickness of the protective layer of the coating, leads to an increase in the time of the beginning of fabric destruction. But the results shown in the table do not show a clearly established regularity of the dependence of the time of the beginning of destruction and the area of tissue damage on the number of layers of impregnation. The content of alcohol in the impregnation composition has a noticeable effect on the changes in the listed properties, which allows us to conclude that the 10 % ethanol content in SiO_2 sols is necessary, despite the fact that this content reduces the survivability of the sols by almost half.

6. Discussion of the results of the influence of the organic solvent on the rheological and flame-retardant properties of the impregnation composition

The buffer effect of the acetic acid used and the sodium acetate formed during the preparation of the protective composition ensures that the pH of the sol is maintained in the range of 5–6. This pH value is necessary for obtaining long-lasting SiO_2 sols of low concentration. An increase in the mass fraction of SiO_2 in the sol leads to an increase in the viscosity of the sol, reduces its fluidity and service life, and complicates the uniform impregnation of the fabric. Therefore, a substance capable of reducing surface tension – ethanol, was added to the composition. But, taking into account that after drying the

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impregnated samples, a certain amount of alcohol remains adsorbed on the surface of the fabric, residual burning or smoldering of the sample may be observed during fire tests. Therefore, it was necessary to determine the minimum amount of alcohol, which would be sufficient to reduce surface tension and improve impregnation, but would not be enough for residual smoldering.

The introduction of small amounts of alcohol probably reduces the buffer capacity of the acetate buffer solution formed at the time of preparation of the sol-gel composition due to the reaction of acetic acid with alcohol with the formation of a complex ether – ethyl acetate. The presence of sodium ions in the sol can also lead to the formation of sodium ethoxide. Simultaneously with the listed reactions in the sol, polycondensation processes of silicic acid occur with the formation of di-, tri- and tetrameters as the main components necessary for the formation of primary SiO_2 particles at the nanolevel. Condensation water can participate in the hydrolysis of ester and sodium ethoxide. During the passage of the listed reactions, a change in the pH value during the induction period of sol maturation is possible and, as a result, a change in the optical density of the sol and the life span. If the alcohol content increases, the effect of changing the buffer capacity is leveled and the pH of the sol is stabilized, which ensures an increase in the survivability of the SiO_2 sol.

Conducting fire studies showed that a non-impregnated sample of cotton fabric burned out in 1 minute. When the source of fire was removed, the fabric continued to actively burn and smolder for 5 minutes until complete destruction, so the area of complete burning was 2340 mm^2.

Applying a silicate coating to the fabric increased its resistance to burning for up to 5 minutes (sample 19), but did not prevent residual decay. When applying diammonium hydrogen phosphate as a 20 % solution to the surface of the fabric, no residual burning or smoldering was observed after fire tests of impregnated samples.

When increasing the thickness of the coating based on sol of 8 % concentration, the time of the beginning of carbonization of the impregnated samples practically did not change, but the time of the beginning of destruction increased significantly (by 3–4 times) regardless of the alcohol content. At the same time, the area of fabric destruction was minimal under the condition of three-time application of the coating with an alcohol content of 5 %.

After exposure to fire for 6 minutes (Fig. 4 a), first of all, only those threads of the fabric that were closer to the fire are charred. As can be seen from the figure, the sample that was in the fire zone for 6 min 42 sec was characterized by a minimal area of destruction (6 mm_2) and had a dense structure. All samples did not lose their elasticity, the coatings did not crumble.

In the case of the introduction of 15 % alcohol, the samples were exposed to fire to a greater extent: the fibers of the threads became thinner (higher shrinkage), the fabric was destroyed, forming a large crack.

When fabric is impregnated with SiO_2 sol of 10 % concentration, the picture changes slightly. Since the sol has a higher viscosity, part of the alcohol probably remains in the fabric after drying, which is the reason for the reduction in the time of the onset of destruction of the impregnated samples.

An increase in the number of fabric impregnations, that is, the thickness of the protective layer of the coating, leads to an increase in the time of the beginning of fabric destruction. But the results shown in the table do not show a clearly established regularity of the dependence of the time of the beginning of destruction and the area of tissue damage on the number of layers of impregnation. The content of alcohol in the impregnation composition has a noticeable effect on the changes in the listed properties, which allows us to conclude that the 10 % ethanol content in SiO_2 sols is necessary, despite the fact that this content reduces the survivability of the sols by almost half.

Taking into account that the resistance of impregnated fabric samples compared to non-impregnated samples increased by 5–7 times, it can be concluded that the use of SiO_2 sols based on liquid glass is promising for fire protection of textile materials.

The developed flame retardant composition does not contain harmful components, is characterized by ease of manufacture, does not require expenditure of energy, and has a low manufacturing cost, which favorably distinguishes it from foreign analogues. The first results obtained will form the basis of further research on determining the optimal composition of the fire-retardant composition in relation to the concentration of SiO_2 and the amount and concentration of flame retardants.

7. Conclusions

1. The composition was developed and SiO_2 sols were obtained based on liquid glass of technical purity by mixing a solution of liquid glass and acetic acid. The effect of ethanol content on the optical density and durability of SiO_2 sols based on liquid glass was investigated. It is shown that small amounts of alcohol lead to a decrease in the buffer capacity of the composition and, accordingly, to a decrease in the viability of the sols. The alcohol content of 15 vol. % significantly increases the durability of the sol.

2. The effect of alcohol content on the fire-retardant properties of impregnated fabric samples was studied. It is shown that, regardless of the concentration of SiO_2 sol, it is necessary to introduce 10 % ethanol into the composition to improve the fire-resistant characteristics of impregnated fabrics. Given the lack of a clear regularity of the influence of alcohol content on the fire-retardant properties of fabrics, it seems appropriate to study the behavior of SiO_2 sols based on liquid glass without alcohol over time and the influence of the concentration of SiO_2 sol on the fire-retardant properties of coatings. The resistance of impregnated fabric samples compared to non-impregnated samples increased by 5–7 times, so it was concluded that the use of SiO_2 sols based on liquid glass is promising for fire protection of textile materials.

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О. Б.Скородумова, *д.т.н., професор, проф. каф.*
О. В. Тарахно, *д.т.н., професор, проф. каф.*
О. М. Чеботарьова, *ад'юнкт*
К. В. Бажанова, *курсант*
Національний університет цивільного захисту України, Харків, Україна

КРЕМНЕЗЕМИСТІ ЗАХИСНІ ПОКРИТТЯ ПО ТЕКСТИЛЬНИХ МАТЕРІАЛАХ НА ОСНОВІ РІДКОГО СКЛА

Вирішено проблему розроблення технологічних принципів одержання кремнеземистих покриттів на основі стійких концентрованих золів SiO_2 на основі рідкого скла. Розроблено склад та одержано золі SiO_2 на основі рідкого скла технічного рівня чистоти змішуванням розчину рідкого скла та оцтової кислоти. Експериментальні покриття по тканинах готували ванним методом. Після нанесення кожного шару покриття і видалення зайвого золю експериментальні зразки сушили при нагріванні в сушильній шафі при (60–80) ºC. Одержаний розчин кремнекислоти характеризується pH 5–6, має достатню стійкість до коагуляції протягом часу близько години. Для підвищення якості просочення ниток тканини композицією в неї додавали етанол у кількості 5–15 об. %. Одержані композиції досліджували спектрофотометричним (КФК-2) та мікроскопічним (Digital Microscope S10) 1000 х методами аналізу. Досліджено поведінку експериментальних золів в індукційному періоді дозрівання та встановлено, що живучість золів зростає з підвищенням вмісту спирту. Показано, що малі кількості спирту призводять до зменшення буферної ємності композиції та, відповідно, до зменшення живучості золів. Вміст спирту 15 об. % значно збільшує живучість золю. Досліджено вплив вмісту спирту на вогнезахисні властивості просочених зразків тканини. Показано, що незалежно від концентрації золю SiO_2 необхідно вводити у композицію 10 % етанолу для покращення вогнезахисних характеристик просочених тканин. Після вогневих випробувань тканини мають досить щільну структуру, не втрачають своєї еластичності, покриття не обсипалися. Стійкість просочених зразків тканини у порівнянні з не просоченими зразками збільшувалась у 5–7 разів, тому зроблено висновок про перспективність використання золів SiO_2 на основі рідкого скла для вогнезахисту текстильних матеріалів.

Ключові слова: рідке скло, кремнеземисті покриття, вогнезахист текстильних матеріалів, вогнестійкість, золь-гель метод

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