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Improving the Fire-Retardant Properties of Cotton-Containing Textile Materials through the Use of Organo-Inorganic SiO_2 Sols

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Abstract. The effect of fire-retardant compositions based on organo-inorganic SiO_2 sols without the use of flame retardants on increasing the fire resistance of cotton-containing textile materials has been studied. Previous studies have shown that the combined use of silicate coatings and flame retardants of simple structure prevents the ignition of impregnated samples of cotton-containing fabric and the proposed chemistry of this process. These studies did not use flame retardants to obtain more information on the effect of the composition and concentration of experimental organo-inorganic SiO_2 sols on the fire resistance of treated tissues. The compositions were prepared by the sol-gel method using a technical product – liquid glass – with a silicate modulus of 2,5 and acetic acid solution. The influence of sols concentration in terms of conditioned SiO_2 on their rheological features has been studied. It was found that the optical density, fluidity and survivability of experimental sols depend on the concentration of SiO_2 in the sol. The compositions were applied to the fabric by the bath method in layers: each layer of the coating was fixed on the surface of the fabric by thermal shock in an oven at temperatures of 80-100 ºC. Untreated cotton fabric caught fire under the influence of fire after 7 seconds. Impregnated tissue samples did not ignite under the action of fire, but gradually charred. To determine the effect of coating thickness and SiO_2 concentration in the sol on the fire resistance of impregnated fabric samples, fire tests at a gas pressure of 0,2 MPa were performed for 8 s and the area of damage to the reverse side of the fabric was determined. Prolonged exposure to the flame resulted in a crack in the center of the charred spot. In the absence of flame retardants in the fire-retardant composition after removal of the fire source, decay was observed. The time for which a crack is formed in the sample under the action of fire was determined at the time of onset of tissue destruction. It is shown that in the case of using a sol of 10% concentration it is necessary to apply three layers of coating, which doubles the fire resistance (from 7 s for untreated fabric to 15 s for impregnated fabric), the start time of destruction increases 5 times (from 1 min to 5,5 min). The area of damage to the reverse side of the fabric not treated and impregnated with the experimental coating is reduced from 632 mm^2 to 0, respectively.

1 Introduction

SiO_2-based coatings obtained by the sol-gel process [1-3] are promising for improving the fire protection of textile materials. In these scientific studies, the processes that occur during the transition of the sol to the gel in the case of the use of organosilicon raw materials are described in sufficient detail [4].

However, in these publications the emphasis is on the processes of gelation, which occur directly on the surface of the fabric in the case of applying layer by layer of components [5]. In this way, it is possible to obtain an extremely heterogeneous coating both in composition and in thickness, which significantly reduces the fire-retardant properties of coatings.

Complex phosphorus-nitrogen-containing organosilicon substances capable of reacting with cellulose of fabric threads and inorganic flame retardant salts are often proposed as starting materials [6]. The main disadvantages in this case are the high cost of the components of the compositions, the rather complex technology of their production, as well as the negative impact on the environment [7-10]. The authors [11] proposed ways to form fire-retardant coatings based on

ethyl silicate gels and well-known simple flame retardants, which greatly facilitates the technology of their production, increases fire protection of textile materials, reduces the release of toxic gases [12], but does not reduce cost due to high prices for organosilicon raw materials, even the technical level of purity. From this point of view, silicic acid sols are interesting, which can be easily obtained by the action of mineral acids on sodium silicate (liquid glass). However, the publications in the technical literature considered the technological aspects of liquid glass processing and the stages of aging of the obtained gels in terms of obtaining gel powders of the desired structure, porosity or pore morphology, but not stable sol [13, 14].

2 Unresolved Issues

Protective coatings based on sodium silicate or liquid glass have long been used in various fields for various purposes: for fire protection of wooden structures [14, 15], for creating foaming or gel-forming compositions for extinguishing flammable liquids [15], etc. The industry produces silica-based silica sols as silicate binders and adhesives, but these sols are stabilized by alkali metal ions to prevent premature coagulation, so they have a large colloidal particle size with deactivated surface and are characterized by high pH values (11-13).

Impregnation of textile materials to increase fire resistance is associated with some technological difficulties, such as the need to preserve the appearance of impregnated fabric and products, maintaining the strength of textile materials, which is a very important indicator of their use as upholstery, protective suits, etc. Of course, high pH values of silica sols do not allow achieving these objectives.

In view of the above, the aim was to develop a stable concentrated sol SiO_2 based on liquid glass in the pH range of 5-6 for fire protection of textile materials from open flame.

The lack of theoretical and experimental data suggests solving the main tasks of this study: to determine the effect of the concentration of developed SiO_2 sols on their rheological characteristics, as well as the ability to fire protection of textile materials.

3 Main Part

Sol based on liquid glass was prepared by mixing aqueous solutions of liquid glass and acetic acid.

It is known that SiO_2 sols in the induction period do not lose their fluidity, do not change externally, but at the nanolevel they undergo significant changes: colloidal SiO_2 particles increase in size due to the effect of recondensation. The size of colloidal particles increases, but their total number decreases. It can be assumed that the optical density of the sol will change at this time, so we studied the change in the optical density of the sols in the time before coagulation, using a photocolorimeter KFK-2.

Increasing the concentration of the SiO_2 sols leads to an increase in the optical density of the sols. The formation of associates in the experimental sols is observed on the optical density curves in the form of steps. The beginning of the formation of spatial structures in the ash (large associates) with increasing concentration of the sol is observed the earlier the higher the concentration of the sol (Fig. 1).

Simultaneously with the measurement of the optical density of the experimental sols, the fluidity was determined every 2 min until the moment of its complete loss, by determining the time in seconds at which the sol flows out of a burette with a capacity of 10 ml and a hole with a diameter of 3 mm. The nature of the curves of changes in the fluidity of experimental SiO_2 sols over time in shape almost coincided with the shape of the curves of changes in optical density.

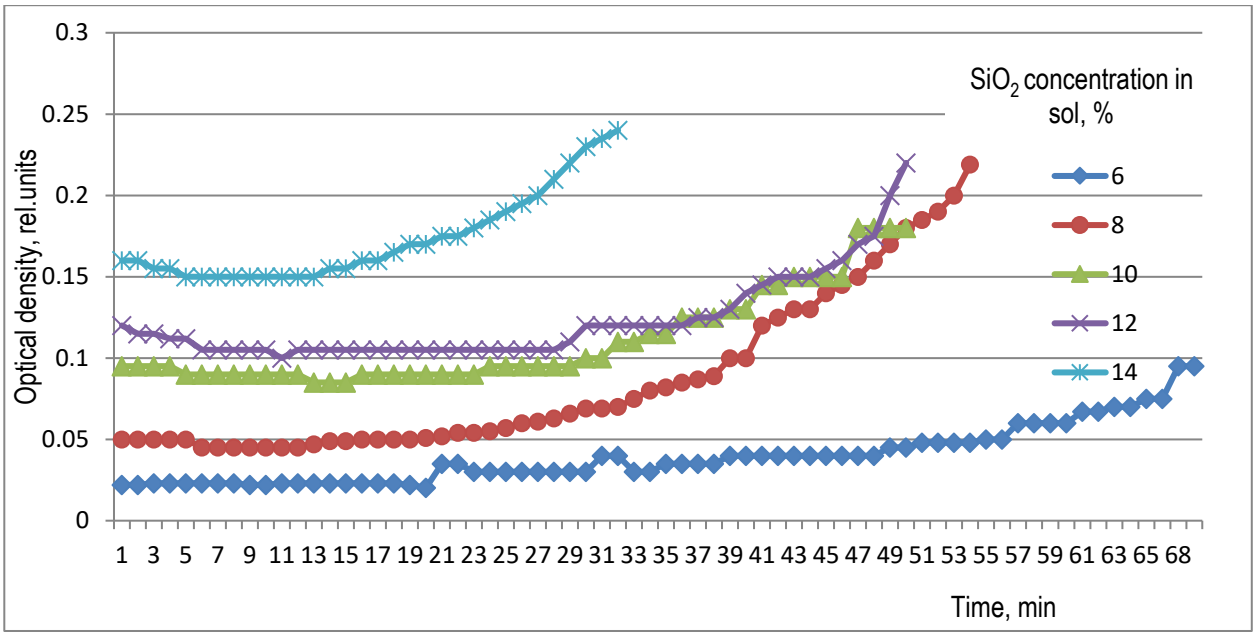

Fig. 1. Changes in the optical density of sols over time

The viability of sols was defined as the time in minutes at which the sols finally lost fluidity (Fig. 2). Increasing the concentration of SiO_2 sol to 14% reduces the survivability of the sol by half (from 70 to 30 minutes). In the range of concentrations of 8-12% SiO_2 survivability practically does not change and is at 50 minutes, which is sufficient to impregnate even a large number of tissue samples.

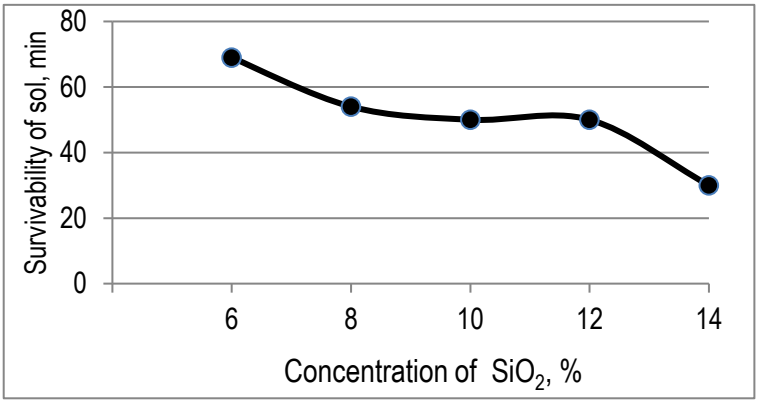

Fig. 2. Dependence of sol survival on SiO_2 concentration

The experimental compositions were applied to the tissue by the bath method in layers. Each layer of coating was fixed on the fabric surface by thermal shock in an oven at temperatures of 80-100 ºC.

Untreated cotton fabric caught fire under the influence of fire after 7 seconds. Impregnated tissue samples under the action of fire (gas pressure was 0,2 MPa) did not ignite, but gradually charred. In the absence of flame retardants in the fire-retardant composition after removal of the fire source, the final decay was observed. 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. Prolonged exposure to the flame resulted in a crack in the center of the charred spot. The time for which a crack is formed in the sample under the action of fire was determined at the time of onset of tissue destruction. The effect of coating thickness and SiO_2 concentration in the sol on the fire resistance of impregnated fabric samples under the action of flame for 8 s was determined as the area of damage to the reverse side of the fabric.

Table 1 shows the results of fire tests of coatings based on sols 10-14% concentration. Given that the silica coating on the fibers of the fabric is very thin, without the use of flame retardants, the final decay is observed.

Table 1. Compositions of impregnating compositions and the results of fire tests of impregnated cotton samples

№ sample	SiO_2 concentration, [%]	Number of layers of coating	Charging start time, [sec]	The area of charring the reverse side at 8 sec	Time of the beginning of destruction, [min]	Area of destruction, [mm^2]
20	not impregnated	-	7	632	1,02	completely burned
21	10	1	6	50	1,36	final decay
22		2	8	22	1,41	
23		3	15	0	5,39	
24	12	1	7	277	1,40	
25		2	9	89	5,18	
26		3	10	0	2,38	
27	14	1	7	299	1,48	
28		2	8	50	2,12	
29		3	9	0	1,47	

As the sol concentration increases, the process of impregnation of tissue samples accelerates. The resistance of impregnated fabrics to the action of flame depends not only on the concentration of the sol, but also on the thickness of the coating, i.e. the number of impregnations of the fabric with sol.

Fig. 3 shows a sharp increase in the time of onset of charring (twice) when using a sol of 10% concentration for three times impregnation of tissue samples.

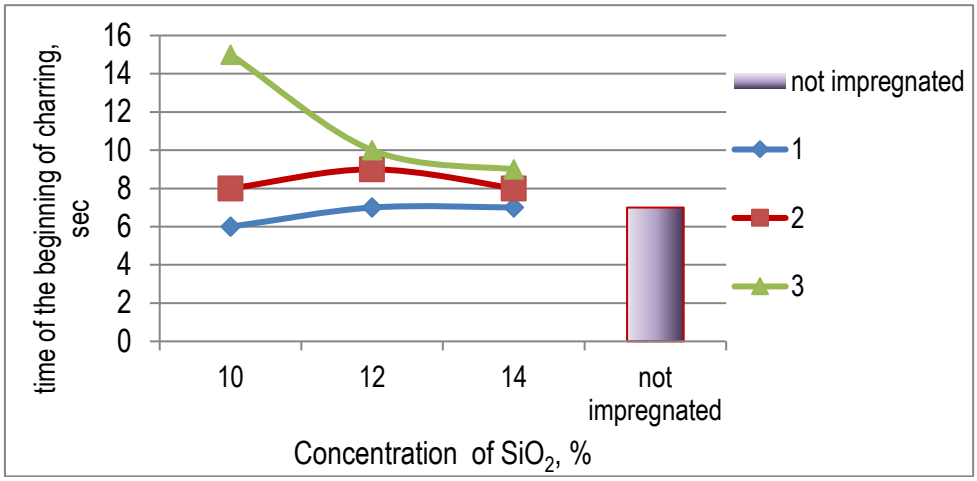

Fig. 3. Influence of SiO_2 sol concentration on the time of onset of charring of impregnated cotton samples

Sol-based coatings of 12% concentration also showed satisfactory results. The time of onset of tissue destruction also depends not only on the concentration but also on the number of layers of the applied coating. There is a pattern here: the higher the concentration of the sol, the less layers of coating are required. As can be seen from Fig. 4, in the case of using 10% SiO_2 sol, the fabric must be impregnated three times. Increasing the concentration to 12% reduces the amount of impregnation to two, which is important to maintain the softness and elasticity of the fabric.

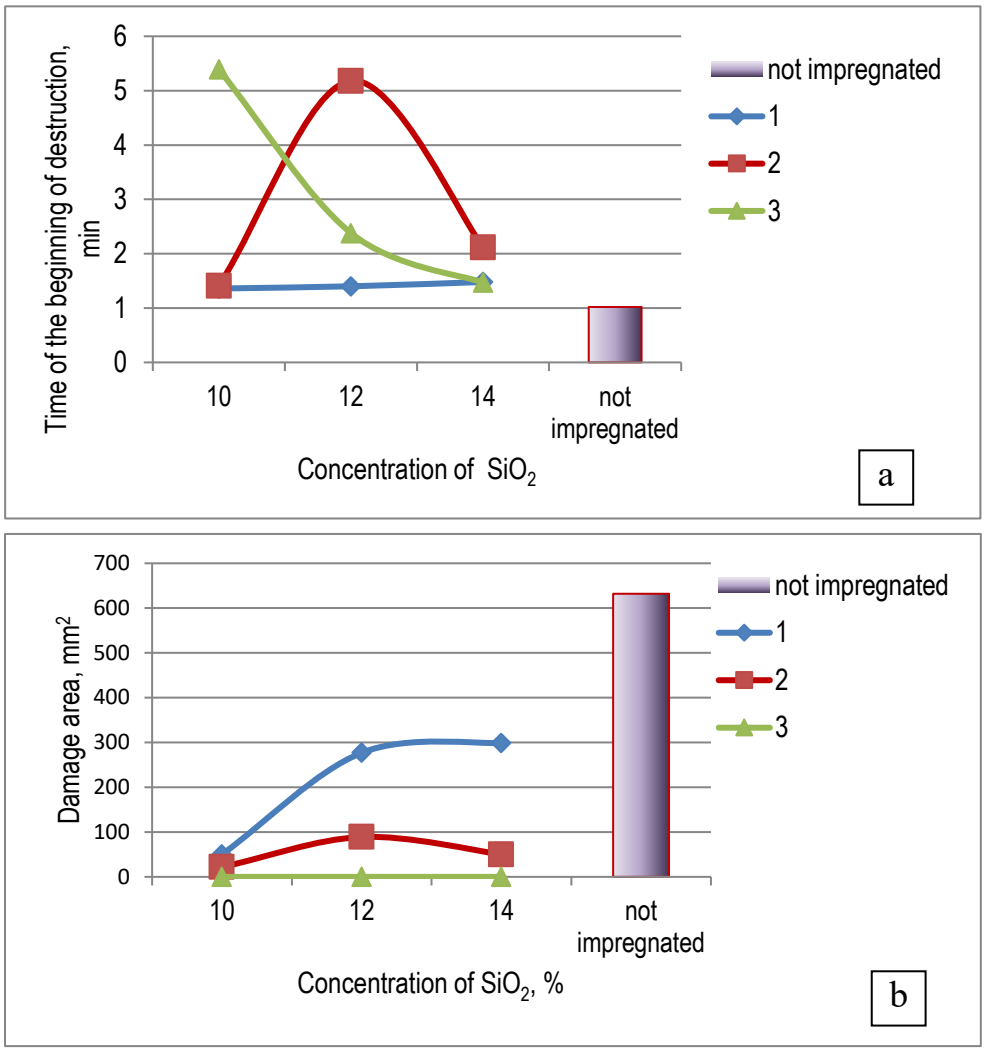

Fig. 4. Dependence of the time of onset of destruction (a) and the area of damage (b) of impregnated samples on the concentration of SiO_2 sol

In the case of short-term fire or under the influence of low-calorie flame sources, the area of damage to the reverse side of the impregnated samples is reduced by half under conditions of single impregnation (Fig. 4). The use of triple impregnation reliably protects the fabric from the effects of fire, so on the reverse side of the fabric thermal changes were not observed at all. Thus, it can be concluded that the optimal range of concentrations of SiO_2 sol based on liquid glass is 10-12%.

4 Conclusion

The composition of SiO_2 sols based on liquid glass was developed and their rheological properties were studied. It is established that with increasing sol concentration the interval of reliable preservation of its rheological properties is 30-50 min.

The influence of sol concentration based on liquid glass on fire-retardant properties of impregnated cotton samples was determined. It is shown that the use of 10% concentration of sol provides an increase in the time of onset of impregnation of the impregnated fabric twice, the time of onset of destruction – 5 times, and the area of damage on the reverse side of the samples can be reduced to almost zero using triple impregnation.

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