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Fire-Resistant Coatings, Obtained by Layer-by-Layer Assembly, in the System of Silicic Acid Gel – Diammonium Hydrogen Phosphate – Urea

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Abstract. The paper discusses the influence of flame retardant compositions obtained in the system of silicic acid sol (SiO_2 sol) – flame retardants on the fire retardant properties of thin dense cotton fabrics and low density voluminous tapestry fabrics. The need to develop the optimal composition of a fire-retardant composition for a specific fabric, or to unify it for two main groups of fabric: thin and bulky low-density ones, is substantiated. Experimental coatings were obtained by applying SiO_2 sol, which was obtained by the reaction between liquid glass and acetic acid, followed by application of flame retardant solutions (diammonium hydrogen phosphate (DAHP) and urea) by spraying or by the bath method. As a result of the optimization, using the central composite uniform rotatable plan of the second order, it was established that the main effect of the flame retardant is exerted by diammonium hydrogen phosphate (DAHP). The content or concentration of urea depends on the concentration of DAHP used: if diluted DAHP solutions (9–10 %) are used, trace amounts of urea (0–0.5 %) must be added. In the case of using a concentrated DAHP solution (18–20 %), the concentration of the urea solution should also be increased to 8–10 %.

1 Introduction

The high tendency of textile materials to ignite often limits their use in places of large crowds. The release of a large amount of heat from the burning of textiles adds to the problems not only during alley fires during its extinguishing. Therefore, an important issue is the development of new ways of reducing the fire hazard from textile materials.

In general, the issue of choosing the optimal method of applying a protective coating on textile materials is widely discussed in the technical literature [1–3]. The most promising are the sol-gel method [4, 5] and layer-by-layer assembly [6, 7], which make it possible to use organosilicon substances, which, as a result of the main stages of the sol-gel transition, become the basis of a silicate coating capable of significantly increasing the fire resistance of the fabric. Usually, the sol-gel method is combined with the coating method - layer-by-layer assembly [8, 9]. This allows to control not only the phase composition, but also the coating process.

Layer-by-layer assembly is performed by various methods: coprecipitation of nanoparticles, the sol-gel method, and their combination by alternately applying layers of a certain chemical composition with subsequent joining of these layers either by chemical reactions or heat treatment [10]. As a result, a multilayer coating with high adhesion to the surface of fabric fibers is formed.

The application of protective coatings is widely used in technology for the production of highly conductive fabrics, fire alarm sensors, electronic textiles, protective suits, etc. [11].

Layer-by-layer assembly was first used for coating acrylic fibers, as a result of which it became possible to change not only the fire resistance of the fabric, but also heat resistance and smoke emission [12]. Depending on the chemical composition of the coating layers, it is possible not only to create a heat-insulating barrier on the fabric fibers, but also to achieve close contact of the coating layers during layer-by-layer assembly, which affects the significant modification of the mechanism of thermal degradation of the fabric [13]. It should be taken into account that the number and sequence of layers applied by layer-by-layer assembly also depends on the chemical composition of the initial components.

The method of layer-by-layer assembly is often used to obtain intumescent multilayer coatings, which can swell under the influence of fire, increasing the thickness of the coating and thermally insulating the fabric [14]. Usually, such coatings are relevant for upholstery and finishing materials, for example, in places with a large crowd of people. But in this case, together with the decrease in the rate of heat release and the decrease in temperature on the surface of the coating, the emission of toxic smoke is still not suppressed.

2 Unresolved issues

During the thermal decomposition of flame retardant compositions, the products of thermal destruction of organophosphorus, nitrogen and halogen-containing flame retardant compounds [15–17] make a large contribution to the level of smoke toxicity. Organophosphorus compounds capable of decomposing into toxic products under the influence of high temperatures have attracted the attention of ecologists from different countries of the world [18–20]. A significant number of publications are known about contamination of the environment [21, 22], soil, air [23, 24], river and sea water [25], including by decomposition products of phosphorus, nitrogen, and halogen-containing flame retardants [26, 27]. The authors [28] consider the possibility of using epoxyamine compositions for building materials based on SiO2, Al2O3, Fe2O3, TiO2, Cr2O3, etc. oxides. At the same time, the cost and flammability are reduced, and it becomes possible to regulate most of the technological and operational properties in a wide range.

Therefore, there is a need to create such fire-resistant compositions that would allow preserving the integrity of the textile material from the action of small fire sources, and in the conditions of a large-scale fire, would not release a large amount of toxic decomposition products [29, 30].

Textile materials of various purposes differ, first of all, in the nature of the interweaving of threads, and this, in turn, creates the problem of high-quality impregnation with a fire-retardant composition. It is known from previous studies that it is better to apply SiO_2 sol to the fabric by the bath method with subsequent removal of excess sol [31, 32]. This is connected with the need not only to wet the surface of all fibers with SiO_2 sol, but also to make a layer of sol more or less uniform in thickness, in which the process of polycondensation is accelerated during drying in the thermal shock mode [33, 34]. The uneven thickness of the sol, which is subjected to accelerated polycondensation, leads to the formation of local stresses, which are the centers of defects that can develop in the form of cracks during fire tests and reduce the fire resistance of the treated fabrics [35].

Flame retardant solutions can be applied both by the bath method and by spraying. First of all, the structure of the fabric is taken into account: its thickness, chemical composition (whether it is mixed), weave density, weave uniformity, etc. Of course, if a thin dense fabric is used for research, such as chintz or pure cotton dense fabric, it is sufficient to use the flame retardant spraying method. At the same time, one flame retardant, or several different ones (in stages or in the form of a mixture) is evenly applied to the surface of the silicate coating layer in the minimum amount necessary to wet the surface of the gel coating.

In the case of impregnation of low-density fabrics (for example, tapestry or wool), spraying does not succeed in evenly applying a layer of flame retardant, so the bath method for applying flame retardants is also used for such fabrics.

Thus, the optimal composition of the fire-resistant coating must be calculated either for a specific fabric, or to unify it for two main groups of fabric: thin and bulky, low density.

The aim of the work was to establish the optimal compositions of fire-retardant compositions of the silicic acid sol (SiO_2 sol) – flame retardants (diammonium hydrogen phosphate and urea) system.

3 Main Part

Thin pure cotton fabric (CF) and mixed tapestry (TF) were used for research. Silicic acid sols, which were prepared by mixing an aqueous solution of liquid glass with a solution of acetic acid, were applied to the fabric by the bath method, the excess sols were removed on wringing rolls and dried at temperatures of 80–100 ºC. SiO_2 sol with a concentration in the range of 6–16 % was applied three times in order to more easily detect the effect of inhomogeneities in the gel coating on its fire-retardant properties.

After drying the gel coating, aqueous solutions of flame retardants were applied to the fabrics in two ways: by spraying (on thin fabric) and by the bath method (on tapestry fabric).

During the fire tests, the time of appearance of signs of charring and destruction of the fabric, as well as the time of final burning and smoldering were determined. Taking into account that the untreated cotton fabric caught fire after 7s of exposure to fire, the area of damage of treated fabric samples was determined by exposing them to fire for 8s. The untreated fabric of the tapestry caught fire after 1 s of exposure to fire, so the area of damage was determined after exposure to fire for 6 s.

Fire tests of low density tapestry fabric (TF). An increase in the concentration of DAHP with the same content of urea (5 %) leads to a decrease in fire-resistant properties (Fig. 1): the time of the beginning of the destruction of the fabric decreases by 5 times, the fabric is more easily destroyed by fire, which is accompanied by an increase in the area of damage (by 2.5 times) . It can be concluded that increasing the ratio of DAHP/urea from 2/1 to 4/1 has a negative effect on the flame retardant properties.

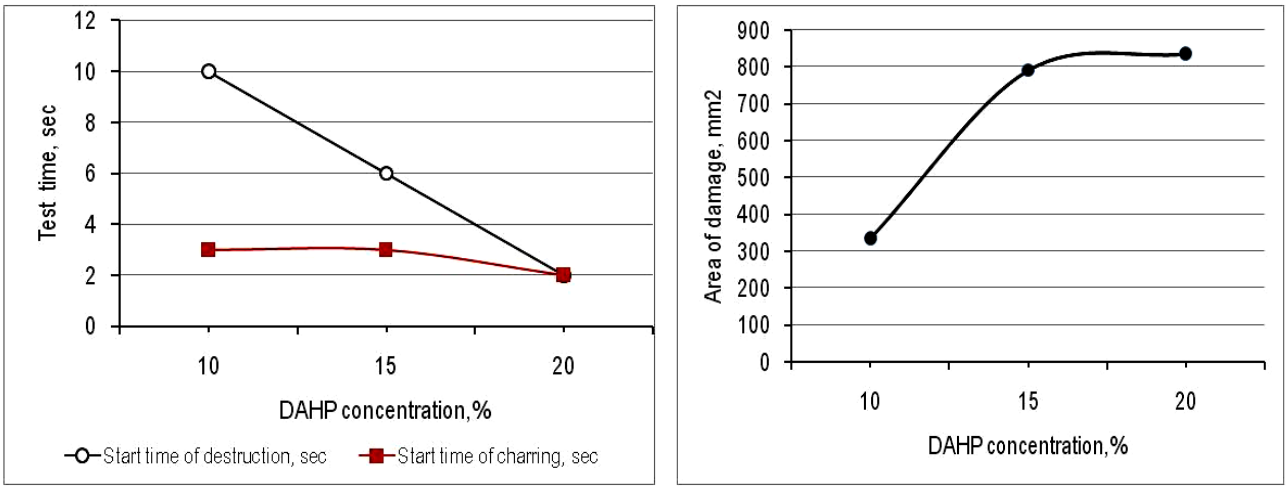

Fig.1. The influence of the concentration of diammonium hydrogen phosphate solution on changes in the fire-retardant properties of TF samples impregnated with SiO_2 sol of 11% concentration.

It should be noted that the time of the start of charring of tapestry fabric samples practically does not change depending on the concentration of the DAHP solution: charring begins almost immediately after the sample is introduced into the fire zone.

Fig. 2 shows the test results of samples of tapestry fabric treated with flame-retardants in the ratio DAHP/urea = 3/1. The time of the onset of destruction largely depends not only on the content of flame-retardants, but also on the concentration of SiO_2 sol. The graph shows that an increase in the concentration of SiO_2 sol leads to a sharp increase in the time of the beginning of tissue destruction. At the same time, the area of damage to the samples is much smaller compared to non-impregnated fabric. Judging by the shape of the graphs, it is possible to conclude that there are two possible ranges of SiO_2 sol concentration in which the fire-retardant properties of coatings have maximum values: 8–10 % and about 16 %.

The obvious influence of the concentration of SiO_2 sol can also be traced on the graph of the change in the time of the beginning of charring. The curves for samples with and without flame retardants have a similar shape (Fig. 2). Comparing the shape of the curves of the beginning of charring in Fig. 1. and 2, it can be concluded that the charring time is more affected by the concentration of SiO_2 sol than by the ratio of flame retardants.

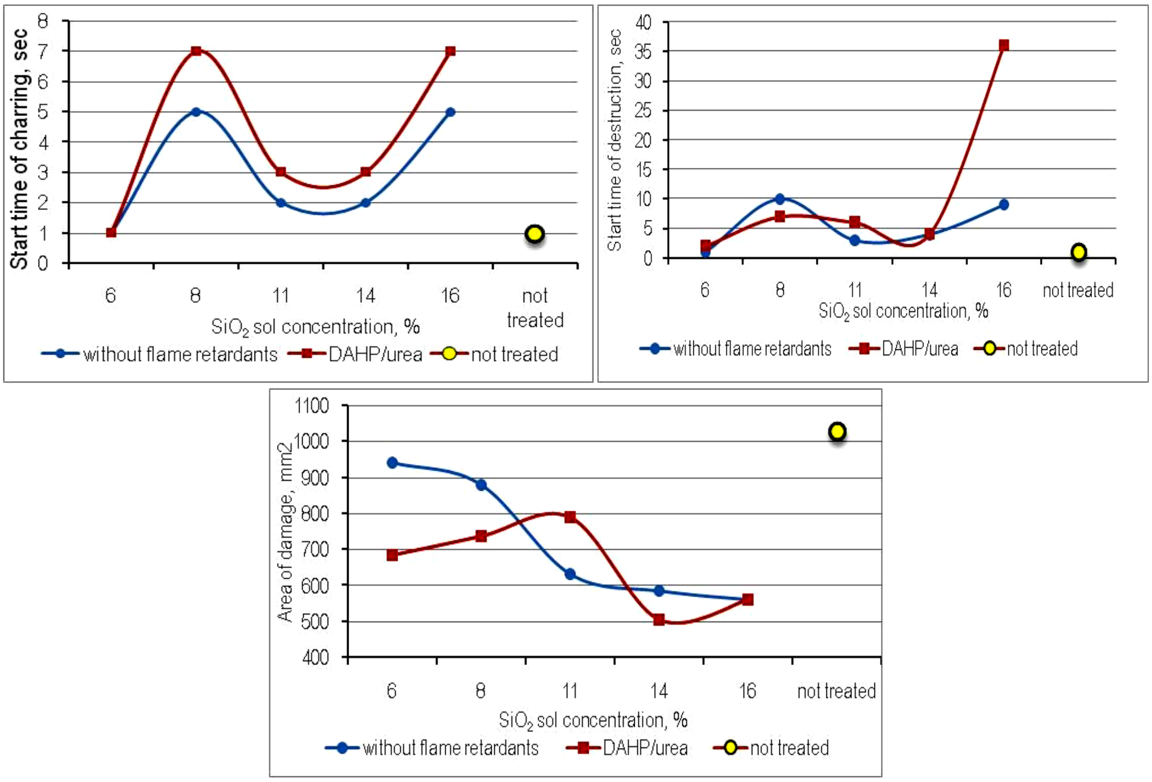

Fig.2. Change in the time of the beginning of carbonization of treated TF samples depending on the concentration of SiO_2 sol.

Fire tests of thin cotton fabric (CF). Fig. 3 shows the dependence of fire-resistant characteristics of cotton fabric on the concentration of SiO_2 sol.

An increase in the concentration of sol leads to a decrease in the time of the beginning of tissue destruction. Of course, even the worst result (21 s) was 2.5 times better than the result of untreated tissue. But the graph clearly shows a sharp decrease (by 2 times) in the time of the start of destruction of coatings based on ash with a concentration higher than 11 %. This may be the result of an increase in the thickness of the coating. Considering that the three layers of the coating are connected to each other due to the condensation of surface hydroxyl groups during heat treatment, the layers are connected to each other unevenly, so inhomogeneities are formed in the coating. The higher the sol concentration, the thicker and less uniform the coating.

The same dependences are observed in the case of tests at the time of the beginning of charring: the curve also has an inflection at the point corresponding to a concentration of 11 %.

The graph of the dependence of the area of damage on the concentration of SiO_2 sol shows that an increase in the concentration of sol to 16 % is not promising: the area of damage increases sharply. The use of flame-retardants mainly prevents the final smoldering of the samples, but does not increase the time of onset of charring and destruction of the tissue. This is probably explained by the uneven detachment of surface OH groups during fire tests, which is the reason for the detachment of flame retardant molecules as well.

Thus, it was established that increasing the coating layers increases fire-retardant properties when using low concentrations of SiO_2 sol (8–10 %) and significantly reduces them when using more concentrated SiO_2 sol (11–16 %).

It is possible to conclude that it is necessary to optimize the impregnation compositions from the point of view of not only the concentration of SiO_2 sol, but also the concentration of flame retardants applied to the surface of the gel coating and their ratio.

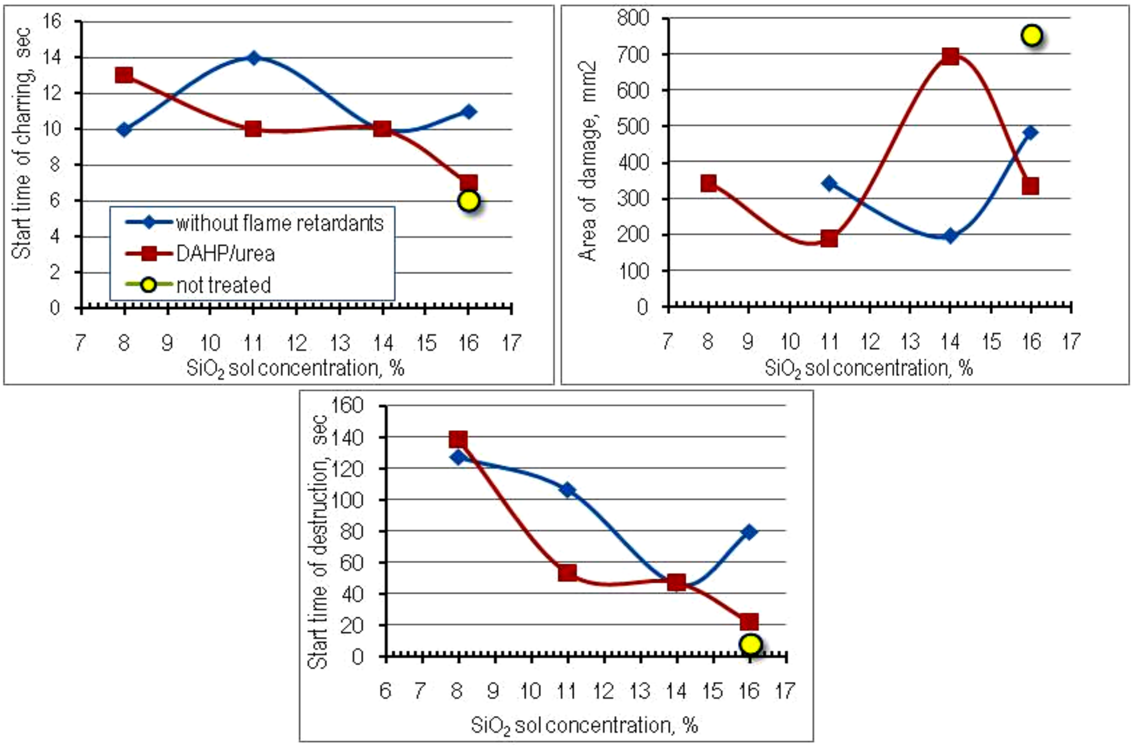

Fig. 3. The influence of SiO_2 sol concentration on the flame-retardant characteristics of treated CF samples.

For this purpose, a central composite uniform rotatable plan of the second order was used. The optimization results are shown in the figure.

To determine the optimal ratio of flame-retardants for thin fabrics, on the example of cotton fabric, the method of alternating spraying of aqueous solutions of diammonium hydrogen phosphate (DAHP) and urea (C) was used, and the content of a 20 % solution of DAHP and a 5 % solution of urea was determined.

To determine the optimal ratio of flame-retardants for voluminous (mixed) fabrics, they were alternately applied by the bath method, followed by removal of excess solution on wringing rollers and drying in a drying cabinet in the thermal shock mode. For the research, approximately the same numbers of solutions but different concentrations were used: for diammonium hydrogen phosphate 10–20 %, for urea 2–10 %.

The target function was the area of tissue damage at a certain point in time of burning and the time of exposure to the flame, during which critical changes appear in the treated tissue: the appearance of signs of charring and destruction. The content of flame retardant solutions and the concentration of SiO_2 sol (for thin fabrics) or the concentration of SiO2 sol and flame retardant solutions (for bulky fabrics) were taken as optimization factors.

The analysis of the obtained surfaces made it possible to conclude that the main effect of flame retardant is exerted by diammonium hydrogen phosphate (DAHP). The content or concentration of urea depends on the concentration of DAHP used: if diluted DAHP solutions (9–10 %) are used, trace amounts of urea (0–0.5 %) must be added. In the case of using a concentrated DAHP solution (18–20 %), the concentration of the urea solution should also be increased to 8–10 %. The optimization results were used to obtain protective coatings on cotton fabric and further test it for water resistance. The samples were kept in clean water for 3 days, replacing the water with fresh water every day, after which they were dried and subjected to fire tests.

After testing for water resistance, the samples did not lose their appearance; the coating was tightly fixed on the surface of the fabric fibers.

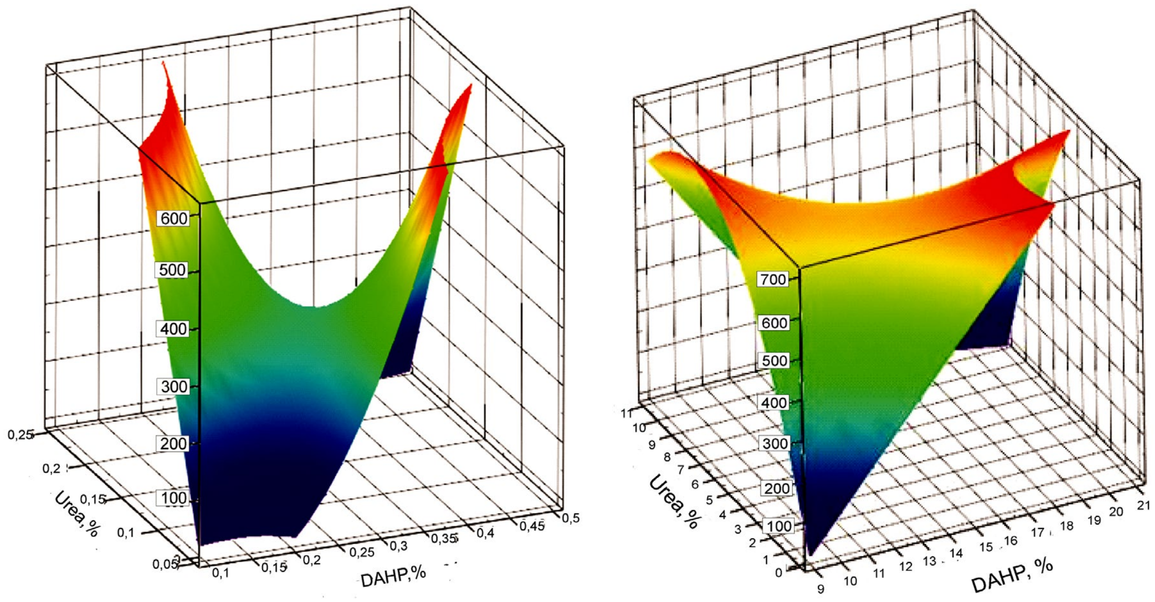

Fig. 4. Dependence of the damage area of treated thin cotton (a) and low-density tapestry (b) fabric samples on the ratio of flame retardants applied to the gel coating obtained on the basis of sol SiO_2 11 % concentration.

After long-term hydration, the fire-retardant properties changed slightly: a decrease in the time of final burning and smoldering was observed on samples without flame retardants, probably due to the hydration of the coating surface and the formation of a layer of hydroxyl groups, the cleavage of which during the action of fire additionally reduces the temperature of the surface of the fabric, which, in turn, reduced the area of tissue damage.

4 Conclusion

Because of the conducted research, it is possible to draw generalized conclusions:

1. The thinner the fabric, the less concentrated SiO_2 sol should be used. Mixed fabrics, which consist of fibers of different composition, require more concentrated SiO_2 sols.
2. It has been established that diammonium hydrogen phosphate (DAHP) exerts the main effect of flame retardant. The content or concentration of urea depends on the concentration of DAHP used: if diluted DAHP solutions (9–10%) are used, trace amounts of urea (0–0.5 %) must be added. In the case of using a concentrated DAHP solution (18–20 %), the concentration of the urea solution should also be increased to 8–10 %.
3. The presence of two zones in which the area of fabric damage after fire tests is the smallest allows to increase the reliability of the technology of applying protective coatings: depending on the type of fabric, adjust not only the concentration of SiO_2 sol, but also change the ratio of flame retardants without leaving the optimum zone.

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