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SILICOPHOSPHATE PROTECTIVE COATINGS FOR REDUCING THE THERMAL SIGNATURE OF TEXTILE MATERIALS

A study was conducted on the possibility of obtaining flame retardant elastic coatings for textile materials with a reduced thermal signature, which can be used to protect against infrared radiation for protective suits of rescuers. The possibility of using silicophosphate flame retardant compositions to reduce the thermal signature of cotton fabrics was investigated. The nature of the influence of the concentration of SiO_2 sol and the content of the orthophosphoric acid additive on the change in the thermal signature of cotton samples was determined. It was established that the flame-retardant coating creates a dense film on the surface of each fiber that forms the fabric threads, preventing the access of oxygen from the air and, accordingly, ignition, but at the same time the fabric is densified, which negatively affects its thermal conductivity. It was shown that the rate of fabric heating decreases with a decrease in the concentration of SiO_2 sol to 8 %. It was found that the effect of modifying additives on the thermal signature is mainly associated with the nature of polycondensation of polysilicic acid in SiO_2 sol. It was shown that H_3PO_4 in small quantities is able to be incorporated into the siloxane framework of polysilicic acid, increasing the fire resistance of the coating. Excess content of this additive provokes the initiation and acceleration of the process of network polycondensation, creating inhomogeneities in the gel coating and reducing its fire-retardant and heat-insulating properties. The effect of long-term action of water on the heat-insulating properties of coatings was determined. It was shown that in the case of using SiO_2 sol of 8 % concentration, long-term action of water improves the heat-insulating properties of coatings. Increasing the concentration of SiO_2 sol to 14 % leads to some swelling of coatings, increasing their thickness and densifying the impregnated fabric.

Keywords: thermal insulation silica-containing coatings, thermal resistance, fire resistance, thermal signature

1. Introduction

In conditions of martial law, the problem of protecting rescuers of the State Emergency Service from unmanned aerial vehicles during rescue operations at night becomes very urgent. The thermal insulation properties of a rescuer's protective suit are increased by using several layers of fabric, each layer of which has its own specific task. The middle layer is usually designed to reflect the heat of a human body and contains specific additives.

The upper layer must be elastic, fireproof and with a reduced thermal signature. Depending on the ambient temperature, the magnitude of the thermal signature also differs: in winter, especially at night, the contours of the rescuer's figure will be clearly visible, but in summer at night these contours will be blurred, which makes it difficult to identify a person next to other heated bodies.

It is known that glass does not transmit infrared radiation, so it can be used as a protective screen for a group of people or equipment. Considering that the basis of glass is silicon dioxide, which is an inorganic polymer that forms Si–O–Si siloxane bonds, it can be assumed that protective coatings based on silica-containing components can reduce the thermal signature of the fabric.

The creation of fire-retardant silica-containing compositions is widely presented in the scientific literature [1, 2]. Organosilicon compounds or liquid glass are most often used as starting components for obtaining protective coatings for textile materials. Organosilicon compounds require careful control of the parameters of hydrolysis and polycondensation reactions, but they provide a thin layer of coating securely fixed on the surface of the fibers of the fabric threads [3]. The main disadvantage of such coatings is the high cost of the starting components and the negative impact on the environment.

The use of liquid glass as a supplier of polysilicic acid for the formation of dense silica-containing coatings allows to eliminate the above-mentioned disadvantages: liquid glass is much cheaper than any organosilicon compound [4]. In addition, the disposal of waste fabrics that have been impregnated with fire-retardant compositions based on liquid glass does not lead to a deterioration of the environmental situation. Polymerization of silicic acid can occur in two ways: through the formation of an oxonium complex in the acidic pH range, as a result of which a network structure of the polysilicic acid gel is formed; the alkaline pH range provokes the polymerization by the ionic mechanism, as a result of which predominantly linear polymers are formed in the gel structure [4]. As previously determined, obtaining elastic coatings occurs under conditions of a weakly acidic or neutral medium [5]. Therefore, it seems appropriate to investigate the influence of the composition of the protective composition based on liquid glass on the thermal insulation properties of the treated fabric.

2. Analysis of literary data and problem statement

The issue of developing technologies for obtaining coatings that reduce infrared radiation from people and vehicles has been the subject of many scientific publications. Military aircraft use so-called stealth coatings to avoid radars. Stealth technology masks ground vehicles from radars and infrared sensors [6]. Graphene, carbon black, carbon nanotubes and carbon fibers are used in the compositions of such compositions [7]. However, the coatings are not fire-resistant, but are capable of self-extinguishing during a single contact with fire. In the event of further fire exposure, fire protection deteriorates sharply.

The paper [8] presents the principles of selecting materials for use as heat-protective coatings. The influence of modifying additives and porosity of coatings on reducing their thermal conductivity coefficient is considered. However, in such compositions, the emphasis is placed on thermal insulation properties, and the fire protection of the treated surface is not considered at all. The authors of [9] consider the basic principles of the formation of ceramic thermal barrier coatings that provide optimal thermomechanical and chemical properties, but are not elastic and cannot be used for fabric impregnation.

In [10], ways to reduce the thermal signature of the fabric by adding staple fibers made of stainless steel, or coated with silver or aluminum are considered. The authors conclude that silver is preferable for obtaining flexible clothing. Such compositions hide the thermal signature of the object, but are too expensive.

In the review [11], materials of the mid-infrared range are considered, which allow contactless adjustment of infrared camouflage and energy efficiency of enclosing structures. The publication is interesting in that it analyzes materials from the point of view of microstructural design, spectral control strategies and application requirements, but does not pay attention to obtaining elastic coatings for textile materials that do not reduce their softness, elasticity and flame-retardant ability.

Therefore, the development of flame-retardant compositions that can simultaneously reduce the thermal signature without changing the aesthetic appearance of the fabric is a topical issue.

3. Purpose and objectives of the study

The aim of the research is to study the possibility of obtaining flame-retardant elastic coatings for textile materials with a reduced thermal signature, which can be used to protect against infrared radiation for protective suits of rescuers. The goal is achieved by solving the following tasks:

1. To investigate the influence of the composition and technological features of the application of the compositions on the change in the thermal signature of the fabric;
2. To investigate the influence of long-term exposure to water on the integrity and thermal insulation properties of the coatings.

4. Materials and research methods

The object of research is the technology of reducing the thermal signature of textile materials. The subject of research is the physicochemical processes occurring in protective coatings during heating.

The main hypothesis of the research is the possibility of using elastic fire-retardant coatings with modifying additives to reduce the thermal signature of textile materials

For the research, fire-retardant compositions based on liquid glass and modifying additives were used.

The compositions were obtained by mixing aqueous solutions of liquid glass and acetic acid. Cotton samples were impregnated with experimental compositions and heat-treated in a drying oven at temperatures of 60–80 °C. The change in the thermal signature was determined using a laboratory setup (Fig. 1).

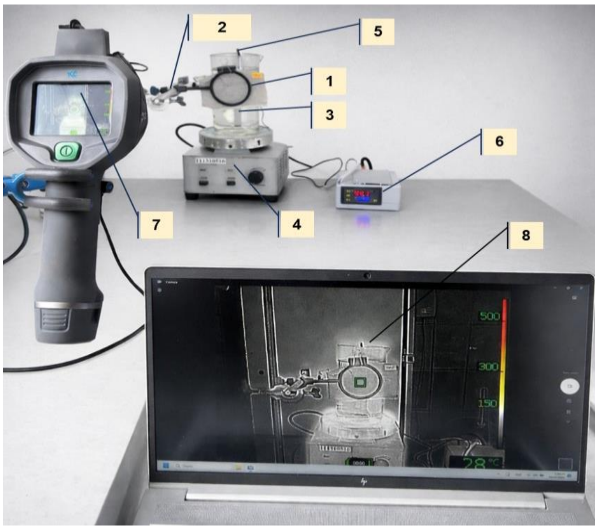

Fig. 1. General view of the laboratory setup for determining the thermal signature of tissue samples: 1 – tissue sample; 2 – tissue sample holder; 3 – beaker with water; 4 – magnetic stirrer with heating; 5 – thermocouple; 6 – temperature controller; 7 – thermal imager; 8 – laptop for recording the course of the experiment

Fabric sample 1 is fixed with a holder 2, in contact with the surface of a glass 3 with water heated to 40 °C. Heating and stirring of water in the beaker is performed on a magnetic stirrer 4 and is controlled by a thermocouple 5 connected to a temperature controller 6. Connecting the magnetic stirrer to the temperature controller ensures switching on/off of stirring and heating when the temperature in the beaker changes to 40±1 °C. The change in the thermal signature over time was observed using a thermal imager 7 connected to a laptop 8, which allows recording a video of the experiment.

As experimental samples, 100 % cotton impregnated with compositions based on liquid glass in 1 or 2 layers, with and without modifying additives, was used. An aqueous solution of a flame retardant – diammonium hydrogen phosphate – was sprayed onto the dried surface of the coating and dried again at temperatures of 60–80 °C.

The outer layer of the rescuer's protective suit, in addition to its fire-retardant purpose, must withstand long-term exposure to water and not peel off during washing. Therefore, the effect of long-term exposure to water for 1–3 days on the change in the integrity and thermal insulation properties of the coatings was additionally studied.

To enhance the hydration effect, the samples were immersed in clean water every day. After exposure to water, the fabric samples were dried in a drying cabinet at temperatures of 60–80 °C and examined on a laboratory installation.

5. Study of the effect of SiO_2 sol concentration on the change in the thermal signature of tissue samples

Thin fabric heats up to the temperature of the heat source quite quickly. Previous studies have shown that untreated fabrics under the influence of high temperatures heat up at different rates depending on the composition of the fabric, its thickness and density. An active increase in the temperature of the fabric is observed in the first 30 s: the back side of woolen fabrics heats up to 400–500 °C, cotton and tapestry mixed fabrics – up to 250–350 °C under the conditions of heating the front side of the fabric to 700–750 °C. Such wide temperature intervals are due to the different thermal conductivity of fabrics, which depends on the degree of non-uniformity of the fabric structure as a result of the presence of defects in the interlacing of threads and their different thickness. The first sharp increase in the temperature of the fabric in the interval of 8–10 s of heating to 100–120 °C corresponds to the ignition of the fabrics. The second temperature jump (16–18 s) is due to its destruction.

The fire-retardant coating creates a dense film on the surface of each fiber that forms the fabric threads, preventing access of oxygen from the air and, accordingly, ignition, but at the same time the fabric is densified, which negatively affects its thermal conductivity.

The heating curves of the fabric impregnated with experimental compositions are practically superimposed on each other, making it impossible to determine the optimal composition of the fire-retardant composition, which has a reduced thermal signature. Therefore, the first section of the curves (up to 30 s) was selected, which was subjected to analysis of the influence of the composition of the fire-retardant coating and the thickness of its application layer on the change in the thermal signature.

Fig. 2 shows the results of determining the thermal signature of samples treated with compositions with different concentrations of SiO_2 sol.

As can be seen from the figures, the samples are heated to lower temperatures under conditions of lower concentrations of SiO_2 sol.

The presence of a complex flame retardant (diammonium hydrogen phosphate and urea) leads to a slight increase in the heating temperature of fabric samples impregnated with a sol with a concentration of 14 % SiO_2 and practically does not affect the thermal signature of samples impregnated with a sol of 8 % concentration. Taking into account the results of previous studies on the technological properties of impregnating compositions (viability, fluidity), compositions of 11 % concentration were used for further studies.

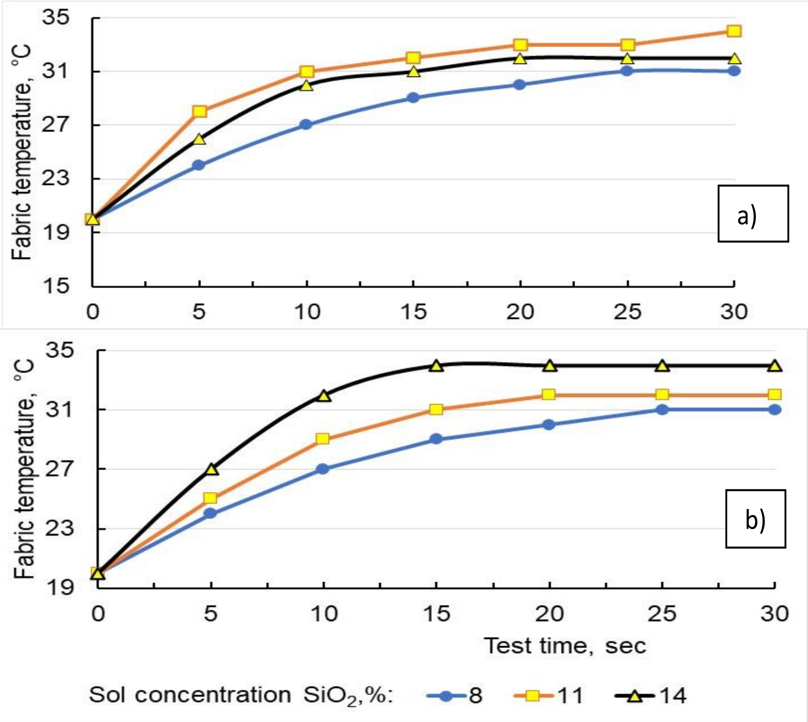

Fig. 2. Change in the thermal signature of cotton fabric samples over time depending on the concentration of SiO_2 sol: a – without flame retardant; b – with flame retardant

Fig. 3. presents the results of determining the change in the thermal signature of the samples depending on the content of the orthophosphoric acid additive.

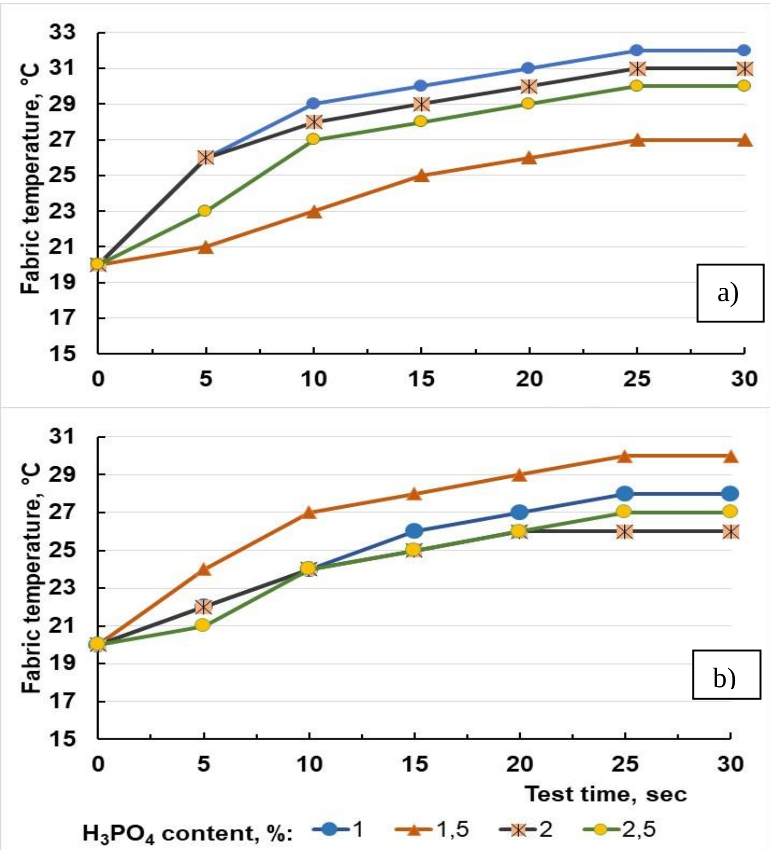

Fig. 3. Change in thermal signature of the fabric over time depending on the content of orthophosphoric acid additive. Sol concentration SiO_2 11%: a – coating applied in 1 layer; b – coating applied in 2 layers

The slowest warming is observed for the fabric with the addition of 1.5 % H_3PO_4 in a single-layer coating (Fig. 3, a). In the case of applying the coating in two layers, the least warming is observed for the fabric with the addition of 2 % orthophosphoric acid.

6. Study of the influence of coating hydration on the change in the thermal signature of impregnated fabrics

Fig. 4. presents graphs of the change in thermal signature over time depending on the hydration time of the coatings. In the case of using a low concentration of SiO_2 sol (8 %), a slowdown in tissue heating is observed with an increase in the time of exposure of tissue samples in water.

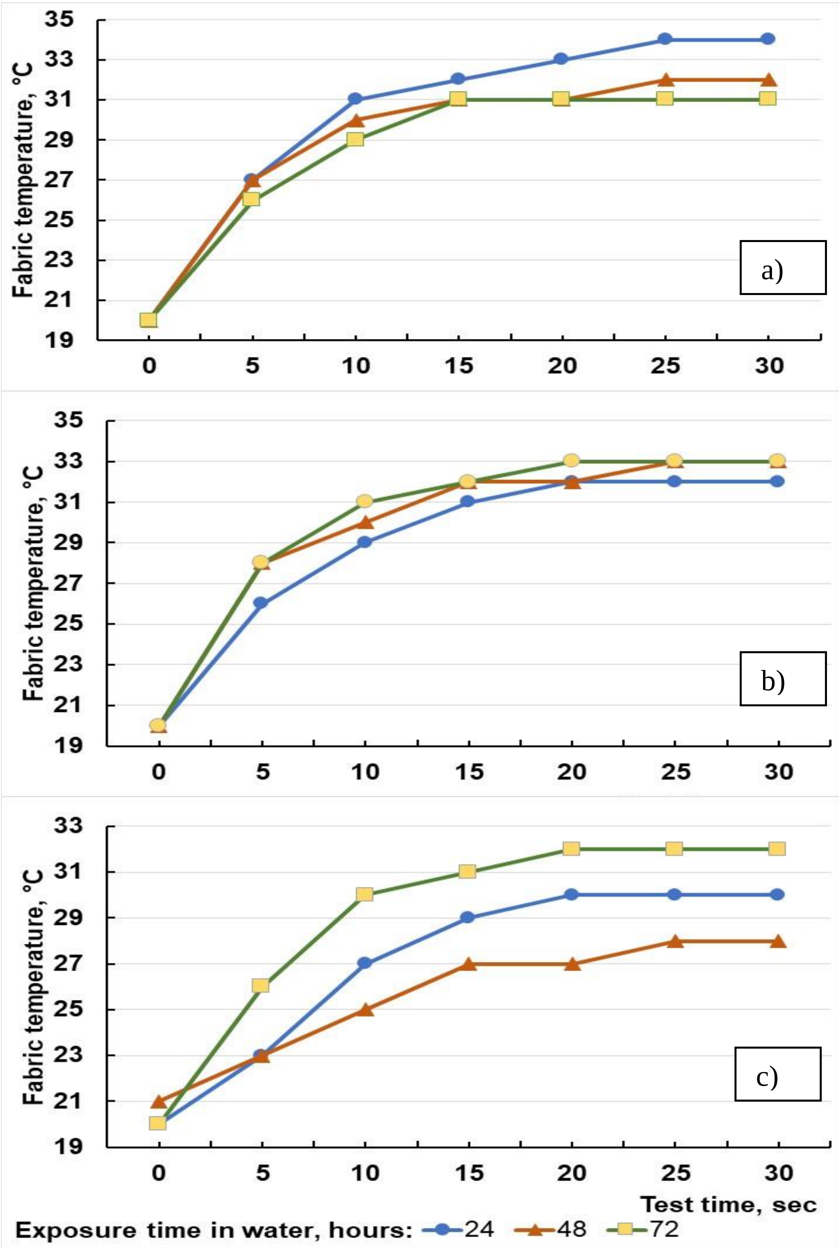

Fig. 4. Change in thermal signature of cotton fabric samples over time depending on the time of exposure in water. Concentration of SiO_2 sol: a – 8%, b – 11%, c – 14%

Holding the impregnated samples in water for 1–2 days leads to a slight decrease in the degree of their heating with increasing SiO_2 sol concentration. Increasing the holding time in water to 3 days changes the picture somewhat: the heating temperature increases with increasing SiO_2 sol concentration.

7. Discussion of the results of the study of the influence of SiO_2 sol concentration and coating hydration

The distribution of curves in Fig. 2 suggests that the rate of fabric heating is affected, first of all, by the thickness of the coating, which fills the free space between the fibers of the fabric threads, compacting and increasing its thermal conductivity. Therefore, the graphs show an increase in the heating temperature with increasing concentration of SiO_2 sol. For the same reason, the presence of a flame retardant slightly increases the heating of the fabric treated with compositions with a 11–14% concentration of SiO_2 sol. The addition of orthophosphoric acid leads to partial incorporation of the phosphate group into the siloxane framework of the SiO_2 gel, as was proven by infrared spectroscopy in [12]. The increased amount of H_3PO_4 can be used for two processes: to initiate the network polycondensation of polysilicic acid and to form a silicophosphate framework. It is likely that the formation of a network structure of the siloxane framework of the gel contributes to a slight decrease in the thermal conductivity of the coating due to the formation of clathrates, which is accompanied by a decrease in the thermal signature in the graphs (Fig. 3,a). This assumption is confirmed by graph 3,b, in which two-layer coatings with a higher content of H_3PO_4 (2–2.5 wt.%) showed a noticeable decrease in the tissue heating temperature. Further increase in the content of H3PO4 leads to acceleration of the process of network polycondensation, which negatively affects the fire resistance of coatings [13].

Hydration of coatings during long-term exposure to water (for 3 days) leads to some swelling of the coating, increasing its thickness and densifying the impregnated fabric. This effect is manifested in the case of using a 14 % concentration of SiO_2 sol. The heterogeneities inherent in the sol with a concentration of 14 % SiO_2 are manifested more brightly, which affects both the fire resistance and thermal insulation properties of the coatings. Even in the case of swelling, the coating does not exfoliate from the fabric. In a thin coating (8 %), on the contrary, hydration of the coating surface (attachment of water molecules by hydrogen bonds to surface Si–O groups) leads to some slowing down of the heating of the fabric (Fig. 4, a). Based on the results of previous studies [12], it can be concluded that not only fire resistance, but also thermal insulation properties depend on the mechanism of polycondensation of SiO_2 sol, the degree of its homogeneity, concentration and number of applied coating layers. The influence of modifying additives on the thermal signature is mainly associated with the nature of polycondensation of polysilicic acid in SiO_2 sol. Based on the analysis (Fig. 2–4), it can be assumed that to reduce the thermal signature, it is necessary to apply sol with a concentration of 8–11 %, with the addition of 2–2.5 % orthophosphoric acid in 1–2 layers. Expanding the above intervals will lead to a decrease in the fire-retardant and thermal insulation properties of the coating.

8. Conclusion

1. The nature of the influence of the concentration of SiO_2 sol and the content of the orthophosphoric acid additive on the change in the thermal signature of cotton samples was determined. It was established that the rate of fabric heating decreases with a decrease in the concentration of SiO_2 sol to 8 %. The content of the orthophosphoric acid additive was determined. It was shown that H_3PO_4 in small quantities is able to be incorporated into the siloxane framework of polysilicic acid, increasing the fire resistance of the coating. Exceeding the content of this additive provokes the initiation and acceleration of the process of network polycondensation, creating inhomogeneities in the gel coating and reducing its fire-protective and heat-insulating properties.

2. The influence of long-term water exposure on the heat-insulating properties of coatings was determined. It was shown that in the case of using 8 % concentration of SiO_2 sol, long-term water exposure improves the heat-insulating properties of coatings. Increasing the concentration of SiO_2 sol to 14 % leads to some swelling of the coatings, increasing their thickness and densifying the impregnated fabric. In further studies, it is planned to determine the effect of additives capable of reflecting infrared radiation on the survivability of fire-retardant compositions, their degree of homogeneity, fire-retardant and thermal insulation properties.

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СИЛІКОФОСФАТНІ ЗАХИСНІ ПОКРИТТЯ ДЛЯ ЗНИЖЕННЯ ТЕПЛОВОЇ СИГНАТУРИ ТЕКСТИЛЬНИХ МАТЕРІАЛІВ

Проведено дослідження щодо можливості отримання вогнезахисних еластичних покриттів для текстильних матеріалів зі зниженою тепловою сигнатурою, які можна використовувати для захисту від інфрачервоного випромінювання для захисних костюмів рятівників. Досліджено можливість використання силікофосфатних вогнезахисних композицій для зниження теплової сигнатури бавовняних тканин. Визначено характер впливу концентрації золю SiO_2 та вмісту добавки ортофосфатної кислоти на зміну теплової сигнатури бавовняних зразків. Встановлено, що вогнезахисне покриття створює щільну плівку на поверхні кожного волоконця, що утворюють нитки тканини, запобігаючи доступу кисню повітря і, відповідно, загорянню, але одночасно відбувається й ущільнення тканини, що негативно відбивається на її теплопровідності. Показано, що швидкість прогрівання тканини зменшується зі зниженням концентрації золю SiO_2 до 8 %. Встановлено, що вплив модифікуючих добавок на теплову сигнатуру, в основному, пов'язаний з характером поліконденсації полікремнієвої кислоти в золі SiO_2. Показано, що H_3PO_4 в невеликих кількостях здатна вбудовуватися в силоксановий каркас полікремнієвої кислоти, збільшуючи вогнестійкість покриття. Перевищення вмісту цієї добавки провокує ініціацію і прискорення процесу сітчастої поліконденсації, створюючи неоднорідності в гелевому покритті і знижуючи його вогнезахисні та теплоізоляційні властивості. Визначено вплив довготривалої дії води на теплоізоляційні властивості покриттів. Показано, що у разі використання золю SiO_2 8 %-ї концентрації тривала дія води покращує теплоізоляційні властивості покриттів. Збільшення концентрації золю SiO_2 до 14 % приводить до деякого набухання покриттів, збільшуючи їх товщину й ущільнюючи просочену тканину.

Ключові слова: теплоізоляційні кремнеземвмісні покриття, термостійкість, вогнестійкість, теплова сигнатура

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