

Mathematical Modeling of the Protective Effect of Ethyl Silicate Gel Coating on Textile Materials under Conditions of Constant or Dynamic Thermal Exposure

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Abstract. The influence of the process of fire-retardant coating on textiles on the degree of fire protection of cotton and blended fabrics is investigated. Physical-chemical properties of impregnated samples of textile materials depending on the composition of ethyl silicate hydrolysates, concentration and content of diammonium hydrogen phosphate on fire resistance of impregnated samples, time of onset destruction and area of charring of samples after fire tests are analyzed.

The obtained experimental data showed the need to build a mathematical model of the protective action of the gel coating based on the laws of heat and mass transfer, which under conditions of maximum simplicity and minimal artificiality takes into account the main processes from the external heat flow processes of heat transfer, thermal decomposition, evaporation and removal of mass, as well as to develop algorithms and software for computer modeling of the protective action of such a coating in conditions of constant or dynamic thermal exposure.

A mathematical model of fire-retardant action of organosilicon coating on cellulose-containing fiber of fabric threads has been developed. It provides predictive estimates of fire safety parameters of textile materials, based on the level of thermal impact on the surface of the protected material.

1 Introduction

The main disadvantage of textile materials is their low ability to resist convective, radiant thermal energy or a combination of two or more. There are three main approaches to improving the fire-retardant properties of textile materials [1]: (a) use textiles that are fire-resistant in nature, containing fibers with high performance; (2) chemically modify existing textiles; (c) use flame retardants in synthetic fibers and / or for special surface treatment.

In recent years, the scientific field of development of refractory coatings for textile materials using the sol-gel method has been actively developing. This technology allows to create new materials with a high degree of homogeneity at the molecular level and with high physical and chemical properties, which differ significantly from the properties of materials obtained by traditional methods [3].

Although this method has been used for a long time, its use to slow down the burning of textiles has only recently begun. Interesting results are given in [2]. It has been shown that the sol-gel process can lead to the formation of nanoparticles of silicon dioxide generated in situ, or coatings of silicon dioxide directly on polyester, cotton fabrics and their mixtures when using different SiO₂ precursors.

In [4] the influence of different TEOS/H₂O ratios on fire protection of cotton fabrics was investigated. However, in some works contradictory data have been published on the influence of the sol-gel process on increasing the fire resistance of tissues of different nature. For example, [5] found that sol-gel treatment selectively acts on different fibers in fabrics: the fire resistance of

polyester does not improve, and in blended fabrics containing 35 wt.% cotton, a significant increase in flash time was detected.

The most successful are complex organo-inorganic compositions containing, in addition to complex organic compounds, inorganic salts that can decompose with endothermic effect or emit gases when heated. The most widely used as an inorganic component is diammonium hydrogen phosphate. There are a sufficient number of publications discussing the effect of the amount and concentration of diammonium hydrogen phosphate in complex flame retardant compositions. It is noted that its action is most effective at a content of not more than 15-20 % [5, 6].

However, there is almost no information on the influence of the conditions of the sol-gel process in the case of fixing the protective coating on the fibers of the fabric on such properties of textile materials as elasticity and preservation of appearance.

The main disadvantages of such technologies are the duration of the process and the complex structure of the synthetic components of the mixture, which are subject to gelation. There is no information in the technical literature on the use of previously obtained hydrolysates of organosilicon compounds as impregnating compositions.

Taking into account the information on the impact of halogenated flame retardants on the ecological situation in the world and human health [7-9], it seems promising to develop a new approach to fabric modification, which would allow using traditional sol-gel process and the simplest harmless flame retardants to protect fabrics from the effects of fire, increase their fire resistance while maintaining their natural elasticity and appearance.

2 Unresolved Issues

The authors of the article in previous studies found that the use of ethyl silicate sols obtained under variable pH conditions can significantly increase the fire resistance of impregnated samples of cotton and blended fabrics [6, 10]. The hydrolysates of technical ethyl silicates were applied to the fabrics by the bath method, after which the coating on the fabric was fixed by heat shock in an oven at 60-80 °C.

After drying, a solution of diammonium hydrogen phosphate was applied to the impregnated samples by spraying and subjected to thermal shock. Fire research has shown that impregnated samples do not ignite, but gradually and slowly char. In comparison with the untreated fabric, the time of the beginning the impregnated samples destruction under the action of fire (the time of the crack formation in the coating) increased from 30 s to 10 min, respectively [10]. Interestingly, the samples did not lose their elasticity. A significant reduction in toxic gases formed during thermal destruction of coatings [6] was recorded.

In the technical literature, the main attention is paid to the mathematical description of changes in the average volume temperature of the gaseous medium in the premises where the fire occurred, the processes of fire development in buildings, operational fire forecasting [11, 12, 13]. The authors [14] investigated the influence of the degree of homogeneity of the SiO₂ sol on the duration of the induction period and the quality of refractory coatings on textile materials. The SiO₂ fire protection system is shown. In [15] the authors present the results of an experimental study of the dynamics of dangerous factors of the environment in the modeling chamber, as well as their autocorrelations and pair correlations at different lags for alcohol, paper, wood and textiles.

Increased demands on materials and products made of them explain creation of new technologies and industries [16, 17]. In works [18, 20, 21] the experimental research of process of occurrence of fire for various rooms and materials is considered. The authors [22, 23] reports the results of research of modern methods of predicting a fire in premises. In [19, 24] attention is paid to the development of a method for detecting arbitrary hazardous pollutants in the air based on the structural function of the current concentrations of pollutants.

Lack of theoretical and experimental research assumes determining the task of this study: based on the laws of heat and mass transfer to build a mathematical model of the protective effect of gel coating, which under conditions of maximum simplicity and minimal artificiality takes into account the main processes that take place in the system "fiber - protective coating - environment", and

describes the processes of heat transfer, thermal decomposition, evaporation and mass removal caused by heating from external heat flow, as well as to develop algorithms and software for computer modeling of protective action of such coating under constant or dynamic heat exposure.

3 Main Part

The gel coating is considered as a porous medium, the basis of which is the solid phase of the siloxane framework of SiO_2 . The spatial scheme of the problem of heating the fiber protected by a layer of gel coating is presented in Fig. 1. At the initial stage, the free pore volume of the siloxane framework is partially filled with components of the sol, which include free water (index "w"), fixed water (index "fw"), and ethanol solvent residues (index "e"). There are two components in the fiber of textile material: carbon (index "C"), which remains in the case of complete pyrolysis of the fiber, and gaseous products of pyrolysis (index "p").

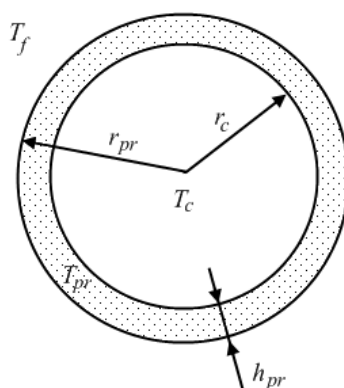


Fig. 1. Scheme of the fiber protected by a layer of gel coating

During heating, the remnants of the organic solvent (ethanol) in the interglobular space and the pores of the coating first turn into a gaseous state. When the temperature reaches about $100\text{ }^\circ\text{C}$, free water evaporates from the coating, and when the temperature reaches about $120\text{-}150\text{ }^\circ\text{C}$, already bound water is removed from the coating. Evaporation processes cool the gel coating, which slows down the heating and subsequent decomposition of the fiber material.

According to the results of differential thermal analysis of gels, exothermic degradation processes with the formation of inert material (associated values are denoted by the index "in") and the parallel release of additional water vapor and ethanol begin when the temperature reaches about $400\text{ }^\circ\text{C}$.

Cellulose fiber is heated under a layer of gel coating due to thermal conductivity and when the critical pyrolysis temperature is reached (for cellulose-containing materials approximately $200\text{-}250\text{ }^\circ\text{C}$) and decomposition processes begin with the formation of carbon residue and gaseous pyrolysis products.

The appearance of additional gases in the system due to the reactions of gas formation under the influence of heating leads to a significant increase in the pressure in the pores of the fiber and the protective coating. According to the laws of hydrodynamics, this causes convective transfer of gases to the outside. Thus, the gases do not have time to react inside the system, and do not allow oxygen from the outside to enter the system, which prevents the oxidation of pyrolysis products and carbon residue.

The penetration of oxygen from the outside, which is exclusively diffusion in nature, becomes possible only after the completion of internal gas formation.

The above circumstances during the simulation allow not to take into account the homogeneous reactions in the gas phase between the gas components leaving the system, but to take into account those components that provide convection flow from the system.

On the basis of the physical model the mathematical model of fire-retardant action of organosilicon covering on cellulose-containing fiber of threads of fabric is created.

Changing the mass of condensed components of the protective gel coating during heating:

$$\frac{dm_i}{d\tau} = -R_i(T_{pr}, m_i); \quad R_i(T, m_i) = k_i m_i \exp\left(\frac{T - T_{i.cr}}{\Delta T_i}\right), \quad (1)$$

where: $i = e, w, fw, si, in$;

R_i – the rate of change of mass of the i -th component;

k_i – characteristic inverse time of the corresponding transformation, s^{-1} ;

$T_{i.cr}$ – characteristic (critical) value of temperature, a significant difference from which leads to the manifestation of the exponential nature of the dependence;

ΔT_i – the width of the transition temperature range, K.

The rates of mass of water and ethanol vapors in the process of **destruction of the solid component** of the coating are equal to:

$$\frac{dm_{w, si-in}}{d\tau} = k_{w, si-in} \cdot k_{si-in}^{gas} \cdot R_{si}(T_{pr}, m_{si}), \quad (2)$$

$$\frac{dm_{e, si-in}}{d\tau} = k_{e, si-in} \cdot k_{si-in}^{gas} \cdot R_{si}(T_{pr}, m_{si}), \quad (3)$$

where: k_{si-in}^{gas} – the proportion of gel coating, which during destruction turns into a gaseous state; $k_{w, si-in}$ i $k_{e, si-in}$ – the fraction of the mass of water and ethanol in the gaseous component released during destruction. These components are dominant, so we can assume that $k_{w, si-in} + k_{e, si-in} = 1$.

The rate of change in the **mass of condensed fiber components** is described by two equations, namely the change in the mass of components m_p , which during pyrolysis are converted into gaseous products CO_2 , CH_4 , and the change in mass of carbon residue m_C remaining after removal of gaseous decomposition products and oxidized environment through a layer of porous coating due to diffusion:

$$\frac{dm_p}{d\tau} = -R_p(T_c, m_p), \quad \frac{dm_C}{d\tau} = -R_C(T_c, \varphi_{O_2}^c, F_C). \quad (4)$$

The rate of **mass generation of individual pyrolysis gases** will be fixed fractions of the total rate of pyrolysis, i.e.

$$\frac{dm_{CO_2p}}{d\tau} = k_{CO_2p} \cdot R_p(T_c, m_p), \quad \frac{dm_{CH_4p}}{d\tau} = k_{CH_4p} \cdot R_p(T_c, m_p), \quad (5)$$

where k_{CO_2p} i k_{CH_4p} – dimensionless coefficients that reflect the mass fractions of pyrolysis products ($k_{CO_2p} + k_{CH_4p} \leq 1$).

The flux density of the i -th gas component between the fiber and the coating is determined by the formula:

$$j_{c-pr}(\varphi_i) = j_{c-pr}^{conv}(\varphi_i) + j_{c-pr}^{diff}(\varphi_i), \quad (6)$$

where i – components CO_2 , O_2 , H_2O , CH_4 , C_2H_5OH , N_2 ;

$j_{c-pr}^{conv}(\varphi_i)$ – convection component of the flow density of the i -th gas component due to the pressure difference of the i -th component in the fiber material and coating,

$j_{c-pr}^{diff}(\varphi_i)$ – the diffusion component of the flux density of the i -th gas component due to the difference in concentrations of the i -th component in the fiber and coating material.

Then, the equations describing the dynamics of gas components in the fiber volume can be represented as the sum of the rate of consumption or formation of the i -th component during the oxidation and mass transfer between the fiber and the coating due to convection and molecular diffusion. For example, the rate of change of CO_2 concentration in the fiber volume is due to the processes of pyrolysis of cellulose-containing fiber material, heterogeneous oxidation of carbon residue and convection introduction from the fiber and has the form:

$$V_{cav}^c \frac{d\varphi_{CO_2}^c}{d\tau} = \beta_{CO_2}^c \frac{R_c(T_c, \varphi_{O_2}^c, F_c)}{\mu_c} + k_{CO_2p} \frac{R_p(T_c, m_p)}{\mu_{CO_2}} - j_{c-pr}(\varphi_{CO_2}) F_{c.cav}, \quad (7)$$

where V_{cav}^c – the volume of pores in the fiber, m^3 ;

$\beta_{CO_2}^c$ – stoichiometric coefficient of carbon oxidation reaction with oxygen;

μ_i – molar masses of the corresponding components ($i = C, CO_2$);

$F_{c.cav}$ – the total cross-sectional area of the pores on the surface of the fiber;

The energy balance in the fiber takes into account the energy consumption for the pyrolysis of the fiber source material and the introduction of a certain amount of heat by convection flow of pyrolysis gases, energy release due to oxidation of carbon fiber residue, and heat fluxes between the protective coating and the protective coating. Then, the change in fiber temperature during heating is described by the equation:

$$\begin{aligned} (m_c c_c + m_p c_p) \frac{dT_c}{d\tau} = & R_p(T_c, m_p) \cdot [\Delta H_p - c_p^{gas}(T_c - T_0)] + \\ & + \beta_{CO_2}^c \frac{R_c(T_c, \varphi_{O_2}^c, F_c)}{\mu_c} \mu_{CO_2} \left[\Delta H_{CO_2} - \left(c_{CO_2} + \frac{\mu_{O_2}}{\mu_{CO_2}} c_{O_2} \right) (T_c - T_0) \right] + \\ & + q_{c-pr}(T_c, T_{pr}) \cdot F_c, \end{aligned} \quad (8)$$

where $F_c = 2\pi r_c L$ – fiber surface area, m^2 ;

ΔH_p – specific heat of pyrolysis of the fiber, taken at temperature T_c , per kilogram of pyrolysis products, $J \cdot kg^{-1}$;

$c_p^{gas} = k_{CH_4p} \cdot c_{CH_4} + k_{CO_2p} \cdot c_{CO_2}$ – isobaric specific mass heat capacity of gaseous pyrolysis products;

$c_{CH_4}, c_{CO_2}, c_{O_2}$ – accordingly, isobaric specific mass heat capacities,

CH_4, CO_2, O_2 , $J \cdot K^{-1} \cdot kg^{-1}$;

ΔH_{CO_2} – specific thermal effect of carbon oxidation reaction, taken at temperature T_c , per kilogram of CO_2 formed, $J \cdot kg^{-1}$;

μ_i – molar masses of the corresponding components ($i = C, CO_2, O_2$).

The last term determines the heat flux from the protective layer of the coating to the fiber due to thermal conductivity and is described by the equation:

$$q_{c-pr}(T_c, T_{pr}) = \lambda_{pr} \frac{T_{pr} - T_c}{h_{pr}}, \quad Wt \cdot m^{-2}, \quad (9)$$

where λ_{pr} – thermal conductivity of the protective coating material;

T_c – the average volumetric value of the fiber temperature.

The equation of *energy balance in the protective coating* sets the temperature changes during heating:

$$\begin{aligned} \left(\sum_i m_i c_i\right) \frac{dT_{pr}}{d\tau} = & \sum_i R_i(T_{pr}, m_i) \cdot \left[\Delta H_i - c_i(T_{pr} - T_0)\right] + \\ & + R_{si}(T_{pr}, m_e) \cdot \left[\Delta H_{si-in} - k_{si-in}^{gas} \cdot c_{si-in}^{gas}(T_{pr} - T_0)\right] - \\ & - q_{c-pr}(T_c, T_{pr}) \cdot F_c + q_{pr-f}(T_{pr}, T_f) \cdot F_{pr}, \end{aligned} \quad (10)$$

F_{pr} - contact area between the protective coating and the external environment, m^2 ;

ΔH_i - specific mass heat of gasification (evaporation) of the i -th component of the protective coating, taken at temperature T_{pr} , $J \cdot kg^{-1}$;

ΔH_{si-in} - specific heat of restructuring of the solid component of the coating, taken at temperature T_{pr} , per kilogram of restructured siloxane framework, $J \cdot kg^{-1}$;

c_i - isobaric specific heat of the i -th component, $J \cdot kg^{-1} \cdot K^{-1}$;

$c_{si-in}^{gas} = k_{w, si-in} c_{sw} + k_{e, si-in} c_{se}$ - isobaric specific heat of gaseous products of carcass restructuring, $J \cdot kg^{-1} \cdot K^{-1}$;

c_{sw}, c_{se} - isobaric specific heat of gaseous drying products (water vapor and ethanol), $J \cdot kg^{-1} \cdot K^{-1}$.

The first component in equation (10) determines the thermal effect of evaporation and deposition from the gel coating of ethanol; free and bound water, the second term takes into account the thermal effect of the process of restructuring the solid siloxane framework of the coating. The next term determines the heat flux from the protective layer of the coating to the fiber due to thermal conductivity (equation 9). The last component of formula (10) reflects the heat flux from the environment to the protective coating. The density of this flow is equal to:

$$q_{pr-f}(T_{pr}, T_f) = \frac{T_f - T_{pr}}{\frac{h_{pr}}{2\lambda_{pr}} + \frac{1}{\alpha}}, \text{ Wt} \cdot \text{m}^{-2}, \quad (11)$$

where α - heat transfer coefficient from the external environment to the surface of the protective coating.

As already mentioned, the penetration of oxygen from the outside to the surface of the fiber through the layer of protective coating is exclusively diffuse in nature and becomes possible only after the completion of internal gas formation. Therefore, it is not necessary to take into account the gas subsystem of this model until the completion of gas formation processes (ethanol removal, evaporation of free water and bound water, pyrolysis of organic fiber mass, gas formation associated with the rigid frame restructuring process). Formally, this means that this stage of the process (until the end of the restructuring of the rigid frame) can be described without taking into account the equations of the gas phase. In addition, the formation of excess CO_2 during the combustion of the carbon residue significantly suppresses the entry of new portions of oxygen into the system, which significantly complicates the oxidation process of the protected fiber compared to unprotected. This means that when describing the heterogeneous combustion of the carbon fiber residue, the consideration of gaseous combustion products becomes mandatory. After pyrolysis, the equation of mass balance of carbon residue, the equation of balance of the main gaseous components: oxygen, carbon dioxide, nitrogen, as well as the equation of energy balance.

In accordance with the developed mathematical model, computer simulation of the heating of a single fiber of a textile material protected by a gel coating was performed. It describes the time interval from the beginning of heating to the moment of possible ignition of the carbon residue of the fiber.

In the simulation used the dependence of ambient temperature on time in the form of the equation:

$$t_f(\tau) = t_{f,\max} - (t_{f,\max} - t_{f,0}) \exp\left(-\frac{\tau}{\tau_f}\right), \text{ } ^\circ\text{C}$$

Where $t_{f,\max}$, $t_{f,0}$ – respectively, the maximum and initial values of ambient temperature, $^\circ\text{C}$;
 τ_f - characteristic time of change of temperature of environment.

Figure 2 shows the dynamics of temperature change of unprotected fiber, fiber with a diameter of $60 \mu\text{m}$ with a gel coating (layer thickness $h_{pr} = 5 \mu\text{m}$), changes in the masses of condensed components and gaseous products released during heating of the system.

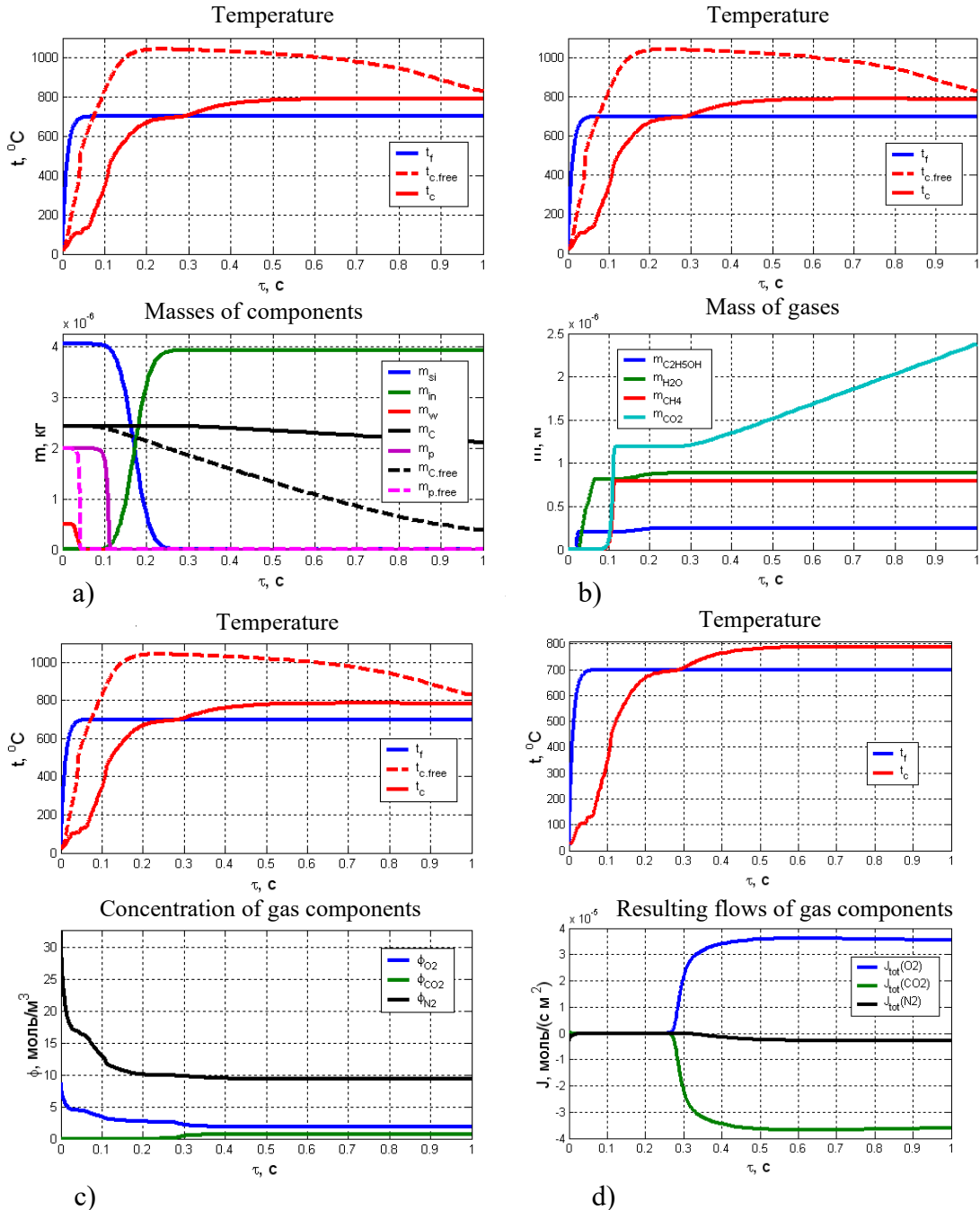


Fig. 2. Changes in ambient temperature, unprotected and protected fibers, as well as the masses of condensed components of the system (a), gaseous products released during heating of the gel-protected fiber (b), the concentration of gases in the protected fiber (c), the resulting flows between fiber and the external environment of gas components (O_2 , CO_2 and N_2) (d) at $t_{f,\max} = 700 \text{ } ^\circ\text{C}$ and time constant $\tau_f = 1 \cdot 10^{-2} \text{ s}$

Unprotected fiber heats up quickly with intense loss of mass of condensed components. First, the pyrolysis part of the fiber decreases, and then the carbon residue begins to decrease intensively. Given that the temperature of unprotected fiber begins to exceed the temperature of the environment, due to exothermic processes, we can conclude that the fiber is burning. The temperature reaches values greater than 1000 °C, which is characteristic of homogeneous flame combustion and is due to the free access of oxygen from the environment to the fiber surface and the free flow of gaseous pyrolysis products.

The gel-protected fiber heats up much more slowly, which is due to the endothermic processes of evaporation and decomposition in the protective layer. In addition, the temperature of the protected fiber begins to exceed the ambient temperature later than that of the unprotected fiber, the rate of decrease in the mass of carbon residue is much lower, so exothermic oxidation of carbon residue when heated is slow due to difficult diffusion of oxygen through the gel coating.

When unprotected fiber is heated to 500 °C, its temperature slightly exceeds the ambient temperature, but the rate of loss of mass of the pyrolysis part of the fiber and carbon residue is much lower than at an ambient temperature of 700 °C. In addition, the fiber temperature does not reach 600 °C, so this indicates that there are exothermic reactions of combustion of cellulose fiber in a heterogeneous mode.

When the protected fiber is heated to 500 °C, the fiber temperature does not exceed the ambient temperature, ie no exothermic combustion processes occur due to the lack of contact of the carbon residue of the cellulose-containing fiber with ambient oxygen. Therefore, the gel coating not only cools the material, but also creates a protective insulating layer.

4 Conclusion

The protective effect of the gel coating based on ethyl silicate sols on cellulose-containing fibers is substantiated and explained with the help of mathematical modeling. The created software can be used as a tool to obtain predictive estimates of fire safety parameters of textile materials, based on the level of thermal impact on the surface of the protected material.

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