1. Introduction

Natural fiber fabrics are increasingly used in various types of building structures, both in permanent and temporary facilities. Living inside and heating such structures may be accompanied by the ignition and rapid propagation of fire since the fabric forms a significant fire load. Since this material is sensitive to high temperatures, it is possible to increase How to Cite: Tsapko, Y., Tsapko, A., Bondarenko, O., Chudovska, V., Sotnikova, I., Sotnikov, D. (2021). Thermophysical characteristics of the formed layer of foam coke when protecting fabric from fire by a formulation based on modified phosphorus-ammonium compounds. Eastern-European Journal of Enterprise Technologies, 3 (10 (111)), 34–41. doi: https://doi.org/10.15587/1729-4061.2021.233479

THERMOPHYSICAL CHARACTERISTICS OF THE FORMED LAYER OF FOAM COKE WHEN PROTECTING FABRIC FROM FIRE BY A FORMULATION BASED ON MODIFIED PHOSPHORUS-AMMONIUM COMPOUNDS

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the level of fire safety of objects where building structures made of fabric materials are used, possibly with the help of their fireproof treatment. An example is a fire that occurred in the structure of the Hartford Circus, Connecticut (USA) during its work, which led to the mass death of spectators.

For the comprehensive protection of cellulose-containing materials from ignition, mixtures of inorganic salts are used, but fire-protective treatment for fabrics is not suitable since the efflorescence formation is observed at the surface. Over time, the material loses its protective properties, which leads to the ignition of combustible structures under the action of a high-temperature flame.

The need for fire protection is also relevant for those objects of mass stay of people that are made of combustible fabrics. In particular, in 2005, in one of the theaters in Cairo (Egypt), an ordinary candle caused the ignition of curtains, wings, and decorations made of paper. One of the reasons for the rapid propagation of the fire was the lack of fire protection of fabric products.

Therefore, it is a relevant task to study the issue related to reducing the level of fire danger from fabrics by forming a layer of foam coke during the thermal decomposition of formulations based on modified phosphorus-ammonium salts.

2. Literature review and problem statement

Work [1] focuses on the performance of textile membranes exposed to fire. Classification tests were carried out with typical textile products. The product range was investigated with two different small flame tests in compliance with the Swedish classification tests. Some products were tested according to EN 13823, which is an average test method for determining the rate of heat release, smoke formation, and flame propagation. In addition, large-scale tests were carried out to obtain information about the behavior of fire in real use and to be used as a benchmark in assessing the information obtained as a result of classification tests. Various testing scenarios were developed that simulated applications in buildings and for weather protection. Large-scale tests have shown the importance of measuring the smoke formation and the occurrence of flame droplets in the classification scheme. However, the relevant physicochemical data on their change in fabric during operation are not given.

Study [2] reports the manufacture of multifunctional protective textiles made of the cotton fabric mixed with aramid, by depositing the three-dimensional polymer coating tetrakis (hydroxymethyl) phosphonium chloride. For this purpose, mixed fabrics with different compositions, weaving structures, thicknesses, and thread density were made, followed by chemical deposition of the polymer coating. The results show that the lowest average length of the coal base at the density of the heat flow of radiation (11.021 kW∙m⁻²), and the highest oxygen index (36.8 %), were demonstrated by the mixed fabric at 40 % of cotton and 30 % of aramid fabric. As regards the water-repellent properties, all the treated samples demonstrated water-repellent properties, expressed as the contact angle of water of up to 141.70° to 151.50°. These results give fundamental grounds for the design of mixed textiles with the necessary functionality. However, the exploitation classes of these substances were not defined.

Fireproof Wired cotton fabric material has been successfully prepared by polymerizing polyaniline doped with new phytic acid by impregnation [3]. The experimental results showed that the limit oxygen index of the source fabric increased to 32 %, and the resistance of the sheet decreased to 1.13 kOhm. During the pyrolysis of the original and finished fabric, the maximum decomposition temperature and the maximum emission of light gas decreased from 380 °C to 290 °C. And the content of carbon residues increased from 12.6 % to 39.2 % at 700 °C, which indicated that the polymerization of aniline doped with polyaniline, led to the fire-retardant properties of the fabric. However, the issues that are associated with the mechanism of formation of foam coke remain unresolved. The reason for this may be the subtleties regarding the formation of a protective layer, which, accordingly, makes such studies difficult.

Article [4] discusses the use of an aqueous-alcohol solution of tetrahydroxysilane, an aqueous solution of sodium silica, phosphorus-containing flame retardants to give fire-protective properties to cellulose textile materials. The speed of heat generation, time, and peak rate of heat generation were investigated, which are the most important factors in predicting the rate of fire growth. However, it is not specified how those formulations can withstand changes in temperature and humidity fields.

Study [5] focused on the use of a new composition based on sodium silica, urea, and sodium phosphate to provide fire-protective properties to cellulose textiles. Untreated fabric measuring 220×170 mm, when tested for flammability during the ignition of 15 s, completely burns out in 60 s. In samples treated with flame retardants, during the ignition time of 15 s, smoldering time is almost reduced to zero. With an increase in the concentration of flame retardant and heat treatment temperature, the loss of material strength, its tensile strength, and the physical appearance of the fabric change slightly. Electron scanning microscopy and energy dispersion microanalysis showed that pure cotton fabric contains 68.77 % of carbon and 31.22 % of oxygen. After modification, sodium particles, 0.02 %, phosphorus, 0.04 %, and potassium, 0.05 %, are formed on the surface of the treated fabric which are distributed rather unevenly. It has been shown that in cellulose materials modified with compositions based on sodium silica and urea, sodium hydrophosphate, fire-protective properties increase. However, the implementation of these operations on the fire protection of fabrics requires special equipment at the stage of manufacturing the materials.

A synthesized series of boron-nitrogen polymers are reported in [6] in order to provide an environmentally friendly alternative to the fire-resistant processing of cotton fabrics. The organic combination of boron, phenylboronic acid was successfully associated with branched polyethylenimine, which was confirmed by analysis. Thermogravimetric analysis revealed that the polymer in the mole ratio of 1:1 of ethylene and flame retardant demonstrates optimal thermal oxidation stability; it is easily applied to cotton fabrics by immersion with high absorption in the acetone medium. Fabric with the addition of 33.8 % by weight demonstrates a self-extinguishing ability. Analysis of the morphology of the carbonated treated fabrics revealed the fire resistance of the coating due to the swelling of the fire protection mechanism.

The search for possible alternatives to traditional flame retardants pushes the scientific and industrial community to design new products that would have a low environmental impact and toxicity, despite the high performance, in contact with the flame [7]. In the cited paper, the suitability and efficacy of some biomacromolecules and products of biological
origin with a specific chemical structure and composition of both flame retardants for natural or synthetic textiles were carefully studied in the laboratory. In particular, various proteins (such as serum proteins, caseins, and hydrophobics), nucleic acids, and extracts from natural sources were selected and used to develop fire-resistant treatment procedures for multiple fibers and fabrics. It was found that the biomacromolecules and products of biological origin, which typically contain key elements (that is, nitrogen, phosphorus, and sulfur), can be applied to textiles using standard impregnation methods. In addition, these products are mainly responsible for the formation of stable protective coal (that is, carbon residue) as a result of the exposure of the fabric to flame. However, nothing was said about the impact of environmental changes on the coating, its destruction over time.

A new fireproof coating layer for historical fabrics has been developed in [8]. Silica nanoparticles were additionally impregnated with organic borate, forming a fireproof composite. The resulting composite is combined with connecting one by mechanical mixing, providing a fireproof paste for the coating. The covering paste is applied to the rear surface of the fabrics. Flammability, thermal resistance, and mechanical properties of workpieces and treated samples of linen fabrics as internal support for historical textiles were investigated. The effect of flame on the reverse-coated fabric samples testified to a high class of flame protection of fabrics with a zero combustion rate, compared to 80.3 mm/min for untreated. At the same time, the synergistic effect of refractivity between the nanoparticles and organic borate was established. The limit of tensile strength for fireproof fabrics was increased by 27 %, while their elongation improved. However, the effects of aging on fire protection and the mechanical properties of reverse-coated fireproof textiles have not been studied.

Work [9] investigated melamine-based resins, which are widely used in fabrics to give fire and heat resistance. Modeled washing experiments suggest that per one pound of washing with water, 76.90 % of melamine was removed from clothing. Therefore, the task is to fix flame retardants in the material.

The decomposition of flame-retardant materials due to an elevated temperature or contact with the flame leads to the emergence of a number of chemical forms, some of which can be quite toxic to humans [10]. Small or bench tests of decomposition products have been conducted in the past but questions have always arisen as to whether the tests were representative of full-scale flash test results. Full-scale testing was carried out to determine whether decay products were sufficient to measure and whether different fire-resistant materials would form a “branded” set of compounds. However, the mechanism of coating swelling was not specified and the operating conditions for the coating were not detected.

The influence of SiO₂ ash homogeneity on the duration of the induction period and the quality of fire-resistant coatings on fabrics was investigated in [11]. Prospects for using IR spectroscopy as an express method for studying the phase composition of the gel coating, the degree of completion of the hydrolysis of the silicon-organic component, and adjusting the parameters for obtaining high-quality fire-resistant binary coating of the ash showed the SiO₂ flame-retardant system. However, the areas of application of those products were not specified.

Thus, our review of the literary sources has revealed that flame-retardant coatings are capable of protecting the surface of textile material from the effects of a fire during operation but require a significant amount of protection and are capable of increasing the rigidity of fabrics. In addition, parameters that ensure resistance to loss of fireproof properties have not been determined. The meagerness of mathematical models to explain and describe the process of fire protection of fabrics, neglect of the use of organic substances for the formation of elastic coatings lead to the inefficient application of protective means. The need to establish parameters for the fabric fire resistance and the effect of coatings on this process has predetermined our research in this area.

3. The aim and objectives of the study

The purpose of this work is to identify patterns in the formation of a layer of foam coke when the fabric that is treated for fire resistance with a formulation based on modified phosphorus-ammonium compounds is exposed to high temperatures. This would make it possible to substantiate the use of a flame-retardant coating on objects that apply fabrics.

To accomplish the aim, the following tasks have been set:

– to model the process that forms a layer of coke foam on the surface of the fabric when decomposed by a composition based on modified phosphorus-ammonium compounds under the influence of high temperature;

– to establish the effectiveness of the fire protection of fabric with the composition based on modified phosphorus-ammonium compounds under the influence of flame.

4. The study materials and methods

4.1. The materials studied, which were used in the experiment

To establish the fireproof efficiency of the fabric treated with the composition, we used samples of sailing fabric measuring 220×170 mm (Fig. 1).

The samples were treated with the modified roofing impregnation solution based on a mixture of organic and inorganic substances “Firewall-Attic” for fire protection of timber (a mixture of urea, 28...30 %, and phosphoric acids, 23...24 %), but modified with starch in the amount of 20 %.

Fig. 1. Model fabric samples for testing

The resulting mass was mixed and applied onto a fabric sample in the amount of 137.0... 140.0 g/m² [12]. In this
case, the fireproof coating penetrated the structure of the fabric and formed an elastic film at the surface of about 20 μm thick.

4. 2. Procedure for determining the indicators of fire protection of fabrics by a composition based on modified phosphorus-ammonium compounds

For the study, we used a setup for determining fabric flammability, which was additionally equipped with a device for measuring the temperature at the surface of the sample during tests using a thermocouple (Fig. 2).

![Diagram](image)

Fig. 2. Setup for testing the thermal conductivity of the fire-proof fabric

A sample of the fabric treated with a fireproof agent was fixed in a holder and inserted into the test chamber (Fig. 2). The sample was fixed so that the end of the thermocouple is pressed against the inner surface of the sample. The burner was lit and brought to the sample, the temperature on the reverse surface was measured. By measured values, thermal insulation properties were determined and changes in the coating on the surface of the fabric were recorded [13].

We studied the modeling of the process of thermal conductivity of fire-proof fabric during thermal action using the main provisions of mathematical physics [14].

5. Results of studying the process of fire protection of fabric by a composition when exposed to high temperature

5. 1. Modeling the process that forms coke foam at the surface of fire-proof fabric under the influence of high temperature

Under the effect of heat flow to the fabric with fire-proof coatings, the material decomposition is directed towards the formation of non-combustible gases and a coke residue with low flammability, which isolates the heat and reduces heat transfer to the fabric.

Determining the thermal characteristics of the fireproof layer of fabric coating is associated with the need to measure the temperature in a thin layer of fire protection (up to 0.5 mm), which poses certain difficulties.

Therefore, in this case, a method for solving the thermal conductivity problem for a two-layer plate with different thermal properties has been proposed. Thus, at the initial moment, a constant heat flow $q_0$ is supplied to the surface of the fire-proof fabric sample, which is maintained constant throughout the entire heating process. In this case, the propagation of temperature occurs through the coating until the critical temperature of the fabric is reached. At the same time, the opposite part of the sample is adiabatic ($q=0$).

The following two regions were considered (Fig. 3):

- 1 – a region of the swollen layer of foam coke, $0<x<R_1$ ($R$ is the coordinate of the transformation of the coating film into a swollen layer of foam coke, m);
- 2 – fabric (a sample of the material with a fireproof solid substance) ($R_1<R_2$), m.

Differential equations of heat transfer at the surface of a two-layer plate are [15]:

- for coating

$$\frac{\partial^2 T_1(x, \tau)}{\partial x^2} + \frac{1}{\phi_1^2} \frac{\partial T_1(x, \tau)}{\partial \tau} = 0$$

($\tau>0; 0<x<R$); (1)

- for fabric

$$\frac{\partial^2 T_2(x, \tau)}{\partial x^2} + \frac{1}{\phi_2^2} \frac{\partial T_2(x, \tau)}{\partial \tau} = 0$$

($\tau>0; R_1<x<R_2$), (2)

under the initial and boundary conditions

$$T_1(x,0)=T_1(x,0)=T_0$$

(3)

$$\lambda_1 \frac{\partial T_1(x, \tau)}{\partial x} = q_0 = \text{const}$$

(4)

$$T_1(R_1, \tau)=T_2(R_1, \tau)$$

(5)

$$\lambda_1 \frac{\partial T_1(R_1, \tau)}{\partial x} = \lambda_2 \frac{\partial T_2(R_1, \tau)}{\partial x}$$

(6)

$$\lambda_2 \frac{\partial T_2(x, \tau)}{\partial x} = 0$$

(7)

where $T_1(x, \tau)$, $T_2(x, \tau)$ is the temperature field of the swollen layer of the foam coke of the coating and fabric at points with $x$ coordinates at the time $\tau$;

$\phi=\sqrt{\phi_1}$: $\alpha_1, \alpha_2$ are the temperature conductivity coefficients of the coating and fabric;

$\lambda_1, \lambda_2$ are the coefficients of thermal conductivity of the coating and fabric;

$R_1(R_2-R)$ is the thickness of the coating and fabric.

Following the mathematical transformations, an analytical solution to the thermal conductivity problem (1) to (7) can be obtained if the temperature field $T_2(x, \tau)$ for fabric at each moment is evenly distributed along the $x$ coordinate (the same in all points $R_1<x<R_2$). In this case, the temperature field $T_1(x, \tau)$ of the swollen layer of foam coke can be recorded in the following form [16]:

$$T_1(x, \tau) - T_1(0, \tau) = \frac{q_0}{\lambda_1} x + \frac{\alpha(\tau)}{2\sqrt{\phi_1}} x^2$$

(8)

where $\alpha(\tau)$ is the rate of change over time of the temperature of the fabric, which depends on the amount of heat flow $q_0$.
the geometric dimensions $R$, $(R_1-R)$, and the volumetric heat capacity of the coating $c_1\rho_1$ and fabric $c_2\rho_2$:

$$\alpha(\tau) = \frac{\partial T_c(x, \tau)}{\partial \tau} = \frac{q_0}{[c_1\rho_1 - R + c_2\rho_2 (R_1 - R)]}. \tag{9}$$

Equating (9) and (10) to determine the volumetric heat capacity of the swollen coke coating layer, we obtain the following equation:

$$c_1\rho_1 = \frac{1}{R} \left[ \frac{q_0(t_2 - \tau_1)}{T_2(R, \tau_2) - T_1(R, \tau_1)} - c_2\rho_2 (R_1 - R) \right]. \tag{11}$$

Hence, based on the measured experimental values of the heat flow, the rate of temperature change, the geometric dimensions of the swollen layer of coke and fabric, and the volumetric heat capacity of the raw fabric, one can calculate the volumetric heat capacity of the swollen layer of foam coke.

If we substitute $x=0$ and $x=R$ in dependence (8), then we obtain the equation:

$$T_1(t_0, \tau) = T_0(t_0, \tau). \tag{12}$$

$$T_1(t_1, \tau) = T_0(t_1, \tau) - \frac{q_0 - \frac{\alpha(\tau)}{\lambda_1} R^2}{2\lambda_1 \rho_1}. \tag{13}$$

Based on $\phi = \sqrt{\frac{a}{c}}$ from (10) and taking into consideration [17], one can obtain a formula for calculating the thermal conductivity of the fabric:

$$a = \frac{(R_1 - R)^2}{2(t_2 - \tau_1)}. \tag{14}$$

Based on the experimental data on temperature difference $\Delta T$ of the examined swollen layer of coke, the dependence in the following form was determined:

$$T_c(t_1, \tau) - T_c(t_0, \tau) = \frac{q_0 - \frac{\alpha(\tau)}{\lambda_1} R^2}{2\lambda_1 \rho_1} c_1\rho_1, \tag{15}$$

since thermal conductivity is the equation

$$a = \frac{\lambda}{c \cdot \rho}. \tag{16}$$

From (15), the equation for calculating the thermal conductivity coefficient was derived:

$$\lambda_1 = \frac{q_0 - 0.5 \alpha(\tau) R^2}{T_1(t_1, \tau) - T_1(t_0, \tau)}. \tag{17}$$

Based on the resulting dependence (17), we calculate the thermal conductivity of the swollen coke layer of the coating. The initial data are the experimental values of the heat flow, temperature difference, and the rate of change in the fabric temperature (equation (10)), the geometric dimensions, and the volumetric heat capacity of foam coke (11).

5.2. Results of determining the surface temperature of timber sample under thermal influence

To establish the flammability of the fabric, we studied its ignition under the action of a burner that mimics a low-calorie source. The study results on the ignition of the non-treated fabric sample under laboratory conditions are shown in Fig. 4.

![Fig. 4. Determining the flammability of a fabric sample under the influence of a burner: a — ignition of the fabric sample; b — fabric burning; c — fabric burnout](image)

The study has shown that the fabric sample, non-treated with a flame retardant, ignited, under the influence of a burner for 5 s, the flame spread throughout the entire vertical, which led to its combustion and weight loss.

Then samples were tested, which were treated with a fireproof formulation. The experimental study results on the flammability of fireproof fabric when a fire protection agent was swollen are shown in Fig. 5.

When the burner flame was applied to the samples of fire-proof fabric, intensive swelling began with a slight increase in temperature at the rear surface of the sample. As a result of our tests, it was established that under the influence of flame for 60 s the swelling of a model sample of fabric treated with a flame-retardant formulation amounted to about 4 mm.

The results of our study involving determining the temperature dynamics of fabric samples experimentally under laboratory conditions are illustrated in Fig. 6, in the form of experimental curves obtained using the above procedure.
Our study has shown that when the flame of the burner was applied to a sample of the non-treated fabric, it ignited, the temperature (curve 1) at the rear surface exceeded 300 °C. Its burning continued over 480 s, the material was burned by more than 40 %. A sample of the fire-proof fabric withstood the temperature effect of the burner flame. Under the action of the heat flow, the coating was swollen, it continued for 60 s, the temperature (curve 2) at the rear surface did not exceed 150 °C, and the weight loss was 2.9 %.

We determined the thermal-physical characteristics of fabric without fire protection according to the procedure given in [18]; the study results are given in Table 1.

### Table 1

<table>
<thead>
<tr>
<th>Material name</th>
<th>Thickness, mm</th>
<th>Mass, g</th>
<th>Density ρ, kg/m³</th>
<th>Temperature conductivity λ, W/(m·K)</th>
<th>Heat capacity, kJ/(kg·K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fabric 170×220 mm</td>
<td>1.2</td>
<td>25.2</td>
<td>560.0</td>
<td>16·10⁻⁶</td>
<td>0.078</td>
</tr>
</tbody>
</table>

Taking into consideration that the heat flow of the gas burner flame is about 10,100 kW/m² [19] and the flame temperature of the gas burner was about 1,000 °C [20]. According to the data on temperature measurements obtained during the tests (Fig. 6), using the derived dependences (11) and (17), we calculated the thermophysical properties of foam coke. Thus, the volumetric heat capacity of the foam coke layer was 427.5 kJ/(m³·K), and the thermal conductivity coefficient was 0.034 W/(m·K), respectively.

The results from our determining the flammability of flame-retardant fabric correspond to the properties of the formed heat-resistant layer of foam coke under the action of high-temperature flame, which testify to the resistance of the flame-retardant formulation against the influence of high flame temperature, as well as material protection.

### 6. Discussion of results of studying the process that forms a layer of foam coke for the fire protection of fabric

It follows from the results (Table 1, Fig. 6) of our study into the process of fire protection of fabric by a formulation based on modified phosphorus-ammonium compounds that it is natural to extend the time of temperature transfer through the fire-proof fabric. This is due to the formation of a layer of swollen coke layer at the surface of fire-proof timber when decomposing flame retardants under the influence of flame, which slows down the processes of heat transfer to the fabric and its combustion.

It should be noted that the presence of modified phosphorus-ammonium compounds leads to the formation of an elastic film at the fabric surface that is fluctuation resistant. Such a mechanism of the influence of an elastic film is likely the factor in regulating the process by which the fire resistance of the fabric is preserved. In this sense, there is an interpretation of the results from determining the nonflammability of the fabric after its exposure to the flame, namely the loss of mass by samples during thermal exposure. The mass loss did not exceed 2.9 %, and the temperature at the rear surface of the sample did not exceed 150 °C. This indicates the formation of a barrier for temperature, which can be identified by the thermal effect on the examined samples.

That means that taking into consideration this fact opens the possibility for effective adjustment of the properties of fire-proof fabric directly during its industrial production.

The comparison of the experimental study into the heat insulation of the foam coke layer for the fire protection of fabric and the theoretical study into the thermal insulation of fabric indicates the inhibition of heat transfer processes. The temperature at the rear surface during the action of the burner did not exceed 150 °C, and the weight loss was 2.9 %.
This does not diverge from practical data known from works [3, 5] whose authors also associate the effectiveness of fire protection with the formation of a layer of coke foam under the influence of a burner flame. However, unlike the results of studies reported in [6, 7], our data on the effects of modified phosphorus-ammonium compounds on the process of inhibition of temperature transfer allow us to assert the following:

1. We have modeled the heat transfer process in the fabric when applying a fireproof coating is exerted by the formation of a layer of coke from an elastic film on the surface of the fabric, resistant to destruction under the action of product oscillations.

2. Features in inhibiting the heat transfer process in the material treated with a composition based on modified phosphorus-ammonium compounds are the formation of a heat-protective layer of coke at the surface of the fabric. Thus, at the surface of the sample, a temperature was achieved that significantly exceeded the ignition temperature of the fabric, and, at the non-heated surface, did not exceed 150 °C.

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