

# IDENTIFYING PATTERNS OF CHANGE IN FIRE PROTECTION OF THE TENT FABRIC TREATED WITH AN EPOXY RESIN-BASED INTUMESCENT COATING

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This study investigates the process of forming a fire-retardant coating based on modified epoxy resin on the surface of tent fabric. The task addressed is to ensure fire protection of tent fabric when treated with a coating based on modified epoxy resin. This is important given the relevance of fire-resistant materials for modern construction.

It has been proven that when treating tent fabric with epoxy resin, the free energy component of the fabric surface decreased by more than 2.3 times, while the polar component increased by 1.6 times, which indicates a change in the surface. At the same time, the tensile strength of tent fabric after treatment with epoxy resin increased by more than 4.1 times.

The study has shown that a sample of tent fabric caught fire when exposed to a burner flame for 5 s and continued to burn for 16 s when the ignition source was removed. The treatment of tent fabric with epoxy resin with flame retardant led to the formation of charring on the sample with a length of more than 70 mm, and the height of the swelling in the flame zone was about 3 ÷ 5 mm. When testing tent fabric samples for the flame spread index, it was found that the sample treated with epoxy resin caught fire in 524 s, the flame spread throughout the sample for 14 s, the burning length of the sample was 300 mm, and the flammability index was 36.3. However, for samples treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide, the flammability indices decreased by more than 30 times. The optimal concentration of components in the coating containing 34 ÷ 35% (wt.) of ammonium polyphosphate and 9 ÷ 10% (wt.) of aluminum hydroxide has been determined.

Thus, there are grounds for the possibility of designing fire-resistant coatings for construction

**Keywords:** tent fabric, epoxy resin, mixture of ammonium polyphosphate and aluminum hydroxide, fire resistance of the fabric, surface energy

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## 1. Introduction

Textile products made of natural fibers are increasingly used in various types of building structures, both in permanent and

in particular in temporary structures. During occupancy and heating of these structures, ignition and rapid spread of fire are possible because the fabric creates a significant fire load. Given the fact that this material is sensitive to the effects of high tem-

perature, it is possible to increase the level of fire safety of objects where building structures made of textile materials are used by means of their fire-retardant treatment.

The need for fire protection is relevant for objects of mass occupancy of people, which are made of flammable textile materials. Below are examples of fires typical of tent camps in the Armed Forces of Ukraine. In particular, in the Carpathian region (Ivano-Frankivsk oblast, Kolomyia city), a strong fire broke out in the tent camp of the 10<sup>th</sup> separate mountain assault brigade on the territory of the military unit (Fig. 1). One of the reasons for the rapid propagation of the fire was the lack of fire protection for such textile products. As a result of the fire, fifteen large 50-person field tents were destroyed where the soldiers lived while the stationary barracks were being repaired, because of the valve opening for its installation being adjacent to the chimney. During the fire, 2 servicemen were injured. The fire destroyed 15 USB-56 tents, as well as the provisions and medical services. Damage from the fire amounted to UAH 8 million 113 thousand 679. Strong winds contributed to the rapid spread of the fire. Five servicemen received minor injuries (light burns to their hands) while saving property as they were taking personal belongings and part of the property and equipment.



Fig. 1. Consequences of a fire in Prykarpattia (Ivano-Frankivsk oblast, city of Kolomyia) in a military camp on the territory of the 10th separate mountain assault brigade

Forecasting the main causes of fires indicates that the following ones will remain: careless handling of fire, violation of the rules for the installation and operation of electrical equipment and household appliances. In addition, because of hostilities in the conflict zone in eastern Ukraine, a new category of characteristic fires in tent camps has emerged, associated with the use of stove heating in the winter period and fires caused by the use of various incendiary mixtures using shock UAVs.

Fire statistics in the Armed Forces of Ukraine indicate the need to build an effective system for ensuring the explosion and fire safety of military facilities, as well as to carry out preventive measures, primarily fire-resistant treatment of cellulose combustible materials.

For comprehensive protection of textile materials from fire, it has been proposed to use mixtures of water-soluble inorganic salts, but fire-retardant treatment with these substances is not suitable for textile materials because the formation of efflorescence is observed on the surface that falls off and, over time, the material loses its protective properties, which leads to the ignition of flammable structures under the influence of high-temperature flames.

Fire-protective coatings based on inorganic binders contain sodium silicate, Portland cement, high-alumina cement,

aluminosilicate binders, and bound water. During heating, the process of water evaporation and the formation of a ceramic phase on the surface of the textile product occurs, which blocks the transfer of heat to the surface of the material. However, these coatings have a large temperature coefficient of linear expansion and low adhesion strength, which makes them ineffective. A promising direction is to design effective and safe reactive coatings for such materials, which, under the influence of high temperature, form a heat-resistant heat-insulating layer of coke.

Therefore, it is a relevant task to perform a study aimed at determining the patterns of fire protection of tent fabrics used for the manufacture of military tents. One promising option is to investigate the formation of a coke layer during fire protection of fabric with a composition based on modified polymer resins.

## 2. Literature review and problem statement

Paper [1] reports a systematic method for the synthesis of a transparent intumescent flame retardant coating based on bisphenol-A epoxy resin. Compared with transparent epoxy resin-based flame retardant coatings described in the literature, the proposed coating exhibits improved performance with excellent flame retardancy. These improvements are achieved by removing urea and adjusting the ratio of phosphoric acid, which leads to simplified synthesis procedures and a significant increase in transparency. It is noteworthy that when the ratio of epoxy resin, diethanolamine, and phosphoric acid is optimized to 1:2:4, a 1.2 mm thick coating achieves optimal flame retardancy. In a large panel simulation method lasting 45 minutes, the backside temperature stabilized at approximately 148°C. However, further development is needed not only in the materials themselves but also in the disposal methods that would allow the devised parameters to be maintained.

A study in [2] provides a thorough evaluation of ethylenediamine ammonium polyphosphate (EDA-MAPP) and foaming agents (CFA) used for the preparation of epoxy resin-based intumescent fire retardant coatings. TGA studies on modified ammonium polyphosphate (MAPP) and ammonium polyphosphate (APP) were performed. The synergistic effect of antimony oxide on the fire retardant coating composition was studied using vertical burning test (UL-94V), thermogravimetric analysis (TGA), limited oxygen index (LOI), and Fourier transform infrared spectroscopy (FTIR). The results (TGA) showed that amalgamation of antimony oxide at each concentration effectively enhances the thermal stability of the fire retardant coating system. Cone calorimeter studies show that antimony oxide successfully minimizes combustion parameters such as peak heat release rate (PHRR) and total heat release (THR). Results of Fourier transform infrared spectroscopy analysis show that antimony oxide can react with MAPP and form a dense charred layer that prevents heat and oxygen transfer, but no optimal solutions for their use have been identified.

In [3], the fire-retardant properties of polyurethane (PU) containing ammonium polyphosphate (APP) and aluminum hydroxide (ATH) were investigated. In addition, flame retardant properties were examined using thermogravimetric analysis, limiting oxygen index (LOI), vertical burning (UL 94), and cone calorimeter. When adding 15 wt% ACE and 5 wt% ATH, the PU/15%ACE/5%ATH sample shows

better thermal stability and flame retardant properties. When 15 wt% ACE and 5 wt% ATH were added, the LOI value of the PU/15%ACE/5%ATH sample was 30.5%, and a UL 94 V-0 rating was achieved. Compared with PU, the peak heat release rate (PHRR), total heat release (THR), and average effective heat combustion rate (av-EHC) of the PU/15%ACE/5%ATH sample decreased by 43.1%, 21.0%, and 29.4%, respectively. In addition, the mechanism of flame retardant action was investigated using a cone calorimeter. The addition of APP/ATH simultaneously exerted a flame retardant effect in both the condensed and gas phases. APP and ATH have synergistic flame retardant properties. However, there is nothing about the expediency of their use in modern construction.

The authors of [4] studied polymer compositions based on epoxy resin cured with polyethylene polyamine, containing ammonium polyphosphate and boric acid as a pore former and carbonization stimulator. The UL 94 method was used to determine the material's ability to burn or extinguish after flame treatment. Weight loss at characteristic temperatures was determined using a Perkin Elmer STA8000 synchronous DTA/THA analyzer. The dependence of heat capacity on temperature was determined (calorimeter ITS-400). It is shown that in the modified compositions containing ammonium polyphosphate and boric acid, the heat capacity changes without significant jumps, which is associated with a calmer course of gas formation. The structure of intumescent coatings was studied using atomic force microscopy. The research was carried out on the P4-Solver NT-MTD device. However, the tests of samples did not show that the addition of boric acid make it possible to obtain a more ordered structure of coke foam and increase the heat capacity of intumescent coatings.

In [5], promising compositions were described that were immobilized in an epoxy binder and formulated with other intumescent additives, such as ammonium polyphosphate (APP) and melamine (MEL), to evaluate the effectiveness in the coating system. These formulations were then evaluated using quantitative cone calorimetry. The specific formulations containing PAA demonstrate peak heat release rates (PHRR) and total heat release (THR) of 283 kW/m<sup>2</sup> and 50.5 MJ/m<sup>2</sup>, respectively, which compare favorably with the data for systems containing BA, in particular PHRR = 229 kW/m<sup>2</sup> and THR = 43.1 MJ/m<sup>2</sup>. The results showed the promise and the need for further research of PAA as a multifunctional additive for use in fire-retardant and intumescent coatings, but the importance of considering specific requirements for practical application was not noted.

In [6], it was reported that aluminum hydroxide nanoplatelets (nATH) were synthesized by a hydrothermal process using an Al(OH)<sub>3</sub> precursor gel. The ATH nanoplatelets were then treated with organic compounds and incorporated into an intumescent flame retardant epoxy system containing polyethyleneimine-modified ammonium polyphosphate (APP@PEI). The resulting nanocomposite achieved a V-0 rating in the UL-94 vertical burning test with a high oxygen index limit of 31.1% and a significant carbon yield of 17.98% at 900°C. In addition, tensile and Izod impact tests showed that the presence of nATH PEI significantly improved the mechanical properties of the APP@PEI composite loading. However, the prospects for the application of intumescent flame retardants in epoxy resins are not noted.

Paper [7] reported experimental results showing that the incorporation of ammonium polyphosphate (APP) sig-

nificantly improves fire resistance and thermal stability. Mechanical properties decrease with increasing APP content; however, the composite containing 20% APP maintains mechanical properties exceeding industry standards for non-load-bearing capacity while exhibiting improved fire resistance. Thermogravimetric analysis (TGA) confirmed that APP promotes char formation, thereby improving thermal stability. Cone calorimetry tests show a decrease in both peak heat release rate (PHRR) and total heat release (THR) with higher APP content. In particular, the composite with 25% APP showed the lowest PHRR, while the composite with 30% APP achieved the lowest THR and smoke generation. In vertical burning tests according to UL-94, the composite modified with 20% APP self-extinguished within 2 seconds, achieving a high level of fire resistance. But the mechanism of the coating has not been revealed.

Study [8] showed that ammonium polyphosphate and aluminum hydroxide were simultaneously added to polyisocyanurate-polyurethane foams as flame retardants. Cone calorimetric tests, limiting oxygen index and scanning electron microscopy were used to study the fire resistance of aluminum hydroxide and ammonium polyphosphate. The results show that the compressive strength of the foams increases after the addition of both aluminum hydroxide and ammonium polyphosphate. The fire resistance of polyisocyanurate-polyurethane foams can be significantly improved under the influence of aluminum hydroxide and ammonium polyphosphate. After the addition of 5-particle aluminum hydroxide and 15-particle ammonium polyphosphate, the oxygen index limit value increases from 21.2% for the control sample to 28.0%, while the peak heat release rate decreases from 159.8 kW/m<sup>2</sup> for the control sample to 76.8 kW/m<sup>2</sup>. However, the interaction between aluminum hydroxide and ammonium polyphosphate occurring in the condensed phase is not mentioned.

In study [9], an environmentally friendly expanded flame retardant coating for polyester (PET) fabrics was designed, based on the flame retardant agent as aluminum diethylphosphinate (P-FR), trisilanolisobutyl-POSS (Si-FR), sericin (SC), and polyvinyl alcohol (PVA) using citric acid (CA) as a chemical crosslinker. Thermogravimetric analysis revealed that SC and Si-FR improved the oxidative stability of carbon. The flame retardant treatment increased the limiting oxygen index (LOI) from 21.1% for the untreated fabric to 31.7% for the treated fabric, while the tensile strength increased and the elongation at break decreased. Notably, after 50 washing cycles, the treated fabrics retained their self-extinguishing properties, although the UL 94 classification was reduced to V-2. However, no information was provided on the degradation of the resulting fabrics, nor on the recycling of the fabrics.

In [10], carboxylated multi-walled carbon nanotubes (COOH-MWCNT) were first aminated to obtain aminated multi-walled carbon nanotubes (NH<sub>2</sub>-MWCNT). Subsequently, NH<sub>2</sub>-MWCNT and ammonium polyphosphate (APP) were incorporated into the epoxy resin by mechanical mixing, thereby creating a synergistic phosphorus-carbon flame retardant system. Compared with the pure epoxy thermosetting material, the EP/17.5APP/0.1NH<sub>2</sub>-MWCNT composite showed a limiting oxygen index (LOI) of 29.6% and achieved a UL-94 V-0 rating. In addition, for the modified composite material, the maximum thermal decomposition rate is 12.4 wt. %/min, the carbon residue at 600°C reaches 44.2%, and the smoke density is 425.8. The impact toughness and ten-

sile modulus increase to 10.1 MPa and 3.0 GPa, respectively, while the compressive strength remains almost unchanged. In addition, the synergistic flame retardant mechanism between phosphorus and carbon has been investigated by analyzing the carbon residue of epoxy resin and its composites. However, their applications in various high-tech industries are not known.

The composition, which is described in [11], consists of two separate components. Component A contains epoxy resin (up to 35%), ammonium polyphosphate (up to 35%), melamine (up to 10%), pentaerythritol (up to 11%), titanium dioxide (up to 5%), flame retardants, and additives (up to 4%). Component B is a polyamide amine hardener. For this purpose, it is proposed to form a nanocomposite: epoxy resin / nanoclay in situ during the production of intumescent paint. Such preliminary modification of the epoxy resin increases the temperature at which the formed expanded layer of foam coke decomposes during exposure to fire and promotes the formation of a heat-insulating carbonized material with increased thermal resistance. The proposed intumescent epoxy composition is applicable as a basis for mass production of fire-retardant compositions for steel structural elements, but tests for assessing their strength were not performed.

Epoxy and unsaturated polyester resins, as noted in [12], are the most widely used thermosetting polymers, but they are flammable. The enhancement of the fire resistance of thermosetting polymers and their composites can be improved by adding flame retardants, but this comes at the expense of their mechanical properties. In this regard, a comprehensive review of recent research papers that studied the fire resistance of epoxy resin, unsaturated polyester resin, and their composites was considered. The fire resistance of various flame retardant/polymer material systems was evaluated in terms of the Fire Resistance Index (FRI), which was calculated based on data obtained during the cone calorimeter test. In addition, flame retardant selection charts were presented, which relate the level of fire resistance to mechanical properties in terms of tensile and flexural strength. However, the mechanism of combustion suppression of polymeric materials, as well as flammability testing methods and the mechanism of action of flame retardants, have not been described.

Our review of the literature [1, 2, 6–10, 12] established that despite extensive research on fire-resistant epoxy composites, limited information is available on their application to cotton-jute fabrics for tents and simultaneous assessment of surface energy, tensile characteristics, and behavior during fire exposure. Therefore, the task to determine changes in textile materials during processing and to define the influence of coating components on the process has necessitated the need for our research.

### 3. The aim and objectives of the study

The aim of our work is to determine conditions for the formation of a fire-retardant coating of tent fabric based on epoxy resin, which is filled with ammonium polyphosphate and aluminum hydroxide. This could make it possible to substantiate the scope of application of these products in construction.

To achieve the goal, it was necessary to solve the following tasks:

- to establish features of change in the energy characteristics of the surface of the tent fabric when treated with epoxy resin;

- to investigate the fire-retardant properties of the tent fabric treated with epoxy resin, filled with a mixture of ammonium polyphosphate and aluminum hydroxide.

## 4. The study materials and methods

### 4.1. The object and hypothesis of the study

The object of our study is the process of forming a fire-retardant coating based on modified epoxy resin on the surface of the tent fabric. The scientific hypothesis is to form a fire-retardant coating from epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydrate on the surface of the tent fabric and experimentally evaluate the effectiveness.

In the process of the study, it was assumed that the process of designing a fire-retardant tent fabric is constant under the influence of external conditions. It was accepted that the temperature, humidity, and pressure of the tent fabric processing do not change.

### 4.2. Tested materials used in the experiment

To study the possibility of fire protection of textile material, we used tent fabric (cotton 50% / jute 50%) produced by TK “Textile Contact” (Ukraine), with a thickness of about 1.5 mm and a density of 88...90 g/m<sup>3</sup> (Fig. 2). To treat the surface of the tent fabric, a two-component epoxy resin “MC-DUR 111 eco (Komponente A)” and a hardener “MC-DUR 111 eco (Komponente B)” produced by MC-Bauchemie (Germany) were used, resistant to moisture, diluted acids, and alkalis, as well as organic compounds, which, when a hardener is added, forms a durable coating. Since epoxy resin is a flammable material (the ignition temperature with an open flame is about 300°C), to reduce flammability and smoke formation, a phosphorus-ammonium flame retardant was added to the resin – a mixture of ammonium polyphosphate, brand APP-201, manufactured by OCEANXEM (China), and aluminum hydroxide, manufactured by KIMLABORREAKTIV (Ukraine), which makes the material more fire-resistant.



Fig. 2. Tent fabric sample for testing

At the first stage, samples of tent fabric treated with epoxy resin in the amount of 6 g and resin hardener 1.5 g were prepared. At the next stage, a composition of epoxy resin together with hardener was prepared and mixed in a 1:1 ratio with flame retardant powder, which was 30 ÷ 35 % (wt.) of

ammonium polyphosphate and 1 ÷ 10% (wt.) of aluminum hydroxide. The resulting composition was mixed to a homogeneous state in a dissolver for 60 s and applied to the surface of the tent fabric with a brush. The total coating consumption for treating the surface of the tent fabric was about  $260 \div 270 \text{ g/m}^2$ . Table 1 gives samples of tent fabric for testing changes in the energy characteristics of the surface, breaking load, and fire protection efficiency of the tent fabric measuring  $220 \times 170 \text{ mm}$ .

Table 2 gives samples for testing the flammability index of a  $300 \times 120 \text{ mm}$  tent fabric treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide.

Tent fabric test samples

Weight of tent fabric sample before application, g	Amount of epoxy resin, g	Amount of epoxy resin hardener, g	Amount of ammonium polyphosphate, g	Amount of aluminum hydroxide, g
untreated	–	–	–	–
27	6.3	1.5	–	–
28	4.5	1.5	3.0	1.0

Table 1

Surface tension; dispersed and polar components for test liquids

Material	$\sigma_{sa}^d$	$\sigma_{sa}^p$	$\sigma_{la}$
water	21.8	50.8	72.6
Ethylene glycol	29.3	19.0	48.3

Table 2

Samples for testing tent fabric treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide for determining the flammability index

Sample number	Weight of tent fabric sample before application, g	Amount of epoxy resin, % (wt.)	Amount of epoxy resin hardener, % (wt.)	Amount of ammonium polyphosphate, % (wt.)	Amount of aluminum hydroxide, % (wt.)
0	27.0	45.10	15.01	–	–
1	27.0	45.05	15.01	33.01	0
2	26.6	45.11	15.00	32.04	1
3	27.1	45.00	15.02	32.01	2
4	26.9	45.09	14.99	33.00	3
5	26.8	45.10	15.00	32.01	4
6	27.0	45.08	15.02	31.00	5
7	27.0	45.07	14.98	31.01	6
8	26.9	45.08	15.00	32.01	7
9	26.8	45.02	14.99	32.00	8
10	26.8	45.01	15.00	31.01	9
11	26.9	45.00	15.00	30.00	10

The obtained samples were dried under laboratory conditions at a temperature of  $20 \pm 2.5^\circ\text{C}$  and a pressure of  $99 \div 101 \text{ kPa}$  for 24 hours. After the formed coating was exposed to air for 3 days under laboratory conditions, the samples were tested for changes in surface properties, strength, and fire resistance of the tent fabric.

#### 4. 3. Methodology for determining the properties of samples

To establish a change in the energy characteristics of the surface of the tent fabric after treatment with epoxy resin, the contact angle was determined according to the methodology from [13]. Its essence was to apply a drop of test liquid to the tent fabric and, after the drop reached an equilibrium state, its height and diameter were determined using a microscope.

The assessment of change in the surface energy of tent fabrics during treatment with epoxy resin was carried out using the Fawkes method, which makes it possible to take into account dispersion, hydrogen and dipole-dipole interactions at the solid-liquid interface [14]

$$(1 + \cos\theta)\sigma_{sa} = 2(\sigma_{sa}^d \cdot \sigma_{la}^d)^{0.5} + 2(\sigma_{sa}^p \cdot \sigma_{la}^p)^{0.5}, \quad (1)$$

where  $\theta$  is the contact angle;

$\sigma_{sa}$ ,  $\sigma_{la}$  are the surface energy of the solid and liquid, respectively; index  $p$  is the component of the total surface energy, which is due to hydrogen and dipole-dipole interactions; index  $d$  is due to dispersion interactions.

This equation has two unknown quantities  $\sigma_{sa}^d$  and  $\sigma_{sa}^p$ ; for practical use, contact angle data for two different test liquids with known surface tensions  $\sigma_{la}^d$  and  $\sigma_{la}^p$  (Table 3) are required.

To determine the tensile properties of coated tent fabric, the tensile load was determined according to ASTM D5035-11 [15]. For this purpose, an Avery (UK) tensile machine was used, which is designed to operate at a speed of  $300 \pm 10 \text{ mm/min}$  with variable loads required to obtain a time to rupture of  $20 \pm 3 \text{ s}$ .

Flammability tests of epoxy-treated tent fabric were carried out according to the working methodology [16], the essence of which was to experimentally determine the combustion characteristics of materials under the influence of flame under controlled laboratory conditions. For testing, at least five samples of one type of tent fabric are prepared for research. For testing, a test rig is used in which a test

sample of textile material ( $220 \times 170 \text{ mm}$  in size) is fixed and a gas burner with a flame height of 40 mm is supplied. The test is carried out under the action of a burner for 5 s; for this purpose, the sample of material is fixed on the spikes of the sample holder, the burner is installed in a horizontal position 40 mm above the lower edge of the sample and moved to the sample at a distance of 17 mm. In the absence of stable combustion, the test is carried out on a new sample, without changing the position of the burner for 15 s. During the test, the duration of residual flame combustion, material burnout, surface flash propagation are recorded; the average length of the charred area is measured to be entered in a table [17].

The essence of the test method for experimental determination of the flammability index of a tent fabric sample is to expose the sample located in the installation to a radiation panel, followed by determination of the ignition process and flame propagation [18]. In this case, the thermal coefficient of the installation is determined, the maximum temperature of the combustion products and the time of its achievement are measured, the time of ignition and passage of the flame front through the surface areas, the length of the burnt part of

the sample, as well as the final combustion after removing the burner for 600 s, which are entered into the table. And according to the data, the value of the dimensionless flammability index is calculated at coefficient  $I$

$$I = \sqrt{\frac{q \cdot Q}{W} \cdot \frac{\Delta T_{\max}}{\Delta T_{he}} \cdot \frac{\tau_{\max} - \tau_0}{\tau_0} \left[ 1 + \frac{60 \cdot l_s}{l} \cdot \sum_{i=1}^n \frac{1}{\tau_i} \right]}, \quad (2)$$

where  $q$  – specific heat of combustion of propane gas (23630),  $\text{kJ} \times \text{L}^{-1}$ ;  $Q$  – gas flow rate of torch burner (0.001),  $\text{L} \times \text{s}^{-1}$ ;  $W$  – power of the electric radiation panel, 0.5 kW;  $\Delta T_{\max}$  – maximum increase in flue gas temperature

$$\Delta T_{\max} = T_{\max} - T_0,$$

where  $T_0$  is the ambient temperature,  $^{\circ}\text{C}$ ;  $T_{\max}$  is the maximum flue gas temperature,  $^{\circ}\text{C}$ ;  $\Delta T_{he}$  is the maximum temperature increase of the heating equipment

$$\Delta T_{he} = T_1 - T_0,$$

where  $T_0$  is the ambient temperature,  $^{\circ}\text{C}$ ;  $T_1$  is the outlet air temperature during operation of the heating equipment,  $^{\circ}\text{C}$ ;  $\tau_0$  is the time of ignition of the sample, s;  $\tau_{\max}$  is the time of reaching the maximum temperature of the flue gases, s;  $\tau_i$  is the time of passage of the flame front through the control sections, s;  $l$  is the length of the sample, mm;  $l_s$  is the length of damage to the sample, mm.

### 5. Results of experimental evaluation of fire-retardant properties of tent fabric treated with an intumescent coating based on epoxy resin

#### 5.1. Results of experimental study of energy characteristics of the surface of tent fabric treated with epoxy resin

The results of determining the contact angle of tent fabric with samples of test liquids are shown in Fig. 3, 4.

The results (Fig. 3, 4) demonstrate that distilled water when wetting tent fabric forms an angle of about  $48^{\circ}$ , and when wetting with ethylene glycol forms an angle of about  $76^{\circ}$ . However, when wetting tent fabric treated with epoxy resin with distilled water, an angle of  $130^{\circ}$  is formed, and for ethylene glycol –  $45^{\circ}$ , respectively.

The results of determining the contact angle of wetting of the tent fabric by test liquids and determining the corresponding components of the surface free energy are given in Table 4.

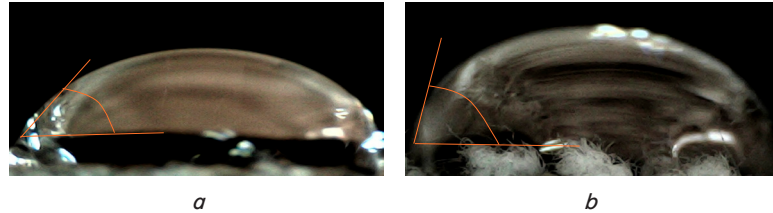


Fig. 3. Test liquids applied to the tent fabric: *a* – distilled water; *b* – ethylene glycol

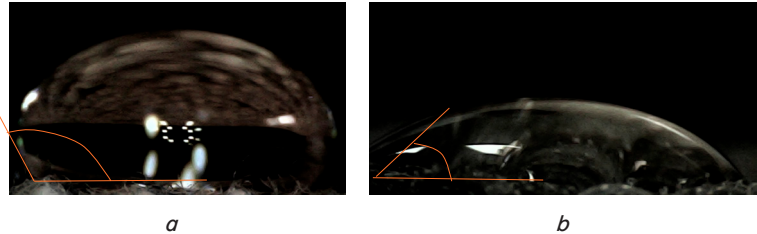


Fig. 4. Test liquids applied to tent fabric treated with epoxy resin: *a* – distilled water; *b* – ethylene glycol

As the results of wetting and the free energy component of the surface of the tent fabric showed, the dispersed component of the free energy of the surface of the fabric after treatment with epoxy resin decreased by more than 2.3 times. On the other hand, the polar component increased by 1.6 times, which indicates a change in the surface of the tent fabric during treatment with epoxy resin.

In order to determine the operational properties of tent fabrics during the construction of modular structures and the erection of buildings, the tensile strength was determined, shown in Fig. 5–7. The results of determining the tensile strength of the tent fabric are given in Table 6.

Table 6

Test results for determining the tearing characteristics of tent fabrics

Tested fabrics	Breaking load, kN
Untreated tent fabric sample	0.58
Epoxy treated tent fabric sample	2.40
Epoxy treated tent fabric sample with flame retardant	2.24



*a*



*b*

Fig. 5. Tent fabric samples for testing: *a* – untreated; *b* – treated with epoxy resin

Table 5

Contact angle and free energy component of the surface of the tent fabric

Tent fabric	Contact angle, $\theta, ^{\circ}$		Surface free energy, $\text{mJ}/\text{m}^2$			Polarity, %
	water	Ethylene glycol	Total	Polar	Dispersed	
Untreated	49	76	41.1	8.3	32.8	20.2
Treated	130	45	27.4	13.3	14.1	48.5

It was found that the tensile strength of the treated tent fabric after treatment with epoxy resin increased by more

than 4.1 times. Filling the epoxy resin with a flame retardant in a ratio of 1:1 reduced the tensile strength by only 7%.

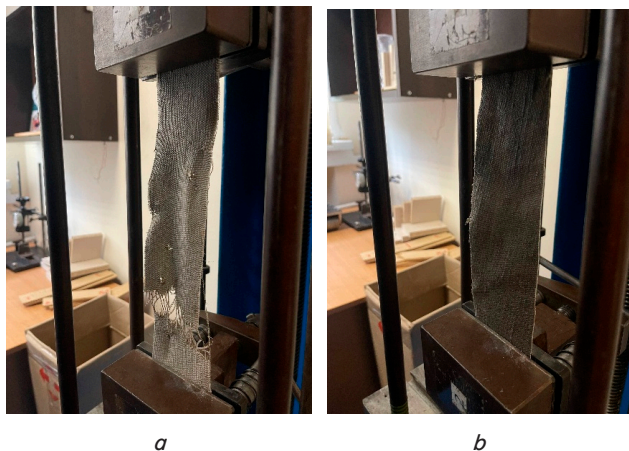


Fig. 6. Tent fabric tensile test: *a* – untreated; *b* – treated with epoxy resin

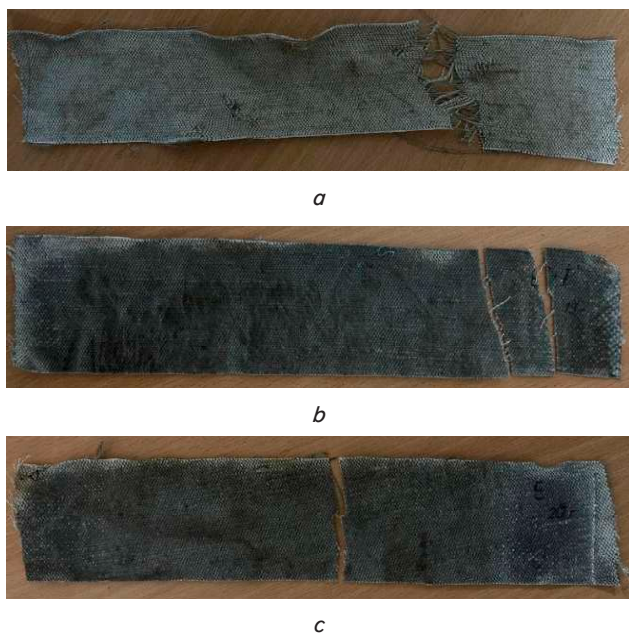


Fig. 7. Destruction of tent fabric during tensile tests: *a* – untreated; *b* – treated with epoxy resin; *c* – treated with epoxy resin filled with flame retardant

**5. 2. Results of investigating the fire-retardant properties of tent fabric treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide**

The results of studies on the ignition of a sample of tent fabric untreated and treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide when interacting with a high-temperature burner flame are given in Fig. 8, 9 and in Table 7.

As can be seen from Table 6, when the thermal action of the burner on the tent fabric sample is 5 s, the sample ignited, and the subsequent residual flame burning for 16 s occurred, which led to the combustion of the sample. In contrast, for the tent fabric sample treated with epoxy resin, when the thermal action of the burner is 15 s, ignition and burning did not occur. However, the length of the charred area exceeded 100 mm, the mass decreased by 21%.

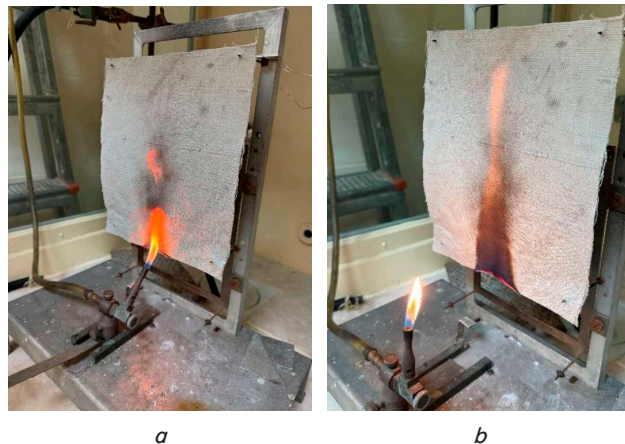


Fig. 8. Ignition of an untreated sample of tent fabric: *a* – exposure to a burner flame; *b* – spontaneous combustion

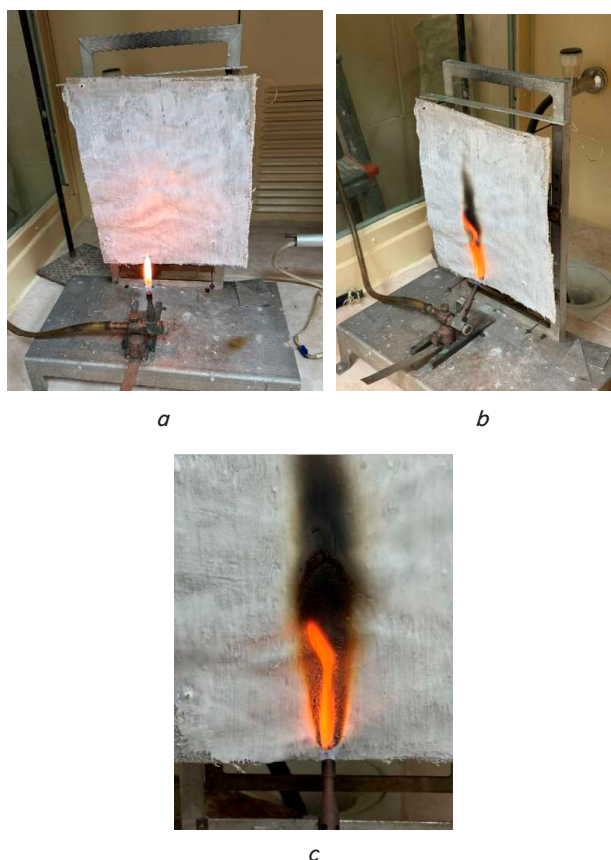


Fig. 9. Ignition of a sample of tent fabric treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide: *a* – sample before testing; *b* – exposure to a burner flame; *c* – swelling of the coating

The sample of tent fabric treated with epoxy resin after 15 s of thermal action did not catch fire, charred at the site of the burner, the flame did not spread, but the length of the charred area was over 100 mm, and the mass loss was 25%. On the surface of the sample of tent fabric treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide, ignition and burning did not occur, charring was formed on the surface with a length of over 70 mm and a width of 25 ÷ 32 mm, and the height of the formed foam coke in the flame zone was about 3 ÷ 5 mm. At the same time, the mass loss was 8%.

Fig. 10, 11 and Table 8 show the process of experimental determination of the flammability index of a sample of tent fabric.

Studies have shown (Fig. 11, 12) that a sample of tent fabric treated with epoxy resin caught fire after thermal exposure. In contrast, a sample of tent fabric treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide did not catch fire.



Fig. 10. Test results from determining the flammability index of tent fabric treated with epoxy resin: *a* – thermal effect on the sample; *b* – ignition of the sample and flame spread

From Table 8 it follows that the sample of tent fabric, which was treated with epoxy resin, caught fire on 524 s, the flame spread throughout the sample for 14 s, the burning length of the sample was 300 mm, the flammability index was 36.3. For samples treated with epoxy resin filled with a mixture

of ammonium polyphosphate and aluminum hydroxide, the flammability index decreased by more than 30 times, but for them both ignition in the first section of the sample for no more than 10 s and flaming of the flame at the final stage of the test for 1 ÷ 2 s were recorded. Thus, under the influence of high flame temperature, a joint decomposition of ammonium polyphosphate and aluminum hydroxide occurs, which release a significant amount of non-combustible vapors and gases, induce the formation of high-temperature phases of aluminum phosphate and obtain a more ordered structure of coke foam; however, it was not found which sample is the most effective.



Fig. 11. Test results from the process that determines the flammability index of tent fabric treated with epoxy resin, filled with a mixture of ammonium polyphosphate and aluminum hydroxide: *a* – thermal effect on the sample; *b* – ignition of the sample and flame spread

Table 7

Flammability test results for tent fabric treated with epoxy resin

Flammability test	Untreated sample of tent fabric	Treated sample of tent fabric with epoxy resin	Treated sample of tent fabric with epoxy resin filled with ammonium polyphosphate
Test time, s	5	15	15
Duration of residual flame combustion, s	16	absent	absent
Material burn-through	burns out	charring	does not burn through
Spread of surface flash more than 100 mm from the ignition point	spreads	not applicable	does not spread
Average length of charred area, mm	–	106	70 (formation of foam coke)
Average mass of samples before testing, g	26.9	31.7	32.4
Average mass of samples after testing, g	2	23.8	29.7

Table 8

Maximum temperature of combustion products and time of its achievement, time of ignition and passage of the flame front through the surface areas, length of the burnt part of the sample during burning of a tent fabric sample

Sample according to Table 2	T °C flue gases,		Ignition time, s	Time of passage of the flame front through sample sections, s									Time to reach maximum flue gas temperature, s	Burning length of the sample, mm	Flammability index	
	T <sub>1</sub>	T <sub>max</sub>		1	2	3	4	5	6	7	8	9				
0	66	383	524	3	2	2	2	1	1	1	1	1	1	541	300	36.3
1	66	100	589	98	–	–	–	–	–	–	–	–	598	30	0.3	
2	67	104	588	99	–	–	–	–	–	–	–	–	596	30	1.1	
2	67	104	589	99	–	–	–	–	–	–	–	–	596	30	1.1	
3	68	106	587	98	–	–	–	–	–	–	–	–	599	30	0.8	
4	68	100	596	–	–	–	–	–	–	–	–	–	599	15	0.05	
5	67	98	598	–	–	–	–	–	–	–	–	–	599	5	0.06	
6	67	109	598	–	–	–	–	–	–	–	–	–	599	16	0.08	
7	68	106	587	97	–	–	–	–	–	–	–	–	589	30	1.1	
8	66	104	587	82	–	–	–	–	–	–	–	–	598	30	0.6	
9	66	108	588	94	–	–	–	–	–	–	–	–	595	6	1.2	
10	67	96	599	–	–	–	–	–	–	–	–	–	600	3	0.002	
11	69	93	599	–	–	–	–	–	–	–	–	–	600	3	0.002	

Note: the expanded uncertainty of the result of reaching the maximum temperature of flue gases is 6.0 s; the expanded uncertainty of the temperature measurement results is 0.2°C; the expanded uncertainty of the burning length measurement results is 0.01 mm. The tests were carried out using measuring instruments calibrated at certified metrological organizations that have proven traceability to international metrological standards.

Based on our studies, the composition for the mixture of ammonium polyphosphate and aluminum hydroxide was optimized for fire protection of tent fabric.

The following variation factors were selected: the amount of ammonium polyphosphate, g (factor  $X_1$ ); the amount of aluminum hydroxide, g (factor  $X_2$ ) – their changes are given in Table 9. As a result of modeling, regression equations were derived and ternary surfaces of changes in the initial parameter were constructed depending on changes in the variation factors (Fig. 12).

The amount of carbonized residue was chosen as the output parameter (response function), the value of which was recorded on samples subjected to thermal destruction. The experimental design matrix and the results of sample testing are given in Table 10.

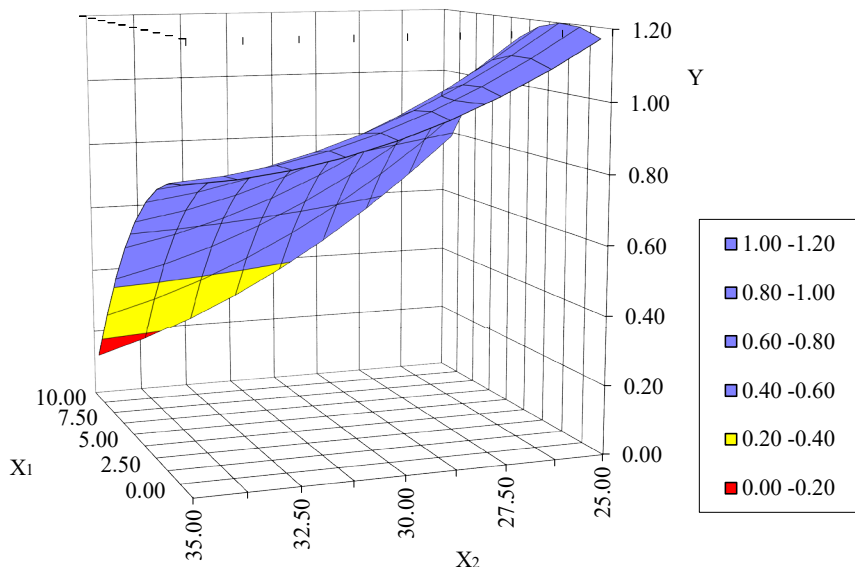


Fig. 12. Results of determining the optimal composition for a flame retardant in the fire-retardant coating applied to linen fabric:  $X_1$  – amount of ammonium polyphosphate, % (wt.),  $X_2$  – amount of aluminum hydroxide, % (wt.),  $Y$  – flammability index

Table 9

Variation factors

Factors	Code	Levels of variation			Variation interval
		-1	0	+1	
Amount of ammonium polyphosphate, % (wt.)	$X_1$	25	30	35	5
Amount of aluminum hydroxide, % (wt.)	$X_2$	0	5	10	5

Table 10

The experiment matrix and its implementation

No. of entry	Factors, form		Planning matrix		Response function	
	$X_1$	$X_2$	Amount of ammonium polyphosphate, % (wt.)	Aluminum hydroxide, % (wt.)	$Y_{actual}$	$Y_{calc.}$
1	1	1	35	10	0.1	0.14
2	1	-1	35	0	0.6	0.82
3	-1	1	25	10	0.9	0.79
4	-1	-1	25	0	1.1	1.17
5	1	0	35	5	0.9	0.64
6	-1	0	25	5	1.1	1.14
7	0	1	30	10	0.3	0.38
8	0	-1	30	0	1.2	0.91
9	0	0	30	5	0.5	0.81
10	0	0	30	5	0.5	0.81
11	0	0	30	5	0.8	0.81

Regression equation

$$Y_{calc.} = 0.811 - 0.250X_1 - 0.267X_2 + 0.083X_1X_1 - 0.167X_2X_2 - 0.075X_1X_2. \quad (3)$$

As a result, the optimal concentration of components in the coating containing 34 ÷ 35% (wt.) ammonium polyphosphate and 9 ÷ 10% (wt.) aluminum hydroxide was determined, which is capable of providing a minimum flammability index during fire protection of tent fabric with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide.

## 6. Discussion of results based on investigating regularities for the modernization of tent fabric with epoxy resin

By comparing studies on the energy characteristics of the surface of tent fabric (Fig. 3, 4; Table 5), it was found that distilled water when wetting the tent fabric forms an angle of about 48°, and when wetting with ethylene glycol forms an angle of about 76°. However, when wetting the tent fabric treated with epoxy resin with distilled water, an angle of 130° is formed, and for ethylene glycol – 45°, respectively. At the same time, as calculations showed, the dispersed component of the free energy of the fabric surface after treatment with epoxy resin decreased by more than 2.3 times. Note that the polar component increased by 1.6 times, which indicates a change in the surface. However, as shown by the test results for determining the tensile characteristics of tent fabrics (Fig. 5-7; Table 6), it is clear that the tensile strength of the treated tent fabric after treatment with epoxy resin increased by more than 4.1 times. Filling the epoxy resin with a flame retardant in a ratio of 1:1 reduced the tensile strength by only 7%.

When the burner thermally acted on a sample of tent fabric, as shown by the results in Fig. 8, 9 and Table 7, the sample ignited within 5 s and the subsequent residual flame combustion lasted for 16 s, which led to the combustion of the sample. In contrast, the treated samples of tent fabric did not ignite after 15 s of thermal action, the flame did not spread, but the length of the charred area for the sample of tent fabric treated with epoxy resin was more than 100 mm, and the mass loss was 25%. On the surface of the tent fabric sample, which was treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide, ignition and burning did not occur, charring over 70 mm long was formed on the surface,

and the height of the formed foam coke in the flame zone was about  $3 \div 5$  mm. At the same time, the mass loss was 8%.

The relevance of the results lies in the suppression of the combustion process of tent fabric treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide. These systems are characterized by the formation of a foam-coke layer on the fabric surface, which prevents ignition. Under thermal exposure, ammonium polyphosphate decomposes and acts as a catalyst, forming an inorganic acid that interacts with the hydrocarbon component and promotes the formation of a char layer. At the same time, aluminum hydroxide decomposes with the release of a significant amount of non-combustible vapors and gases, which facilitate the formation of high-temperature aluminum phosphate phases and stabilize the structure of the foam coke [19].

Analysis of the results of our studies given in Table 8 revealed that the sample treated with epoxy resin caught fire on 524 s, the flame spread throughout the sample for 14 s, the burning length of the sample was 300 mm, and the flammability index was 36.3. For samples treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide, the flammability index decreased by more than 30 times, but for them both ignition in the first section of the sample for no more than 10 s and flaming of the flame at the final stage of the test for  $1 \div 2$  s were recorded. This demonstrates the mechanism of action of the fire-protective coating, making it possible to identify this process.

Therefore, based on our review of the literature [1, 4, 7, 8], which are aimed at the formation of a fire-retardant coating, this paper reveals the mechanism of action of a mixture of ammonium polyphosphate and aluminum hydroxide. Namely, the decomposition of ammonium polyphosphate, which releases phosphoric acid, which induces the destruction of the biopolymer (fabric) and the dehydration of aluminum hydroxide with the formation of foam coke with the use of flame retardants, as well as their application to the surface of the tent fabric, which are well known.

However, in contrast to the results reported in [20] regarding the mechanism of fire protection of tent fabric, we can state the following:

- the basic process of inhibiting the thermal destruction of tent fabric is the formation of a heat-protective layer of coke on the surface of the textile material, which prevents the penetration of high temperature because aluminosilicate materials during thermal decomposition do not inhibit the flame and give off only water;

- there is an essential process of protecting the textile material, which is treated with a polymer resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide, which creates an elastic film on the surface of the tent fabric that is resistant to thermal action.

The determination of the high-temperature resistance of the fire-protected fabric is confirmed by experimental studies and substantiates its applicability under operating conditions, which constitutes an advantage of the present research. However, experimental data on determining the fire resistance of tent fabric are limited because the use of epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide for them requires an increased number of tests.

In addition, the combustion behavior of epoxy resin under thermal exposure has been investigated only to a limited extent, which restricts the scope of comparison. Another limitation is that part of the experimental data is presented

in graphical form; however, these results make it possible to reveal the role of the fire-protection system. Subsequent studies on the development of fire protection will refine the optimal composition of the coating.

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## 7. Conclusions

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1. Our studies have shown that distilled water, when wetting tent fabric, forms an angle of about  $48^\circ$ , and when wetting with ethylene glycol, forms an angle of about  $76^\circ$ . However, when wetting tent fabric treated with epoxy resin with distilled water, an angle of  $130^\circ$  is formed, and for ethylene glycol –  $45^\circ$ , respectively. At the same time, the dispersed component of the free energy of the surface of the fabric after treatment with epoxy resin decreased by more than 2.3 times. On the other hand, the polar component increased by 1.6 times, which indicates a change in the surface. Thus, the breaking load for an untreated sample of tent fabric was 0.58 kN. However, the breaking strength of the tent fabric after treatment with epoxy resin increased by more than 4.1 times. Filling epoxy resin with flame retardant in a 1:1 ratio reduced the tensile strength by only 7%.

2. As a result of our tests of the flammability of the fabric, it was found that when the thermal action of the burner on the tent fabric sample is 5 s, the sample ignited; and the subsequent residual flame combustion lasted for 16 s, which led to the combustion of the sample. In contrast, for the tent fabric sample treated with epoxy resin, when the thermal action of the burner is 15 s, ignition and burning did not occur, but the length of the charred area exceeded 100 mm, and the mass decreased by 21%. The tent fabric sample treated with epoxy resin did not ignite after 15 s of thermal action, charred at the site of the burner action, the flame did not spread, but the length of the charred area was over 100 mm, and the mass loss was 25%. On the surface of the tent fabric sample treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide, ignition and burning did not occur, charring with a length of more than 70 mm and a width of  $25 \div 32$  mm was formed on the surface, and the height of the formed foam coke in the flame zone was about  $3 \div 5$  mm. At the same time, the mass loss was 8%.

From the results of experiments to determine the flammability index, it was established that the tent fabric sample treated with epoxy resin caught fire in 524 s, the flame spread throughout the sample for 14 s, the burning length of the sample was 300 mm, and the flammability index was 36.3. For samples treated with epoxy resin filled with a mixture of ammonium polyphosphate and aluminum hydroxide, the flammability index decreased by more than 30 times; however, both ignition in the first section of the sample for no more than 10 s and flame flaring at the final stage of the test for  $1 \div 2$  s were recorded. The optimal concentration of components in the coating containing  $34 \div 35\%$  (wt.) ammonium polyphosphate and  $9 \div 10\%$  (wt.) aluminum hydroxide was determined, which is capable of providing a minimum flammability index for fire protection of tent fabric.

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## Conflicts of interest

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The authors declare that they have no conflicts of interest in relation to the current study, including financial, personal, authorship, or any other, that could affect the study and the results reported in this paper.

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**Data availability**


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All data are available in the main text of the manuscript.

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**Use of artificial intelligence**


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**Authors’ contributions**


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**Yuriy Tsapko:** Conceptualization, Methodology (supervisor) writing – review and editing (supervisor), Formal analysis (supervisor); **Aleksii Tsapko:** Conceptualization (assistant), Research – conducting experiments and data analysis, writing – original draft; **Oksana Berdnyk:** Research – conducting experiments and data analysis, Writing – original draft (assistant), Formal analysis; **Ruslan Likhniovsky:** Methodology (assistant), Data curation, Validation; **Tetiana Nehrii:** Resources, Verification, Funding; **Oksana Kasianova:** Resources, Supervision, Verification; **Yurii Feshchuk:** Validation, Financing, Visualization; **Vasyl Lomaha:** Raising funding, Verification, Visualization; **Olga Bedratiuk:** Investigation, Financing, Resources; **Kseniia Bielikova:** Resources, Supervision, Verification.

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