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DEVELOPMENT OF FIRE RESISTANT COATING FOR THE PROTECTION OF ELECTRICAL CABLES OF FIRE IN A CLOSED SPACE

Наводяться результати оптимізації складу вогнестійкого покриття для захисту кабельної ізоляції під час пожежі в замкнутому просторі. Компоненти покриття вибрані за вимогами вогнестійкості та токсичності. Обґрунтовано передбачуваний механізм роботи вогнестійкого покриття під час пожежі. Проведено лабораторні випробування зразків вогнестійких покриттів. Отримано часову залежність температури на зворотному боці вогнестійкого покриття від складу композиції.

Ключові слова: *нетоксичне вогнестійке інтумесцентне покриття для захисту кабельної ізоляції, компоненти складу, методика випробувань.*

1. Introduction

The development of fire-resistant materials and coatings in Ukraine is a strategic direction in the sphere of rational use and protection of material and fuel-energy resources. The relevance of the composition optimization of fire-resistant intumescent coatings is due to the fact that they are relatively thin-layered, do not emit a significant amount of toxic substances when heated, and have a high flame retardant efficiency. And also can be applied to the protected surface by different mechanized methods. The thickness and volume of fire-resistant coating increase tens of times at the action of high temperature due to the formation of a non-flammable and solid foamed layer (coke) with a density of $3\cdot10^{-3}$–$3\cdot10^{-2}$ g/cm^3 and a of thermal conductivity coefficient like thermal conductivity of air.

The fire-resistant coatings are especially effective in fires in confined spaces of engineering structures – ship rooms, nuclear and thermal power stations, at petrochemical enterprises and other potentially dangerous objects.

2. The object of research and its technological audit

The object of research is the composition of the fire-resistant swelling coating.

The main problem of electric cables fire protection in a confined space is a high concentration of toxic substances in the composition of combustion products, which makes it difficult fire extinguishing works. This is due to the fact that the cables polymer insulation (polyethylene, polyvinyl chloride) and traditional flame retardant coatings based on epoxy and phenolic resins contain potentially hazardous substances. These substances during combustion are form toxic compounds (chlorine, carbon monoxide, hydrogen cyanide).

Experimental research of different compositions the fire-resistant intumescent coatings on the basis of a potentially safe silicone resin was carried out to reveal the peculiarities operation of the coating during a fire.

On the initial stage of development of fire-resistive coating composition, «Pentelast 712 A» (or its analogue) the silicone resin, was chosen as a binder. This resin has good technological characteristics (low viscosity and hardening under temperatures of 20–30 °C) not toxic and water resistant. Its disadvantages are: the low mechanical (1.5–3 MPa) and adhesive strength (up to 0.3 MPa) which can be increased by primer (special base coat for better adhesion). For the fire-resistive coating swelling under the sharp temperature rise, the following mineral and polymeric fine divided fillers are proposed to introduce into the composition:

– alkaline earth metal hydroxides $Al(OH)_3$ и $Mg(OH)_2$ are thin white powder, water-insoluble, in most (under 0.12–0.16 %), under temperatures of 190–230 °C and 270–300 °C, respectively, decomposes chemically with water escape, absorbing the great amount of heat energy. This proves that $Al(OH)_3$ and $Mg(OH)_2$ to be the effective fire-retardant. $Al(OH)_3$ и $Mg(OH)_2$ are not toxic;

– pentaerythritol $C(CH_2OH)_4$ and Melamine $C_3H_6N_6$ are a synthetic products, water-insoluble (under 6 % and 0.5 %, respectively, at 20 °C). Temperature of chemical decomposition of pentaerythritol is 275 °C, Melamine is 354 °C. These are carbon-bearing and foaming components in the composition of fire-resistant coating necessary for coked cellular layer formation. Pentaerythritol and Melamine are low toxic products;

– ammonium polyphosphate $(NH_4PO_3)_n$ under $n>1000$ is a synthetic product, practically water-insoluble (0.25 %). Temperature of chemical decomposition is 270 °C. This is a gas-forming agent in the composition of fire-resistant coating. It is needed for coked cellular layer formation and as a source of phosphoric acid which prevents the combustion of carbon-bearing components. Ammonium polyphosphate is low toxic product.

Therefore, initially all the basic components of intumescent fire-resistant coating meet the requirements for heat resistance, low water absorption and low toxicity. Besides, the small size of fillers allows to create a coating thickness

300–500 μm, this will make a positive impact on technical and economic characteristics of fire-resistant coating.

Mechanism of work of the fire-resistant coating under the chosen composition will as follows:

On the initial stages of temperature rise (up to 190–200 °C) the fire-resistant coating will have no visually observed changes of material structure because of high heat resistance of silicone resin. Later, with the growth of temperature decomposition of $Al(OH)_3$, starts with water escape and partial fire-resistive coating foaming.

When temperature rises to 270 °C chemical processes involving decomposition of $(NH_4PO_3)_n$ and $C(CH_2OH)_4$, start with intensive gas escape and foaming of upper layers of fire-resistant coating. The coke layer starts to form.

Almost at once, under temperature of 290 °C the thermal decomposition of $Mg(OH)_2$, magnesium hydroxide, starts with water escape, additional foaming of the fire-resistant coating and heat release.

The newly formed MgO strengthens the coke layer. Under temperature of 350 °C melamine $C_3H_6N_6$, the last active component, decomposes. It is a film-forming agent which finally consolidates the coke layer of the intumescent fire-resistant coating.

It should be noted that under open flame impact temperature raises too fast, therefore all thermal and chemical process happen almost simultaneously, touching the thin outer layer of the fire-resistive coating. Due to foaming the thickness of fire-resistive coating increases in 20–30 times, forming the thick and incombustible coked cellular layer of low thermal conductivity; this layer secures the inner layers of the material. Therefore, usually there is no need in making the fire-resistive coating thicker than 1–2 mm.

Under fire condition the function of the active components of fire-resistant coating is carried out finely dispersed fillers and the silicone resin is thermally decomposed to silicon oxide and is chemically neutral.

It is of interest in this connection to optimize the composition of fire-resistant coating and maximize increase the filler content, but without compromising the technological characteristics of the composition.

3. The aim and objectives of research

The aim of research is optimization of the fire-resistant coating composition for increase in its fireproof properties. At the same time the fire-resistant coating has to be adapted for drawing on electric cables directly in situ of their installation in enclosed space.

To achieve this aim, it is necessary perform the following objectives:

1. To define influence of composition the fire-resistant coating on the basis of silicon resin, namely the ratio of the components of the intumescent complex – ammonium polyphosphate, pentaerythritol and melamine, on the fireproof properties.
2. To reduce the toxicity of both the components of fire-resistant coating and the products of its thermal destruction in case of fire.

4. Research of existing solutions of the problem

The fire-resistant coatings are multicomponent composite materials. At influence of a flame in process of temperature increasing the various physical and chemical processes are consistently flows in the intumescent fire-resistive coating. Various physical and chemical processes are consistently flow in the intumescent fire-resistive coating at influence of a flame in process of temperature increasing. A stable foam-coke layer with a low thermal conductivity is formed, as a result, which protects the structure from action of high temperatures a sometime. Intumescent technologies are the most effective means of fires protection [1].

In recent years the direction on creation of fire-resistant coating was created, which do not contain halides because volatile products of their thermal decomposition are toxic and corrosion-active substances [2].

The fire-resistant coatings based on mineral binders (cement, aluminosilicate) are relatively inexpensive, but have a large coefficient of temperature expansion and low water resistance, so they have high adhesion to various surfaces during operation [3].

Application of various polymers in the form of synthetic resins and their aqueous dispersions as binders of fire-resistant coatings allows them to significantly increase their water resistance and adhesion strength, to reduce the mass and thickness of the coatings. However, at the same time, the amount of toxic substances and smoke released during a fire increases, especially when epoxy resins are used in fire-resistant coatings [4]. Application of aqueous dispersions of polymers [5] reduces the water resistance of the fire-resistant coatings composition. Especially unfavorable conditions for the exploitation of coatings are the tropical climate that narrows geography and the sphere of their application.

Swelling of coatings perhaps in the presence in their composition intumescent complex and consist of ammonium polyphosphate, pentaerythritol and melamine. The degree of swelling depends on the relationship between these components, the type of binder and the presence of other functional fillers [6], and the thickness of the coating can be increased tens of times.

It is difficult to establish the mechanism of interaction between individual components of intumescent coatings, in view of the high rate of their heating during a fire. Various reactions can occur between the coating components, especially at high temperatures. It is difficult to predict the direction of high-temperature reactions.

Interaction between components of intumescent complex is usually reduced to their successive endothermic decomposition with the release of a large number of noncombustible gases – carbon dioxide, ammonia and water vapor, which carry off a significant amount of heat, and the solid phase of the products of chemical reactions forms a foam-coke.

The foam-coke is a solid porous structure with a low coefficient of thermal conductivity, which is able to protect the surface from the effects of the flame for some time. Efficiency of such protection is largely determined by the initial thickness of the coating and the swelling coefficient [7].

The components of the standard intumescent complex begin to swell at temperatures above 250 °C, while the temperature of beginnings of inflaming of wood and the softening of most polymers is less than 200 °C.

Therefore, mineral fillers which contain bound water are introduced in composition; these are alkaline earth metal hydroxides (aluminum, magnesium), which release water at temperatures of 190–270 °C and protect the surface at the initial stage of heating [8, 9]. Mineral fillers are non-toxic, effective and relatively inexpensive, which increases the interest for their using as flame retardants.

The composition of most developed fire-resistant coatings [10, 11] contains in various proportions the components of the intumescent complex and functional fillers, and as a binder either toxic resins or moisture resistant polymeric dispersions are used.

Non-toxic and water-resistant organosilicon resins are used infrequently [12], due to low adhesion strength, so it is promising to study the properties of fire-resistant coatings based on silicones.

5. Methods of research

5.1. Planning experiment. The basic composition for experimental samples of fire-resistant coating was chosen as it follows [13–18]; the constituents' content by weight varies in the range:

– silicone resin with curing agent by weight of fire-resistant coating – 30 %;
– fine fillers by weight of fire-resistant coating – 70 %;
– ammonium polyphosphate – 65±5 % by fine fillers weight;
– pentaerythritol and melamine taken as three-to-two – 25±5 % by fine fillers weight;
– magnesium hydroxide and aluminum trihydrate taken as one-to-one – 10±5 % by fine fillers weight.

For the manufacture of samples fire-resistant coating the experiment was planned with three variable parameters:

x_1 – ammonium polyphosphate $(NH_4PO_3)_n$ at $n>1000$ content;

x_2 – pentaerythritol $C(CH_2OH)_4$ and melamine $C_3H_6N_6$ as three-to-two content;

x_3 – magnesium hydroxide $Mg(OH)_2$ and aluminum trihydrate $Al(OH)_3$ as one-to-one content.

Plan-matrix of the experiment is listed in Table 1.

Table 1

Plan-matrix of the experiment for determining the optimal composition of fire-resistant intumescent coating

Number of experimental composition	Content of ammonium polyphosphate $(NH_4PO_3)_n$, %	Content of pentaerythritol $C(CH_2OH)_4$ and melamine $C_3H_6N_6$ (3:2), %	Content of magnesium hydroxide $Mg(OH)_2$ and aluminum trihydrate $Al(OH)_3$ (1:1), %
1.1	65	30	5
1.2		27.5	7.5
1.3		25	10
2.1	60	32.5	7.5
2.2		30	10
2.3		27.5	12.5
3.1	55	35	10
3.2		32.5	12.5
3.3		30	15

5.2. Method for determining the fireproof properties of coatings. The experimental samples consisted of steel plates 0.25×150×150 mm in size with a 1 mm thick of fire-resistant coating (Fig. 1).

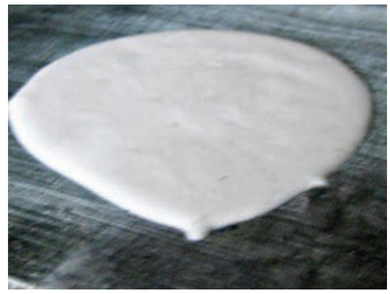
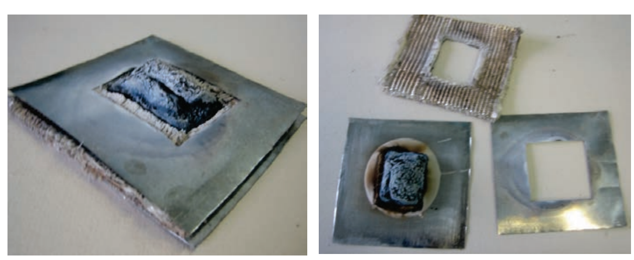


Fig. 1. Sample of fire-resistive coating

All samples of fire resistant coating were tested under the following conditions:

– the common gas burner was used as a fixed source of flame (temperature); its capacity is 1.9 kW and flame temperature is under 1350 °C;
– the experimental sample of fire resistant coating together with heat resistant glass-fabric of 4 mm thickness and steel plate of 0.25 mm thickness with overall dimensions are 150×150 mm was collected in a package. In the center of the heat resistant fabric and steel plate there was a square hole 70×70 mm for open flame access. The experimental samples of fire-resistive coating were covered by a layer of heat resistant fabric and steel plate (Fig. 2). This way stacked sample together with the temperature measuring unit was fixed in a frame and was vertically mounted at the laboratory stand;
– the temperature measuring (Fig. 3) unit is an outlet of 5 thermoelectric transducers 3 from the side not exposed to the flame;
– the temperature on the surface of the fire-resistant coating was monitored by one thermoelectric transducer 4 (type K). The readings of all thermoelectric sensors during the tests were registered in an automatic mode and processed electronically using a measuring complex (Fig. 3);
– the temperature on the surface of fire-resistant coating of the experimental samples was kept at the level of 600±30 °C, duration of testing was 15–16 minutes, at the mean.

This set of equipment allows to give a comparative evaluation of the fireproof properties coatings of various compositions.



a *b*

Fig. 2. Experimental sample after fire resistance testing: *a* – in a fixing frame; *b* – disassembled

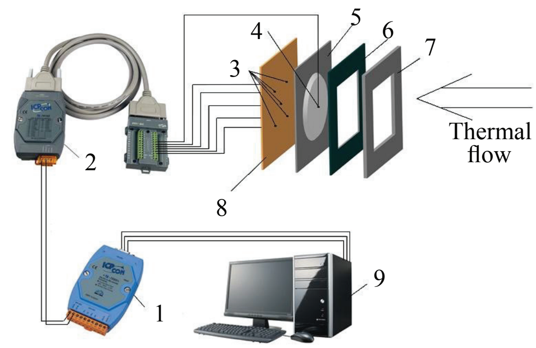


Fig. 3. Measuring complex:
1 – converter 17 520 A; 2 – external expansion module (EEM);
3 – temperature sensors T2...T6; 4 – temperature sensor T1;
5 – sheet with fire resistant coating; 6 – intermediate sheet;
7 – front protective sheet; 8 – sheet with temperature sensors;
9 – computer

6. Research results

The photo exposures of samples surface changes due to swelling when exposed to an open flame in the course of experiments were made (Fig. 4).

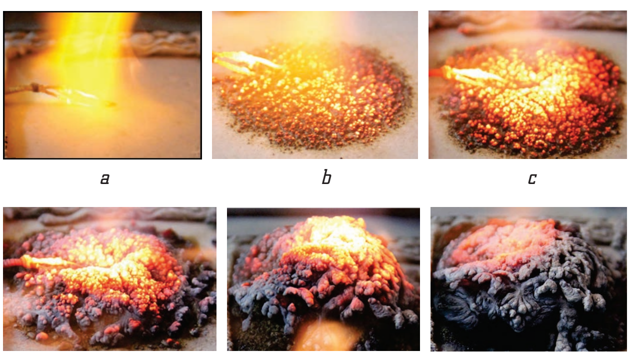


Fig. 4. Change of samples surface with a fire resistant coating when exposed to a flame during a time τ at a temperature on a back surface T:
a – τ=5 sec; T=26.3 °C; *b* – τ=50 sec; T=60.2 °C;
c – τ=300 sec; T=110.3 °C; *d* – τ=450 sec; T=127 °C;
e – τ=780 sec; T=135 °C; *f* – τ=900 sec; T=142 °C

The thickness changes (Fig. 5) and the swelling coefficients of fire-resistant coating (a relative increase in thickness) were established in the course of the experiments. The swelling coefficient was calculated by the formula:

$$K=\frac{t_f}{t_s}, \qquad (1)$$

where t_f – coating thickness after experiments, mm; t_s – coating thickness before experiments, mm.

The swelling coefficient of fire resistant coating for the sample batches is the range 29–52, at the mean (Table 2) and does not significantly depend on the composition.

Tests executed for 9 different compositions of fire-resistant coating (Table 1) and 5 samples for each composition. Statistical processing of experimental results was carried out.

The main function of the response is the temperature (°C) on a back surface of experimental samples. The averaged values of the temperatures for each composition are shown in Fig. 6.

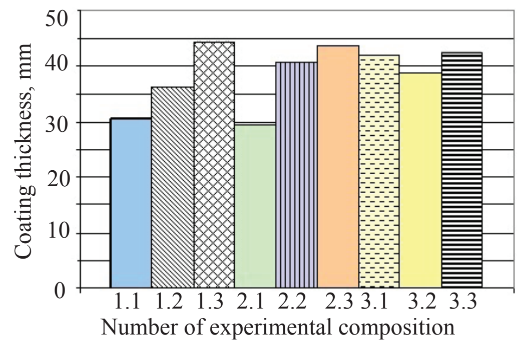


Fig. 5. The thickness of the fire-resistant coating of different compositions after the experiments

Table 2

Swelling coefficient for different compositions

Number of experimental composition	Swelling coefficient
1.1	29.1
1.2	38.9
1.3	42.2
2.1	31.2
2.2	39.2
2.3	38.4
3.1	51.9
3.2	39.2
3.3	46.6

The empirical response function T_{emp} is approximated by a linear relationship with respect to the content of components (parameters of variation) and supplemented with the work of factors to determine the optimal composition:

$$T_{emp}(b_0, b_1, b_2, b_{12}, x_1, x_2)= \\ = b_0 + b_1 \cdot x_1 + b_2 \cdot x_2 + b_{12} \cdot x_1 \cdot x_2, \qquad (2)$$

where b_0, b_1, b_2, b_{12} – are unknown empirical coefficients; x_1 – relative ammonium polyphosphate content; x_2 – relative pentaerythritol and melamine as three-to-two content.

The sum of squares deviations of empirical response function T_{emp} from the average temperature in experiments for each experimental composition was adopted as target function D:

$$D(b_0,b_1,b_2,b_{12},x_1,x_2)= \\ =\sum_{i=1}^{9}\left(T_{emp\,i}\cdot(b_0...x_2)-T_{exp\,i}\cdot(b_0...x_2)\right)^2. \qquad (3)$$

Minimization of the target function D was performed using the standard Minimize function in MathCAD 14 program.

As a result of minimization, the values of the empirical coefficients for (2) are obtained: b_0=535; b_1=–845; b_2=–1624; b_{12}=3275.

The correlation coefficient between the value calculated from the empirical formula (2) and the value of the temperature in the experiments is $K(T_{emp},T_{exp})$=0.966.

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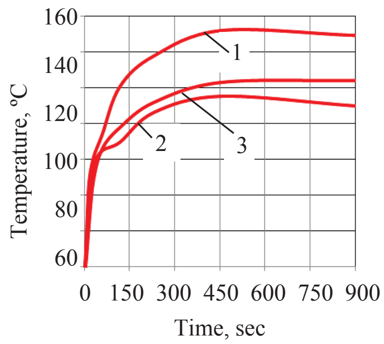
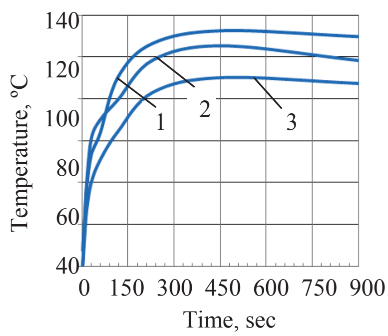


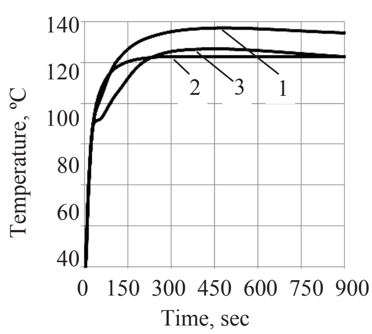
1 – Composition 1.1; 2 – Composition 1.2; 3 – Composition 1.3

a



1 – Composition 2.1; 2 – Composition 2.2; 3 – Composition 2.3

b



1 – Composition 3.1; 2 – Composition 3.2; 3 – Composition 3.1

c

Fig. 6. The dependence of temperature change on a back surface of the sample from the time: *a* – Composition 1; *b* – Composition 2; *c* – Composition 3

The graph of response function $T_{emp}(b_0, b_1, b_2, b_{12}, x_1, x_2)$ is shown in Fig. 7.

Experimental compositions of fire-resistant coating showed good fire-retardant properties. The temperature on a back surface of the sample during the tests did not exceed 160 °C at the mean, which is much lower than the temperature of destruction polymer insulation of electric wires.

The proposed simple test method makes it possible to give a comparative evaluation of the performance characteristics of fire resistant coating.

For the first 3–4 minutes of exposure to an open flame, the surface layers of fire resistant coating foamed, and the temperature on a back surface of sample different experimental compositions stabilized at 110–150 °C.

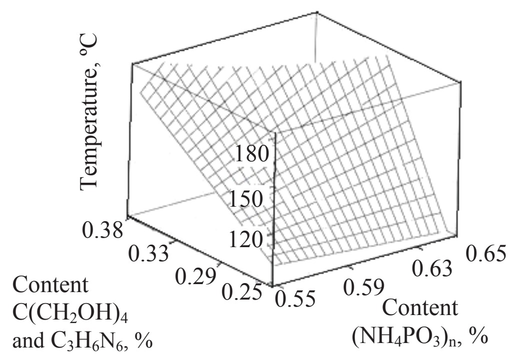


Fig. 7. The dependence of temperature on a back surface of the sample from content of composition

It is marked that temperature on a back surface of sample decrease with increasing content of ammonium polyphosphate (and correspondingly reducing the content of pentaerythritol and melamine), which is due to increased formation of phosphoric acids that act as flame retardants. At the same time, a decrease in the content of carbon-containing components led to a deterioration in the structure of coked cellular layer.

Aluminum and magnesium hydroxides actively remove heat at the first moment of heating the fire-resistant coating at 190–270 °C, but do not give a significant effect of swelling. Their content in the composition should not exceed 10 %.

Based on the results of experimental studies of fire resistant coating, dependence was obtained:

$$T(x_1, x_2) = \\ = (5.35 - 8.45 \cdot x_1 - 16.24 \cdot x_2 + 32.75 \cdot x_1 \cdot x_2) \cdot 100\ °\text{C}, \quad (4)$$

that adequately reflects the functional dependence of temperature on a back surface of the sample from content of composition.

The coefficient of correlation between empiric dependence (4) and the test results of samples of fire resistant coating was 0.966 that allowed defining the optimal content of composition.

Perspective composition of fire resistant coating contains: ammonium polyphosphate 65±2 %; pentaerythritol – 15±2 %; melamine – 10±2 %; aluminum and magnesium hydroxides – 5±1 % by fine fillers weight. This composition as a whole meets the requirements for fire resistance and toxicity.

7. SWOT analysis of research results

Strengths. Among strong parties of this research it is necessary to mark using for carrying out tests on the fire-resistance of open flame of gas burner, as compared to traditional tests in a heat chamber that anymore corresponds to the terms of the real fire.

In favor of this statement is the fact that laboratory tests in heat chamber can't recreate the conditions of a fire, as evidenced by an analysis of the modern world scientific periodicals on fire protection problems. More reliable test results can be obtained according to IEC 60332-3-10:2000, when tests are carried out in test chambers equipped with gas burners.

The properties of fire resistant coating in the experiments were investigated, using relatively optimal ranges of concentration of fire resistant coating components, which made it possible to solve the problem of choosing a rational composition.

On the one hand the temperature on a back surface of the samples and structure of the formed foam coke, on the other hand, were the criteria for choosing the optimal components.

Weaknesses. The weaknesses of this study are related to the fact that the flame temperature of the gas burner during the experiments was not constant, in view of the gas flow reduction when the gas filling cartridge was used. Therefore, it was necessary to regulate the supply of gas in manual mode. In other words, the role of the subjective factor in the strict reproduction of experimental conditions for various experimental compositions of fire resistant coating is increasing.

At the same time, it is well known that under significant temperature differences on the front side of the coating, the thermochemical reactions between the coating components can pass through different mechanisms, which can effect on the characteristics of the of fire-resistant coating.

As a result, an error arises in determining the real relationship between the concentrations of the components of the fire-resistant coating and the flame retardant properties.

Opportunities. Further studies can develop in the direction of modification a silicone base to increase the adhesion properties of fire-resistant coating, studying the interaction between its components and optimize the formulation, taking into account the introduction of refractory fillers, dispersing and plasticizing additives.

Threats. The difficulties in implementing the results of the research are related to two main factors.

The first of them – only one brand of silicone resin was used in experiments to determine the optimal composition of fire-resistant coating. It is well known that the mechanical and adhesive properties of silicone resins can vary over a wide range. Therefore, the results obtained in this study cannot automatically be extended to all of fire-resistant coating with silicon resin.

The second factor – silicon resins are more expensive, in comparison with traditional epoxy and phenol-formaldehyde resins. The using of fire-resistant coating with silicone resin can be justified for the protection of objects with an increased level of fire hazard.

Thus, SWOT analysis of research results allows to identify the main directions for the successful achievement of the research objective. Among them:

1. Modification of existing silicone resins to improve the adhesion properties of fire-resistant coating to the surface to be protected.

2. Improvements of the proposed method for testing coatings for fire resistance, which will stability, support the influence of fire conditions (temperature and gas flow) during the whole experiment.

8. Conclusions

1. Influence of composition the fire-resistant coating on the basis of organic silicon resin on fireproof properties was determined. The properties were estimated on temperature on back surface of experimental samples. It was established that the increasing of ammonium polyphosphate content from 55 to 65 % allows reducing the temperature on the on a back surface of the sample from 150 to 110 °C.

2. It was succeeded to reduce the toxicity of gases which generated by the action of an open flame on the fire-resistant coating to the limited allowable concentrations of 0.1 mg/m^3 of chlorine, 0.08–0.12 mg/l of carbon oxide (II), 0.3 mg/m^3 of hydrogen cyanide. This was achieved by using the silicon resin that does not contain chlorine and nitrogen as a binder in the fire-resistant coating composition.

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РАЗРАБОТКА ОГНЕСТОЙКОГО ПОКРЫТИЯ ДЛЯ ЗАЩИТЫ ЭЛЕКТРИЧЕСКИХ КАБЕЛЕЙ ПРИ ПОЖАРЕ В ЗАМКНУТОМ ПРОСТРАНСТВЕ

Приводятся результаты разработки и оптимизации состава огнестойкого покрытия для защиты кабельной изоляции при пожаре в замкнутом пространстве. Компоненты покрытия выбраны по требованиям огнестойкости и токсичности. Обоснован предполагаемый механизм работы огнестойкого покрытия при пожаре. Проведены лабораторные испытания образцов огнестойких покрытий. Получена временная зависимость температуры на обратной стороне огнестойкого покрытия от состава композиции.

Ключевые слова: нетоксичное огнестойкое интумесцентное покрытие для защиты кабельной изоляции, компоненты состава, методика испытаний.

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RESEARCH OF MINERALOGICAL COMPOSITION, STRUCTURE AND PROPERTIES OF THE SURFACE OF UKRAINIAN ASH MICROSPHERES

Досліджено властивості (мінералогічний склад, змочуваність, пористість, питому поверхню та її енергетичний стан) зольних мікросфер різних ТЕС України, що отримуються в результаті спалювання вугілля Донецького (Трипільська, Курахівська, Криворізька і Придніпровська ТЕС) та Львівсько-Волинського вугільних басейнів (Бурштинська ТЕС). Проаналізовано вплив властивостей поверхні зольних мікросфер на їх потенційну здатність використання в якості наповнювачів для будівельних матеріалів.

Ключові слова: *зольні мікросфери, питома поверхня, мінералогічний склад, порошкоподібний матеріал, аморфна фаза.*

1. Introduction

The development of industry, especially in recent years, requires the creation of new building materials with improved properties. First of all, these materials must have increased strength, heat resistance, reduced thermal conductivity, as well as a lower cost in comparison with analogues present in the construction market of Ukraine.

With the development of scientific and technical progress, systematic identification of factors determining the operational properties and cost of building materials and, as a consequence, the potential possibilities of their regulation become more and more urgent [1].

Significant interest in this direction is represented by ash microspheres. These are hollow ash pellets with an average size of 20 to 500 μm with solid, non-porous walls