










Geopolymer-Based Mineral Mixtures for Fire and Heating Protection of Concrete and Steel Products of Nuclear Power Plants

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Abstract. The article presents the results of the use of fireproof coatings and fire-insulating mixtures based on geopolymer to protect concrete and metal structures and products of nuclear power plants from fire and self-heating temperature from the heat release of radionuclides during their decay. According to the results of fire tests, it was determined that the treatment of the concrete surface with a fireproof geopolymer coating with thicknesses from 6 to 18 mm prevents critical heating of the surface of coated products (265.5–334 °C) to a critical temperature - 380 °C. It was determined that the temperature of concrete at the depth of reinforcement embedding (25 mm) warms up from 101.8 to 122.6 °C, which is 4.08–4.91 times less than the critical temperature of metal heating. It has been shown that 6 mm of coating is sufficient for fire protection of external concrete surfaces. And for interior concrete surfaces - 18 mm. The specified thickness of the geopolymer fire protection coating provides protection against the permissible thermal load (250 °C) on concrete from the heat release of radionuclides during their decay. The developed composition of the geopolymer coating provides a fire resistance class of concrete structures and products not lower than R180. The fire protection effectiveness of mineral mixtures on a geopolymer basis for the protection of metal structures and products has been determined. It is shown that at a thickness of 25 mm, the fireproof coating provides a fire resistance class of R90-R120 and group III of fire protection efficiency. Based on the calculation data in accordance with Eurocode 3, it is established that the coating thickness of 30 mm provides a fire resistance class of R120 and group II of fire protection efficiency. Below.

Keywords: Coating · Container · Concrete · Geopolymer binder · Fire protection · Fire test · Mineral mixture · Nuclear power plants · Steel structures · Temperature

1 Introduction

In today's reality, namely, military operations, there is a certain threat of fire at nuclear power plants as a result of rocket and artillery fire, fire failure of control equipment, etc. A fire can negatively affect the concrete and metal structures of plants and radioactive waste disposal containers, significantly reducing their load-bearing capacity and integrity. According to works [1, 2], the critical temperatures for concrete samples are 280 °C and for metal samples - 500 °C. As a result of exposure to elevated temperatures in concrete and reinforced concrete structures, there is a probable loss of bearing capacity due to deformations caused by asymmetric changes in the physical and mechanical properties of the material as a result of uneven heating in the cross-section of the bearing elements; reduction of the design height of the section due to heating of concrete and reinforced concrete to high temperatures; sliding of reinforcement along the support when the contact layer of concrete and reinforcement is heated to a critical temperature, etc.

In the case of concrete containers, as a result of heat generation during radioactive decay of radionuclides, the self-heating temperature can reach values from 400 to 700 °C (in some cases up to 1000 °C) [3]. As a result of such a temperature increase, due to an increase in the degree of radionuclide delocalization, their transition to a gaseous state with an increase in pressure inside the container, destruction of the cement matrix crystalloid structure due to radiolysis, its internal brittle destruction occurs. As a result, there is a high probability of leaching of radionuclides into the environment. Therefore, the permissible load temperature for concrete is 200–250 °C. This temperature limit helps to limit the internal dimensions of waste containers.

In the case of metal containers, depending on steel grades, the critical temperature values are 450–500 °C. At such temperatures, large plastic deformations occur, as a result of which the metal ceases to perceive the forces of external and internal loads. Given that during radioactive decomposition of radionuclides, the specific heat emission is above 0.1 W/m³ and the metal has high thermal conductivity, it is likely that heat and radioactive gases will be released into the external space with subsequent negative impact on this environment [4].

Therefore, it is important to design safety and fire protection systems for nuclear power plant structures and products for radioactive waste immobilization and disposal, taking into account various fire and heat release scenarios [5–8]. These measures are primarily aimed at preventing limit states of both concrete and reinforcement in it when exposed to fire and metal products.

Among the variety of fire and heat protection materials used in the aspect under consideration are coatings on cement [9, 10] and geopolymer bases [11, 12].

A common disadvantage of the proposed solutions is the increase in the additional distributed load on concrete and metal structures due to the increase in the mass and thickness of the material, as well as the low fire resistance limit, which does not exceed 1.5 h in a standard fire [13–15]. No such information is available on the fire and heat protection of containers.

The aim of this work is to determine the suitability and effectiveness of fire and thermal insulation mixtures based on geopolymer for the protection of concrete and

metal structures of nuclear power plants in case of fire, and containers for immobilization of radioactive waste from self-heating temperature during heat generation during radioactive decay of radionuclides.

This goal is achieved by solving the problems of fire and thermal insulation of these structures and products by conducting fire tests to assess their effectiveness.

2 Materials and Methods of Research

2.1 Materials

To achieve these goals, an aluminosilicate binder of the composition $\text{Na}_2\text{O} - \text{Al}_2\text{O}_3 - \text{SiO}_2 - \text{H}_2\text{O}$ was used in the form of a dispersion, with the ratio of structural oxides being $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 1.0$, $\text{SiO}_2/\text{Al}_2\text{O}_3 = 6.0$ and $\text{H}_2\text{O}/\text{Al}_2\text{O}_3 = 20$ (hereinafter – geopolymer), which was characterized by a density of 1.427 g/cm^3 , a dynamic viscosity of 1987 cP at 25 rpm and a surface tension of 46.9 mN m .

The calculation of the ratios between oxides in the geopolymer binder for synthesis of zeolites was carried out with taking into account the recommendations given in [16, 17].

Aluminosilicate granules based on zeolite-like geopolymer matrices of the above composition with a particle size of $0.63 \dots 4.0 \text{ mm}$ was also used as an intumescent filler. These granules were obtained by granulation of the geopolymer binder in CaCl_2 solution ($\rho = 1350 \text{ kg/m}^3$), the appearance of these granules is shown in Fig. 1 [18].

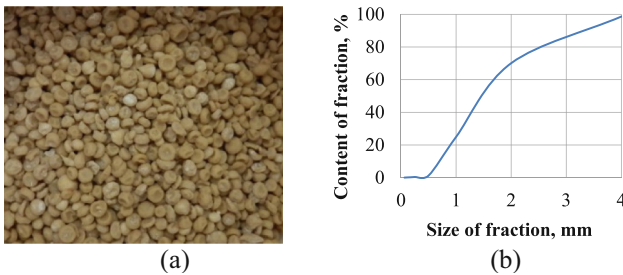


Fig.1. Appearance of the granules (a) and their distribution by fraction (b)

Metakaolin was used as a solid phase component of the binder. The composition of metakaolin, % by mass of mixture: $\text{CaO} - 0.52$, $\text{SiO}_2 - 53.67$, $\text{Al}_2\text{O}_3 - 43.61$, $\text{Fe}_2\text{O}_3 - 0.77$, $\text{MgO} - \text{traces}$, $\text{TiO}_2 - 0.74$, $\text{K}_2\text{O} - 0.75$, $\text{Na}_2\text{O} - 0.25$, other – 0.14, $\text{LOI} < 0.5$. The specific surface area of the ground metakaolin was $300 \dots 350 \text{ m}^2/\text{kg}$ (by Blaine).

Sodium silicate solution with a silicate modulus $M_s = 3.05$ and $\rho = 1420 \pm 10 \text{ kg/m}^3$ was used as an alkaline component.

Modification of the zeolite-like cement matrices has been done by adding NaOH , KOH and a rotten-stone (by mass, %: $\text{CaO} - 3.6$, $\text{SiO}_2 - 88.4$, $\text{Al}_2\text{O}_3 - 2.3$, $\text{Fe}_2\text{O}_3 - 1.1$, $\text{MgO} - 0.9$, $\text{TiO}_2 - 0.2$, $\text{K}_2\text{O} - 0.9$, other – 0.6, $\text{LOI} < 2.0$). The specific surface area of the ground rotten-stone is $250 \dots 280 \text{ m}^2/\text{kg}$ (by Blaine).

Limestone (by mass, %: CaO – 44.15, SiO₂ – 7.6, Al₂O₃ – 2.92, Fe₂O₃ – 2.64, MgO – 2.89, TiO₂ – 0.22, K₂O – 1.18, LOI < 39.71) was used as an intumescent and a filler [19, 20]. Its specific surface area after grinding is 70...80 m²/kg (by Blaine).

Perlite (by mass, %: SiO₂ – 70, TiO₂ – 0.3, Al₂O₃ – 12.5, Fe₂O₃ – 0.6, FeO – 0.95, CaO – 1.0, Na₂O – 3, K₂O – 2.8, H₂O – 5, LOI < 3.85, granules a size of 0.16–1.25 mm, density 2.30 - 2.39 g/cm³, porosity 1.8 - 70%) was used as a heating filler [20–22].

Organic thickeners and adhesion promoters based on polyacrylates were used to regulate the rheological characteristics of the mixtures and increase the adhesion to the substrates.

For fire protection of concrete products (containers) for radioactive waste disposal the rationally selected composition of the mixture consisting of geopolymer, geopolymer granules and limestone was applied.

For fire and heat protection of metal products (barrels) for radioactive waste disposal the rationally selected composition of the mixture consisting of geopolymer, geopolymer granules, limestone, perlite and polymer additives was applied. These compositions are the authors' know-how and are not disclosed in this paper.

2.2 Testing Methods

After mixing the geopolymer-based matrices with fillers in appropriate proportions, the resulting coating was applied by hand using a trowel to the vertical surface of concrete cube samples (150 mm) with a thickness of 6 mm at 18 mm. After the coating had cured, 2 TT-K-24-SLE type K thermocouples were installed, temperature range 20...1100 °C, accuracy ± 0.05 °C (Czech Republic). These three thermocouples (T_s, T_c and T_a) (Fig. 2), connected to a KIMO HD 200 HT multifunction device (France), were inserted into specially drilled holes in the concrete samples to a depth of 75 mm and at a distance of 25 mm of reinforcement. A DT 8867H infrared pyrometer (Germany) was used to measure the temperature (T_s) on the surface of uncoated and coated concrete specimens as well as in the depth of the concrete specimen (temperature field distribution). A gasoline burner with a flame temperature at the coating surface of not more than 1100 °C

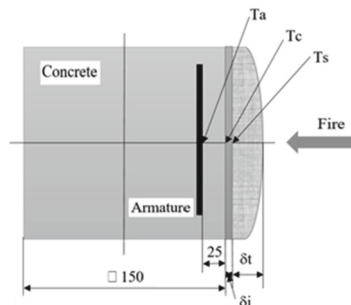


Fig. 2. Location of thermocouples in standard fire tests: T_s - temperature at the surface of the coating; T_c - temperature at the surface of the concrete under the coating; T_a - temperature of the concrete at the depth of laying the reinforcement

was used in the fire test (Fig. 2). The distance of the burner flame to the coating surface was 200 mm [18].

The surface heating temperature T_s , which corresponds to the standard fire temperature, was determined by the formula [18] and provided by changing the distance of the flame of the gasoline burner within 160...200 mm:

$$T = 20 + 345 \log_{10}(8t + 1) \quad (1)$$

where T is the flame temperature, °C; t is the duration of thermal exposure during the fire test, min.

For fire and heat protection of metal products (containers) dry and liquid components of the mixture were mixed with a construction mixer in a container for 3 min. Fire tests were carried out on a hot-rolled I-beam made of steel No. 20 GOST 8239–89 (Fig. 3).

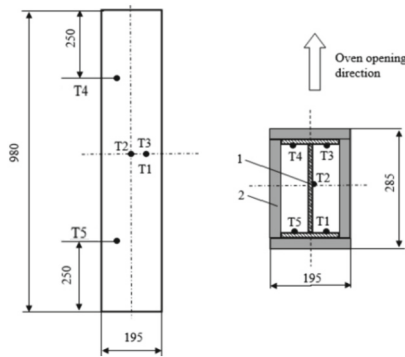


Fig. 3. Schematic representation of the insulated I-beam and of the layout of thermocouples set on it: 1 - I-beam; 2 fire-heat-insulating geopolymer coating; T1-T3 - the main thermocouples for the I-beam; T4-T5 - additional thermocouples for the I-beam.

The anti-corrosion treatment of the shelves and walls of the I-beam was carried out using the “Contrrust” converter (PE “Ruslan and Lyudmila”, Kyiv, Ukraine). After rust transformation, a thin layer of geopolymer suspension was applied to the chelated film with a brush to improve the adhesion of the fire-heat-insulating mixture to metal surfaces.

The fire resistance limits of steel structures protected from fire by materials for general construction purposes were determined based on the results of fire tests in accordance with national standards (DSTU B V.1.1-4-98* Fire protection. Building structures. Fire test methods. General requirements; DSTU B V. 1.1-14: 2007 Fire protection - Columns - Fire test method (EN 1365-4: 1999, NEQ)) and calculation methods in accordance with the European design line standards according to the Eurocodes: DSTU-N B EN 1993-1-2: 2010 Eurocode 3. “Design of steel structures. Part 1-2. Basic provisions. Calculation of structures for fire resistance” (EN1993-1-2: 2005, IDT); DSTU-N B EN 1991-1-2: 2010 Eurocode 1. “Actions on structures. Part 1-2. General actions. Actions on a structure during a fire” (EN 1991-1-2: 2002, IDT).

The calculation of the fire resistance limit of steel structures according to Eurocode 3, for which constructive protection methods (plastering) are used as fire protection, is carried out using the thermophysical characteristics of fire-retardant materials in the calculations. The calculation method is based on determining the temperature rise $\Delta\Theta_{g,t}$ over a period of time Δt for a uniform temperature distribution in the cross section of the protected steel structure:

$$\Theta_{a,t} = \frac{\lambda_p A_p / V (\Theta_{g,t} - \Theta_{a,t})}{d_p c_a \rho_a (1 + \phi/3)} \Delta t - (e^{\phi/10} - 1) \Delta \Theta_{g,t} \quad (2)$$

at $\Delta \Theta_{a,t} \geq 0$, if $\Delta \Theta_{g,t} > 0$,

$$\phi = \frac{C_p \rho_p}{C_a \rho_a} \cdot \frac{d_p A_p}{V}, \quad (3)$$

where: A_p/V – section coefficient of steel structures coated with fire retardant materials (m^{-1}); A_p – surface area of fire-retardant material per unit length, m^2 ; V – volume of structures per unit length, m^3 ; C_a – specific heat capacity of steel, $\text{J/kg}\cdot\text{K}$; C_p – specific heat capacity of a fire-retardant material, independent of temperature, $\text{J/kg}\cdot\text{K}$; d_p – fire retardant thickness, m ; t – time interval, while $\Delta t \leq 30$, s ; $\Theta_{a,t}$ – steel temperature at time t , $^{\circ}\text{C}$; $\Theta_{g,t}$ – temperature of the environment (nominal fire) at the moment of time t , $^{\circ}\text{C}$; $\Delta \Theta_{g,t}$ – increase in the temperature of the environment (nominal fire) at the moment of time Δt , $^{\circ}\text{C}$; ρ_a – steel density equal to 7850 , kg/m^3 ; λ_p – thermal conductivity coefficient of a fire retardant system, $\text{W/m}\cdot^{\circ}\text{C}$; ρ_p – density of fire-retardant material, kg/m^3 .

Equations for calculating the fire resistance limits of protected steel structures:

$$R = \left[C_1 \left(\frac{17W}{P_s} \right) + C_2 \right] d_p / 25.4 \quad (4)$$

where: R – fire resistance limit, min ; W – specific weight of a steel column, kg/m ; d_p – the thickness of the sprayed fire-retardant material, mm ; P – heated perimeter of the steel column, mm ; C_1 and C_2 – coefficients characterizing the thermal conductivity of the sprayed material. For geopolymer-perlite thermal insulation mixtures $C_1 = 33$ and $C_2 = 100$.

The main technological operations for applying the thermal insulation geopolymer-based mixture to the I-beam are shown in (Fig. 4).

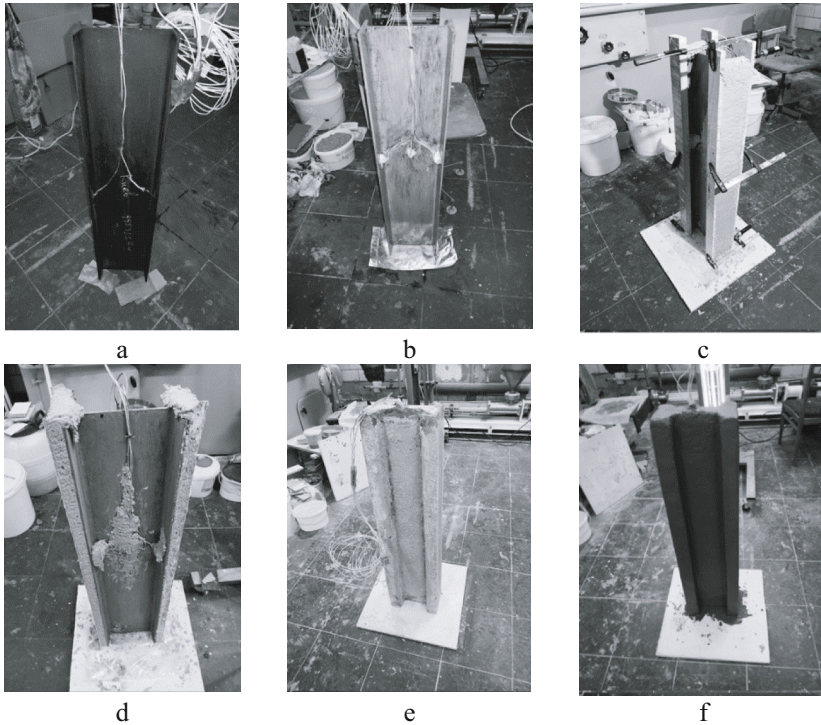


Fig. 4. Scheme of geopolymer mixtures application on an I-beam: a - treatment of the I-beam with Contrast converter; b - painting of the beam with geopolymer suspension to increase adhesion [23]; c-e - application along the rails with fire and thermal insulation mixture; f - painting the structure with mineral paint on geopolymer-base [24, 25], thickness 125 μm

3 Research Results

At the first stage of the fire tests, concrete samples protected by a geopolymer fire protection coating containing geopolymer granules and limestone were examined. The coating was applied to concrete surfaces with thicknesses of 6, 12, and 18 mm, respectively. The average temperature on the surface of the fire protection coating was 1100 °C during the entire fire test period. The temperature was recorded on the surface of the concrete under the fire protection coating (Fig. 5) and at the depth of reinforcement embedding (Fig. 6).

As can be seen from Fig. 5, the maximum temperature (T1) on the surface of concrete 1110 °C was recorded after 1 h 21 min of heating. This is 4.44 times higher than the temperature of the permissible thermal load on concrete from the heat release of radionuclides during their decay. After completion of the fire test, the concrete surface was characterized by melting and cracks with a width of 1.5 to 2 mm. The cracks were formed as a result of the release of physically bound water from the concrete structure and from the thermal destruction of the hydrosilicate phases of cement hydration products. At the depth of reinforcement embedding (25 mm) (Fig. 6), the maximum temperature

value was recorded - $267.8\text{ }^{\circ}\text{C}$ after concrete heating for 1 h and 52 min, which is 1.1 times higher than the permissible thermal load on concrete from heat release of radionuclides during their decomposition.

The temperature on the concrete surface (T2) under the 6 mm thick coating reached the maximum values - $334\text{ }^{\circ}\text{C}$ after 2 h and 32 min of fire tests (Fig. 5), which is 1.34 times lower than the critical temperature for unprotected concrete and 1.34 times higher than the temperature of the permissible thermal load on concrete from the heat release of radionuclides during their decomposition. It should be noted that in the time interval from 0 to 1 h and 16 min, the value of heating of the concrete surface under the coating did not exceed $250\text{ }^{\circ}\text{C}$.

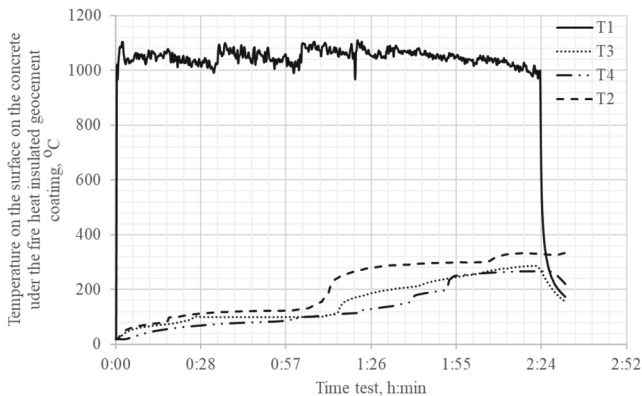


Fig. 5. Evolution of temperature on the concrete surfaces: T1 - temperature on the surface of uncoated concrete; T2 - temperature on the surface of concrete with a coating of 6 mm; T3 - temperature on the surface of concrete with a coating of 12 mm; T4 - temperature on the surface of concrete with a coating of 18 mm

At the depth of reinforcement embedding (25 mm) (Fig. 6), the maximum temperature T2 was recorded at $122.6\text{ }^{\circ}\text{C}$ after concrete heating for 2 h 32 min, which is 2.04 times less than the permissible thermal load on concrete from heat release of radionuclides during their decay.

A similar dependence is observed for the values of concrete temperature (T3) under a 12 mm thick coating (Fig. 5). At the depth of reinforcement embedding (25 mm) (Fig. 6), the maximum value of temperature T3 was recorded - $122.6\text{ }^{\circ}\text{C}$ after concrete heating for 2 h and 32 min, which is 2.04 times less than the permissible thermal load on concrete from heat release of radionuclides during their decay.

The temperature on the concrete surface (T4) under the 18 mm thick coating reached the maximum value of $265.5\text{ }^{\circ}\text{C}$ after 2 h and 21 min of fire tests (Fig. 5), which is 1.43 times lower than the critical temperature for unprotected concrete and 1.06 times higher than the temperature of the permissible thermal load on concrete from the heat release of radionuclides during their decomposition.

At the depth of reinforcement embedment (25 mm) (Fig. 6), the maximum temperature T4 was recorded at $101.8\text{ }^{\circ}\text{C}$ after concrete heating for 2 h and 32 min, which

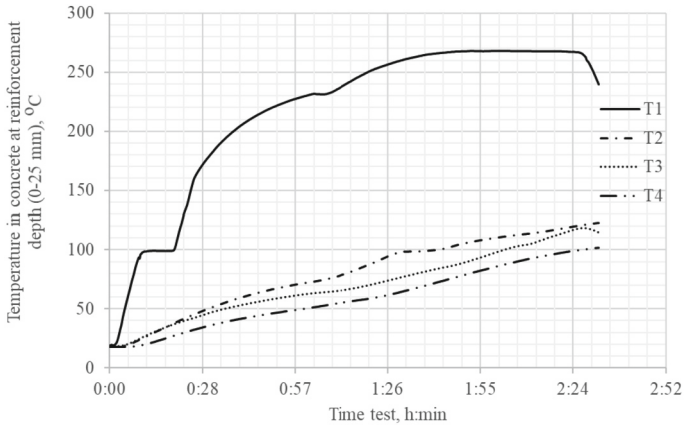


Fig. 6. Distribution of concrete temperature at the depth of reinforcement embedding (25 mm from the sample surface): T1 - uncoated concrete; T2 - 6 mm thick coated concrete; T3 - 12 mm thick coated concrete; T4 - 18 mm thick coated concrete

is 2.47 times less than the permissible thermal load on concrete from heat release of radionuclides during their decay.

The results obtained can be used for fire protection of concrete containers against both external temperature and internal self-heating of immobilized radioactive waste (Fig. 7).

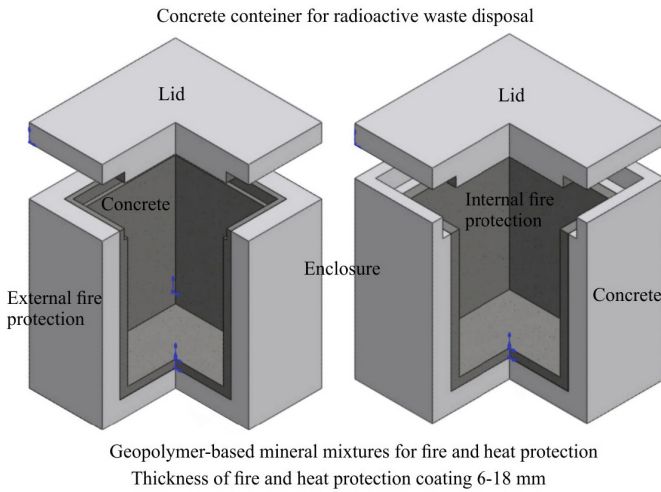


Fig. 7. Schemes of fire protection of external and internal surfaces of concrete containers

In Fig. 7 shows two options for fire protection of concrete surfaces against temperature effects with geopolymer-based fire protection coatings. Thus, in the case of external fire protection, it is recommended to apply a 6 mm thick coating to concrete surfaces; for

internal fire protection (in case of self-heating of radioactive waste), it is recommended to treat surfaces with a coating of 18 mm thickness. This coating thickness will not significantly reduce the active volume of radioactive waste.

At the second stage of the fire tests, a sample of the I-beam was examined, which was protected by a fire and heat-insulating geopolymer mixture containing, in addition to the geopolymer binder, geopolymer granules, limestone, perlite sand, and organic additives-modifiers. After mixing the components with a construction mixer, the mixture was manually applied to the shelves and walls of the 25 mm thick I-beam.

After hardening of the geopolymer-based mineral mixtures, the I-beam was placed in a furnace to undergo firing tests along a standard fire curve. In Fig. 8 shows a graph of the furnace temperature change. It should be noted that the temperature regime was maintained, no significant deviations from the standard temperature curve were recorded, which is confirmed by the data given in the work [1].

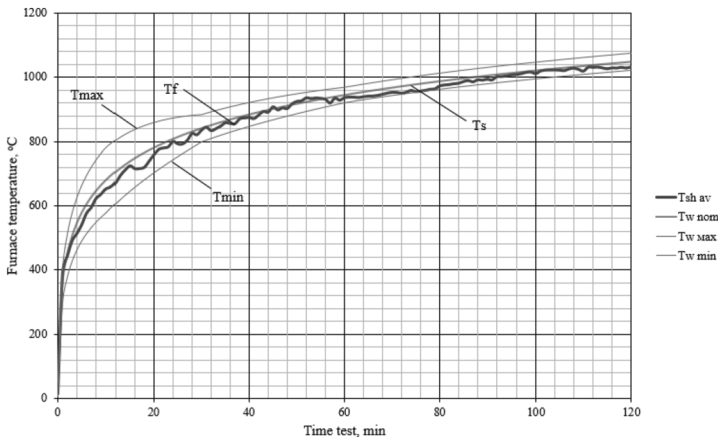


Fig. 8. Variation of the average furnace temperature (T_f) with time. Standard temperature regime (T_s), maximum temperature (T_{max}) and minimum temperature (T_{min}), are indicated.

In Fig. 9 shows a graph of the temperature distribution on the side flanges and the I-beam wall.

After the completion of the firing tests, a comparison was made between the experimental and calculated data given in the Table 1 and for the given parameters of the mixture.

Experiment data: reaching the critical temperature of heating the I-beam up to 500 °C with a fireproof coating thickness of 25 mm is carried out for 113 min of fire tests; the fire resistance class of the steel column ranges from R90 to R120.

Estimated data for Eurocode 3 and Eq. 3: reaching the critical temperature of heating the I-beam up to 538 °C with a fire-retardant coating thickness of 25 mm is carried out for 116 min; the fire resistance class of the steel column ranges from R90 to R120.

To ensure the fire resistance class R120, in the future, it is necessary to increase the thickness of the geopolymer-based mineral mixtures to 30 mm, which will increase the fire efficiency of the proposed fire-retardant material.

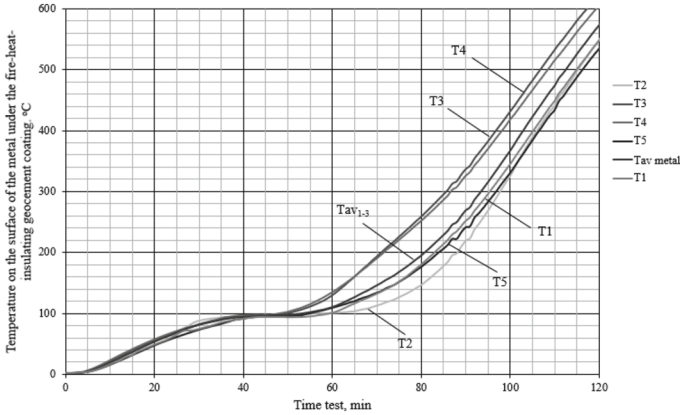


Fig. 9. Evolution of temperature on the metal surfaces of the I-beam: T1-T3 – the main thermocouples adapted on the I-beam; Tav1–3 – average values of thermocouples T1-T3; T4-T5 – additional thermocouples.

Table 1. Comparison of the minimum thicknesses of geocoement perlite containing thermal insulation mixture (d_{ρ} , mm)¹

Geopolymer-based mineral mixtures, $\rho=0.560$ [g/cm ³], $\lambda_{\rho}(\lambda_c)=0.1$ [W/m·°C] ² , $C_p=1130$ [J/kg·K]						
Profile section coefficient, A_m/V , [m ⁻¹]	Calculation method	Fire resistance class				
		R60	R90	R120	R150	R180
345-140	Eurocode 3	12-18	18-24	24-32	30-37	36-44
	Equation 3	12-16	18-22	24-29	30-35	34-41



¹Contour protection

²According to Equation 1, the thermal conductivity of a fire-retardant system is temperature dependent. The calculation took into account the data obtained during tests according to DSTU B V1.1.-17: 2007

The results obtained can be used for fire protection of metal containers against both external temperature and internal self-heating of immobilized radioactive waste (Fig. 10).

In Fig. 7 shows two options for fire protection of metal surfaces against temperature effects with geopolymer-based mineral the fire protection mixtures. Thus, in the case of external fire protection, it is recommended to apply a 25 mm thick coating to metal surfaces; for internal fire protection (in case of self-heating of radioactive waste), it is recommended to treat surfaces with a coating of 30 mm thickness. This mineral the fire protection mixtures thickness will not significantly reduce the active volume of radioactive waste.

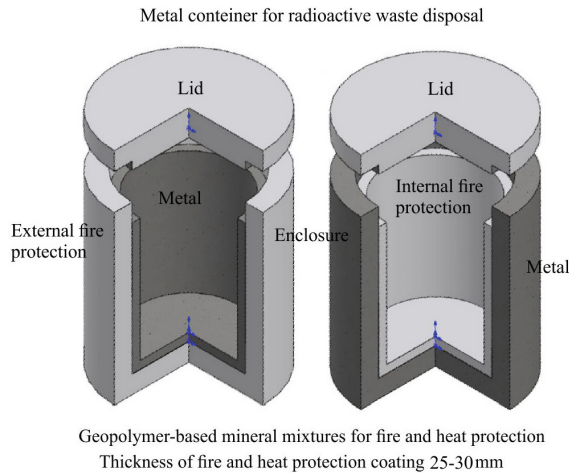


Fig. 10. Schemes of fire protection of external and internal surfaces of metal containers

4 Conclusions

According to the results of fire tests, it was determined that the treatment of concrete surfaces with a fire-retardant geopolymer coating with thicknesses from 6 to 18 mm prevents critical heating of the surface of the coated products (T_{fact} from 265.5 to 334 °C) to the critical temperature - 380 °C and prevents brittle destruction of its surface. It has been determined that the temperature of concrete at the depth of reinforcement embedding (25 mm) warms up from 101.8 to 122.6 °C, which is 4.08–4.91 times less than the critical temperature of metal heating. It was shown that 6 mm of coating is sufficient for fire protection of external concrete surfaces. And for internal concrete surfaces - 18 mm. The specified thickness of the geopolymer fire protection coating provides protection against the permissible thermal load (250 °C) on concrete from the heat release of radionuclides during their decay. The developed composition of the geopolymer coating provides a fire resistance class of concrete structures and products not lower than R180.

The use of geopolymer fire- and heat-insulating mixtures in the protection of metal structures from fire from both the outside and the temperature from inside metal containers provides a fire resistance class of R90-R120 and a fire protection efficiency group III. It is shown that with a fire-insulating coating thickness of 25 mm, the metal surface of an I-beam is heated to a critical temperature of 500 °C during 113 min of testing. Based on the calculation data in accordance with Eurocode 3, it was found that the critical heating temperature of an I-beam to 538 °C with a fire protection coating thickness of 25 mm is reached in 116 min, which ensures a fire resistance class of R90 - R120 and a fire protection efficiency group III. For fire protection of the outer surfaces of metal containers, 25 mm of coating thickness is sufficient, and for the inner surfaces, 30 mm of coating thickness is sufficient to ensure a fire resistance class of R120.

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