ISSN 1213–2489
e-ISSN 2787–9402

DOI: 10.21062/mft.2023.026 http://www.journalmt.com

Fire Resistance Test of Geopolymer Coatings on Non-Metallic Underlying Substrates

Filip Mamoń (0000-0002-5658-9534), Martin Jaskevič (0000-0003-3205-3177), Jakub Mareš (0000-0003-0272-6238), Jan Novotný (0000-0001-7676-8495)
Faculty of Mechanical Engineering, J. E. Purkyne University in Usti nad Labem. Pasteurova 3334/7, 400 01 Usti nad Labem. Czech Republic. E-mail: mamon@iic.cas.cz

This article summarizes the results from the fire resistance test of geopolymer suspensions in the form of coatings on non-metallic substrates. Their heat resistance was evaluated based on the burn-through time compared to the uncoated substrate. Non-metallic substrate materials, extruded polystyrene (XPS) and chipboard (DTD) were chosen as the underlying substrates for GP suspension research. Parameters from the fire resistance test as fire test duration of geopolymer coatings and percentage increase in burn time of geopolymer coatings on an XPS and DTD substrates compared to an uncoated substrate were evaluated. One of the discussed point was also the addition of $CaCO_3$ and $Al(OH)_3$ for studding flame retardancy effect.

Keywords: geopolymer, coatings, fire resistance test, non-metallic substrate, flame retardancy

1 Introduction

Fire-resistant coatings are one of the solutions to protect materials structures from a fire. The loss of mechanical properties during a fire can cause catastrophic consequences [1, 2]. At present, fire events that are caused by the ignition or explosion of hydrocarbon based fuels in the built environment are common. These events induce a rapid rise in temperature from the flashover state to more than 1100 °C within a few minutes [3]. This can be helped by geopolymer coatings based on alkaline silicates, which create a protective barrier and thus increase the protective time for building evacuations [4]. The great potential of geopolymer as fire-resistant and thermal insulators captures the increased researcher's interest. Geopolymers can be applied in constructions, automotive industry as bulk material, and as coatings. The chemical composition of the designed geopolymers depends on the desired application [5]. Geopolymer coatings can be applied to various surfaces, such as wood, polymers, metals or concrete [6-9]. Their main advantages of using alkaline silicate coatings as passive fire protection are less environmental concerns, resistance to microorganisms and UV radiation. Geopolymer based coatings have inherently better fire resistance compared to Portland cement and organic polymer-based systems. The fire resistance of cement coatings is controlled by a number of factors. Geopolymer coatings are essentially inorganic based and are considered non-flammable and do not emit any toxic fumes when exposed to fire [10]. To ensure the good structural performance of geopolymers subject to high-temperature heating, geopolymer must perform well at micro-scale (i.e., the stability of chemical structure), at meso-scale (i.e., resistance to deformation) and macro-scale (i.e., strength endurance and spalling resistance) [11]. Other publications state that physical examinations of the degree of cracking, spalling, brittleness, and loss of strength of geopolymers upon exposure to high temperatures and during fires provide an indicator of their resilience to such conditions [12]. Compared to Portland cement-based coatings, geopolymers retain a significant level of structural stability after fire exposure and show little spalling, which is attributed to an interconnected pore system that allows the easy passage of volatiles, mainly water, through the geopolymer structure when a temperature gradient is applied [10]. These coatings can be used both outdoors and indoors, but due to their sensitivity to carbon dioxide and moisture, indoor applications are more common [7]. When heated, silicate coatings swell due to the release of water vapor and form a porous layer that can prevent the spread of fire [10]. As a very interesting part of the fire resistance geopolymer suspensions research is the creating mixtures of geopolymer suspensions with $CaCO_3$ and $Al(OH)_3$ solutions as an active component for flame retardancy. $Al(OH)_3$ is more expensive than $CaCO_3$, but due to its good workability $Al(OH)_3$ finds more applications e.g. additive to plastic rubber in wire, cable, insulators, copper clad laminate, or acrylic boards [13].

2 Materials and methods

The fire resistance of geopolymer suspensions for non-metallic substrates was analyzed on extruded polystyrene (XPS) and chipboard (DTD) substrates. The test was carried out using a propane-butane burner, where the inlet pressure was appropriately chosen to achieve the ideal burner performance according to the used underlying substrate. The flame was directed perpendicular to the substrate to the center of the sample. The mouth of the burner was placed at a certain constant distance from the surface of the substrate. After igniting and placing the sample in the rack, the time it took for the sample to burn completely and for the flame to penetrate through the sample was measured. Due to the different properties of the underlying substrates, different input conditions were chosen for the ideal test procedure. For both analyzes (XPS and DTD substrate), the same burner with a diameter of 14 mm and a maximum power of 1.7 kW was kept. The initial conditions that affect the course of the test depending on the selected underlying substrate were changed by changing the distance of the burner from the surface of the substrate and by changing the gas inlet pressure.

2.1 XPS underlying substrate

Extruded polystyrene FIBRAN XPS ETICS GF-I (EN 13164) [14] with a thickness of 50 mm was chosen for the fire resistance analyses. FIBRAN XPS ETICS GF-I is a thermal insulation board made of extruded polystyrene with a roughened surface (waffle) for good application of mortar and adhesives. The board has straight edges. Thermal insulation provides perfect protection in humid environments and under higher mechanical loads. Mainly used as protection of exterior walls, insulation of facade plinths, window and door sashes, corners or as interior thermal insulation of wall surfaces.

2.2 DTD underlying substrate

Raw chipboard (DTD) with a thickness of 10 mm comply with the ČSN EN 312 (P2) [15]. DTD substrate was chosen as another non-metallic underlying substrate for fire resistance analyses. Raw chipboards are non-load-bearing boards designed for use in dry interior conditions with high dimensional and shape stability. They show a low degree of swelling in thickness and are easy to work with common woodworking tools. The surface is smooth, suitable for further surface treatment (lamination, veneering, etc.) The same degree of strength in all directions. The boards are pressed from the wood material of coniferous and deciduous trees, which are connected with a high-quality and harmless to health urea-formaldehyde resin. They are natural boards, where the fine drawing of wood chips is visible. The wood chips are thermally pressed and the chips are stored in three layers in all directions. Chipboards are only used in dry areas (indoors). The chipboard is sanded and has a low amount of releasable formaldehyde (E1).

2.3 Geopolymer suspensions

Two types of geopolymers (GP) suspensions were selected for the non-metallic underlying substrates. Due to the nature and use of this material, the research on the non-metallic substrate will be focused on the fire protection of polystyrene and chipboard using GP suspensions. Geopolymers B and E have the same basic composition, as do geopolymers C and F. Suspensions E and F also contain $CaCO_3$ and $Al(OH)_3$ as an active component for flame retardancy shown in the table 1. Thus, B and C serve as comparative samples to compare the flame retardancy effects of the active ingredients. Furthermore, by mixing suspensions E and F in a 1:1 weight ratio, suspension E+F was created to verify the functionality of the suspension, which contains both active components, where one component is activated at a temperature above 200 °C and the other is activated at a temperature above 700 °C.

Tab. 1 *GP suspensions for non-metallic underlying substrates*

Designation GP suspension	**Active folder**
B	-
C	-
E	$CaCO_3$
F	$Al(OH)_3$
E+F (1:1)	$CaCO_3 + Al(OH)_3$

3 Results

3.1 Fire test of XPS substrate

Since the XPS material is very flammable and its burning occurs after only a few seconds, a larger distance of the burner from the surface was chosen and at the same time the inlet pressure was reduced, to reduce the burner performance and flame temperature.

Tab. 2 *The following input conditions were chosen for the fire analysis of geopolymer coatings on the underlying XPS substrate.*

Substrate	**XPS**
Burner diameter, mm	14
Propane butane inlet pressure, bar	1.5
Distance of the burner from the substrate, mm	100

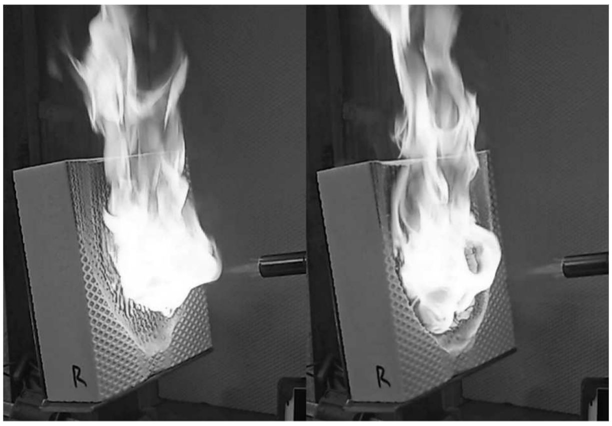


Fig. 1 *XPS (R) underlying substrate at the 5th second of the test and at the time of burnout*

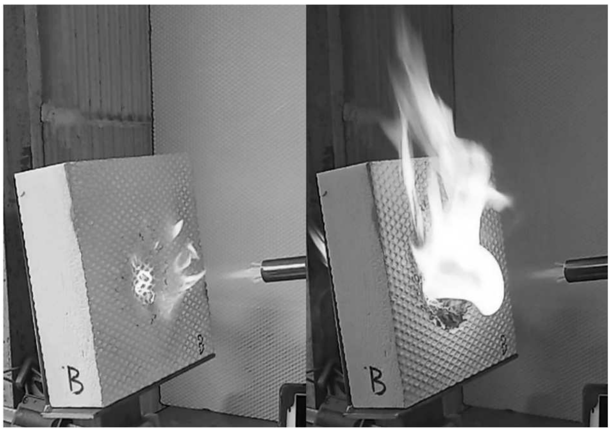


Fig. 2 *GP substance B at the 5th second of the test and at the time of burnout on XPS substrate*

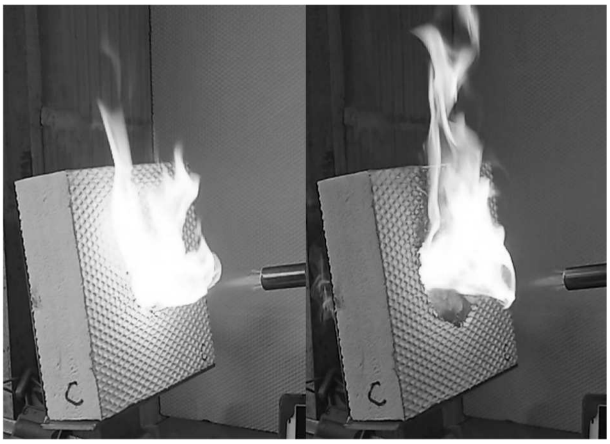


Fig. 3 *GP substance C at the 5th second of the test and at the time of burnout on XPS substrate*

From the course of the individual tests, the course of the burning and the extent of damage can be seen for the XPS substrate without coating and individual GP coatings. In the case of a substrate without a coating, there is an almost immediate ignition both in the volume and, above all, in the area where the flame of the torch falls and a large destruction of the sample occurs. For samples with GP coatings, a small circular area of the GP coating always collapses after a certain time, where the flame falls, and only then does the fire spread in the volume of the sample. However, there is no spread of fire over the surface of the substrate and the extent of damage is always lower for samples with GP coatings.

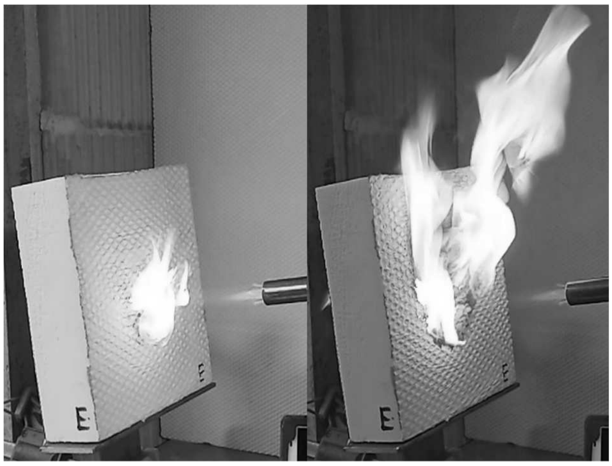


Fig. 4 *GP substance E at the 5th second of the test and at the time of burnout on XPS substrate*

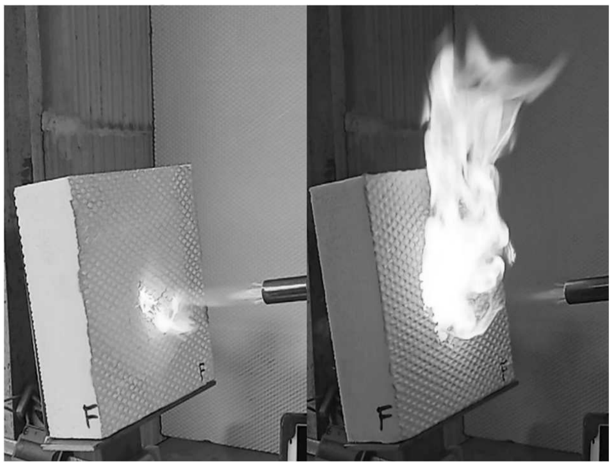


Fig. 5 *GP substance F at the 5th second of the test and at the time of burnout on XPS substrate*

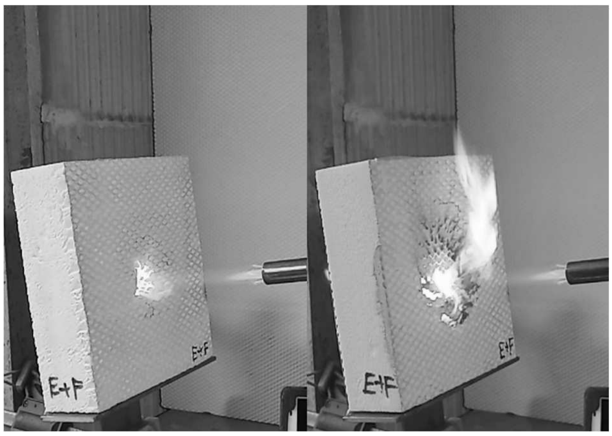
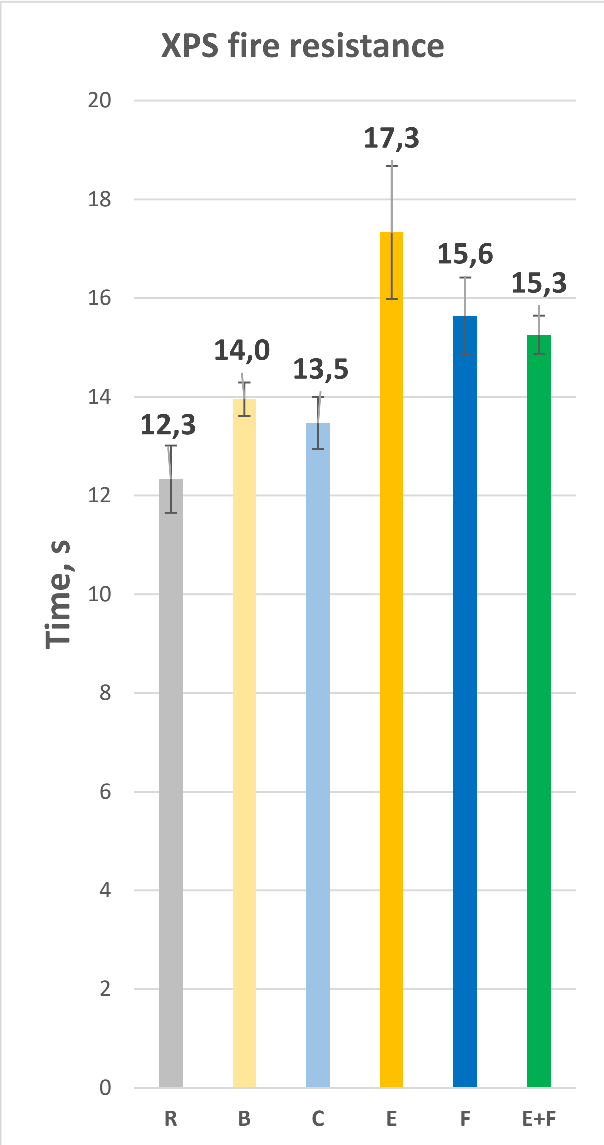


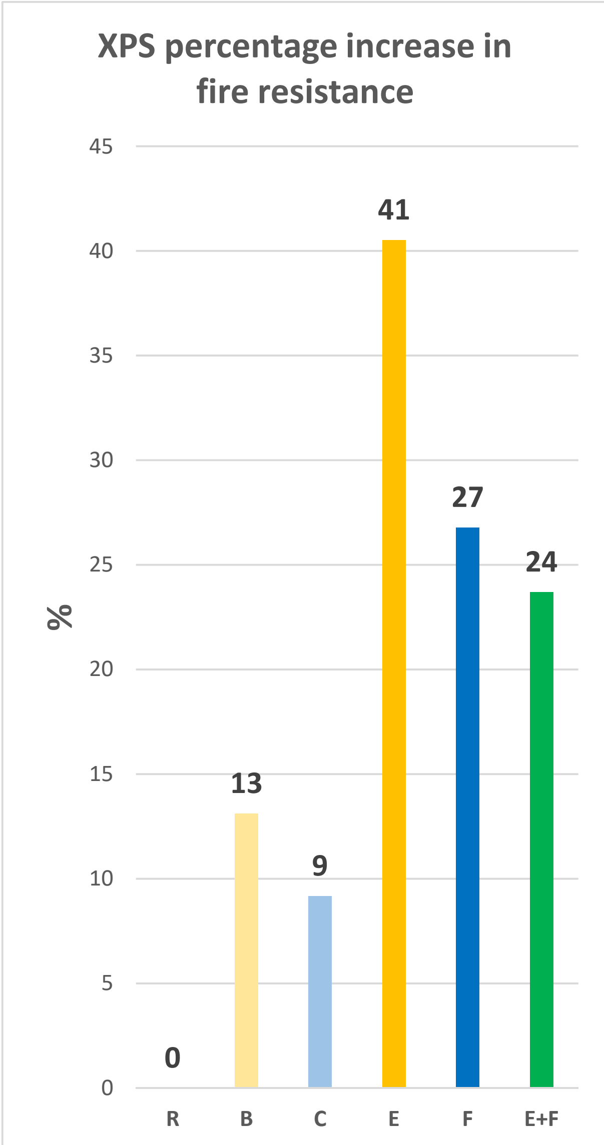
Fig. 6 *GP substance E+F at the 5th second of the test and at the time of burnout on XPS substrate*

Tab. 3 *Fire test duration of GP coatings (burn-through) on XPS substrate and percentage increase compared to uncoated substrate (R)*

Substrate XPS	Designation of geopolymer suspension						
n	**R**	**B**	**C**	**E**	**F**	**E+F**	**units**
1	12.6	13.5	13.2	19.0	14.7	15.8	s
2	13.0	14.1	13.0	17.3	16.6	14.9	s
3	11.4	14.3	14.2	15.7	15.6	15.1	s
Time average	12.3	14.0	13.5	17.3	15.6	15.3	s
Standard deviation	0.7	0.3	0.5	1.3	0.8	0.4	s
Percentage increase	0	13	9	41	27	24	%



Graph 1 *Fire test duration of GP coatings (burn-through) on XPS substrate and uncoated substrate (R)*



Graph 2 *Percentage increase in burn time of GP coatings on an XPS substrate compared to an uncoated substrate*

The fire test duration graphs show the times when the XPS substrate will burn out and the percentage increase in burn time compared to the uncoated substrate. The sample without GP coating burned in 12.3 s. Higher fire resistance was achieved by suspensions B (14.0 s) and C (13.5 s) without active ingredient, an increase of 13 and 9%, i.e. very similar results. The best result was achieved by suspension E (17.3 s) with the active component $CaCO_3$ (activation above 700 °C), and that is an increase in burn-in time by 41%. This was followed by suspension F (15.6 s) with the active component $Al(OH)_3$ (activation above 200 °C) with an increase in burn-in time by 27%. By mixing suspensions E and F, an improvement of 24% (15.3 s) was achieved. It is evident from the results that in the case of XPS coatings, the most appropriate use is GP suspension with $CaCO_3$ admixture. The suspension with $Al(OH)_3$ admixture also achieved an improvement in fire resistance, but to a lesser extent. The progress of the test is recorded in the images below. Due to the great damage to the XPS substrate after burnout and the continuation of burning after the end of the test, I recorded the progress of the test every 5 seconds from the start of the test (first image) and the moment when burnout occurred (2nd image).

3.2 Fire test of DTD substrate

DTD material is not as flammable as XPS and burn-through occurs only after a few minutes. In this case, the power of the burner was increased by increasing the gas inlet pressure and at the same time the distance of the burner from the surface of the substrate was reduced.

Tab. 4 *The following input conditions were chosen for the fire analysis of geopolymer coatings on the DTD underlying substrate.*

Substrate	**DTD**
Burner diameter, mm	14
Propane butane inlet pressure, bar	2.0
Distance of the burner from the substrate, mm	60

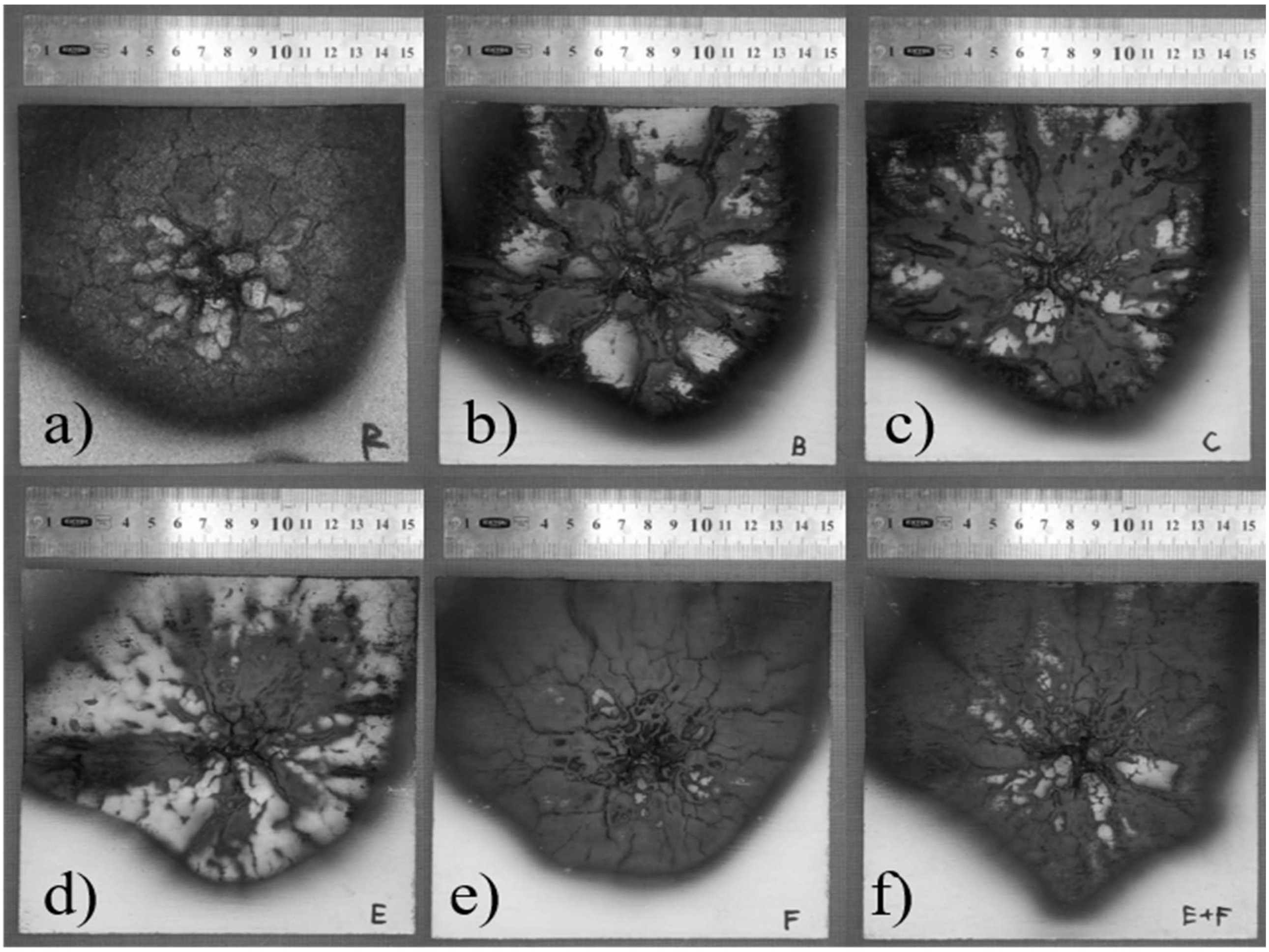
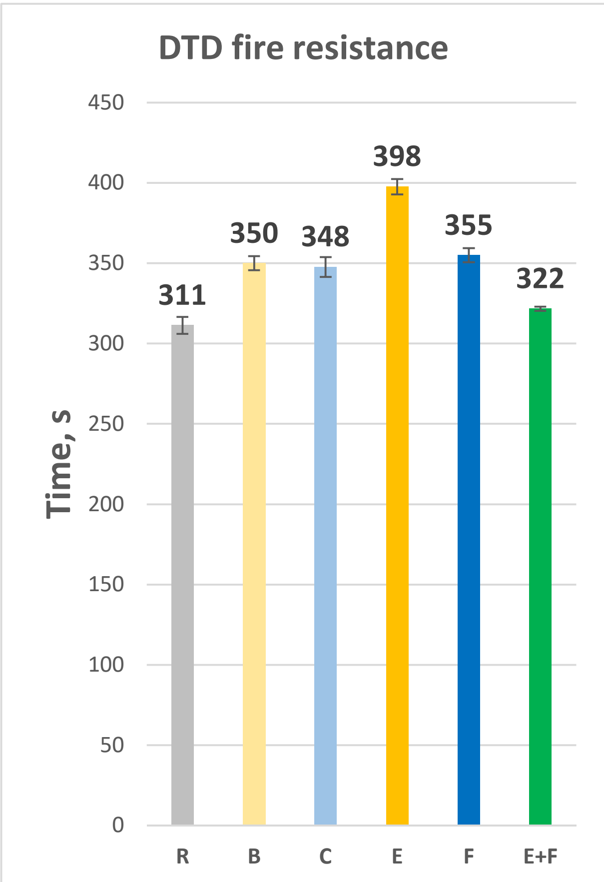


Fig. 7 *Details of DTD substrate in the burn trough moment of samples a) DTD without GP coating, b) DTD with coating of GP substance B, c) DTD with coating of GP substance C, d) DTD with coating of GP substance E, e) DTD with coating of GP substance F, f) DTD with coating of GP substance E+F*

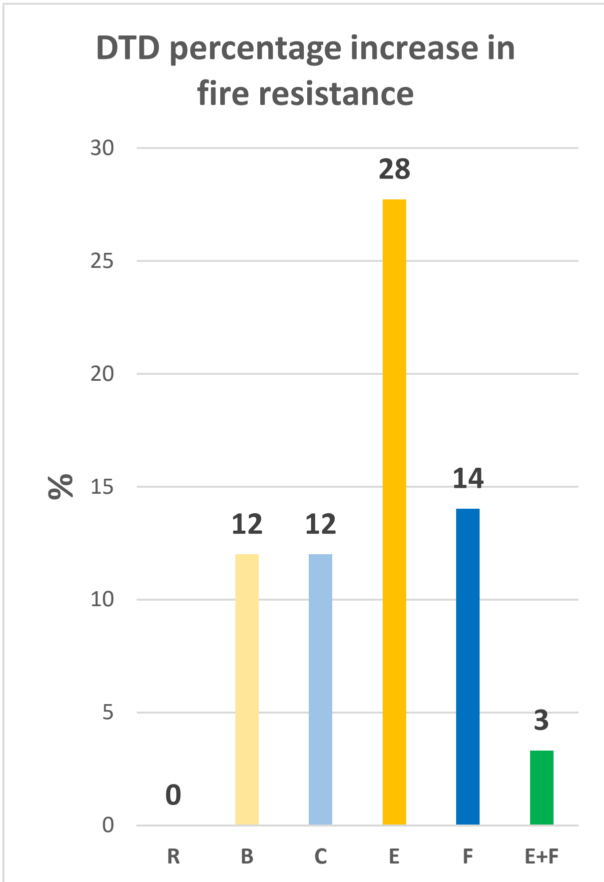
From the course of the individual tests, the course of burning and the extent of damage can be seen for the uncoated DTD substrate and individual GP coatings. On the uncoated substrate, visible surface damage occurs over a larger area than on samples with GP coatings that protect the surrounding surface. Here too, similarly to the XPS substrate, the circular surface of the GP coating is first destroyed, where the flame falls, and then the flame penetrates the substrate. The circular area after burning through the flame is always smaller for the substrate with GP coatings, and especially in the area on the surface of the sample that is protected by the GP coating, there will be lower damage caused by the flame and the prevention of fire spread on the surface. The total damage to the sample with GP coatings is thus always lower.

Tab. 5 *Fire test duration of GP coatings (burn-through) on DTD substrate and percentage increase compared to uncoated substrate (R)*

Substrate DTD	**Designation of geopolymer suspension**						
n	**R**	**B**	**C**	**E**	**F**	**E+F**	**units**
1	311	356	356	402	359	323	with
2	318	348	341	400	349	320	with
3	305	346	346	391	357	322	with
Time average	311	350	348	398	355	322	with
Standard deviation	5	4	6	5	4	1	with
Percentage increase	0	12	12	28	14	3	%



Graph 3 *Fire test duration of GP coatings (burn-through) on DTD substrate and uncoated substrate (R)*



Graph 4 *Percentage increase in burn time of GP coatings on DTD substrate compared to uncoated substrate*

The fire test duration graphs show the times when the DTD substrate will burn through and the percentage increase in burn time compared to the uncoated substrate. The trend of increasing the fire resistance of GP suspensions on the DTD substrate almost copies the trend observed on the XPS substrate. The sample without GP coating burned out in 311 s. Suspensions B and C show an identical increase in burn-out time, an improvement of 12% (350 and 348 s). The best fire protection was again achieved by suspension E (398 s) with $CaCO_3$ with an increase of 28%. Suspension F with $Al(OH)_3$ also achieved an increase in burn-in time (355 s), i.e. an increase of 14%, but the increase is not as striking compared to the basic suspension C without active ingredient, as in the case of the XPS substrate. The lowest increase in burn-in time was achieved by the E+F suspension mixture (322 s), an improvement of only 3%. This result does not correlate with the result obtained with the XPS substrate and more thorough testing of this mixture would be required. Here, too, it is evident from the results that the most suitable admixture of GP suspensions for fire-fighting purposes is $CaCO_3$, followed by the admixture $Al(OH)_3$, which is also apparently suitable for protection at lower temperatures (apparently the layer will be quickly destroyed by high temperatures and the protective function will be lost). The progress of the test is recorded in the images below. The progress of the test was recorded at the 2nd minute (1st picture), 4th minute (2nd picture) and at the moment of burnout (3rd picture). Furthermore, an image of the burned-out sample after the end of the test to compare the damage (4th image).

4 Conclusions

Used geopolymer suspensions is covered by the patent [16]. Thys suspensions is usable for insoluble heat and corrosion resistant coatings. Our previous research also shown that the selected geopolymer suspensions adhesion was at a very high level [17]. The fire resistance of geopolymer suspensions for non-metallic substrates was analyzed on XPS and DTD substrates. The resistance of substrates without coatings and with coatings with the active component $CaCO_3$ (activation above 700 °C) and $Al(OH)_3$ (activation above 200 °C) was investigated. The results show that geopolymer coatings with an active component significantly protect the underlying substrate against burnout. Suspension E with the active component $CaCO_3$ achieved the best result on the underlying XPS substrate, where the burnout time increased by 41%. Suspension F with the active ingredient $Al(OH)_3$ showed an increase of burnout time by 27%. The trend of increasing the fire resistance of GP suspensions on the DTD substrate almost copies the trend observed for the XPS substrate. The best fire protection was again achieved by suspension E with $CaCO_3$, with an increase in burnout time by 28%. Suspension F with $Al(OH)_3$ achieved an increase in burnout time by 14%, but the increase is not as significant compared to basic suspension C without active ingredient.

Acknowledgement

This research was supported by U21 Q-GRANT MSMT No. CZ.02.2.69/0.0/0.0/19_073/0016947.

References

[1] PURI, R.G.; KHANNA, A.S. (2016). Intumescent coatings: A review on recent progress. *J. Coat. Technol. Res.* 14, pp. 1–20

[2] LUCHERINI, A.; MALUK, C. (2019). Intumescent coatings used for the fire-safe design of steel structures: A review. *J. Constr. Steel Res.* 162, 105712

[3] DAS, A.K. (2020). Principles of Fire Safety Engineering: Understanding Fire and Fire Protection. *PHI Learning Private Limited: Delhi, India*

[4] BEH, J.H.; YEW, M.C.; SAW, L.H.; YEW, M.K. (2020). Fire Resistance and Mechanical Properties of Intumescent Coating Using Novel BioAsh for Steel. *Coatings*, 10, 1117

[5] AZIZ, I.H., AL BAKRI ABDULLAH, M.M., YONG, H.C., MING, L.Y., HUSSIN, K., SURLEVA, A., AZIMI, E.A. (2016). Manufacturing parameters influencing fire resistance of geopolymers: A review. Proceedings of the Institution of Mechanical Engineers, Part L. *Journal of Materials: Design and Applications*, 233

[6] KAZMINA, O.; LEBEDEVA, E.; MITINA, N.; KUZMENKO, A. (2018). Fire-proof silicate coatings with magnesium-containing fire retardant. *J. Coat. Technol. Res.* 15, pp. 543–554

[7] CHEN, S.N.; LI, P.-K.; HSIEH, T.H.; HO, K.S.; HONG, Y.M. (2021). Enhancements on Flame Resistance by Inorganic Silicate-Based Intumescent Coating Materials. *Materials*, 14, 6628

[8] BRICIN D., KRIZ A., NOVOTNY J., SPIRIT Z. (2022). The Effect of Boriding And Heat Treatment on the Structure and Properties of 100Cr6 Steel. *Manufacturing Technology,* 22, pp. 2-9

[9] HREN I., MICHNA S., NOVOTNY J., MICHNOVA L. (2021) Comprehensive analysis of the coated component from a FORD engine. *Manufacturing Technology*, 21, pp. 464-470

[10] VICKERS, L.; VAN RIESSEN, A.; RICKARD, W.D.A. (2015). Fire-Resistant Geopolymers: Role of Fibres and Fillers to Enhance Thermal Properties. *Springer*, ISSN 2192-1105

[11] LAHOTI, M., TAN, K. H., YANG, E.-H. (2019). A critical review of geopolymer properties for structural fire-resistance applications. *Construction and Building Materials*, 221, pp. 514–526

[12] AMRAN, M., HUANG, S.S., DEBBARMA, S., RASHID, R.S.M. (2022). Fire resistance of geopolymer concrete: A critical review. *Construction and Building Materials,* 324

[13] SAPUTRA, A.H., SUNGKAR, F. (2017). Synthesis and characterization of non-halogen fire retardant composite through combination of epoxy resin, $Al(OH)_3$ additive and filler. *In Proceedings of the American Institute of Physics Conference, Proceedings of the 3rd International Symposium on Applied Chemistry*, Jakarta, Indonesia, pp. 23–24

[14] FIBRAN XPS ETICS GF-I (EN 13164), Extruded polystyrene

[15] ČSN EN 312 (P2), Raw chipboard

[16] MICHNA, S., NOVOTNY, J., JASKEVIC, M., CERNY, Z., ROSYPAL, P. (2022). PV 2021-103. Geopolymer suspension and preparing it. University J. E. Purkyne in Usti nad Labem

[17] NOVOTNY, J., JASKEVIC, M., MAMON, F., MARES, J., HORKY, R., HOUSKA, P. (2022) Manufacture and Characterization of Geopolymer Coatings Deposited from Suspensions on Aluminium Substrates. *MDPI Coatings,* 12, 1695