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Original Article

Influence of the Manufacturing Method on the Fire Resistance of Geopolymer Materials Based on Mining Slag

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Abstract - *Developing geopolymer materials based on waste is being promoted as an approach to reduce landfilling and encourage a circular economy. In this regard, high-performance geopolymers based on mining slag are developed for fire protection products, where the manufacturing method could have an influence. Accordingly, this paper assesses the fire resistance performance of two geopolymer products based on the same slag but produced considering two different manufacturing processes (precast and 3D printed), mainly focused on their use for tunnels. Furthermore, it studies other fire resistance evaluation methods (laboratory tests at different scales, in-situ tests, and computer based simulations), identifying their suitability for product development or research phases. On the one hand, results show that the production method affects the fire resistance performance since tested geopolymers reveal different thermal transmittance and mechanical behavior in prolonged or extreme fire exposure due to the diverse nature of the geopolymer material itself the first one is ductile material while the second a brittle material. In this sense, the 3D printed material shows a better thermal performance, but this can be significantly affected by the fastening configuration used. On the other hand, a step-by-step methodology based on the combination of the different fire resistance evaluation methods is presented to facilitate the product assessment during the various product development stages and for different system configurations or end-use applications.*

Keywords - *Production methods, Geopolymer products, Fire performance, Waste valorization, 3D printing.*

1. Introduction

Implementing a circular economy model in the European Union requires an elevated level of commitment from companies to create collaborative and inter-company solutions that promote eco-innovation. In this sense, work is being done on developing geopolymer materials based on waste for construction products, [1] such as transforming mining slag into geopolymer products, [2] or recycling these same materials.

An approach opens new horizons for the extractive and processing industries in the raw materials sector in terms of minimizing landfill costs and valorizing waste into value-added products. In turn, the infrastructure sector can source more sustainable materials, optimizing the life cycle of its products and reducing their environmental impact. However, these materials must meet or respond to specific performance requirements, such as fire protection, and market demands, such as installation times and costs. Geopolymers' competitive fire resistance performance with recycled fibers has already been proven. [3] Beyond this, the technological parameters influencing the thermal behavior or fire resistance properties of geopolymer-based materials have also been studied, and possible applications have been defined depending on their composition. [4] Geopolymer-sprayed concrete, for example, has been proposed as a fire protection coating for tunnel linings. [5] Geopolymers can be suitable for various uses and sectors, such as tunnel linings, protection of concrete structures, or service installations.

Still, the manufacturing process and the related specific requirements will also directly affect the final applications of a geopolymer. In this regard, the 3D printing method has already been compared with the conventional casting method in terms of environmental impact, concluding that the environmental benefits of using waste materials might be diminished for the 3D printing due to the higher activator content used and, hence, its potential increases with the level of building complexity. It is more suitable for non-repetitive freeform products. [6] Accordingly, 3D printed geopolymers at a small scale or product scale and a building scale have already been presented [7, 8], and methodologies have already been proposed for formulating geopolymer-based materials for the requirements and demands of commercially available printers. [9] Accordingly, geopolymers have been used as binder systems in binder jet-based additive manufacturing by reacting with the reactive component (metakaolin) in the powder bed. At the same time, the liquid reactant (alkaline solution) is deposited through nozzles [10].

Nevertheless, a research gap has been identified related to the fire resistance performance of the products created from different production methods since the influence of the manufacturing process in this aspect has not yet been assessed. This paper evaluates the fire resistance performance of geopolymer products produced with different manufacturing methods to comprehensively respond to the market using standard or customized products according to market needs. For that purpose, new geopolymer products were created using two different technologies in this research: prefabricated molds (precast) as conventional technology that lays the foundations for comparison with competing standard products and 3D printing as an innovative approach to additive manufacturing as an ad-hoc means of production for customized products.

Fire resistance is the ability of a constructive system or a structural element to contain or resist a fully developed fire. Provides an idea of the time you may have to evacuate the building or to protect your assets or belongings before the total or partial collapse of the structure. However, the fire resistance evaluation method will depend on the purpose of the assessment or evaluation since either small-scale tests, full-scale tests both in laboratory or in situ and/or computer-based simulations can be used for the mentioned purpose. Laboratory tests can be carried out at different scales depending on the target: small scale to compare formulations and define fire scenarios that will be linked to the final use of the product; experimental scale to compare different products (manufacturing processes or product characteristics) and real scale to check the installation methods or different configurations and determine final fire resistance performance for product validation/certification.

The latter are mainly based on European standards according to the selected end use and fire scenario, [11, 12] but specific protocols are also available for particular applications, such as tunnel uses. [13] Several types of small-scale fire resistance tests have already been reviewed for tunnel linings considering the method used, the fire scenario selected, the specimen size, or the specimen or the external loading of the specimen, [14] which may considerably affect the test result. Experimental tests have also been used to validate the behavior of new or recycled lining materials in a tunnel fire scenario. [15] Finally, large-scale concrete tunnel lining fire tests have been recently researched to evaluate concrete spalling, highlighting the importance of replicating the end-use loading condition of the tunnel (surrounding soil and/or water pressure). [16] In this regard, different fire-temperature curves have been defined to simulate the fire scenarios that could occur in a tunnel (Figure 1). The ISO 834 fire curve, or the cellulosic curve, is based on the burning rate of materials found in general building components and contents, and it is used as a standard fire scenario for building uses. It is also used for tunnels with limited traffic to cars and/or vans but does not depict burning petrol or chemicals. [17, 18] Accordingly, when higher fire resistance levels are required (e.g., all vehicles are allowed), different thermal exposures are used depending on the country as fire scenarios, such as HCM in France, RWS in the Netherlands, or RABT in Germany.

The Hydrocarbon (HC) fire curve is applicable where small petroleum fires might occur considering a maximum temperature of 1100˚C, while the Modified Hydrocarbon (HCM) increased this temperature to 1300˚C due to a possible rapid ascension of temperature in a tunnel fire. The Rijkswaterstatt (RWS) fire curve assumes that in a worst-case scenario, a 50 m³ fuel, oil, or petrol tanker fire with a fire load of 300 MW could occur, lasting up to 120 minutes and reaching a maximum temperature of 1350˚C.

Finally, the Richtlinien für die Ausstattung und den Betrieb von Straßentunneln (RABT) curves include a rapid rise of temperature, up to 1200˚C within 5 minutes, but with a shorter maximum temperature duration than in other curves, with a temperature drop off starting to occur at 30 minutes for highway and 60 minutes for railway tunnels, and a cooling period of 110 minutes.

In situ fire resistance tests validate the fire resistance performance of existing constructions or infrastructures and protection elements. There have been already performed for tunnel linings based on the large scale of the EFNARC´s Specifications and Guidelines for Testing of Passive Fire Protection for Concrete Tunnels Lining [19] or on the updated Efectis R0695 Fire Testing Procedure for Concrete Tunnel Linings and Other Tunnel Components, which includes a specific section with a test protocol for mobile furnace tests to perform in-situ fire resistance tests [13].

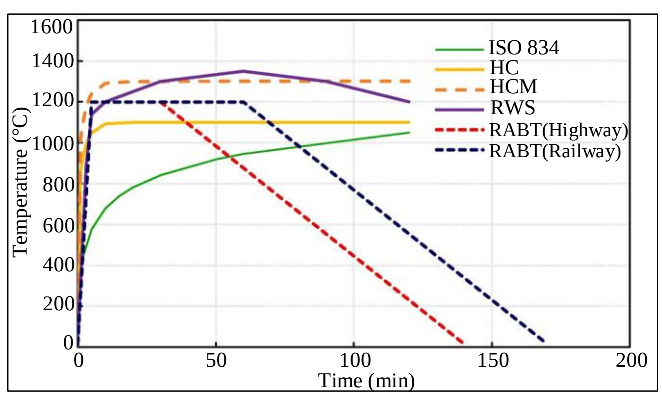


Fig. 1 Standard fire curves [17]

Computer simulations can complement fire resistance tests to extend the field of application of the test results obtained. Accordingly, the use of computer simulations to assess the thermomechanical behavior of structures has been developed, for example, to predict temperatures in a product or in a specific element that could limit the structure's performance in case of fire.

[20] In this regard, the evaluation of the thermo-mechanical response of concrete tunnel linings, considering different evaluation approaches towards numerical simulation, has already been studied [17], where commercially available software has already been implemented. [21] Additionally, simulations have already been used to simulate specific phenomena, such as spalling, considering the concrete as damaged or spalled above a selected critical temperature. [22, 23]

Therefore, this article studies, on the one hand, the fire resistance performance of geopolymer materials that are based on the same mining slag considering the manufacturing process (precast and 3D printing) to respond to the lack of knowledge about the influence of the manufacturing method of a geopolymer in its fire resistance performance. On the other hand, it compares and evaluates different fire resistance assessment methods (laboratory tests at different scales, in-situ tests, and computer simulations) to identify the most appropriate method for each product development step or end-use application, adapting them to the needs of each case, mainly focused on their use for tunnels.

2. Materials and Methods

This research evaluates the fire resistance performance of two geopolymer products based on copper slag but manufactured with different technologies. One is produced by the precast manufacturing method, a conventional technology that uses mobile modular production units.

In contrast, the second product uses additive manufacturing technology (3D printing), an innovative technology for ad-hoc production. Both products are manufactured in 40 mm thick panels. They are installed using nail anchors (FNA II 6 x 30/50 RB Fischer) as direct fixations to concrete with a spacing of 40 cm between nails and 10 cm from nail-to-board edges and with no protection materials between panel joints.

Different fire resistance evaluation methods are proposed to evaluate the products' development and validation process, each adapted to each phase's needs. Accordingly, different evaluation tools are presented at different scales, mainly fire resistance tests in laboratories at different scales, but also in-situ fire resistance tests and computer simulations. Fire resistance tests are performed according to existing standards, depending on the end use or fire scenario selected.

The following tests are proposed:

- Medium-scale laboratory tests in an experimental furnace according to the 2020-Efectis-R0695 fire testing procedure for concrete tunnel linings and other tunnel components [13] and following the ISO 834 fire curve or the cellulosic heating curve, shown in Section 5.1.1 of EN 1363-1:2020 [11].
- Real-scale laboratory test in a horizontal furnace according to EN 13381-3:2015 test methods for determining the contribution to the fire resistance of structural members Part 3: applied protection to concrete members [24] based on the ISO 834 fire temperature curve.
- Small-scale laboratory tests in a small furnace are according to the EFNARC´s specifications and guidelines for testing passive fire protection for concrete tunnels lining [19] and based on the RABT fire temperature curve.
- In-situ fire resistance test using a mobile experimental furnace according to the EFNARC´s specifications and guidelines for testing passive fire protection for concrete tunnels lining [19] and based on the ISO 834 fire temperature curve.

Computer simulations of heat transfer within the concrete slabs are also proposed to complement some of the previous tests and to determine the temperature within the concrete at various depths, as suggested in 2020-Efectis-R0695 [13] using FEM heat transfer calculations. ANSYS software is proposed to create models, while Eurocodes are used for material properties [25, 26].

3. Results

This section studies the fire resistance performance of different geopolymer materials considering the manufacturing process (precast vs. 3D printing) using other evaluation methods: small-scale fire resistance tests for product development, medium-scale laboratory tests in an experimental furnace; computer simulations of concrete slab behavior based on laboratory tests; accurate scale laboratory tests for specific uses; small scale fire resistance tests for different fire scenarios; and in-situ tests with a mobile furnace.

3.1. Medium-Scale Laboratory Tests in an Experimental Furnace

A medium-scale thermal insulation test is performed in an experimental furnace (Figure 2), evaluating the fire resistance performance of geopolymer panels as fire-protective concrete tunnel lining elements. The test is performed according to the 2020-Efectis-R0695 fire testing procedure for concrete tunnel linings and other tunnel components [13] (Figure 2) and following the ISO 834 fire curve or the cellulosic heating curve, shown in Section 5.1.1 of EN 1363-1:2020 [11], which follows the relationship (Equation 1):

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

Where T is the average furnace temperature in degrees Celsius, and t is the time in minutes. According to Clause 4.7 of 2020-Efectis-R0695, the recommended limiting temperatures are the following: [13]

- T95% < 250ºC in the steel reinforcement.
- T95% < 380ºC in the exposed side of the sample.

$T_{95\%}$ is defined as 95% of the characteristic value of all the values of each level (Equation 2):

$$T_{95\%} = T_{ave} + t \times \sigma \quad (2)$$

Where T_{ave} is the average temperature, σ is the standard deviation, and t is obtained depending on the nº of measurements (t (6)=1.943; t (7)=1.895; t (8)=1.860; t (9)=1.833; t (10)=1.812; t (15)=1.753; t (>20)=1.725). Two tests are performed, one for the precast geopolymer panel and another for the 3D printed panel, both of 4cm thickness and directly fixed in horizontal position to the concrete slab using commercial anchors (Figure 3).

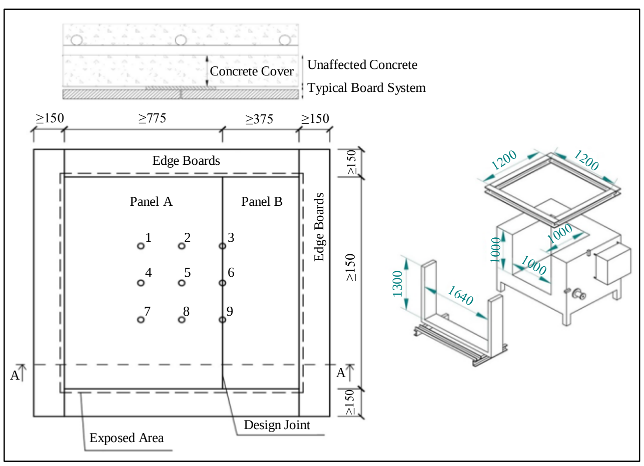


Fig. 2 Testing configuration and thermocouple positioning for concrete tunnel lining elements according to the standard [12] (left) and experimental fire resistance furnace (right)

Test results are included in Table 1. The differences between the two systems in terms of surface temperature vary measurements in the reinforcements, the system's temperature with 3D-printed protective panel is always lower, not even reaching the limit temperature in 4 hours of testing. In the precast case, the first failure point is in the reinforcement, but both failures occur in a similar testing period (184 minutes in the reinforcement while 193 minutes on the concrete surface). In the case of the 3D printed sample, the surface failure occurred slightly (199 minutes) later than the precast sample, but there was no failure in the reinforcement during the 4 hours of testing. Therefore, there is a higher heat transmission within the material in the precast system than in the 3D-printed system.

Figure 5 shows the status of the samples after the test. The precast sample begins to deform and gradually melt, while the 3D-printed sample cracks and breaks. Therefore, the temperature distribution in the concrete surface is not uniform in the 3D printed sample once the cracks appear (Figure 4). The breaks occur mid-distance between two fasteners but do not appear when the distance is small enough (Panel B). It should be added that there is no material loss, which the fasteners hold in place. All this could lead to a different performance of the materials in high classification requirements or more severe fire scenarios.

Fig. 3 Samples and mounting of fire resistance tests: precast (left) and 3D printed (right)

Table 1. Temperatures obtained during the experimental fire resistance tests

	Temperature on the Concrete Surface		**Temperature on the Lower Steel Reinforcement**	
	Precast	**3D Printing**	**Precast**	**3D Printing**
$T_{95\%}$ after 60 Minutes	98,4	104,0	79,2	58,9
$T_{95\%}$ after 90 Minutes	171,3	101,2	99,2	72,5
$T_{95\%}$ after 120 Minutes	241,9	209,5	142,4	104,8
$T_{95\%}$ after 180 Minutes	357,2	340,0	244,2	167,1
$T_{95\%}$ after 240 Minutes	452,7	483,2	332,8	233,5
$T_{surf/reinf}$ as 95% of the Characteristic Calculated Temperature	Recommended Requirement ($T_{surf} \leq 380$ ºC)		Recommended Requirement ($T_{reinf} \leq 250$ ºC)	
	193 min	199 min	184 min	No Failure (240 min)

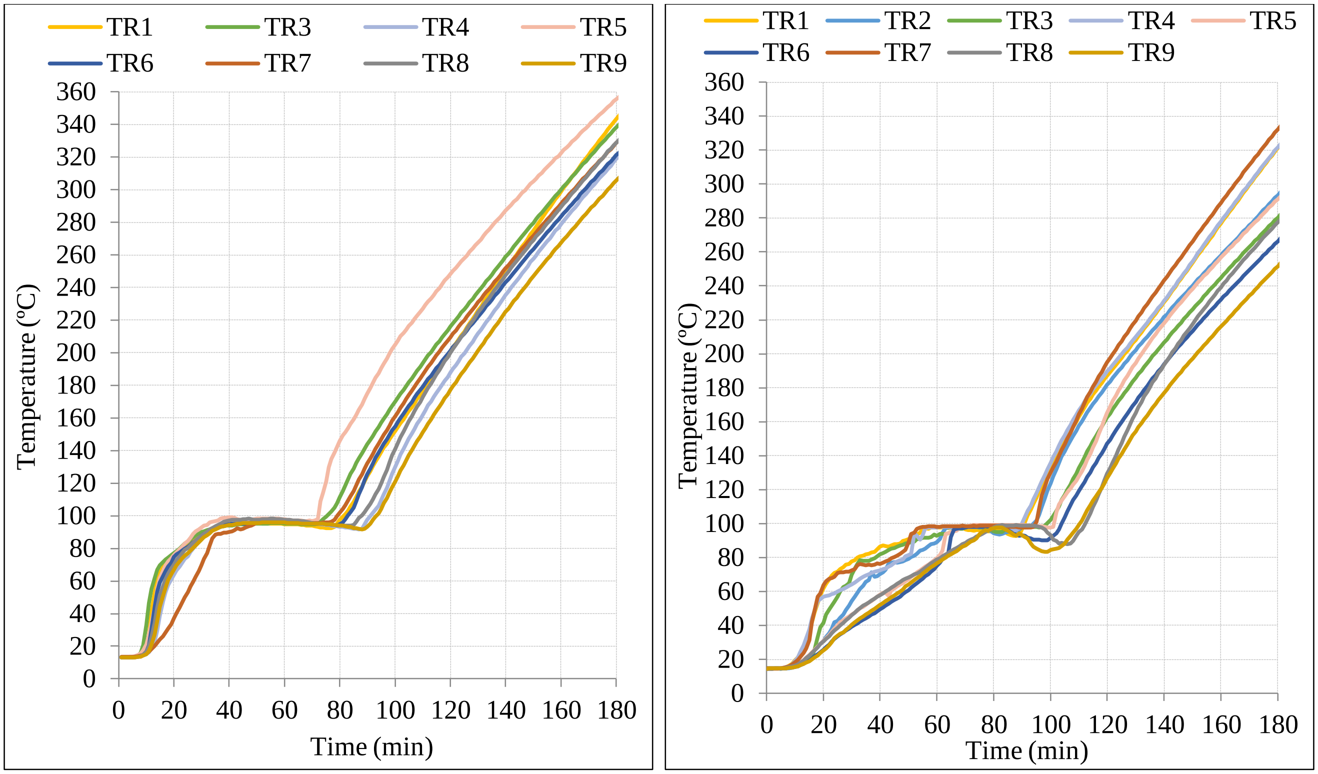


Fig. 4 Evolution of maximum temperatures in the concrete surface: precast (left) and 3D printing (right)

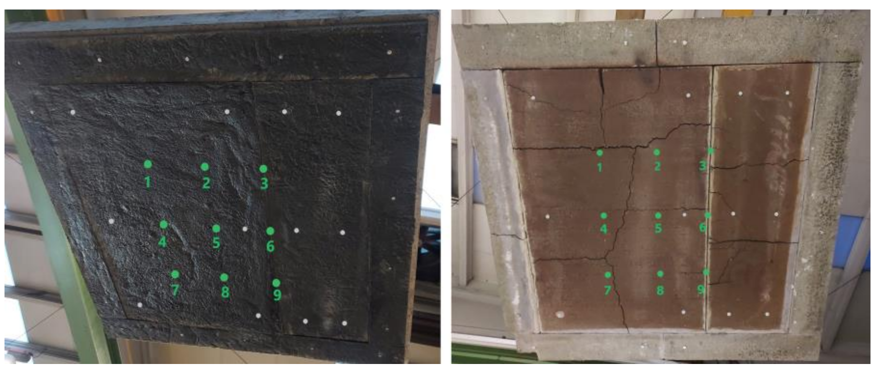


Fig. 5 Status of precast (left) and 3D printed (right) samples after fire resistance tests. Fixations are marked in white, and thermocouples in green.

3.2. Computer Simulations of Concrete Slab Behavior Based on Laboratory Tests

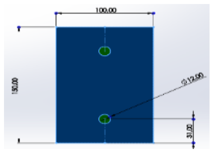
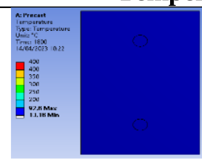
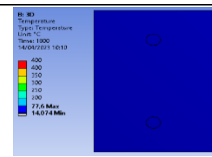
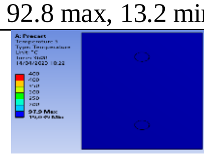
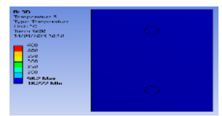
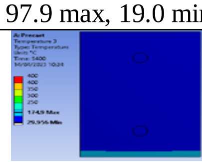
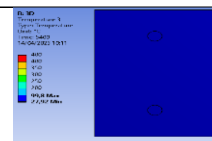
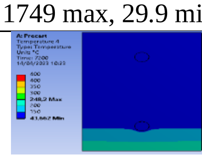
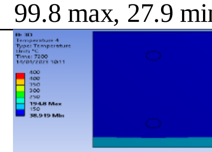
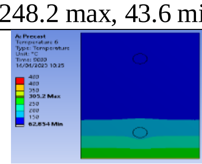
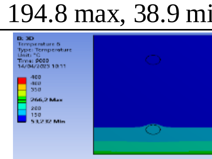
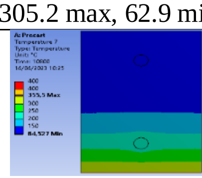
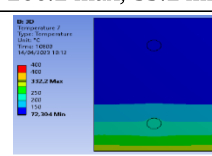
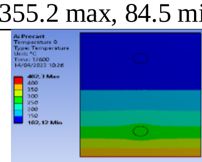
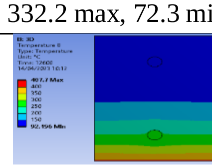
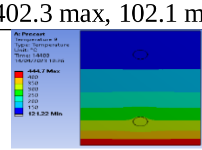
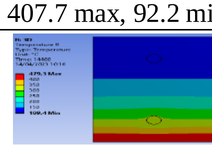
According to 2020-Efectis-R0695, [13] temperatures within the concrete at various depths can be determined using FEM heat transfer calculations based on concrete surface temperatures measured during a fire resistance test and considering the material thermal properties of the Eurocodes. In this regard, temperature distribution within the concrete slabs has been simulated within the same fire scenario based on the results of the experimental fire resistance tests for each 30 minutes.

The drawings have been created in solidworks and the models in ANSYS, using a 2.5 mm modeling mesh for concrete 1 mm mesh for steel and considering a bonded type contact between them. Thermal properties of concrete and steel have been determined from Eurocodes 2 (EN 1992-1-2) and 3 (EN 1993-1-2), respectively, [25, 26]. In contrast, temperatures on the lower edge of the slab have been determined as the maximum concrete surface temperatures recorded during the experimental fire resistance tests.

Modeling of each testing period is represented in Table 2 including the testing period, the model, and the maximum and minimum temperatures simulated within the concrete slab and in Table 3 including the temperatures for different depths of the concrete slab and different testing periods.

As a result, the simulations confirm a faster thermal transmission in the concrete slab for precast material compared to 3D printing material (Table 2). In this regard, temperatures in the concrete surface for the 3D printed protective elements are lower than for the precast case except for the final stage of the test or simulation (240 min. period), mainly due to the formation of cracks and a more direct affection of the fire to the concrete. This tendency is stabilized (lower temperatures for the 3D-printed protective element) in deeper areas of the concrete slab (Figure 6).

Table 2. Temperature distribution in the concrete slab for each testing period according to the FEM

Model	**Precast**	**3D Printing**
		
Time (min)	**Temperature Distribution**	
30	 92.8 max, 13.2 min	 77.6 max, 14.1 min
60	 97.9 max, 19.0 min	 98.2 max, 18.2 min
90	 1749 max, 29.9 min	 99.8 max, 27.9 min
120	 248.2 max, 43.6 min	 194.8 max, 38.9 min
150	 305.2 max, 62.9 min	 266.2 max, 53.2 min
180	 355.2 max, 84.5 min	 332.2 max, 72.3 min
210	 402.3 max, 102.1 min	 407.7 max, 92.2 min
240	 444.7 max, 121.2 min	 479.3 max, 109.4 min

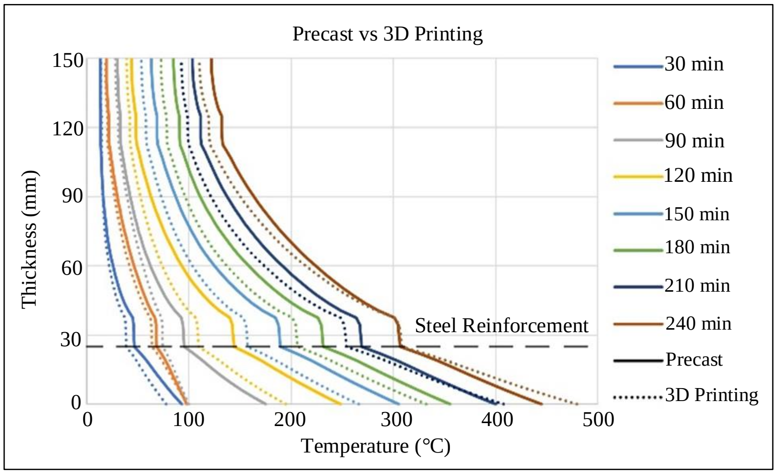


Fig. 6 Temperature distribution in the concrete slab for the different test periods and protection materials

Table 3. Temperatures obtained by computer simulations (FEM) at different depths of the concrete slab

Time (min)	Temperature (˚C) on the Concrete Surface		T (˚C) on the Lower Steel Reinforcement (25 mm)		T (˚C) at 75 mm		T (˚C) at 125 mm	
	Precast	3D Print	Precast	3D Print	Precast	3D Print	Precast	3D Print
30 min	92.8	77.6	46.697	38.747	19.659	17.865	13.648	14.290
60 min	97.9	98.2	68.404	63.941	31.136	33.463	21.672	20.338
90 min	174.9	99.8	95.276	74.397	50.905	46.426	33.032	30.820
120 min	248.2	194.8	144.13	109.65	75.835	61.510	48.280	42.147
150 min	305.2	266.2	189.510	157.52	102.55	85.902	68.761	57.978
180 min	355.5	333.2	231.44	206.250	130.84	112.730	90.653	78.139
210 min	400.1	407.7	269.060	254.170	160.475	143.230	111.287	98.712
240 min	444.7	479.3	306.68	308.170	190.110	177.750	131.920	119.32

3.3. Real-Scale Validation Tests in A Horizontal Furnace

Fire resistance assessment or final product validation has been done utilizing real-scale laboratory tests for specific uses. These end uses have been selected to address different niche market opportunities (benchmarking of existing products for precast and new markets for 3D printing) and considering the fabrication/production characteristics or features of each material (precast material could be suitable for panel uses while 3D printing for ad hoc pieces). Accordingly, precast panels have been evaluated as protection elements for concrete members, and 3D printed material has been assessed as customized pieces for service installation ducts, both tested in a horizontal furnace (Figure 7). This article only includes real-scale results for the precast case and compares them with those obtained at medium scale.

The precast geopolymer material is tested according to EN 13381-3:2015 Test methods for determining the contribution to the fire resistance of structural members –Part 3: applied protection to concrete members [24] based on the ISO 834 fire temperature curve with a 4 x 3 m2 exposed surface of protective material directly fixed to a concrete slab and supporting a load 48.15 kN load during the fire resistance test (Figure 8). The load is removed because the maximum deflection is reached at minute 316, and the test is stopped at minute 360. Pictures of the sample's installation and dismantling are included in Figures 9 and 10.

Throughout the test, surface and internal temperatures of the concrete and its reinforcement are measured, characteristic temperature curves are specified, and observations are made to provide fire resistance results and classification.

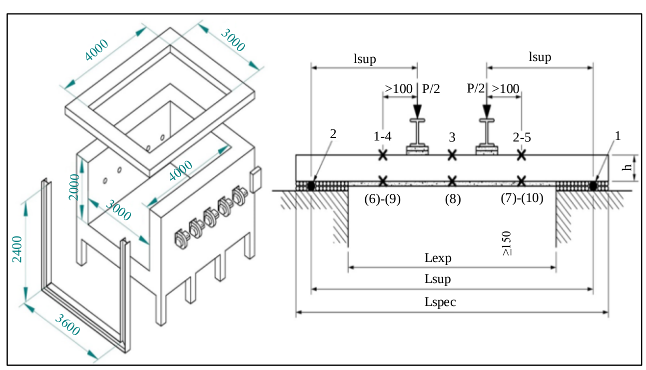


Fig. 7 Horizontal furnace drawing (left) and testing configuration drawing [24] (right)

The research objective of the test, however, is to compare the fire resistance performance of the protective boards in an accurate scale (4 m x 3 m) loaded test and an experimental scale test (1 m x 1 m). In this regard, temperatures registered in the concrete surface and the steel reinforcements during the whole real-scale horizontal furnace test are included in Figure 10. The diagrams show that temperatures are homogenized after the typical humidity loss of concrete, but they are highly increased once the protective panels lose effectiveness.

In this regard, although it is concluded that there is no loss of stackability regarding the requirements of the standard, it is seen in Figure 9 (right) that significant detachment of the protection system occurs during the test, which is also identified in the rapid increase of the temperature in the concrete surface that starts in minute 271 (Figure 10 left).

Fig. 8 Precast sample installation of the fire resistance test for the protection of concrete structural members

Fig. 9 Deflection of the concrete slab during the test (left) and state of the precast sample after the test (right)

3.4. Small-Scale Fire Resistance Tests for Different Fire Scenarios

Small-scale tests help find the most suitable formulation for each material and test different fire scenarios, such as more severe time-temperature curves specific to tunnel requirements. For the latter purpose, several small-scale tests were carried out for the RABT curve, where the temperature rises rapidly to 1200˚C. The tests are performed according to the EFNARC´s Specifications and guidelines for testing of passive fire protection for concrete tunnels lining, where the fire resistance requirement is that the temperature of the concrete reinforcement should not exceed 300˚C. [19] The sample consists of a 400 x 400 mm^2 exposed area, tested in a vertical orientation, where temperature measurements are taken in different locations and depths of the concrete slab (Figure 11). In this case, points 1, 5, and 9 are only used since an estimated performance wanted to be evaluated using an easy-to-mount system.

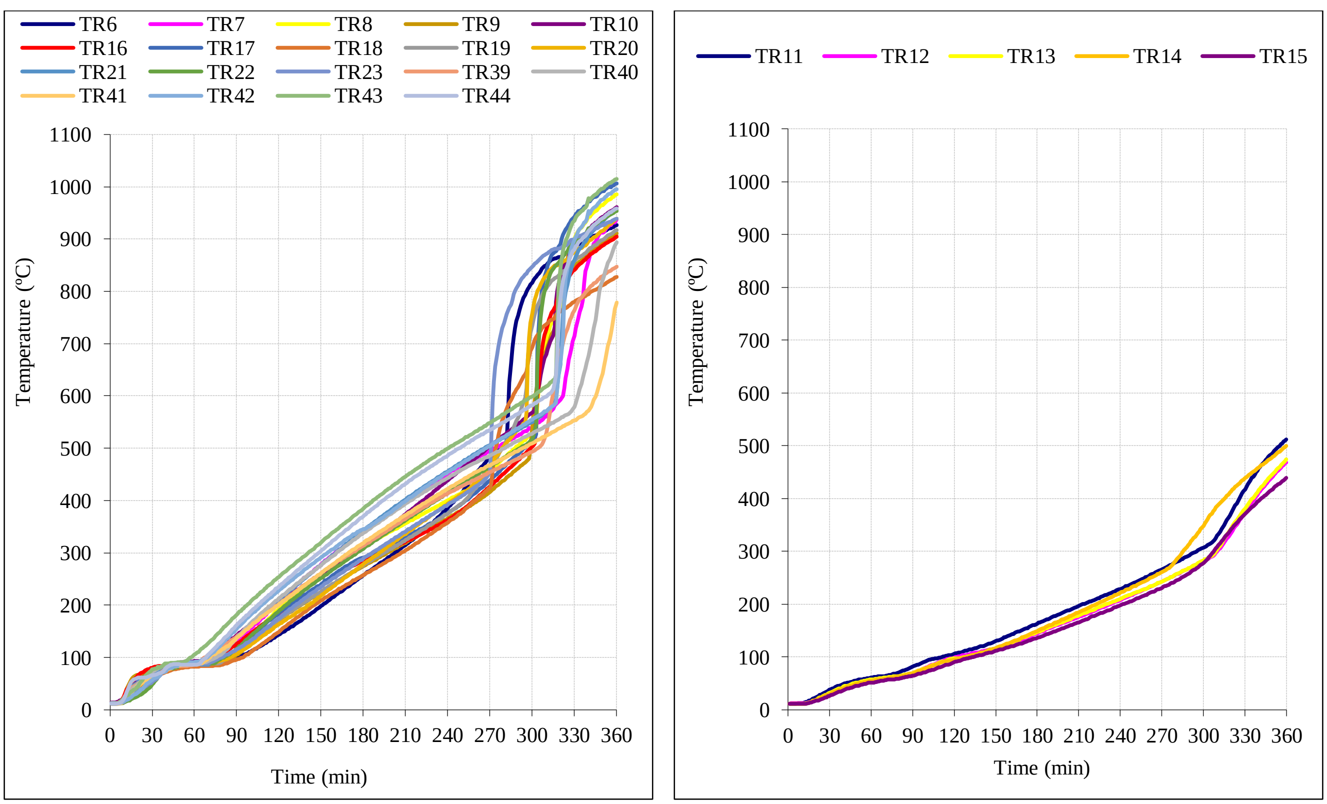


Fig. 10 Maximum temperatures measured in the concrete surface (left) and steel reinforcements (right)

Different formulations of each material (precast and 3D printing) are tested, including different slag percentages or curing times for precast and different lightening additives (Leca or Cenosphere) or protection thicknesses for 3D printing. The results of four of them are presented in Figure 12. The first test of the precast protection material was stopped due to the risk of furnace damage since too high temperatures were recorded too soon during the test, and the specimen was bent (Figure 13).

Although the whole test was performed for the second case, the maximum temperature limit was also reached before the end of the test, concluding that the developed precast geopolymer is unsuitable for more severe fire scenarios. Meanwhile, temperature values obtained in the 3D printed geopolymer tests have not reached the temperature limits established by the EFNARC´s procedure, [19] so results are considered valid, and the material might be suitable for more severe fire scenarios (Figure 14).

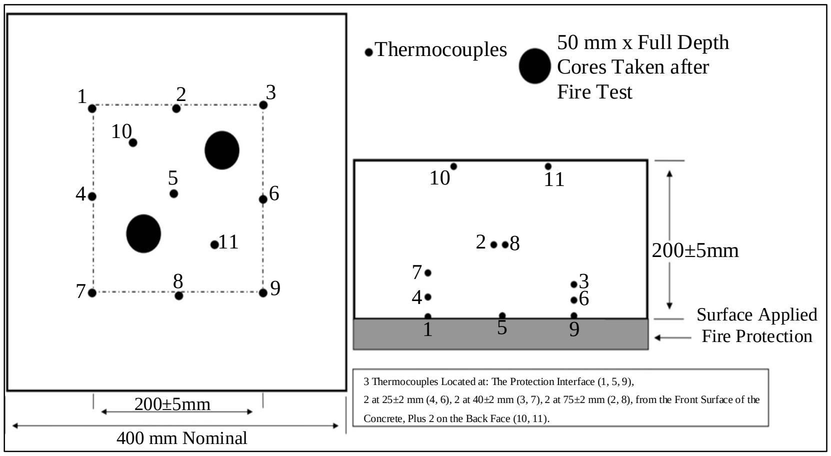


Fig. 11 Small-scale testing configuration and positioning of thermocouples [18]

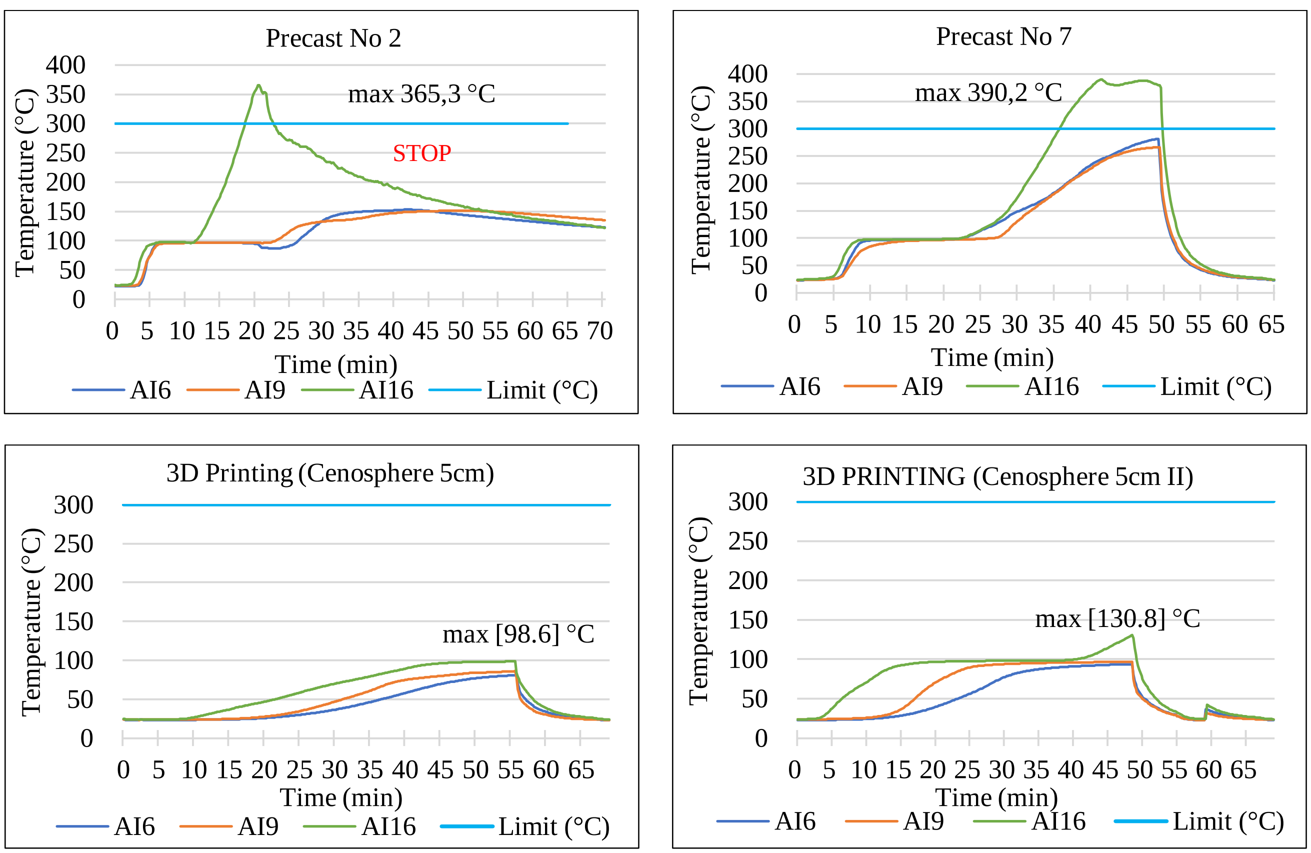


Fig. 12 Maximum temperatures in the concrete reinforcement in the RABT fire scenario for precast and 3D printed geopolymer protective panels

Fig. 13 Status of precast panels after small-scale fire resistance tests: test 1 (left and middle) and test 2 (right)

Fig. 14 Status of 3D printed panels after small-scale fire resistance tests for 4 and 5 cm thick panels

3.5. In-Situ Fire Resistance Tests

In-situ fire resistance tests have also been carried out according to the large-scale test of EFNARC´s specifications and guidelines for testing of passive fire protection for concrete tunnels lining [19] and following the ISO 834 fire curve to confirm the fire performance of developed geopolymers in a demo tunnel (Figure 15) in Lavrio (Greece). Tests have been performed in a vertical configuration for two hours using precast panels of 4 cm and 3D printing panels of 5 cm thick, without including any joint.

Fig. 15 Large-scale test according to EFNARC´s specifications and guidelines for testing of passive fire protection for concrete tunnels lining [18]

Maximum temperatures measured during the tests are included in Table 4 but are not comparable due to the differences in the panel thickness. Moreover, obtained values differ from the experimental scale laboratory test results, where the non-inclusion of joints could have some implications. It should be added that protective panels did not suffer any damage during the testing period, as expected after the experimental scale laboratory tests.

Table 4. Temperatures obtained during the in-situ fire resistance tests

Max. Temperature (˚C) on the Concrete Surface	Precast	3D Printing
30 Minutes	153,3	88,7
60 Minutes	174,1	116,5
90 Minutes	176,4	118,4
120 Minutes	177,7	126,5
Maximum T_{surf}	Failure Limit ($T_{surf} \leq 180$ ºC)	
	no Failure (120 min)	no Failure (120 min)

4. Discussion

The previous section presents results for materials according to all sets of test experiments and simulations. Hence, it is possible to identify suitable end uses for each of them based on the differences and specifications in the performance related to each material.

Accordingly, the geopolymer materials developed present a favorable fire performance of 3 hours for the precast material and 4 hours for the 3D printed material for their use in tunnels in an ISO 834 curve fire scenario, considering the temperatures measured on the reinforcements during the experimental fire resistance laboratory tests. However, the surface temperatures exceed the limit criteria by 3 hours in both cases. Furthermore, comparing the results from the computer simulations carried out with the experimental data, in the case of precast, the simulations show similar temperatures in the lower reinforcement to those measured in the test; however, the temperature values of the 3D printed material test are lower than those simulated (Table 5). Therefore, in the case of 3D printing, the laboratory test shows an even lower transmission than expected in the simulation.

Table 5. Temperatures obtained by means of computer simulations (left) and during the experimental fire resistance tests (right)

Max. Temperature (˚C) on the Lower Steel Reinforcement	FEM data		Experimental Data	
	Precast	3D Printing	Precast	3D Printing
60 minutes	68.404	63.94	79,2	58,9
90 minutes	95.276	74.397	99,2	72,5
120 minutes	144.13	109.65	142,4	104,8
180 minutes	231.44	206.25	244,2	167,1
240 minutes	306.68	308.17	332,8	233,5

Hence, experimental data and computer simulations show better thermal performance for the 3D printed material. Still, its performance is poorer when cracks appear and fire is inserted within the panels. Regarding the physical behavior of the material, the precast sample begins to deform and gradually melt, while the 3D-printed sample cracks and breaks.

The first is ductile, while the second is brittle, as shown in a flexural test diagram (Figure 16). Therefore, the distance between fixations becomes an essential variable for the 3D printed geopolymer material, where no cracks appear in small distances (175 mm tested), cracks appear in medium distances (475-575 mm tested), and material loss could appear in higher distances. Moreover, cracks or breakages may also occur during installation, so fixing elements must be applied carefully.

All this could lead to a different performance of the materials in high classification requirements or more severe fire scenarios. In this sense, small-scale tests have shown that 3D printed material can work for more severe curves related to higher standards in tunnels, but not the precast material.

Therefore, the most suitable end-use application for the geopolymer material manufactured by the precast method may be focused on protecting flat and large surfaces with not very severe requirements for tunnels, such as light vehicle traffic (car and van traffic). Meanwhile, the geopolymer material manufactured by the 3D printing process may be more suitable for more severe uses (truck traffic) in tunnels or specific shapes due to its fire resistance performance, manufacturing time, and cost, and considering its environmental impact.

[6] Furthermore, beyond the classification limits for fire resistance obtained in the experimental furnace, a full-scale test in the horizontal furnace has shown that the precast material melts, with almost no material remaining inside the furnace at the end of the test (after 6 hours). It should be added that the test results were favorable, as material fallout is not considered a failure criterion, and the material is valid for the protection of concrete slabs.

Furthermore, from comparing the experimental scale and real scale loaded tests for the precast protective material, small differences in the fire resistance performance can be identified based on the temperatures measured. Although the general performance is similar, a faster start of temperature increase is detected in the real scale (minutes 50-80 in real scale vs. minutes 70-90 in experimental scale) and at a lower temperature (85˚C in real scale vs. 95˚C in experimental scale) with the loss of water from the concrete slab (Figure 17 and Table 6).

After 240 minutes, maximum temperatures increase to 499˚C on real scale, while up to 452,7˚C on experimental scale, showing slightly poorer results in the real scale test. Hence, it can be concluded that applying load or system configuration (the panels' dimensions and the distance between fixations) can affect the fire resistance performance.

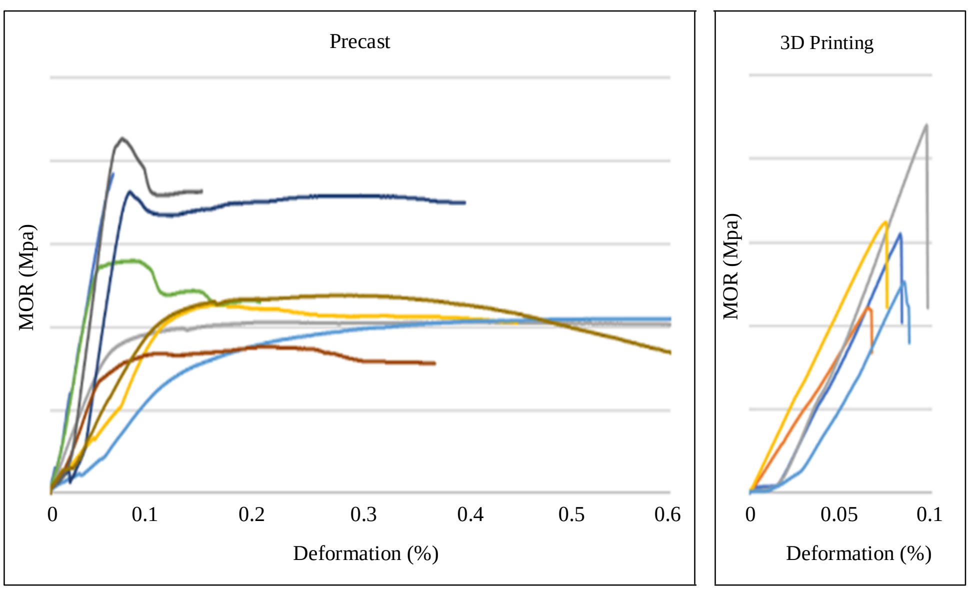


Fig. 16 Flexural performance of developed geopolymer materials

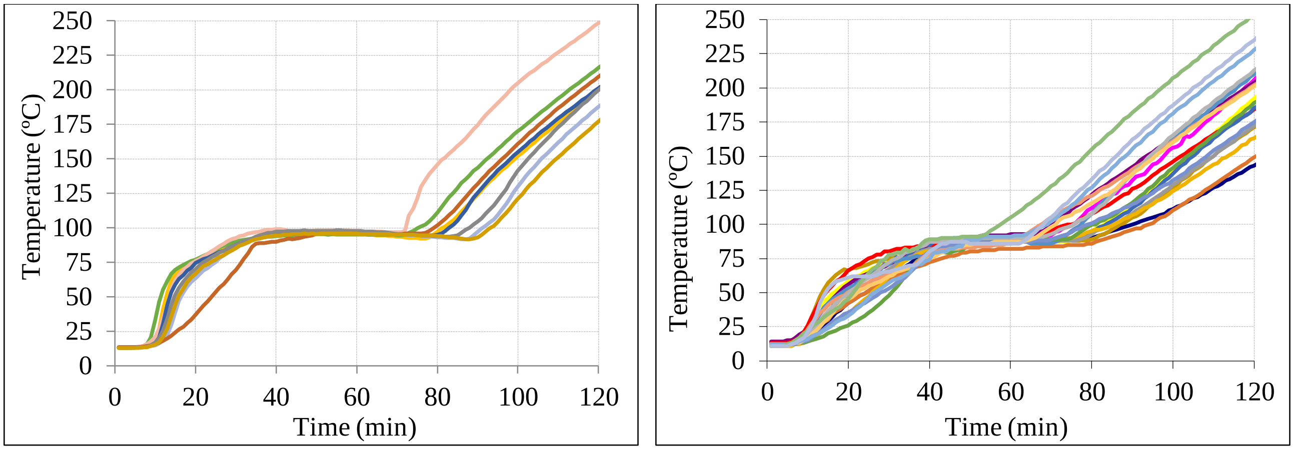


Fig. 17 Maximum temperatures measured for the precast product in experimental (left) and real-scale (right) tests

Table 6. Maximum temperatures obtained during the experimental and real-scale fire resistance tests

	Maximum Temperature (˚C) on the Concrete Surface	
	Experimental Scale	**Real Scale**
60 minutes	97.9	105
90 minutes	174.9	182
120 minutes	248.2	254
180 minutes	355.5	385
240 minutes	444.7	499

5. Conclusion

This article contributes to the background by showing the fire resistance performance of geopolymer materials, in this case, two materials based on the same mining slag but produced using two manufacturing processes, where their fire resistance behavior and material characteristics have been identified. Additionally, it studies different fire resistance evaluation methods and assesses their suitability for other product development or research phases.

Research results demonstrated that the production method of the geopolymer materials that are based on the same mining slag affects their fire resistance performance since, although the same classification can be obtained for some of the uses and scenarios, they present a different thermal transmittance and different mechanical behavior in long or extreme fire exposures due to the diverse nature of the geopolymer material itself (ductile vs brittle material).

Accordingly, different end uses or use scenarios can be associated with each manufacturing method and associated product: precast geopolymer material in panel format for tunnel uses (low fire resistance performance) and 3D printed geopolymer material in panel format or customized pieces for tunnel uses (high fire resistance performance). It should also be added that developed products (precast and 3D printing processes) have a relatively high density compared to benchmark products, so auxiliary lifting devices are needed for installation. Different fire resistance evaluation methods have been used to achieve different objectives in each research phase. Small-scale fire resistance tests have been used to compare different material formulations, but mainly to test the possibility of withstanding more severe fire scenarios related to tunnel requirements, showing a favorable performance for the materials produced by the 3D printing method.

However, the effect of the installation method is not assessed since the samples are not big enough to include real distances between fixations. Hence, installation or mounting methods must be evaluated at full scale since the inclusion of joints, the dimension of protection material, the application of the load, or the fixing systems and elements could vary the result of each developed product. That is why experimental and full-scale fire-resistant tests have been used to check the suitability of each product regarding the selected end use: experimental scale fire resistance test based on Efectis R0695 protocol for tunnel uses and full-scale fire resistance tests based on European standards for other specific end uses.

Additionally, in-situ fire resistance tests are considered useful for already installed protective materials to assess the fire resistance performance of installed products but not for newly developed materials, or at least the damage of the tunnel needs to be considered in that case (if the test is performed until failure). Finally, it is not feasible to test all the configurations or combinations in each product, such as thickness, dimensions, installation methods, or distances between fixings.

However, they may significantly affect the result of the fire resistance test. In this regard, computer simulations can provide a suitable tool to validate other configurations or to evaluate specific points or areas of the fire resistance test. In this case, thermal-mechanical simulations have considered the thermal transmittance of the two products during the test to understand the differences in the results, showing a slower thermal transmittance in the 3D-printed protective panels and to anticipate the use of these products in other fire scenarios.

However, they have not considered the formation of cracks or holes that appeared during the tests in the 3D printed boards, so actual experiments are needed to improve the model and get more accurate results. Hence, it can be concluded that combining the different evaluation methods can facilitate product assessment during the different product development stages and for different end-use applications. Only accurate scale fire resistance tests can evaluate all variables considered. Still, smaller scale tests are considered valuable to make an initial assessment of the fire resistance performance of the different parameters to be selected during the design phase, such as panel dimensions or thickness, type of fixations and distances between them, or the protection material itself considering the manufacturing method or the formulation.

This step-by-step methodology has been applied to evaluate new high-performance geopolymer materials based on mining slag and to evaluate the influence of the manufacturing process. However, it can apply to incorporating other components to the formulations, such as other by-products, waste, or recycling materials, or evaluating different product parameters, such as installation methods or product thickness. Finally, it should be added that concrete spalling has not been assessed using a specific test method. Still, this phenomenon has not been observed after accurate scale experiments.

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