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Innovative Inorganic Insulation Material (Mineral Slag Based Geopolymers) *in Situ* Production as Thermal Insulating Screed with Fire Resistant Properties

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Abstract

In a previous study, we developed a low density inorganic polymer based on industrial minerals and waste designed for *in situ* applications in the construction sector. In this paper, further testing and development of this new screed with thermal insulation and fire resistance properties is presented. The large scale tests of *in situ* production and fire-resistant properties are also discussed. The newly developed material succeeded at large scale fire test under the RABT-ZTV (car) fire curve and also was used *in situ* in large scale test in a typical screed application in a renovation building project using a typical screed pump.

Subject Areas

Material Experiment

Keywords

Inorganic Polymers, Insulation Materials, Industrial Waste, Fire Resistant Screed

1. Introduction

With the price increase of construction products during the last years, the continuous demand for new products with improved properties and the market trend of 2 in 1 solutions, the proposal of innovative materials with improved environmental footprint valorising industrial waste is a promising alternative to traditional approaches. The 2022 annual growth rate of construction producer prices for new residential buildings is 11.9% for EU (11.5 for EA-20) [1]. At the same time landfilling of wastes, the corresponding taxes, and the EU policy for the diversion of waste from landfills pose a significant opportunity to explore new alternatives [2] [3]. As described in a previous study [4], industrial minerals and wastes, due to their aluminosilicate content, are excellent precursors to inorganic polymers, offering at the same time enchant fire resistance properties. In the previous study, we reached a low density of 401 Kg/m^3 but with an increased cost due to the high price of metakaolin. In this study we explore a different route, valorising a mineral byproduct (GGBFS) to produce lightweight [5] and fire-resistant [6], inorganic polymer for screed applications. At the same time project target is to develop a screed product of "*just add water*" type with thermal insulation and fire resistance properties which can be produced *in situ* in the construction site using typical construction equipment (e.g. concrete/plaster pumps).

2. Large Scale Experiments

2.1. Materials

As described in a previous study [4], initially inorganic polymers were produced with grinded (−100 μm) ferronickel granulated EAF slag from reductive smelting of laterites with mean particle size (d_{50}) of 15.05 μm, metakaolin (AGS Mineraux) with mean particle size d_{50} of 13.68 μm, 2 M sodium hydroxide solution (pellets, Merck, 99.5% purity) and 0.7%w/w hydrogen peroxide as the foaming agent. An anionic wetting and air-entraining surfactant (Hostapur OSB, Clariant) was also used to retain porosity and prevent the gel from collapsing. Trying to achieve even lower porosity and cost, since the target material is a fire-resistant screed, Ground Granulated Blast Furnace Slag (Ecocem) with mean particle size d_{50} of 11 μm was used, along with 7 M potassium hydroxide solution (>90%, Kalochem) and aluminum powder (Alfa Aesar), −325 mesh, 99.5% (metals basis) as foaming agent, to produce lightweight inorganic polymer.

GGBS is an excellent precursor for the production of inorganic polymers due to its chemical composition rich in Al_2O_3 and SiO_2. (**Table 1**)

2.2. Experimental Procedure

To achieve low density and based on the targeted material specifications and literature review, we used only GGBFS without adding any other slag or sodium silicate and aluminum powder was chosen as the foaming agent, as it is a common practice in aerated concretes and does not require special additional equipment as required for the production of aerated concrete and the addition of foam.

Table 1. Ecocem GGBFS chemical composition (percentage average).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	SO_3	Cl^-	S_2^-	Na_2O	K_2O	Na_2O_{eqv}
37.7	10.2	0.6	43.8	6.4	0.7	0.1	0.02	0.7	0.21	0.28	0.37

Different potassium solutions were used (4 M, 6 M, 7 M, 8 M, 10 M) and different amounts of aluminum powder (with the higher workable solid-to-liquid ratio of 1.6), in order to achieve the optimum lower density and higher mechanical strength.

From this study of adding different percentages of aluminum powder in order to achieve low density, it emerged that the amount of aluminum powder should be 0.25% in relation to the solids. From the analysis made on the various samples produced, it appeared that the continuous increase in the amount of aluminum does not lead to a linear decrease in density, but there is a limit above which further increase in the amount leads to the collapse of the material as the creation of many and of large pores with ambient temperature curing does not result in stable materials. So the optimum synthesis was the one with 7 M potassium solution, S/L ratio to 1.6 and 0.25% w/w aluminum powder reaching a 28 day compressive strength of 2.0 MPa and a lower density of 401 Kg/m^3.

2.3. Large Scale *in Situ* Production

As mentioned before, the project's goal is to valorise industrial waste or by-products with polymerization technology to develop an *in situ* produced screed with thermal and fire-resistant properties. The upscaling of the lab process was performed using the industrial pump for spray concrete/plaster (screw type). The activating alkaline solution was prepared ahead of the test, so the actual practice of spray concrete/plaster application was replicated.

The *in situ* production in real conditions with GGBFS and industrial pump, took place in a small roof warehouse with an area of 5 m^2. The average thickness applied was 5 cm. The process was the same as the laboratory setup: mixing in the mixer of solids and liquids and with a suitable pump and pipe applying the material on the floor. The foaming agent (aluminum powder was premixed with the GGBFS at the defined ratio, so the actual lab scale formula was replicated). A suitable foam tape was applied around the walls to prevent cracking during the curing of the material and the openings (door and window) were covered with opaque material to protect it against direct exposure to light and air. (**Figure 1**)

Cubic samples of the *in situ* produced screed were casted on 15 × 15 × 15 cm metal moulds for testing mechanical and other essential properties at 7 and 28 days. Also *in situ* produced material was casted in 100 × 50 × 4 cm moulds to test it in a small scale fire test according to RABT-ZTV (for validating the positive previous lab scale fire test results). (**Figure 2**)

2.4. Fire Testing

Based on its inorganic nature and high SiO_2 content, GGBFS is a very common material in the production of foamed [7] geopolymers [8] and thus it was decided to carry out a large-scale test based on the EFNARC guidelines (Specification and Guidelines for Testing of Passive Fire Protection for Concrete Tunnels Linings, 2006) [9] on a concrete slab coated with the developed material to investigate the behaviour of the material in fire exposure when the temperature on

(a) (b)

(c)

Figure 1. (a) and (b) application of the newly developed screed on 5 m^2 floor. (c) the screw-type pump used for the test.

(a) (b)

Figure 2. Samples from the large scale *in situ* production of the screed for further testing of the material.

the exposed surface follows the RABT-ZTV time-temperature curve (for cars). The concrete slab had dimensions of 1.5 × 1.5 m and a thickness of 20 cm with Φ20 reinforcement placed 5 cm from the "face" of the slab, while the GGBFS inorganic polymer was applied to a thickness of 5 cm.

The RABT curves were developed in Germany as a result of a series of test programmes, such as the Eureka project. In the RABT ZTV curves the rise in temperature is very rapid, up to 1200˚C within 5 minutes. However, the duration of 1200˚C exposure is shorter than in other curves. With the temperature drop off starting to occur at 30 minutes for car fires. The drop off for train fires only starts at 60 minutes. A 110-minute cooling period is applied to both fire curves. The failure criterion for specimens exposed to the RABT-ZTV time/temperature curve is that the temperature of the reinforcement should not exceed 300˚C. There is no requirement for a maximum interface temperature. (**Figure 3**)

The specific test was conducted by using a furnace with a combustion area of 100 × 100 cm². The furnace used and its set-up for the implementation of the test are shown in the following photos **Figure 4** and **Figure 5**.

Real time monitoring of the process is assured by a data logger (connected to the thermocouples) and a camera that transmits image from inside the furnace. The gas burning furnace is controlled by a central unit with a PLC easily programmable to the desired fire curve. (**Figure 6**)

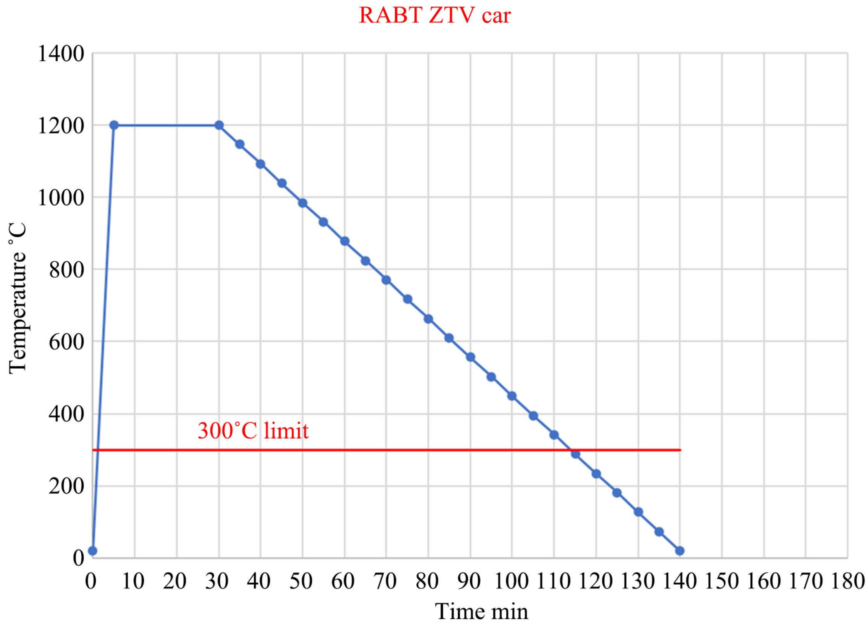


Figure 3. RABT-ZTV (car) time temperature curve.

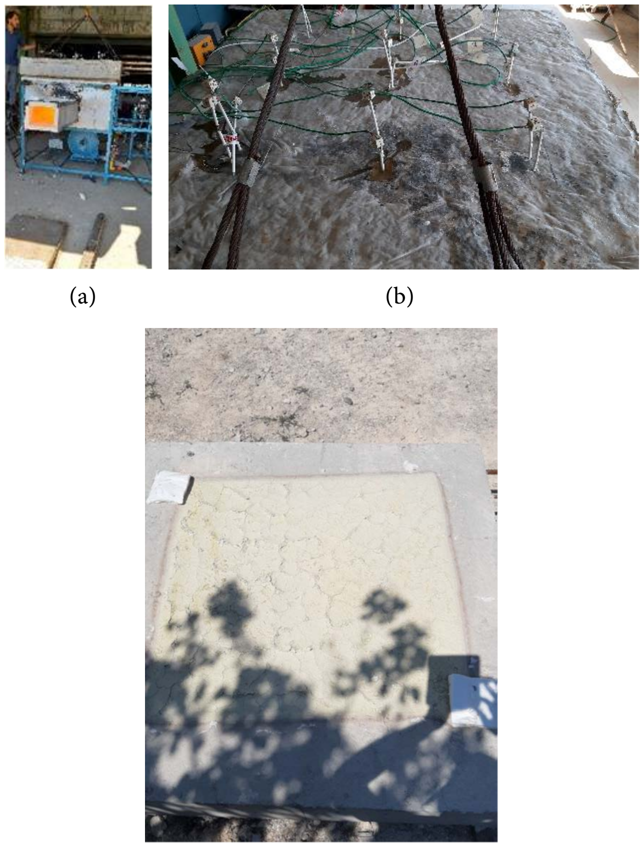
(a)

(b)

(c)

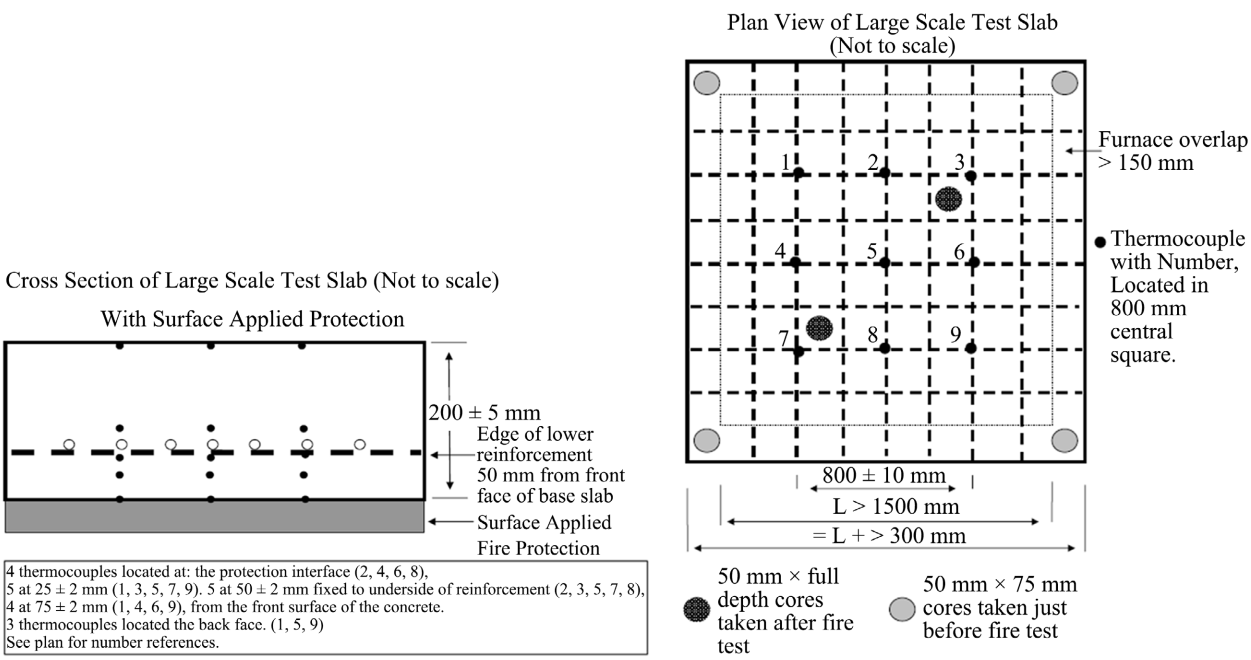
(d)

Figure 4. Furnace set-up for the implementation of the fire resistance test. (a) data recorder, central PLC unit and laptop connected to furnace camera, (b) furnace with the specimen and all thermocouples installed-connected to data logger, (c) specimen before the fire test (d) side view of the furnace with the specimen ready for the test to begin.



(a) (b) (c)

Figure 5. Furnace set-up for the implementation of the fire resistance test. (a) gas exhaust, (b) thermocouples installation, (c) specimen after the test and cool down time.



(a) (b)

Figure 6. Thermocouple setup acc. to EFNARC guidelines for large scale setup with surface applied protection.

3. Results

The samples taken from the *in situ* production of the screed were tested for compressive and flexural strength (EN 13892-2), wear resistance (EN 13892-3) and thermal resistance (by means of guarded hot plate, EN 12664).

The 28 days compressive and flexural strength reached 2.8 and 0.8 MPa respectively, wear resistance the product can be classified as A22 and the thermal conductivity was measured 0.057 W/mK.

Moreover, the material successfully passed the large-scale fire resistance test using the RABT-ZTV time-temperature curve for cars, since all thermocouples in the depth of the reinforcement did not exceed the temperature limit of 300˚C. In fact, the temperature at these points did not exceed 105˚C on average (only a single thermocouple reached 150˚C, Figure 7 thermocouple AI1) which is significantly lower than the limit [10].

In Figure 6 the evolution of temperature in the reinforcement depth inside the specimen is presented, where it is clearly seen that the highest temperature of all four thermocouples installed is lower than the limit set by EFNARC and in Figure 8

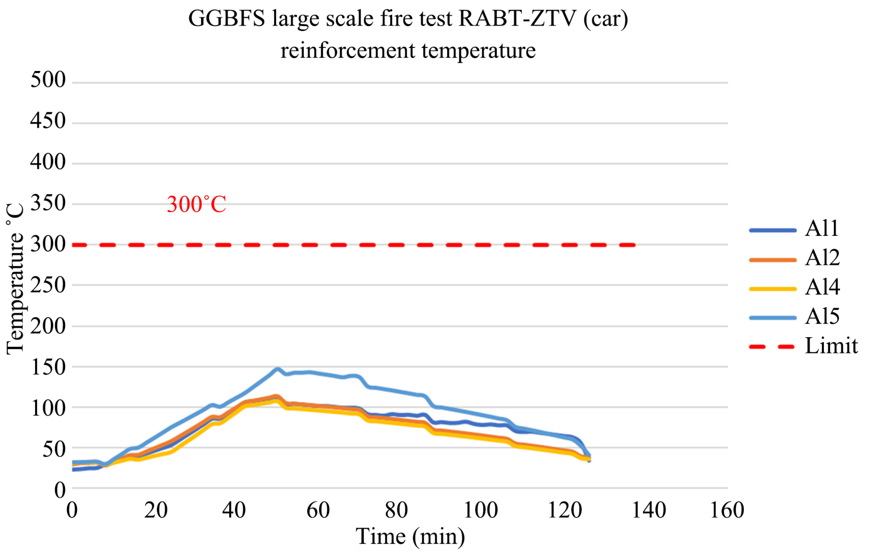


Figure 7. Large scale fire results in the reinforcement depth.

(a) (b)

Figure 8. Specimen after the test. (a) right after cooling and (b) 48 hr later.

the specimen after the test finished and cooled down. The powdered screed was removed 48 hr after test completed to measure the depth of the layer impacted by the test and also to inspect the concrete slab condition. The average thickness of the screed that was affected and removed was 3 mm (measured at 25 points inside the square defined by the furnace opening (in **Figure 8**, photo b presents the furnace opening that defines the area of the specimen subjected to testing).

4. Conclusions

In this study, a thermal insulation screed with fire resistance properties was developed with the geopolymerization of GGBFS. The material was successfully produced *in situ* at the construction site using a large scale screw type pump for spray concrete/plaster. Samples casted from the fresh material were later tested according to EN 13813: 2002 "screed material and floor screeds-Screed material-Properties and requirements" and the product can be classified as C3-F1-A22 screed for internal use.

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Conflicts of Interest

The authors declare no conflicts of interest.

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