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UTILISATION OF FeNi - SLAG FOR THE PRODUCTION OF INORGANIC POLYMERIC MATERIALS

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Abstract

This paper deals with the utilisation of slag for the production of inorganic polymeric materials. The performed work includes the optimisation of the slag-based geopolymeric system through the study of the effect of the synthesis parameters on the properties of the materials and the production of fire resistant materials utilising slag as a raw material. The obtained results showed that the materials produced, presented high compressive strength. Then, the materials were tested for their resistance under high temperatures (up to 1350°C). From the results it is also concluded that these geopolymers perform well under various fire scenarios, without spalling.

Introduction

The geopolymerisation is a rapidly developing and innovative technology, which utilises solid aluminosilicate materials (naturally occurring minerals, industrial by-products or waste) to produce a wide range of revolutionary materials, the geopolymers. Geopolymers are inorganic polymers that possess excellent physical, chemical and mechanical properties such as high compressive and flexural strength, high surface hardness, micro- or nano-porosity, low water absorption and permeability, negligible shrinkage, thermal stability, fire and chemical resistance. Geopolymerisation involves a heterogeneous chemical reaction between aluminosilicate solid materials and alkali metal silicate solutions, under highly alkaline conditions. The reaction takes place at atmospheric pressure and temperatures below 100°C resulting in the formation of amorphous to semi crystalline polymeric structures, which consist of Si–O–Si and Si–O–Al bonds.

Experimental

The chemical analysis of the slag (Table 1) was performed by fusion with a mixture of $Li_2B_4O_7/KNO_3$ followed by nitric acid digestion. According to Table 1, the slag is a siliceous material very rich in iron oxides and rich in alumina. It also contains substantial amounts of trivalent chromium, magnesium and calcium oxides, as well as traces of nickel.

Table 1: Chemical analysis of Slag

Species	% w/w
SiO_2	41.14
Al_2O_3	13.79
FeO	34.74
Cr_2O_3	5.41
MgO	3.59
CaO	0.71
Ni	0.14

Four different kinds of materials (a, b, c, d) were produced according to the following three stages procedure. A homogeneous viscous paste was initially prepared by mixing mechanically the ferronickel slag with the strongly alkaline aqueous phase. Then, the paste was moulded in appropriate open plastic (ERTASETAL) moulds and then vibrated for 1 min on a vibrating table. The last stage is the curing of the produced materials. After curing, the specimens were de-moulded and the properties of materials were measured through a set of tests. In the first material that was produced the research was focused on finding optimum synthesis conditions which will create compact, rigid and high compressive strength materials. The parameters that define the synthesis of geopolymers are the solid to liquid (*S*/*L*) ratio and the chemical composition of the strongly alkaline aqueous phase, namely the initial sodium hydroxide and silica concentrations. The effect of the above parameters on the properties of the slag-based geopolymers was studied through three independent series of experiments.

Results and Discussion

The optimum conditions for the synthesis of ferronickel slag-based geopolymers that were experimentally determined in this work were *S*/*L* ratio equal to 5.4 g/mL, initial NaOH concentration in the aqueous phase equal to 7 M, initial SiO_2 concentration in the aqueous phase equal to 4 M and curing in a laboratory oven for 48 h at 60°C. The geopolymers produced under the optimum conditions presented compressive strength equal to 120 MPa, water absorption capacity equal to 0.7% and apparent density equal to 2480 kg/m^3. The other three materials were designed in such a way in order to work as fire resistant materials. The scope of this research was to develop materials which can resist in all standard fire scenarios-curves used for testing the fire-resistance rating of passive fire protection systems. It is already known that when concrete is subjected to excess temperatures causing severe spalling and serious damage to concrete structures with significant economic cost and high potential risk to human life safety are taking place. Although a variety of fire-protection methods exist, there is always a need for the development of new materials with improved thermophysical properties and low cost. Inorganic polymeric materials are promising from this point of view. In order to test the performance of a material under the conditions of the standard fire scenarios, a small sample of each material was thermally treated in the maximum temperature of each scenario for two hours.

The preparation and curing procedures of the slag-based materials are described below. The first material is a sodium activated geopolymer with *S*/*L* ratio equal to 4 g/mL and initial NaOH concentration in the aqueous phase equal to 7 M. The curing of the material took place at ambient conditions for 4 hours. This material resisted till the temperature of 1100°C which means that can fulfill the first two curves (ISO – 834, RABT – ZTV (train)), of the standard fire scenarios. The second material is a potassium activated geopolymer, doped with alumina with *S*/*L* ratio equal to 2.73 g/mL and initial KOH concentration in the aqueous phase equal to 9 M. The curing of the material took place at 50°C for 4 hours. This material resisted till the temperature of 1200°C which means that can reach the first four curves (ISO – 834, RABT – ZTV(train), RABT – ZTV (car), HC) of the standard fire scenarios. The third material is a potassium activated geopolymer doped with higher amount of alumina with *S*/*L* ratio equal to 2.73 g/mL, initial KOH concentration in the aqueous phase equal to 5.85 M. The curing of the material took place at 70°C for 48 hours. This material resisted till the temperature of 1350°C which means that can reach all curves (ISO – 834, RABT – ZTV (train), RABT – ZTV (car), HC, HCM, RWS) of the standard fire scenarios. The syntheses of all the materials and the possible application of each material are shown in Table 2.

Table 2: Syntheses of all materials

	Material a	Material b	Material c	Material d
Slag (%w.w)	79.22	75.76	55.15	35.98
Al_2O_3 (%w.w)			2.03	21.21
SiO_2 (%w.w)	13.04			
KOH (%w.w)			10.67	6.87
NaOH (%w.w)	2.76	5.30		
H_2O (%w.w)	4.98	18.94	32.15	35.94
S/L (g/ml)	5.4	4	2.73	2.73
Curing(°C /h)	(60 /48)	(25/4)	(50/4)	(70/48)
Application	Building material	Fire resistant (2 curves)	Fire resistant (4 curves)	Fire resistant (6 curves)

In Figure 1 is shown the best fire resisting material before and after the exposure at 1350°C. It is clearly observed that the material did not spall and the dimensions kept stable. The results of the laboratory-scale tests proved that the FeNi slag-based inorganic polymer has a good thermal insulating capacity which may render it a promising material for passive fire protection of constructions. Although the results are indicative of its large-scale behaviour, the fire resistance of this external fire protection system as well as

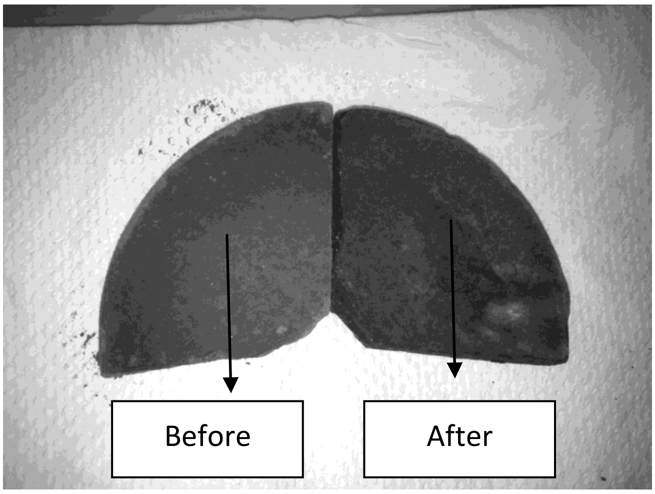

Figure 1: Inorganic polymeric material before and after the thermal exposure

its application and fixing methods have to be demonstrated in large-scale testing including all kinds of mechanical loading before concluding for its effectiveness and suitability for passive fire protection of underground constructions.

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