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Modification of mechanical and thermal properties of fly ash-based geopolymer by the incorporation of steel slag

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ABSTRACT

Geopolymeric binders (GB) were produced using fly ash (FA) and electric arc furnace slag (EAFS). The slag has been added in the range of 0–40%. The effects of slag content on the strength, microstructure and thermal resistance were evaluated. It was found that the amount of EAFS up to 30% positively affects the strength evolution of GB. The main reaction product of FA/EAFS blends was amorphous N–(C)–A–S–H gel along with geopolymer-type gel (N–A–S–H). Thermal resistance of GB was considered from the standpoint of their mechanical and dimensional stability after heating in the temperature interval of 600–800 °C. The changes in mechanical and thermal properties of GB after heating are attributed to the changes in their structure. The results have shown that EAFS negatively affects the thermal resistance of GB above 600 °C due to the phase transition and morphological transformation of the amorphous gel phase.

1. Introduction

The role of FA in the production of GB is a novel way for utilizing by-products from the construction industry, especially for its environmental benefits. In general, geopolymers are produced by the alkali activation of aluminosilicate materials (fly ash and metakaolin) with a highly alkaline activator, which leads to the formation of an amorphous sodium aluminosilicate (N–A–S–H) gel as a reaction product [1]. Alkali activation of slag results in a different reaction product, calcium alumino silica (C–A–S–H) gel [2]. Properties of both, fresh and hardened FA-based GB can be improved by the addition of Ca-rich materials like blast furnace slag, which leads to the formation of an additional C–A–S–H gel, as a result of slag hydration during the alkali activation process [3]. The addition of slag to the FA affects the amount of the reaction product as well as the silicate structure, leading to a highly polymerized structure [4] and thus improving the properties of GB [5].

The aim of this research was to investigate the influence of EAFS addition on the strength and thermal resistance of FA-based GB. Thermal stability of blended binders containing FA and EAFS was through their mechanical and dimensional stability at elevated temperatures.

2. Experiment

The FA and EAFS, with the average grain size (d_{50}) 82 μm and 24 μm, respectively, were used for the synthesis of GB. Total pore volume and BET (Brunauer–Emmett–Teller) surface area of fly ash were 0.042 g/cm^3 and 36 m^2/g while for slag these values were 0.005 g/cm^{3}and 4 m^2/g, respectively. The main oxide constituents of FA were 55.62% SiO_2, 20.11% Al_2O_3, 9.44% CaO, 5.78% Fe_2O_3, 4.32% K_2O, 1.9% MgO, while EAFS comprises of 46.5% CaO, 23.5% FeO, 12.2% SiO_2, 6.5% MgO, and 7.24% Al_2O_3. Four binary mixtures of FA and EAFS (FA/EAFS with mass ratio of 100/0, 80/20, 70/30 and 60/40; denoted as F/S-100/0, F/S-80/20, F/S-70/30, F/S-60/40, respectively) were prepared by mixing known masses of FA or FA/EAFS mixtures with an alkali activator at a solid to a liquid to solid ratio of 0.77 . The alkali activator was prepared by mixing sodium water glass ($SiO_2/Na_2O=3.2$) with 10 M NaOH solution in a mass ratio of 1.5. The SiO_2/Na_2O ratio of final alkali solution was 1.23.

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Geopolymeric paste was cast in a cylindrical mold, cured for 48 h at 65 °C, removed from the mold and left to rest for an additional 4 weeks at ambient temperature. For the thermal treatment the samples were heated to 400, 600 and 800 °C for period of 60 min

The normal consistency of the geopolymer paste was determined by the Vicat apparatus. The GB samples were tested for compressive strength. Measurements of total pore volume and pore size distribution were carried out using N_2 adsorption/desorption isotherms. Microstructural investigations were carried out using the FEI 235 DB focused ion beam system, equipped with the EDAX energy dispersive spectrometer (EDS).

The phase analysis was performed by X-ray powder diffraction (XRPD) technique. XRPD patterns were obtained on a PHILIPS PW 1710 diffractometer using monochromatized CuKα radiation (λ=1.54178 Å) and step-scan mode (2θ range was from 4 to 90°2θ, step 0.02°2θ, time 0.8 s).

The thermal behaviour was studied by simultaneous TG–DTA (Setsys, SETARAM Instrumentation, Caluire, France) in the temperature range from 30 to 900 °C. The samples were placed in an Al_2O_3 pans and heated at a constant heating rate of 15 °C min^{-1} under an air flow (20 cm^3 min^{-1}). Shrinkage data were collected on thermo-mechanical analyzer (TMA, Setsys, SETARAM Instrumentation, Caluire, France) in the same experimental conditions used for TG-DTA study.

3. Results and discussion

The results have shown that compressive strength of FA-based geopolymers can be improved by increasing the EAFS amount up to 30% (Fig. 1(a)). This is attributed to the increase of CaO content in the initial solid mixture, which is known to improve the strength of GB [3]. The inclusion of CaO in a geopolymer mixture by the incorporation of slag, introduces soluble Ca^{2+} ions, which favour the formation of C-A-S-H gel in addition to geopolymer N-A-S-H gel, which leads to the increase in strength of the geopolymer structure [6,7]. In blended fly ash/slag geopolymeric systems, C-A-S-H precipitates act as nucleation sites and promote rapid geopolymerization [8–10] and the formation of a higher amount of the reaction product which occupy pore space and thus lead to the higher strength of GB [3,11]. The sample with the highest strength was the one comprised of 30% FA and 70%EAFS *i.e.* the sample labeled as F/S-70/30. The sample with the EAFS content above 30% exhibited strength decrease.

The strength change with the EAFS addition correlates well with the change of porosity of F/S samples. The values of total pore volume of F/S samples with the slag contents of 0, 20%, 30% and 40% of EAFSS were 0.154; 0.142; 0.135 and 0.149, respectively. The continual pore volume decrease with the slag addition up to 30% is in agreement with the strength increase of F/S samples. However, the EAFS addition in amounts of 20% and 30% has no influence on the pore size. In Fig. 1(b) peaks show that the majority of the pores are about 18 nm in width for samples F/S-100/0, F/S-80/20 and F/S-70/30. The EAFS addition in amounts above 30% shifts the pore size peak to a higher value (23 nm) and induces the increase of total pore volume which is sccompained by the strength decrease of GB, probably due to the excess of water in the starting mixture which was not consumed during the geopolymerization process.

The normal consistency of F/S-100/0, F/S-80/20, F/S-70/30 and F/S-60/40 GB pastes were 43.0%, 41.5%, 40.0% and 36.0%, respectively. These findings indicate that EAFS addition, in amounts of 20% and 30%, to the fly ash leads to the slight decrease of water demand for a standard consistency of GB paste. The highest decrease of normal consistency was observed in a mixture with 40% of slag, which indicates the lowest water demand. The FA is characterized by higher pore volumes and surface areas than EAFS and more liquid was adsorbed on the FA pores than in EAFS during the dissolution process. Thus, with the EAFS replacement for FA, the decrease of the amount of porous FA in FA/EAFS blends is observed, which reduces the water demand for geopolymerization process. Given that the same amount of alkali activator was used in all GB samples it is most likely that, when the EAFS content was above 30%, the excess of water, which was not consumed during the hydration process, caused the increase of porosity and the decrease in strength of GB.

Since the F/S-70/30 sample displayed the highest strength, it was selected for the investigation of thermal resistance of blended FA/EAFS binder. The results presented in Table 1 have shown that EAFS addition has no significant influence on the thermal resistance of FA-based GB during the heating up to 600 °C although above this temperature, EAFS addition negatively affects thermal resistance of GB.

The microstructure of F/S-70/30 sample (Fig. 2(a)) before heating is characterized by the presence of unreacted FA and EAFS (A) embedded in the reaction product of geopolymerization of the FA/EAFS blend (B). The results of EDS analysis of the reaction product (Fig. 2(b)) indicate the formation of N–(C)–A–S–H gel with a low Ca content along with geopolymer-type gel (N–A–S–H), as observed previously [12]. Heating up to 600 °C has no significant influence on the microstructure of the blended FA/EAFS binder (Fig. 2(c) and (d)). After heating to 800 °C (Fig. 2(e)) the development of a highly porous structure was observed which was accompanied by a high strength loss as shown by the mechanical investigation (Fig. 2(e)).

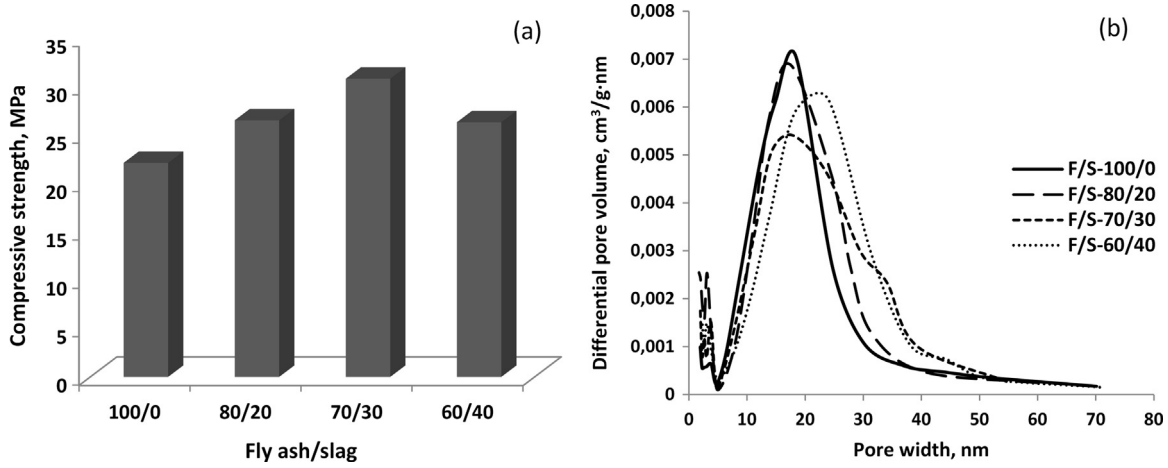

Fig. 1. Compressive strength (a) and pore size distribution (b) of F/S binders with different FA/EAFS.