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Geopolymeric concretes based on fly ash with high unburned content

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HIGHLIGHTS

- Alkali-activated concretes based on fly ash with 21% of unburned material were produced.
- The compressive strength of FA concrete was 20 MPa after 28 days of curing.
- The compressive strength is increased 115% with the addition of 20% GBFS.
- The alkaline-activated concretes FA and FA/GBFS were produced at room temperature (25 °C)

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ABSTRACT

This study used fly ash (FA) with a high amount of unburned content (21%) to produce simple (Geo FA) and binary (Geo FA/granulated blast furnace slag [GBFS]) geopolymeric concretes. The effect on the mechanical strength of the SiO_2/Al_2O_3 and Na_2O/SiO_2 molar ratios and the percentage of GBFS (% GBFS) added to the blend were determined. Using the optimal parameters of alkaline activation, concretes with strengths up to 48 MPa were obtained after 28 days of curing at room temperature (25 °C). The results of this study are complemented by the microstructural characterisation of the geopolymeric pastes using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) techniques.

1. Introduction

Thermal power plants usually use fossil fuels such as coal to produce electricity and generate significant amounts of fly ash (FA), approximately 800 million tonnes worldwide [1]. Because only a small portion of this ash is currently used (20–30%), this waste creates several environmental problems, including air and water pollution as well as pollution mostly due to poor or inadequate disposal. This waste has motivated the search for new and diverse fields of application. Most notable are the recent studies that have used FA as precursors in the production of alkali-activated cements or geopolymers, obtaining materials with similar or even superior cementitious characteristics to those of ordinary Portland cement [2–7]. Importantly, this field of application, in addition to the economic and environmental benefits of using waste, would reduce the use of traditional OPC and thereby contribute to reducing greenhouse gas (CO_2) emissions and lower the consumption of energy and non-renewable natural resources.

The alkaline activation of FA is a process through which the material (FA) is mixed with certain activators (alkaline solutions), and after curing at low temperatures, the material solidifies. In this process, the vitreous phase present in FA is dissolved and transformed into a three-dimensional macromolecular structure [3,8–10]. This process is known as "geopolymerisation", and the result is a cement with suitable mechanical strength. In addition, the material finally obtained has numerous physical properties including thermal stability, hardness, chemical resistance, and high adhesion to different surfaces. The characteristics of FA (e.g., oxide composition, particle size distribution, alkali metal content, vitreous phase content, among others) depend on different factors related to the type of coal used, its particle size, and the proper monitoring of the combustion process. Consequently, FA from different sources has different reactivities that modify the final properties of the geopolymer obtained [11,12]. The significant heterogeneity of FA has made the commercial application of alkaline-activated FA-based cements difficult in certain countries.

In general, the process of FA geopolymerisation requires thermal curing to achieve favourable properties [13–16]. Some studies

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recommend temperatures between 60 °C and 80 °C [17] and curing times of up to 96 h [18], although the quality of FA can be appropriate (less than 1% level of unburned material).

One solution for improving FA quality and avoiding thermal curing might be the incorporation of other materials such as granulated blast furnace slag (GBFS), OPC, or both [19–21]. GBFS is a by-product of iron manufacture, consisting essentially of vitreous and highly reactive SiO_2, Al_2O_3, CaO, and MgO oxides. The blend of GBFS with FA after alkaline activation creates C—S—H gel (C—A—S—H), which improves the fresh and hardened properties of alkali-activated cements based on FA as well as avoids the need for thermal curing [6,22,23]. Shi and Day (1999) determined that the addition of a small amount of GBFS significantly increased the initial strength of FA/GBFS blends activated with NaOH and sodium silicate [23]. Chi and Huang (2013) and Puertas et al. (2000) evaluated the compression strength (CS) of 10% NaOH-activated FA/GBFS blends and reported that the maximum CS is obtained by adding 50% GBFS [24,25]. Nath and Sarker (2014) and Deb et al. (2014) studied the behaviour of FA/GBFS alkali-activated concretes that were cured at room temperature and reported a sufficient workability, suitable setting time, and acceptable mechanical behaviour, with a mechanical strength of up to 60 MPa [26,27]. However, most studies have used FA with a low unburned content (~3%), and in many thermal plants and boilers, ash is produced with an unburned content that can reach up to 30% due to the type of coal, its granulometry, or the inadequate control of combustion parameters.

Therefore, this study evaluated the use of high unburned-content FA (21%) to determine the optimum parameters for its alkaline activation and the effect of the addition of GBFS on mechanical strength, taking into account the SiO_2/Al_2O_3 and Na_2O/SiO_2 molar ratios as well as the slag content in the blend; furthermore, based on optimised systems, this study sought to validate their use in the production of simple (Geo FA) and binary (Geo FA/GBFS) geopolymeric concretes. The study is complemented by the microstructural characterisation of the geopolymeric pastes using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) techniques.

2. Experimental methodology and materials

2.1. Materials

A Colombian FA was used from a boiler located in a paper mill, and GBFS was obtained from a steel factory as raw materials for the production of geopolymer material. The chemical composition of these materials, determined by X-ray fluorescence (XRF) using a Phillips MagiX-Pro PW-2440 spectrometer fitted with a rhodium tube with a maximum power of 4 KW, is included in Table 1. The table shows that approximately 56.5% of the FA was composed of silica, aluminium, and iron oxides as well as a low content of calcium oxide (6.68%); a notably high amount of unburned content (loss on ignition, LOI = 20.67%) that exceeded the standard specification defined in ASTM C618 (6% maximum) as well as a relatively high content of sodium oxides (7.94%) were also found. For its part, GBFS was mainly composed of silica, aluminium, and calcium oxides.

Table 1
Chemical composition of the materials used.

Characteristics, %	FA	GBFS
SiO_2	28.53	31.99
Al_2O_3	19.18	14.54
Fe_2O_3	8.80	1.12
Na_2O	7.94	0.23
CaO	6.68	46.86
S	2.71	0.82
MgO	2.24	1.05
TiO_2	1.62	0.54
Others	1.65	1.03
LOI	20.67	1.82

The crystalline phases identified using XRD in FA were quartz (Q), mullite (M), haematite (H), anhydrite (A), and analcime (An). In GBFS, calcite (C), Q, and Gelenite (G) were observed (Fig. 1). Of these two materials, the greater amorphous phase content in GBFS stood out, represented by a baseline lift (amorphous halo) between 22 and 36° 2Θ. The mean particle sizes D (4.3), determined using the laser granulometry technique with a Mastersizer 2000, were 22.1 μm and 17.8 μm for FA and GBFS, respectively.

A mixture of commercial sodium silicate ($Na_2SiO_3{\cdot}nH_2O$), with the composition by mass of SiO_2: 32.24%, Na_2O: 11.18% and H_2O: 55.85%, and 96.7% pure industrial sodium hydroxide (NaOH) was used as alkali activator. NaOH pellets were dissolved in water and after 30 min was added the silicate solution (SS); the proportion of each one was selected to obtain the molar ratios SiO_2/Al_2O_3 and Na_2O/SiO_2 specified in the mix.

2.2. Experimental design

To statistically analyse the information obtained by this study, a response surface modelling methodology was applied. For the 100% FA geopolymers, two study factors were considered: the molar ratios SiO_2/Al_2O_3 and Na_2O/SiO_2 (Table 2). For the binary geopolymers, FA/GBFS, the Na_2O/SiO_2 ratio and the percentage of GBFS addition (Table 2) were considered. Each of the treatments was performed at random under identical conditions. The response variable in the statistical analysis was the CS at 28 days of curing at room temperature.

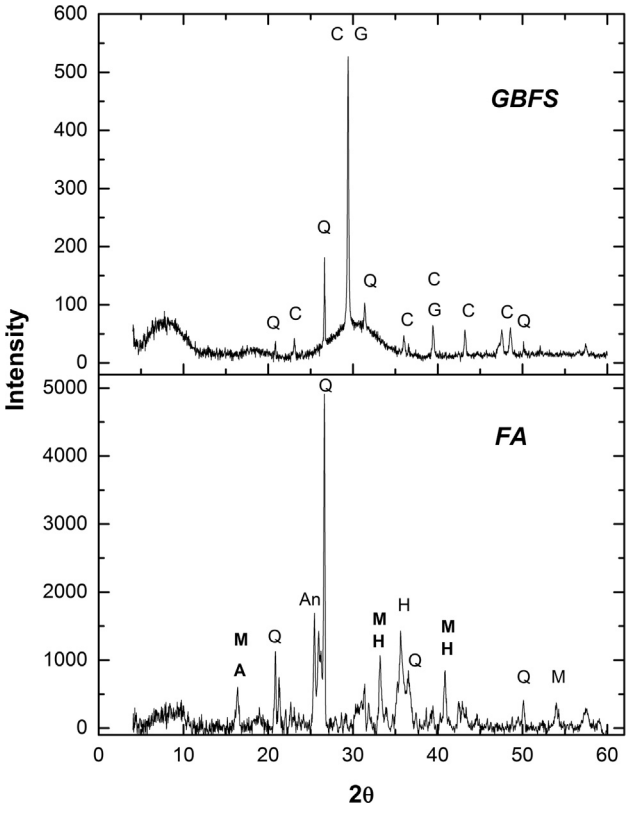

Fig. 1. Mineralogical composition of the raw materials (XRD).