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Performance of blended metakaolin/blastfurnace slag alkali-activated mortars



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ABSTRACT

This paper studies the effect of silicate content on the mechanical and durability-related properties of metakaolin (MK) and metakaolin/blastfurnace slag (BFS) alkaline activated mortars. A reference mortar based on the alkaline activated MK was compared to 60/40 MK/BFS mortars containing different SiO₂/Na₂O molar ratios in the activator. The properties assessed were compressive strength, porosity (water saturation), porosity and pore size distribution by Mercury Intrusion Porosimetry (MIP) and water capillary sorption. The microstructure was assessed using SEM and x-ray computerized micro-tomography (μ -CT). Results show that the addition of BFS significantly alters the microstructure of alkali-activated mortars, promoting a reduction of porosity and capillary sorption. In addition, an optimum SiO₂/Na₂O molar ratio in the activator is required to produce better durability mortars, which however do not necessarily present the highest mechanical strength.

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1. Introduction

Alkali-activated materials (AAM) have been studied worldwide as alternative binders for civil engineering, as well as other industrial applications [1,2]. The production process starts with an aluminosilicate powder or a blend of different materials rich in both SiO₂ and Al₂O₃. These precursors are mixed with a high alkaline solution, usually composed of an alkali metal hydroxide and alkali silicate. The high pH of the activating solution dissolves the solid aluminosilicate source and the resulting paste undergoes a series of chemical reactions that give rise to a solidified material, which hardens and develops mechanical strength comparable to or higher than that of Portland cement-based materials. A good review of the activation process is given by Provis & Bernal [3].

Much attention has been given to the development of AAM in the last two decades, given that some of those materials may offer great advantages in terms of mechanical strength [4]; chemical durability [5–7]; thermal resistance [8] and immobilization of toxic and hazardous wastes [9]. These advantages offer significant

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Another reason for the technical and social interest in AAM is the possibility to significantly reduce the environmental impact when used as construction materials for civil engineering [10]. This is based on the fact that (i) SiO_2 and Al_2O_3 – rich residues and wastes may be used as raw materials, and (ii) the production process of AAM requires much less energy than that of Portland cement, as well as reduces greenhouse gas emissions. This is the subject of current research on life cycle assessment (LCA) of AAM and Portland cement-base materials, although researchers do not necessarily agree on the same LCA figures to confirm the environmental benefits of AAM [11–16].

Two important raw materials for alkali-activation are pulverized fly ash (PFA) and metakaolin (MK). Their choice is based on the fact that they are both amorphous sources of SiO₂ and Al₂O₃ in a single material. The alkaline activation of these and other low-calcium or calcium-free materials produces a matrix composed of a Na₂O–Al₂O₃–SiO₂–H₂O gel (N-A-S-(H)), also described as geopolymer gel. On the other hand, calcium-rich activated materials will also produce a CaO–Al₂O₃–SiO₂–H₂O gel (C-A-S-H), resembling the composition of Portland cement paste (C–S–H gel like structure) [17]. A common example of the latter is the alkaline activation of ground granulated blastfurnace slag (BFS).





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In fact, the activation of two aluminosilicates (one rich in calcium) may present advantages over the activation of a single calcium-free aluminosilicate, such as the acceleration of the condensation reaction (and consequently setting and hardening) [18], better residual strength after high temperature exposure as well as significant improvement in the mechanical strength [19]. The latter results from the coexistence of C-A-S-H and N-A-S-(H) gel in blended activated systems [18,20], with C-A-S-H- gel filling the pores and voids in the geopolymer network, as well as bridging the gaps between different reaction products and unreacted particles [17].

Bernal et al. [19,21,22] have studied several AAM containing both MK and BFS, reporting the benefits of that binary blend. The engineering and durability properties, however, were only addressed for small inclusions of MK (up to 20%) in activated BFS [22].

Higher amounts of MK in MK/BFS activated mortars, as well as lower silicate content in the activator may be of special interest to avoid excessive autogenous and drying shrinkage, which are commonly reported in the activation of BFS with higher silicate contents [23,24]. In addition, the reduction of sodium silicate may bring environmental benefits, given that this activator plays an important role in the CO₂ emissions of AAM [12].

To the authors' knowledge, there is little information in the literature regarding the engineering properties and microstructure changes in alkali-activated MK/BFS with high contents of MK, as a function of the changes in the silicate content in the activator. This paper compares a reference MK geopolymer with MK-BFS activated mortars, with a focus on the microstructure and mechanical and durability-related properties of the different formulations. A single 60/40 ratio between MK and BFS mortar is studied, but different amounts of sodium silicate are employed, thus reducing the overall SiO₂/Na₂O molar ratio of the activator from 1.22 to 0.75.

2. Materials and methods

2.1. Raw materials and formulation of the alkali activated mortars

The alkali-activated matrices studied are based on the activation of metakaolin (MK) and ground granulated blastfurnace slag (BFS) with sodium silicate and sodium hydroxide. MK was supplied by Metacaulim do Brasil Itda, Jundiaí, Brazil, and BFS from Central IBEC, Matozinhos, M.G., Brazil. Table 1 shows the chemical composition (main oxides) for the raw materials, as well as some particle size parameters obtained from LASER particle size distribution (Fig. 1). Fig. 2 shows the XRD patterns for both MK and BFS.

Both aluminosilicates are mostly amorphous; MK contains small peaks of quartz and muscovite, which are normal impurities in kaolins and metakaolins. A small peak at $2\theta \cong 26^{\circ}$ shows that a small fraction of kaolinite did not turn into MK during the calcination. BFS presents crystalline peaks for the mineral gehlenite, which is usually found in pig iron slags. BFS is coarser than MK; so, it is expected that the replacement of MK with BFS affects the

Table 1

Chemical composition and particle size distribution of MK and BFS.

Oxides (%)	МК	BFS
SiO ₂	55.54	34.95
Al ₂ O ₃	44.16	12.63
CaO	0.08	45.10
K ₂ O	0.90	1.24
Na ₂ O	0.05	0.22
Particle size >45 μm	0.02%	11.0%
Particle size <2 μm	31.3%	12.4%







Fig. 2. XRD diffraction patterns for the starting materials, MK and BFS. Q = quartz; K = kaolinite; M = muscovite; Gn = gehlenite.

workability of the MK/BFS activated mortars, which has been also observed by Bignozzi et al. [25].

The activation solution used in this work was composed of 40% vol. NaOH solution (14.6 M) mixed with sodium silicate (Na₂SiO₃, SiO₂ = 28.5% Na₂O = 8.70%; H₂O = 62.8%; Ms ~ 3.3). The ratio between the silicate and hydroxide solutions was such that the activating parameters (molar ratios) documented in other research could be achieved [19,26–29], thus enabling the production of alkali-activated concretes for structural applications.

Table 2 shows their mix design (in mass, per 1 kg of binder), as well as the activating parameters (molar ratios) of the formulations studied. The reference formulation (REF) is a plain MK-based geopolymer; its high solution to binder ratio (s/b) is a consequence of the high fineness and particle shape of MK [30]. Borges et al. [29] studied the activation of the same MK and found that MK-geopolymers with good mechanical strength (28–46 MPa) were produced with s/b ratio ranging from 1.3 to 1.5; the best results,

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