



# Effect of the activator dose on the compressive strength and accelerated carbonation resistance of alkali silicate-activated slag/metakaolin blended materials



Susan A. Bernal

Department of Materials Science and Engineering, The University of Sheffield, Sir Robert Hadfield Building, Mappin St, Sheffield S1 3JD, United Kingdom

## HIGHLIGHTS

- The concentration of activator influences the kinetic of reaction of slag.
- The addition of MK to AAS increases the total setting time.
- At high activator concentrations addition of MK increased strength.
- At high activator concentrations addition of MK reduced accelerated carbonation.

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## ABSTRACT

The effects of activator dose on the properties of alkali-activated slag/metakaolin blends, were studied in fresh and hardened states: heat evolution, strength and accelerated carbonation. High activator concentrations affect the slag dissolution rate, reducing compressive strength when this is the sole precursor. An increased activator concentration favours metakaolin reaction, promoting high strengths and reduced permeability. Metakaolin addition, and increased activator concentrations reduce the susceptibility to carbonation, associated with the refinement of the pore network under extended CO<sub>2</sub> exposure. The effect of adding an aluminosilicate precursor to an alkali-activated slag system is strongly dependent on the activator concentration.

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## 1. Introduction

Alkali-activation technology is attracting the attention of industry and academy as a suitable alternative for valorisation of large streams of industrial wastes and by-products, for the production of environmentally friendly cementitious materials. Alkali-activated binders can develop advantageous properties for application in the construction sector, such as high mechanical strength at early times of curing, high resistance to acid attack, and high performance when exposed to elevated temperatures, depending on the nature and dose of the precursor and activator used, and the curing conditions adopted [1–3]. The main raw materials typically used as precursors for alkali-activation are those which are also used as supplementary cementitious materials (SCMs) in Portland cement blends, such as blast furnace slag from the iron making industry, fly ashes from coal combustion, and thermally

treated clays such as metakaolin [4,5]. The nature of these raw materials is highly variable from source to source, and therefore, the production of alkali-activated binders requires higher quality control than conventional Portland cements, in order to develop specific desired properties.

Alkali-activated slag binders can develop high mechanical strengths at early times of curing, with a lower permeability than identified in Portland cements [6]; however, blast furnace slags are already extensively exploited by the construction industry for the production of blended Portland cements. Consequently, in some parts of the world different precursors are needed for the production of alkali-activated binders. As a potential solution, the development of alkali-activated binders using blends of two or more precursors has been carried out over the past decades, including blended systems of fly ash/slag [7–10], fly ash/metakaolin [11] and slag/metakaolin [12–16]. These binders usually present improved properties compared to systems where the aluminosilicate precursors are activated alone, with a microstructure including co-existing Ca-rich and Na-rich reaction products,

E-mail address: [s.bernal@sheffield.ac.uk](mailto:s.bernal@sheffield.ac.uk)

depending on the fraction of the Ca-rich precursor incorporated in the blend.

There is not yet a standardised methodology for dosing the alkali activator when producing alkali-activated binders. This is a critical factor controlling the properties of these materials, and in the case of blended activated systems it is essential to consider the differences in chemistry of slag and aluminosilicate precursors, and to control the kinetics of dissolution and promote the reaction of each component of the blend activated binder. Typically, when a new precursor or blend is going to be activated, preliminary studies are carried out to identify the amount of activator that allows the production of a workable binder, with moderate initial setting time, that develops compressive strengths within a desirable range, as specified by the needs of the application of the final product. This means that the activator dose varies from study to study, and therefore there is not a good understanding of the role of the concentration of activation in the microstructural development or durability of alkali-activated materials.

Carbonation of cementitious materials is understood as the chemical reaction taking place between the hydration products composing the binders and the CO<sub>2</sub> from the atmosphere, leading to the formation of carbonates. This has been identified as one of the potential disadvantages of alkali-activated binders, compared with Portland cement, as the earlier studies assessing the susceptibility of degradation of these cements, via acceleration carbonation tests, showed higher potential to develop carbonation problems than conventional Portland cement [17–19]. Recent studies have demonstrated that the mechanism of carbonation in alkali-activated binders is strongly dependent on the type of the precursor used (aluminosilicates or granulated blast furnace slag) [9,20,21], the nature of the activator [17] and the accelerated carbonation testing conditions such as relative humidity [22], and CO<sub>2</sub> concentration [23]. Therefore, the general statement that alkali-activated materials will carbonate more than Portland cement is inaccurate, as there are too many variables controlling how and when carbonation of these binders is going to occur, and limited correlation has been identified between natural and accelerated carbonation results for alkali-activated slag materials [23].

In order to gain a better understanding of the effect of the alkali concentration on the fresh paste properties of alkali-activated slag/metakaolin blends, Vicat testing and isothermal calorimetry were carried out in this study. Compressive strength evolution of the pastes was also tested. Mortars were produced with selected paste formulations, and their resistance to accelerated carbonation was evaluated after 28 days of curing.

## 2. Experimental programme

### 2.1. Materials

The primary raw material used in this study was a granulated blast furnace slag (GBFS; Table 1) with a basicity coefficient ( $K_b = \text{CaO} + \text{MgO}/\text{SiO}_2 + \text{Al}_2\text{O}_3$ ) and quality coefficient ( $\text{CaO} + \text{MgO} + \text{Al}_2\text{O}_3/\text{SiO}_2 + \text{TiO}_2$ ) of 1.01 and 1.92, respectively. Its specific gravity was 2900 kg/m<sup>3</sup> and Blaine fineness 399 m<sup>2</sup>/kg. The particle size range, determined through laser granulometry, was 0.1–74 μm, with a  $d_{50}$  of 15 μm.

The metakaolin (MK) used was generated in the laboratory by calcination of a kaolin containing minor quartz and dickite impurities. Calcination was carried out at 700 °C in an air atmosphere, for 2 h. The particle size range of the metakaolin

**Table 1**  
Composition of the GBFS and MK used, from X-ray fluorescence analysis. LOI is loss on ignition at 1000 °C.

Precursor	Component (mass% as oxide)						
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Other	LOI
GBFS	32.3	16.3	42.5	2.4	2.9	1.7	1.9
MK	50.7	44.6	2.7	–	–	1.0	1.0

was 1.8–100 μm, with a  $d_{50}$  of 13.2 μm and  $d_{10}$  of 4 μm, and Blaine fineness 391 m<sup>2</sup>/kg. Alkaline activating solutions were formulated by blending a commercial sodium silicate solution with 32.4 wt.% SiO<sub>2</sub>, 13.5 wt.% Na<sub>2</sub>O and 54.1 wt.% H<sub>2</sub>O, together with 50 wt.% NaOH solution, to reach overall desired molar ratios (SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> (S/A) and Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub>).

### 2.2. Sample synthesis and test procedures

#### 2.2.1. Pastes

Pastes formulated with an overall (solid fraction in the activator + solid precursor) SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> (S/A) molar ratios of 3.6, 4.0 and 4.4, GBFS/(GBFS + MK) ratios of 0.8 (20 wt.% MK), 0.9 (10 wt.% MK) and 1.0 (0 wt.% MK), with a constant water/(GBFS + MK + solid fraction in the activator) ratio of 0.23, were produced in accordance with the standard procedure ASTM C305-06 [24]. The water/solid ratio of these pastes was determined accordingly to the procedure ASTM C187 [25]. The relationship between the overall oxide ratios and the concentration of the activator (expressed as Na<sub>2</sub>O wt.% relative to the amount of precursor to activate), is presented in Table 2. The modulus of solution ( $M_s = \text{molar ratio SiO}_2/\text{Na}_2\text{O}$ ) of the activators used is between 0.9 and 1.5.

These activation concentrations are considered high for the sole activation of slag, taking into account that the conventional concentrations of activation of slag with sodium silicate solution are usually between 3 wt.% and 7 wt.% Na<sub>2</sub>O [26]; however, production of concretes with these formulations has been achieved [27,28], which motivates the detailed understanding of the structure developed in these materials.

In fresh pastes, setting time was determined using the Vicat apparatus by following the standard procedure ASTM C191-08 [29]. The setting process of these mixes was also assessed by isothermal calorimetry (JAF calorimeter) at 25 °C, over the first 40 h of reaction. Fresh paste was mixed externally (~40 g of total mix), weighed into polystyrene vessels, and immediately placed in the calorimeter.

For compressive strength testing, five specimens were cast in a cylindrical mould (40 mm height and 20 mm diameter) and stored in hermetic containers at a relative humidity of 90% and a temperature of 27 ± 2 °C for 1, 7, 28, 56 and 180 days.

#### 2.2.2. Mortars

Mortars were produced with similar formulations to the pastes (Table 2), following the standard procedure ASTM C305-06 [24]. River sand with a specific gravity, absorption and fineness modulus of 2450 kg/m<sup>3</sup>, 3.75% and 2.57 was used as fine aggregate. All samples were formulated with a constant water/(slag + metakaolin + solid fraction in the activator) ratio of 0.47 and a binder/sand ratio of 1:2.75. This water/solids ratio is significantly higher than the used for producing pastes, however, it was selected in order to replicate the content of water used for producing concretes with similar binder formulations [27]. The specimens were cast in cubic moulds with dimensions of 50.8 × 50.8 mm, and stored under controlled humidity (relative humidity (RH) ~85%) and ambient temperature (~25 °C) for 24 h. Samples were then demoulded and cured under RH of 90% and a temperature of 27 ± 2 °C for 28 days.

### 2.3. Accelerated carbonation

After 28 days of curing the mortar specimens were removed from the humidity chamber, dried at 60 °C for 24 h, and then the top ends of the specimens were covered using an acrylic resin (Acronal <sup>®</sup>), applying a minimum of 4 layers, to direct the ingress of CO<sub>2</sub> through the selected faces of the cubes during testing. Samples were then transferred to the carbonation chamber for CO<sub>2</sub> exposure, without application of an intermediate drying or conditioning step. This was done to minimise the potential microcracking and differences in sample maturity, which would be observed if they were conditioned for extended periods at the testing relative

**Table 2**  
Equivalence between overall oxide ratios (precursor + activator) and activation concentration (% Na<sub>2</sub>O by mass of GBFS + MK) used for the preparation of the pastes assessed.

GBFS/(GBFS + MK)	Overall SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> ratio	Activation concentration (wt.% Na <sub>2</sub> O)
1.0	4.4	10.6
	4.0	9.9
	3.6	9.1
0.9	4.4	12.5
	4.0	11.6
	3.6	10.5
0.8	4.4	14.5
	4.0	13.0
	3.6	12.0

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