



# Chemical acceleration of a neutral granulated blast-furnace slag activated by sodium carbonate



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## ABSTRACT

This paper presents results of a study on chemical acceleration of a neutral granulated blast-furnace slag activated using sodium carbonate. As strength development of alkali-activated slag cements containing neutral GBFS and sodium carbonate as activator at room temperature is known to be slow, three accelerators were investigated: sodium hydroxide, ordinary Portland cement and a combination of silica fume and slaked lime. In all cements, the main hydration product is C-(A)-S-H, but its structure varies between tobermorite and riversideite depending on the accelerator used. Calcite and gaylussite are present in all systems and they were formed due to either cation exchange reaction between the slag and the activator, or carbonation. With accelerators, compressive strength up to 15 MPa can be achieved within 24 h in comparison to 2.5 MPa after 48 h for a mix without an accelerator.

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## 1. Introduction

Alkali-activated slag (AAS) concretes are considered as a possible alternative to ordinary Portland cement (OPC) concretes. There are four main activators usually used to activate slags, namely sodium silicates, sodium hydroxide, sodium sulfate, and sodium carbonate.

Sodium silicates provide the highest strength [1–4] and chemical resistance [5], but their usage is associated with high shrinkage [1,4,6,7], challenging rheology of fresh concrete mix, relatively high cost of the concretes and high amounts of associated CO<sub>2</sub> emissions [8]. Sodium silicates and their combinations with sodium hydroxide provide fast setting, resulting in limited time for casting and finishing of AAS concrete [7].

Sodium hydroxide is another commonly used abundant alkali, but its usage for slag activation provides relatively low strength [2,3], high porosity and coarse pore structure [9,10]. Sodium hydroxide solution has the highest pH value amongst the activators. It is therefore highly corrosive, and safety during production of AAS concretes becomes an issue.

Sodium sulfate is the cheapest amongst the four activators used for AAS concretes but it provides the lowest strength, especially at an early age, and poor resistance to frost and carbonation [2].

Sodium carbonate provides high strength, good durability, long setting times and the lowest shrinkage [1,4,11,12]. Sodium carbonate solution is much less corrosive than sodium silicate/hydroxide, making for safer work environments. However, a major shortcoming of sodium

carbonate as activator, is the slow strength development of AAS concretes containing acid or neutral slags hardened at ambient temperature [13,14].

Recent research on AAS concretes has mainly focused on sodium silicate and sodium hydroxide activators and their combinations [10,15,16]. Thus, literature on AAS concretes containing sodium carbonate as the main activator is limited, especially on acceleration of strength development of AAS concretes containing acid or neutral slags hardened at ambient temperature.

The aim of this research is to add knowledge on AAS concretes containing sodium carbonate and neutral GBFS. Three accelerating admixtures were used in this study, and their effect on hydration products and strength development was investigated.

## 2. Experimental setup

### 2.1. Materials

A granulated blast-furnace slag was used to produce AAS concretes. The chemical composition of the slag is presented in Table 1. The GBFS was a neutral slag with a basicity coefficient ( $K_b = (\text{CaO} + \text{MgO}) / (\text{Al}_2\text{O}_3 + \text{SiO}_2)$ ) of 0.92 based on the chemical composition as indicated in Table 1. X-ray diffraction analysis shows that the slag is a glassy phase with minor inclusions of gehlenite. The particle size range of slag, determined by laser granulometry, was 0.4–152 μm, with a mean ( $d_{50}$ ) of 16.8 μm and 10% of particles finer than 1.8 μm.

Commercially available sodium carbonate (99.0% purity) was used to prepare the activator solutions. Tap and distilled water was used to prepare alkaline solutions for AAS concretes and pastes respectively.

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**Table 1**  
Chemical composition and physical characteristics of used materials.

Material	Chemical composition, %										Physical characteristics	
	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>	LOI, %	Blaine, m <sup>2</sup> /kg	Relative density
Slag	34.87	0.72	14.38	0.89	8.03	37.05	<0.01	0.72	1.96	0.16	425	2.90
Cem I 52.5N	22.30	0.50	4.98	3.35	1.92	61.50	0.21	0.26	2.92	1.77	480	3.17
Silica fume	84.00	0.03	0.75	1.96	1.08	2.22	0.17	3.43	0.09	5.56	n.a.	2.30
Slaked lime	4.01	0.03	0.37	0.36	1.67	67.80	0.00	0.08	0.11	25.0	1060	2.33

Sodium hydroxide flakes (98.5% purity), OPC CEM I 52.5N, a combination of silica fume and slaked lime were used as admixtures to promote early strength development of AAS concretes at room temperature. The chemical composition and physical characteristics of the materials are shown in Table 1. The particle size distribution of CEM I 52.5N was 0.4–93 μm, with a mean (d<sub>50</sub>) of 16.4 μm and 10% of particles finer than 3.5 μm.

Crushed dolomite stone and dolomite sand were used as aggregates. The relative density of the aggregates was 2.86. The maximum size of coarse aggregate was 9.5 mm. The sand had a fineness modulus of 3.86 with 9% particles smaller than 75 μm.

## 2.2. Mix designs

Based on the results of previous research indicating that it provided good durability [15] and compressive strength [16], all AAS concrete mixes (Table 2) contained 500 kg/m<sup>3</sup> of binder (slag + OPC + silica fume + slaked lime).

The coarse to fine aggregate weight ratio of 65:35 and water to binder ratio of 0.35 were kept constant in all mixes.

## 2.3. Sample synthesis and test procedures

Setting times were measured according to BS EN 196-3:2005 [17] in a temperature and relative humidity controlled room at 25 °C and 97 ± 2%. The water/binder ratio was kept constant at 0.26 [18] and the moulds filled with AAS paste were not submerged into water between measurements to avoid alkali leaching.

The concrete preparation procedure consisted of mixing dry materials in a pan mixer, the addition of aqueous activator solution, and a further one and a half minute of mixing. Workability (slump, Vebe test) was tested following the standard procedure of BS EN 12350:2009 [19], before placing the fresh concrete mix in 100 mm plastic cube moulds. Compaction was done by vibration. Cast samples were cured at 25 °C and >90% humidity for the first 24 h. Samples of the control mix without accelerating admixtures (SC, Table 2) were cured for 48 h as they could not be de-moulded at 24 h due to slow strength development. After de-moulding, all samples were stored in a room with constant conditions: 25 °C and 55% relative humidity. Concrete samples were tested for compressive strength according to the standard procedure BS EN 12390-3 [20] at 1, 28, and 91 days.

**Table 2**  
AAS concrete mix designs and properties.

Mix code	Components, kg/m <sup>3</sup>							Initial set, min	Final set, min	Slump: mm/Vebe: s
	Slag	Cem I 52.5N	Silica fume	Slaked lime	Stone	Sand	Activator <sup>a</sup>			
SC	500				1200	650	3.5/0	455	>600	10/11
SH20	500				1200	650	2.8/0.9	125	195	10/11
SH50	500				1200	650	1.8/2.3	155	330	5/12
SH100	500				1200	650	0/4.7	90	175	5/12
OPC3	485	15			1203	651	4.6/0	55	120	0/12
OPC4	480	20			1203	651	4.6/0	40	105	0/12
OPC5	475	25			1203	651	4.7/0	40	90	0/15
SFSL	470		15	15	1196	649	4.5/0	45	165	0/18

<sup>a</sup> Sodium carbonate/sodium hydroxide in Na<sub>2</sub>O wt.% of slag.

Analysis of pore solution chemistry was done on pore liquid expressed from 100 ml paste samples. AAS paste mixes were obtained by excluding aggregates from corresponding concrete mix designs (Table 2). The paste samples were cast into plastic bottles and cured under controlled conditions at 20 ± 1 °C. The expression of pore solution from hardened AAS pastes was performed using a device similar to one described by Barneyback and Diamond [21]. Elemental composition of the pore solutions was studied by a Spectro-Across Inductively-Coupled Plasma Optical Emission Spectroscopy (ICP-OES) instrument.

AAS pastes were prepared for mineralogical and microstructural characterization with X-ray Diffractometry (XRD), Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR), Scanning Electron Microscopy (SEM), Electron Backscattered Scanning Electron Microscopy (BSE) and Energy Dispersive Spectroscopy (EDS). Paste samples were cured at 25 °C and >90% humidity for the first 24 h and at 25 °C and 55% relative humidity afterwards.

XRD analysis was done at 1 and 28 days. Paste samples were crushed into small pieces with a maximum size of 5 mm and submerged into ethanol to prevent further hydration. Crushed samples were then dried at 50 °C for 12 h and milled in a tungsten carbide mill. The powdered samples were analysed using a PANalytical X'Pert Pro powder diffractometer in θ–θ configuration with an X'Celerator detector and variable divergence and receiving slits with Fe filtered Co-Kα radiation (λ = 1.789 Å). The specimens were collected in the 2θ range 5–90° with a scanning rate of 17°/s and a step size of 0.017° 2θ. The phases were identified using X'Pert Highscore plus software.

Samples of pastes for SEM investigation were crushed at 1 and 28 days, washed with ethanol, and placed into a desiccator. Freshly fractured surfaces were coated with carbon and studied using a Zeiss Ultra Plus SEM at an accelerating voltage of 1 kV.

BSE/EDS studies were carried out on 1-day pastes. Samples were submerged into ethanol for 24 h to remove the capillary water, dried in a desiccator before epoxy resin impregnation in vacuum. Samples were ground and polished using diamond pads (66, 28.8 and 16 μm) and diamond suspensions (9, 3, 1 and 0.25 μm). Polished sections were coated with a single carbon layer to provide a layer of minimal thickness which would be undetectable during EDS scanning. A JEOL JSM 5800 scanning electron microscope (20.0 kV) fitted with a solid-state BSE detector and a Thermo Scientific NSS7 energy dispersive X-ray analyser (EDS) was used for the study.

Samples for ATR-FTIR analysis were prepared as for XRD. Infrared spectra were recorded using a Golden Gate diamond ATR cell (Bruker),

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