



Alkali-activated slag concrete: Fresh and hardened behaviour



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ABSTRACT

The behaviour of fresh and hardened alkali-activated slag (AAS) and OPC concretes was compared and the effect of mixing time assessed. OPC and AAS concrete slump and rheological results proved to differ, particularly when the slag was activated with waterglass (WG). The nature of the alkaline activator was the key determinant in AAS concrete rheology. Bingham models afforded a good fit to all the OPC and AAS concretes. In OPC and NaOH-activated AAS concretes, longer mixing had an adverse effect on rheology while improving hardened performance only slightly. In WG-AAS concrete, longer mixing times, improved mechanical properties and also rheological behaviour was enhanced, in which those conditions were required to break down the microstructure. Longer mixing raised thixotropy in OPC and NaOH-activated AAS concretes, but lowered the value of this parameter in waterglass-activated slag concrete.

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1. Introduction

For over 20 years, the study and development of alkaline activated materials (AAMs), mainly cements and concretes has been the subject of great interest by the scientific community [1–6]. That is largely due to the enormous promise of these products as sustainable alternatives to portland cement-based binders. AAMs are natural or artificial aluminosilicates activated by hydroxide-, silicate- or alkaline carbonate-solutions. Their manufacture not only generates lower GHG (especially CO₂) emissions, but is more economically and environmentally sustainable, for the raw materials or precursors used are often valorised industrial by-products [7–10]. The manufacture of some alkaline activators, sodium silicate or waterglass (WG) especially, also carries adverse environmental impacts, however [11]. The production of possible alternative activators by processing different types of waste (such as glass or rice husk ash) would consequently constitute a further step toward sustainability [12–15] in new binder development.

Alkali-activated slag (AAS) cements and concretes are among the most prominent AAMs, for their manufacture emits less GHGs than portland cement and consumes less energy and water [16].

These AAS systems are also characterised by high mechanical strength and durability [17–22]. Nonetheless AAS cement, mortar and concretes exhibit two shortcomings that hamper their standardisation and more general use, namely high drying shrinkage [2,23–25] and quick setting when activated with waterglass solutions [26–28].

The reason for such quick setting lies in the initial and speedy formation of a primary C-S-H gel, which hardens sodium silica-activated AAS pastes. Shi and Day [29] used isothermal conduction calorimetry to study slag activation with different activators. They observed two contiguous signals in the pre-induction period in sodium silicate-activated pastes. The first was attributed to slag dissolution and the second to the formation of a primary C-S-H gel as a result of the presence of [SiO₄]⁴⁻ ions in the solution. This latter signal intensified as the WG silica modulus rose. Primary C-S-H formation was also observed in constant shear rate rheology tests [28,30] on waterglass-activated AAS pastes. After an initial rise, shear declined and flattened, attendant upon paste deflocculation possibly attributable to primary C-S-H flocks gel breakdown. Lengthening mixing time from 3 min to 10 min was also shown to retard initial setting by 40 min and final setting by over 4 h. At a mixing time of 30 min, initial and final settings were also delayed [26].

More recently, the intensity and timing of this rheological signal at constant shear rate in WG-activated AAS pastes have been shown

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to depend on the Na₂O concentration and the SiO₂/Na₂O modulus in the activating solution used. The higher the latter and consequently the greater the silicon content in the solution, the earlier and more intense the signal [31].

Such rapid setting obviously affects rheology and therefore on-site paste and concrete casting. As the literature shows [28,30,31], rheological studies contribute to an understanding of the reasons for such behaviour and the pursuit of possible solutions. Few studies have focused on researching sodium silicate-activated AAS system rheology, however.

Palacios et al. [28,30] found that NaOH-activated AAS pastes fit a Bingham model, whereas WG-activated materials conform to the Herschel-Bulkley model. Studies comparing OPC and AAS rheology based on minislump test-determined workability [31,32] showed that WG-activated pastes were less workable than NaOH-activated and OPC pastes. Kashani et al. [33], studying hydroxide- and alkaline silicate-activated AAS paste rheology, found that the latter had lower initial shear stress than the former due to the adsorption of silicate ions on the particles, which had a fluidising effect as a result of the increase in the double electrical layer. When these results were studied in conjunction with isothermal conduction calorimetry findings on degree of reaction, the times at which yield stress rose were found to be correlated to the heat release pattern. Those data were consistent with the results reported by Varga et al. [31], where initial yield stress was observed to be lower in WG- than in NaOH-activated AAS pastes. Unlike Varga et al., however, Kashani et al. [33] observed that yield stress was lower in the first 20 min in WG- than in alkaline hydroxide-activated AAS pastes. Different authors may on occasion report contradictory findings due to variations in the activators used and paste mixing history, as well as in the specific rheological study conducted.

Fluidity loss has been shown to be greater in sodium silicate-activated AAS mortars than in OPC and NaOH-activated AAS mortars [2,32]. Waterglass-activated AAS mortars have also been found to exhibit lower workability and greater fluidity loss than alkali-activated fly ash mortars [34,35].

Sight should not be lost of the fact that the end product is concrete, however. An understanding of its workability and rheology is consequently of cardinal importance [36,37]. Empirical findings such as yielded by the slump test, V-funnel or J-ring [38] not only furnish very valuable information on concrete workability, but can be readily and economically conducted. As operator-sensitive, single-point tests [39,40], they may nonetheless find two very different concretes to be in the same workability class. AAS concrete systems should therefore be studied with multi-point tests that furnish information on rheological parameters such as yield stress, plastic viscosity and information about thixotropy [39–42].

Portland cement concrete rheology has been exhaustively studied and found to fit a Bingham model (Equation (1)):

$$\tau = \tau_0 + \mu \cdot \dot{\gamma} \quad (1)$$

where $\dot{\gamma}$ is shear rate (s⁻¹), τ_0 is yield stress (an indication of the force needed to initiate flow) and μ is plastic viscosity (Pa·s) or the material's resistance to flow [40,43]. OPC thixotropy has also been explored by many authors [44–46]. Thixotropy is the property of these systems whereby viscosity gradually declines with rising shear rate and the initial structure is recovered when shear is removed [40,47]. It should not be confounded with the irreversible 'structural breakdown' in cement pastes defined by Tattersall and Banfill [40].

Any number of studies have shown that portland cement concrete rheology, thixotropy and workability vary with the presence of additions [48–50], the presence and composition of

superplasticisers [51–53] and the composition and particle size distribution of the aggregates [54–57].

In contrast, rheological studies on alkali-activated concretes are scant to non-existent. The addition of ultrafine fly ash [58] or lime slurry [59] has been observed to improve AAS concrete workability (lower lost of slump). No information has yet been forthcoming on AAS concrete rheological parameters, however. More specifically, no studies comparing NaOH- or sodium silicate-activated AAS to OPC concrete rheology have been published to date. As noted, moreover, as longer mixing times may induce initial breakdown of the primary C-S-H gel in WG-AAS systems, improving the workability as has been proven in previous studies on AAS paste and mortars [31,60]. The motivation of this study was to know the effect of longer mixing time of AAS concrete rheology with different alkaline activators (NaOH and Wg) and to compare the results with OPC concretes. Mixing time must be factored into experimental designs to determine NaOH- and WG-activated AAS concrete workability and rheology.

2. Experimental procedure

2.1. Materials

CEM I 52.5 R portland cement (OPC) and ground-granulated blast furnace slag (S) were used as binder to prepare the concretes studied. The chemical composition of these materials was listed in Table 1 and determined by a PHILIPS PW-1004 X-Ray Fluorescence (XRF) spectrometer, together with the loss on ignition (found further to European standard EN-196-2:2014). The ground-granulated blast-furnace slag used in this study had a Blaine specific surface of 346 m²/kg and a specific gravity of 2880 kg/m³. The cement used, the chemical composition for which is given in Table 1, had a Blaine specific surface of 420 m²/kg and a density of 3100 kg/m³. The rolled sand fines had a specific gravity of 2600 kg/m³ and an absorption of 0.23%, both coarse siliceous aggregates had a specific gravity of 2640 kg/m³, with 0.36% absorption in the 4–8 mm material and 0.40% absorption in the 8–12 mm gravel (Table 2). The particle size distribution of the coarse aggregates is shown in Fig. 1.

2.2. Mixture proportions and mixing protocols

The blast furnace slag and cement concretes were designed to a total content of 357 kg/m³. The OPC concrete was mixed with water and 0.1% (cement weight) chemical admixture, whereas the alkali-activated slag concretes were mixed with one of two solutions (Table 3):

- Panreac 98% pure, 10 M NaOH (5% Na₂O by slag mass)
- Sodium silicate hydrate or waterglass (5% Na₂O by slag mass, SiO₂/Na₂O modulus of 1.2), prepared with Merck sodium silicate (SiO₂: 27 wt%; Na₂O: 8 wt%; H₂O: 65 wt%).

A slump of 120 mm or larger was sought in all cases.

Two mixing protocols were deployed to prepare both the OPCC and AASC systems (see Fig. 2). Protocol 1 corresponds to AAS concrete dosage previously determine in RILEM TC 247-DTA (the stability and good mechanical strenght behaviour were the main factors considered). In protocol 2 a longer mixing time of all concrete components were considered according to previous rheological studies [26,31,60].

In the first (protocol 1), the aggregates were blended for 2 min in the mixer with 1.65% of the water. The aggregates were then allowed to stand for 8 min to absorb the mixing water. The binder (portland cement or ground-granulated blast furnace slag) were

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