



Rheology of alkali-activated slag pastes. Effect of the nature and concentration of the activating solution



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ABSTRACT

An understanding of the rheological behaviour of OPC-based products has been widely studied, for it is essential to determining and predicting the fresh and hardened characteristics and properties of pastes, mortars and concretes. The rheology of alkali-activated material (AAM) systems has been much less intensely researched, however.

The present study aimed to ascertain the effect of factors such as the nature and concentration of the alkaline activator on the rheological behaviour of alkali-activated slag (AAS) pastes, with a comparison between the rheological parameters and fluidity of these pastes to the same parameters in OPC. More specifically, the study explored how paste rheology was affected by the nature of the alkaline activator (NaOH, 50/50 wt% NaOH/Na₂CO₃ or waterglass – Wg), its concentration (3–5% Na₂CO₃ of slag weight) and, in the waterglass solution, the SiO₂/Na₂O ratio.

The findings showed that AAS paste rheology is affected by the nature of the activator. The rheological behaviour in AAS pastes activated with NaOH alone or combined with Na₂CO₃ was similar to the rheology observed in OPC pastes, and fit the Bingham model. Conversely, the AAS pastes activated with waterglass fit the Herschel–Bulkley model and their rheology proved to depend on both the SiO₂/Na₂O ratio and the Na₂O concentration. Moreover, regardless of the activator used (NaOH, Na₂CO₃ or waterglass), an increase in Na₂O concentration implies a raise of shear stress.

The formation of primary C–S–H gel in Wg–AAS and its effect on paste rheology were confirmed. Gel formation was likewise shown to be related to the SiO₂/Na₂O ratio and activator concentration.

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1. Introduction

Rheology is the study of the fluidity and deformation of matter. An understanding of the rheological properties of cement pastes, mortars and concretes is requisite to ascertaining their consistency and workability and consequently their ease of casting or placement [1]. Cement system rheology also affects the microstructure and with it the mechanical behaviour and durability of the material [2].

A number of methods are in place for determining the fluidity of binders, including slump tests, the compactness factor and the Vebe consistometer. The advantages of these primarily empirical procedures are their low cost, on-site applicability and simplicity. As single measurement, single result tests, however, they may lead to erroneous conclusions, such as assuming that the same slump value indicates similarity between two pastes that may in fact be different. Another drawback to these methods is their high

sensitivity to the tester and test preparations. Methods able to characterise the rheology of these materials on the grounds of physical parameters are therefore required. Cement system fluidity can be determined from physical parameters such as viscosity or yield stress measured with viscometers or rheometers. Many authors have studied Portland cement paste rheology as a preliminary to determining the rheology of the respective mortars and concretes [3–9].

Rheological testing on Portland cement pastes, normally conducted with viscometers, consists of subjecting the pastes to shear at a given rate to break down the flocs formed mainly due to the hydration of cement grains. The shear rate (rotor speed) is then gradually lowered. As a rule, the down ramp on the shear rate vs shear stress (τ) curve is a straight line that fits the Bingham model (Eq. (1)): i.e., where the slope is the plastic viscosity (μ) and the y-intercept the yield stress (τ_0). Plastic viscosity is related to the number and size of the flocs, while the yield stress is a measure of the strength and number of inter-floc interrelations that are broken down when shear is applied [5].

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

The literature [1] nonetheless describes other models for cement paste rheological behaviour, such as the Ostwald de Waele and Herschel–Bulkley models (Eqs. (2) and (3), respectively).

$$\text{Ostwald de Waele model : } \tau = K \dot{\gamma}^n \quad (2)$$

$$\text{Herschel – Bulkley model : } \tau = \tau_0 + K \dot{\gamma}^n \quad (3)$$

where k is the consistency coefficient ($\text{Pa}\cdot\text{s}^n$) and n is the dimensionless fluidity index.

Very few studies have been conducted on alkaline activated cement paste, mortar or concrete rheology, however. These cements, which are more eco-efficient than conventional portland cements, are defined as binders resulting from the chemical interaction between highly alkaline solutions and a number of aluminosilicates of variable compositions (i.e., CaO content) and origin (natural or artificial). The term alkaline cements covers a wide variety of materials [10] whose differences depend on the origin and composition of their starting aluminosilicates. Alkali-activated slag and fly ash cements are among the most widely known for their availability and high performance. Due to the differences in the chemical composition of the starting material (vitreous blast furnace slag and aluminosiliceous fly ash, respectively), the reaction products and microstructures of these cements vary considerably. These features determine the properties of the resulting cements and concretes, which have been shown to exhibit suitable strength development and good durability [11–13]. Those properties, along with drying shrinkage, have likewise been proven to depend heavily on the nature of the activating solution [14,15].

For blast furnace slag, the activating solutions traditionally used include alkaline or alkaline-earth compounds such as hydroxides and siliceous salts. Of these, NaOH, Na_2CO_3 and sodium silicate ($\text{Na}_2\text{SiO}_3 \cdot n\text{H}_2\text{O}$) (waterglass) are the most effective alkaline activators from the standpoint of mechanical and other properties [16]. Wang et al. [17] and Fernández–Jiménez and Puertas [18] studied the effect of these alkaline solutions (effect of activator type and concentration and $\text{SiO}_2/\text{Na}_2\text{O}$ ratio) on the mechanical properties, durability and activation kinetics of alkali-activated slag (AAS) cements. Their research showed that, for the best mechanical results, the optimal activator concentration ranged from 3.0% to 5.5% Na_2O (referred to slag mass), and for waterglass, a $\text{SiO}_2/\text{Na}_2\text{O}$ ratio of 1.0–1.5.

In a review of the setting time and mechanical strength behaviour of slag cement pastes activated with different alkaline solutions, Fernández–Jiménez and Puertas [19] demonstrated the key role of solution pH in slag dissolution and subsequent activation. These same authors [20–22] studied the effect of the type of alkali on the structure of the reaction products, reporting that the degree of condensation of the gels varied depending on the activator used. They attributed the differences in mechanical strength found by other authors to such variations.

As noted, very few studies have been conducted on alkaline cement rheology. Preliminary research on the rheological behaviour of sodium carbonate-activated slag pastes was conducted by Stryczek and Gonet [23]. Palacios et al. [24,25] also studied the rheology of AAS pastes and mortars activated with waterglass and NaOH (4% Na_2O). Their results showed that paste rheology depended on the nature of the activator used: in waterglass-activated slag pastes and mortars, the extensive structural breakdown under shear made the Herschel–Bulkley model a better fit to the down ramp of the flow curve, while NaOH-activated pastes and mortars, like Portland cement pastes and mortars, behaved like Bingham fluids. They noted that the problem of undesirably short setting times for waterglass-activated slag mortars and concretes could be overcome by lengthening the mixing time up to 30 min. They also identified significant knock-on effects, for the resulting

improvements in matrix cohesion and compactness raised mortar mechanical strength by approximately 11%. Drying shrinkage in these mortars, in turn, was 16% lower than in mortars mixed for shorter times, due essentially to the decline in the percentage of pores smaller than $0.05 \mu\text{m}$. Lastly, longer mixing times were not observed to modify the chemical or mineralogical composition of the reaction products [26].

AAS system rheology may also be affected by the presence of superplasticisers. To date, however, the studies conducted by different authors have yielded contradictory results, with admixtures sometimes reported to have a fluidising effect and at others to exhibit anomalous behaviour due to their instability in basic media [27–33].

The factors involved in alkali-activated system rheology and workability have yet to be ranked by importance. The present study aimed to ascertain the effect of factors such as the nature and concentration of the alkaline activator on the rheological behaviour of alkali-activated slag (AAS) pastes.

2. Experimental

2.1. Blast furnace slag (BFS) and alkaline activators

The materials used in this study were: a commercial Portland cement, CEM I 52.5R (OPC), used as a reference and a vitreous blast furnace slag (BFS). The chemical composition, Blaine fineness (as per European standard EN 196–6), particle size distribution and 10%, 50% and 90% size cut-offs (by volume) for the materials are given in Table 1.

The alkaline solutions used to activate the BFS were prepared with a commercial sodium silicate (Merck waterglass: $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio = 3.37; density = 1.36 g/mL), NaOH pellets (Panreac, 98% pure) and Na_2CO_3 (Panreac, 99.5% pure).

Three solutions were used to activate the BFS:

- NaOH (N).
- 50/50 (wt%) NaOH/ Na_2CO_3 (NC).
- Waterglass ($\text{Na}_2\text{SiO}_3 \cdot n\text{H}_2\text{O}$ + NaOH) (Wg) with $\text{SiO}_2/\text{Na}_2\text{O}$ ratios = 0.5, 0.8, 1.2, 1.5 and 2.

The effect of activator concentration (3%, 4% and 5% Na_2O by slag mass) on paste rheology was determined for all the samples. Table 2 lists the physical and chemical characteristics of the solutions used, along with the sample labels. The OH^- ion concentration was found by HCl titration.

2.2. Sample preparation and tests conducted

2.2.1. Variations in shear stress in AAS pastes at a constant shear rate

The OPC pastes were prepared by mechanically stirring a mix of 80 g of cement with water at a liquid/solid ratio of 0.5 for 3 min. The same procedure was used for the AAS pastes, although here the liquid/solid ratio used was 0.55 to compensate for the density of the solutions (see Table 2). All the pastes were tested for 30 min on a Haake Rheowin Pro RV1 rotational rheometer fitted with a serrated cylindrical rotor and operating at a constant shear rate of 100 s^{-1} .

2.2.2. Determination of rheological parameters (yield stress and plastic viscosity)

Paste rheological behaviour was characterised by determining yield stress and plastic viscosity, using the aforementioned rotational rheometer.

The procedure consisted of pre-shearing at 100 s^{-1} for 2 min, followed by ramping up from 0 to 10 s^{-1} in 1 min and from 10 to

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