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Alkali-activated mortars: Workability and rheological behaviour

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ABSTRACT

Workability and rheology in alkali-activated materials (AAMs) have been scarcely studied and differ substantially from ordinary Portland cement (OPC) systems. This study aims to explore the workability and rheology of alkali-activated slag (AAS) and fly ash (AAFA) mortars, ascertaining the effect of the precursor, the nature and concentration of the alkaline activators and the aggregate content. Mortars were prepared varying the aggregate/binder and liquid/binder ratios. OPC mortars were used as a reference. Mortar workability was determined by flow table measurements. The rheological tests included the stress growth test and torque at constant shear rate. Mortars were tested for 7 day compressive strength.

AAS and AAFA mortar workability was more sensitive to changes in the liquid/solid ratio than OPC mortars. In AAM mortars, fluidity was found to be proportional to the liquid/solid ratio and dependent upon aggregate content. When in a plastic state, AAS and AAFA mortars activated with waterglass solutions exhibited larger spreads and greater workability than OPC mortars, confirming the fluidising effect of waterglass. Rheology of AAS mortars was more influenced by the nature and concentration of the alkaline activator than that of AAFA mortars. Rheological behaviour of AAS and AAFA mortars fits the Bingham model.

1. Introduction

In the construction sector, quality and innovation requirements are increasingly present in building materials, not only from criteria for optimization of existing materials, but also in the search for new, more eco-efficient and sustainable materials. One outcome has been a growing number of papers dealing with new types of cements and concretes [1,2].

The requirements in place for mortars and concretes include mechanical strength in keeping with their use, volume stability over time and durability to ensure the longest possible service life. In addition, these materials must exhibit the necessary workability for transport and placement at a minimum cost while ensuring an optimal surface finish [3] and performance. Empirical trials to determine consistency, such as slump, L-box or K-ring tests [4] provide valuable information on mortar and concrete workability. Nonetheless, concrete mixtures that are a priori similar in empirical tests may behave differently when cast [5]. Hence, rheological studies are necessary to assess key physical parameters, such as yield stress and plastic viscosity [6]. Many studies have been conducted over the years to enhance the understanding of OPC paste rheology [5,7–10]. The factors with the greatest effect on rheology and hence on the workability of these systems include: cement fineness [5,11,12], the liquid/solid ratio or solids content [5,7,13], aluminate presence and content [12,14,15], mineral additions [16–19] and chemical admixtures [20–22].

Rheology is more complex in OPC mortar and concrete than in the respective pastes because of the variable nature of the aggregates as well as their particle size and fineness [23–26]. In these mixtures, the higher the percentage of fine aggregates, the higher the yield stress and the greater the amount of liquid needed to reach a given consistency. Viscosity and yield stress are also affected by aggregate shape [24]. In this regard, Hafid et al. [25] concluded that the more spherical the particles, the greater their packing and the lower the yield stress and viscosity induced.

As OPC mortars are deemed to be associated with Bingham-type fluids [27,28], if they are tested in mortar rheometers that measure torque in relation to speed, Bingham's equation may be expressed as:

$$T = g + hN$$

where T is torque (N·mm), g is a variable associated with yield stress (N·mm) and h is a variable linked to plastic viscosity (N·mm·min).

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As noted earlier, in addition to quality and innovation requirements, materials are now expected to meet sustainability and eco-efficiency demands. Alkali-activated materials (AAMs), also known as alkaline cements, should have environmental advantage over Portland cements in that respect [1,29–31]. The mechanical properties and durability of these systems make them particularly apt for certain applications [29,31–33]. In order for these AAMs to compete effectively with OPC systems, however, the advantages and disadvantages of transport and casting of these materials must be determined.

AAM mortars and concrete are highly versatile, for they can be prepared with a wide spectrum of precursors, such as blast furnace slag (BFS), type F fly ash (FA), metakaolin and FCC wastes; alkaline activators, such as alkaline hydroxides, alkaline silicate hydrates, alkaline carbonates or any mix of these products; and aggregates. Precursors, activators and aggregates all affect AAM system rheology. Generally speaking, alkali-activated slag (AAS) pastes, mortars and concrete have higher initial fluidity than their OPC and alkali-activated fly ash (AAFA) counterparts [34,35]. However, AAS-Wg systems present quick setting due to formation of a primary C-S-H gel [34], affecting concrete rheology and workability.

Recent studies [36] have shown that AAFA concrete workability (slump) declined as the percentage of BSF additioned rose, as a result of the angular morphology of slag and the fact that it is activated more quickly than fly ash. Huseien et al. [37] observed that workability improved in AAS mortars when up to 20% of the slag was replaced with fly ash.

A number of approaches have been adopted to monitor and modify workability in AAS systems. Collins and Sanjayam [38] studied the effect of adding lime slurry to solid or liquid waterglass (Wg)-activated AAS concrete. Their results showed that solid Wg-activated AAS concretes containing lime slurry exhibited better and longer-lasting workability than liquid Wg-activated, lime slurry-bearing slag concrete. That was explained by the lower initial reactivity of solid sodium silicate. Palacios et al. [39] proved that lengthening Wg-activated AAS mortar mixing time improved workability, lengthened setting times and raised mechanical strength. Such extended mixing prompts the de-clustering of the C-S-H gel initially formed.

The effect of superplasticisers on alkaline mortar and concrete rheology and workability has likewise been studied. The admixtures normally used to prepare OPC concretes have often been found to be unstable at the high alkalinity prevailing in AAM systems [40]. BNS (naphthalene)-based admixtures have been shown to be effective in both AAS (if the activator is a NaOH solution) [35,41] and AAFA [42] systems. Admixtures based on polycarboxylate ethers have exhibited very divergent effects, however, depending on the PC admixture, the activator and the alkalinity of the medium [35,42,43].

The studies conducted to date appear to indicate that NaOH- [39,40] and NaOH/Na_2CO_3-activated AAS pastes [34] and NaOH- [43,44] and NaOH- and Wg-activated AAFA pastes fit the Bingham model [44,45], whereas Wg-activated AAS pastes fit the Herschel-Bulkley model [34,39]. Constant shear rate rheological tests have shown that the initial shear stress in AAS and AAFA is lower than in OPC, although it rises with activator concentration, irrespective of the activator used or its SiO_2/Na_2O ratio [34,44].

Moreover, activator nature and concentration play an instrumental role in yield stress and plastic viscosity values. Both Kashani et al. [17] and Puertas et al. [34] found that yield stress was lower in waterglass-activated AAS paste than in both alkaline hydroxide-activated and OPC pastes. In the latter study [34], the authors observed that in constant shear rate tests, shear stress first rose and then declined in Wg-activated AAS pastes. The respective signal was associated with the formation and subsequent de-clustering of an initial C-S-H gel formed in the interaction between the silicate ions in the activator and the calcium ions dissolved in the slag, the mechanism deemed to be responsible for the low workability of Wg-activated AAS mortars. The timing and intensity of that signal, which was not present in NaOH- or Na_2CO_3/NaOH-activated AAS pastes, were shown to be related to the concentration of the Wg solution and its silica ratio.

The presence of Si in the alkaline activator is a determinant for AAM system fluidity [29,44,46–48], although the results are not always consistent because the shear history and preparation of such mortars affect the rheological findings substantially. Laskar and Bhattacharjee [45] showed that in NaOH/$Na_2O{\cdot}SiO_2$-activated AAS mortars yield stress and viscosity rose with solution molarity. In AAFA concretes with different $NaSiO_2$/NaOH ratios, in turn, slump and fluidity declined when the $NaSiO_2$/NaOH ratio rose [49].

In light of the results of these earlier studies, the objective pursued here was to explore the effect of the nature and concentration of the alkaline activator and aggregate content on AAM mortar workability and rheology against the backdrop of the analogous OPC mortar characteristics.

2. Experimental

2.1. Solid materials

A blast furnace slag (BFS), a Coal fly ash (FA) and a CEM I 52.5R cement (OPC) were used as binders or precursors. EN 196-1:2016 [50]-compliant standardised siliceous sand was the aggregate used throughout.

The chemical composition of the materials, found on a PHILIPS PW-1004 X-ray fluorescence (XRF) spectrometer, is given in Table 1, together with the loss on ignition (found further to Spanish and European standard UNE-EN 196-2:2014 [51]) and insoluble residue (determined as per Spanish standard UNE 80230:2010 [52]) data. The McMaster method as modified by Hooton and Emery [53], applied to determine the vitreous content in the glassy slag, yielded a value of 99%. The fly ash had a vitreous content of 87.75%, found with a selective attack with 1% HF [54].

Starting material mineralogy was determined on a Bruker AXS D8 Advance X-ray diffractometer and the Rietveld method was applied to quantitatively analyse the OPC and sand samples. The XRD findings are shown in Fig. 1. The diffractogram for the slag (Fig. 1a) contained an amorphous hump with a peak at around 2θ = 28–31°, confirming the high vitreous content in the sample. The hump covered the region where the most intense diffraction lines for akermanite (2θ = 31.14°, 28.87° and 51.78°) and gehlenite (2θ = 31.42°, 29.13° and 52.09°) lie. These are the two majority minerals in crystallised blast furnace slag [35,55]. The diffractogram (Fig. 1b) for the essentially amorphous coal fly ash [56] also exhibited a hump, at 2θ = 20°–35°. Certain crystalline phases such as quartz (Q), hematite (H), mullite (Mu) and magnetite (M) were also observed. XRD pattern for Portland cement (OPC) is shown in Fig. 1c. According to the Rietveld analysis, the sample contained 64% alite (C_3S or A), 14.5% belite (C_2S or B), 9% tricalcium aluminate (C_3A or C), 5.5% ferritic phase (C_4AF or F), 3% gypsum ($CaSO_4{\cdot}2H_2O$ or G) and 3.5% limestone. Lastly, the diffractogram in Fig. 1d shows that the sand used contained 98.3% quartz (SiO_2) and a 1.7% of microcline ($KAlSi_3O_8$) (Rietveld analysis).

Table 1
Chemical composition (wt%) of slag (BFS), fly ash (FA), cement (OPC) and sand.

wt%	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	SO_3	Na_2O	K_2O	TiO_2	P_2O_5	LoI*	IR**
BFS	39.21	10.36	0.33	0.21	7.65	40.28	1.04	0.67	0.35	0.36	0.04	-0.5	0.0015
FA	44.65	24.50	6.85	0.09	1.88	3.88	1.73	0.75	3.40	1.04	0.49	10.74	0.1300
OPC	21.69	5.88	2.55	0.03	1.56	59.01	4.15	0.79	1.24	0.13	0.12	2.75	0.007
Sand	96.80	1.51	0.43	–	–	0.10	–	–	0.55	–	–	0.61	–

* LoI: Loss on ignition.
** IR: insoluble residue.