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# Role of Sodium Concentration, Waiting Time and Constituents' Temperature on the Rheological Behavior of Alkali Activated Slag Concrete

Muhammet M. Erdem, Erdoğan Özbay, Ibrahim H. Durmuş, Mustafa Erdemir, Murat Bikçe, Müzeyyen Balçıkanlı

***Abstract*—**In this paper, rheological behavior of alkali activated slag concretes were investigated depending on the sodium concentration (SC), waiting time (WT) after production, and constituents' temperature (CT) parameters. For this purpose, an experimental program was conducted with four different SCs of 1.85, 3.0, 4.15, and 5.30%, three different WT of 0 (just after production), 15, and 30 minutes and three different CT of 18, 30, and 40 °C. Solid precursors are activated by water glass and sodium hydroxide solutions with silicate modulus (Ms = $SiO_2/Na_2O$) of 1. Slag content and (water + activator solution)/slag ratio were kept constant in all mixtures. Yield stress and plastic viscosity values were defined for each mixture by using the ICAR rheometer. Test results were demonstrated that all of the three studied parameters have tremendous effect on the yield stress and plastic viscosity values of the alkali activated slag concretes. Increasing the SC, WT, and CT drastically augmented the rheological parameters. At the 15 and 30 minutes WT after production, most of the alkali activated slag concretes were set instantaneously, and rheological measurements were not performed.

***Keywords*—**Alkali activation, slag, rheology, yield stress, plastic viscosity.

## I. Introduction

DEVELOPING industry caused to emission of $CO_2$, and greenhouse gases are obligated to reduce energy consumption by authorities. Based on this, cement manufacturers resorted to find eco-efficient binding materials [1]-[5]. Therefore, after 1940's, the material known as alkali activated or geopolymer is recognized as a cementitious material [1], [6]-[8]. Due to noticing the impressive mechanical and durability properties of these types of concretes, researches on them are increased significantly. In this regard, several materials are studied such as alkali activated blast furnace slag as an alternative to Portland cement. In literature, many research papers might be found related to the effect of activator type, activator concentration, curing temperature, materials used, and so on.

As an activator, sodium hydroxide, water glass, sodium meta silicate, sodium sulfate, sodium carbonate, etc. have been used extensively. Findings of literature demonstrate that alkali activated slag concretes (AASC) activated by water glass have better properties with respect to initial strength, heat of hydration, acid, seawater, sulfate, and corrosion resistance [9], [10], [18], [19]. However, such negative side of AASC as sudden setting prevents it from becoming widespread use [11], [12], [20]. So, poor workability complicates the site applications of AASC. It is considered that clarifying the rheological behavior of AASC on site conditions makes its usage on site possible.

The process stages of fresh concrete until hardening are pumping, spreading, molding, and compaction, respectively. These mentioned stages are performed by utilizing rheological properties of concrete [13]. Rheological behavior of Portland cement pastes, mortars, and concretes are investigated extensively. However, there is dearth of study on rheological behavior of the water glass activated slag concretes. A few researches have been performed about the effect of nature of activator, activator concentration, and CT on the properties of alkali activated slag mortars and concretes; however, in those studies, the effects of those mentioned parameters are investigated independently [14]-[16]. It might be valuable to find out the effects of those parameters concurrently to clarify the rheological behavior of AASCs.

Correspondingly, the objective of this research is to investigate the combined effects of activator concentration, WT, and CT on the rheological behavior of AASCs. Moreover, it could be considered that the combination of these effects illustrates the site conditions of water glass activated slag cement concrete.

## II. Experimental studies

### A. Materials, Mixture Proportions and Basic Mechanical Properties

In this study, Iskenderun Iron-Steel factory slag which has a 99% vitreous content and 540 $m^2$/kg surface area was used. By using Malvem Mastersizer 2000, particle size distribution of slag was determined and presented in Fig. 1. Table I shows the chemical compounds of slag which is obtained by using XRF. Blends of sodium silicate (water glass) solution and crystals of sodium hydroxide were used as activator by keeping the silicate modulus constant. Chemical and physical properties of water glass and sodium hydroxide are given in Tables II and III, respectively.

Crushed limestone coarse and fine aggregates were used as 25% between 11 and 22 mm size, 25% between 4 and 12 mm size, and 50% between 0 and 4 mm size by weight. Fineness modulus of aggregates are 2.817, 2.505, and 6.054, and

M. M. Erdem, E. Özbay, M. Bikçe, and M. Balçıkanlı are with the Iskenderun Technical University, Civil Engineering Department, Iskenderun, Turkey (e-mail: musab.erdem@iste.edu.tr, erdogan.ozbay@iste.edu.tr, murat.bikce@iste.edu.tr, muzeyyen.balcikanli@iste.edu.tr).
İ.H. Durmuş is with the Adana Cement Industry Inc., Adana, Turkey.

specific weights are 2.62, 2.66, and 2.68, water absorptions by percentage are 2.7, 0.5, and 0.5, respectively. Gradation curves of each aggregate are given in Fig. 2. Four different mix designs were conducted depending on the SC by keeping constant the slag content at 490 kg/m$^3$ and (water + activator solution)/slag ratio at 0.53. The ingredients of each mixture are presented in Table IV.

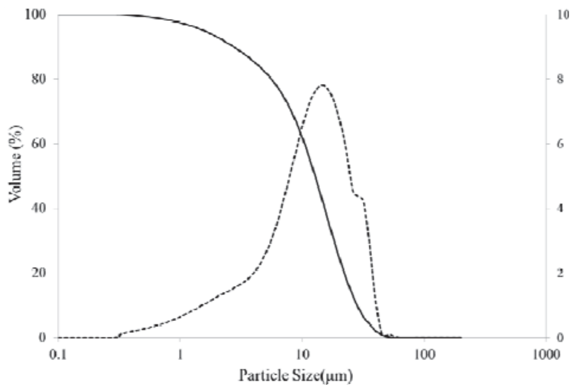


Fig. 1 Particle size distribution of ground granulated blast furnace slag

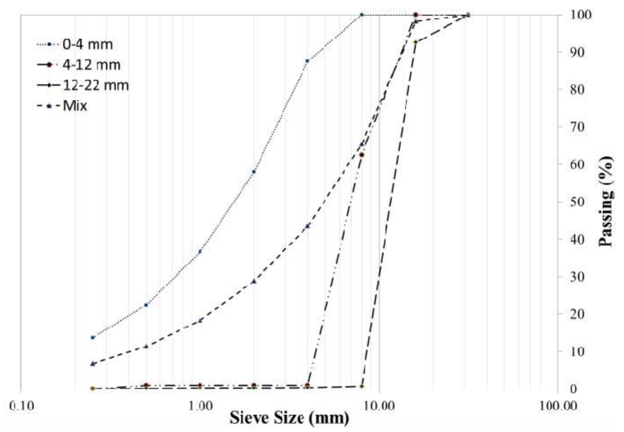


Fig. 2 Particle size distribution of aggregate

TABLE I
CHEMICAL COMPOSITION OF GROUND GRANULATED BLAST FURNACE SLAG

| Oxide | Composition by weight (%) |
|---|---|
| $SiO_2$ | 43.08 |
| Insoluble residue | – |
| $Al_2O_3$ | 11.34 |
| $Fe_2O_3$ | 0.74 |
| CaO | 36.25 |
| MgO | 6.1 |
| $SO_3$ | 0.6 |
| Sulfide Sulfur as S-2 | 0.51 |
| Loss on ignition | 0 |
| $Na_2O$ | 0.28 |
| $K_2O$ | 0.75 |
| $Na_2O + 0.658K_2O$ | 0.77 |
| Free Lime | – |
| $SiO_2$ | 43.08 |
| demagnetizing factor | $1 \rightarrow 1/(4\pi)$ |

TABLE II
PHYSICAL AND CHEMICAL PROPERTIES OF WATER GLASS

| Physical and chemical properties | Analysis result |
|---|---|
| Appearance | Filtered, colorless, tempered liquid |
| Module by weight (SiO2/Na2O) | 3.19 |
| Module by molecule ($SiO_2/Na_2O$) | 3.3 |
| Be (at 20 °C) | 39.4 |
| Density (at 20 °C, g/cm$^3$) | 1.373 |
| $Na_2O$ (%) | 8.52 |
| $SiO_2$ (%) | 27.09 |

TABLE III
PHYSICAL AND CHEMICAL PROPERTIES OF NAOH

| Physical and chemical properties | Analysis result |
|---|---|
| Total alkalinity (NaOH) (g/kg) | ≥990 |
| Sodium carbonate ($Na_2CO_3$) (g/kg) | ≤4 |
| Sodium sulfate ($Na_2SO_4$) (mg/kg) | ≤80 |
| Sodium chloride (NaCl) (mg/kg) | ≤200 |
| Iron (Fe) (mg/kg) | ≤10 |
| Mercury (Hg) (mg/kg) | ≤0.1 |
| Arsenic (As) (mg/kg) | ≤1 |
| Cadmium (Cd) (mg/kg) | ≤1 |
| Chrome (Cr) (mg/kg) | ≤1 |
| Lead (Pb) (mg/kg) | ≤0.5 |
| Antimony (Sb) (mg/kg) | ≤5 |
| Selenium (Se) (mg/kg) | ≤5 |
| Nickel (Ni) (mg/kg) | ≤2 |
| $SiO_2$ | ≥990 |
| demagnetizing factor | ≤4 |

TABLE IV
MIX PROPORTIONS OF AASC

| Material | Amount (kg) in 1 m3 Concrete | | | |
|---|---|---|---|---|
| | 1.85% SC | 3.00% SC | 4.15% SC | 5.30% SC |
| Water | 227.4 | 210.3 | 193.3 | 176.2 |
| Slag | 490.0 | 490.0 | 490.0 | 490.0 |
| Fine Aggregate | 982.3 | 982.3 | 982.3 | 982.3 |
| Mid-size aggregate | 508.3 | 508.3 | 508.3 | 508.3 |
| Coarse aggregate | 504.5 | 504.5 | 504.5 | 504.5 |
| NaOH | 8.2 | 13.3 | 18.3 | 23.4 |
| $Na_2SiO_3$ | 43.7 | 70.8 | 98.0 | 125.1 |

### B. Apparatus

In this study, ICAR Rheometer produced by German Instruments is used. Rheometer is composed of a vane having 125 mm diameter and height, a container having 286 mm diameter, and vertical strips at inner side wall. The vane rotates at desired angular velocities. The vertical strips prevent slippage between the concrete and container wall. Rheometer is controlled by computer software. Before the test, initial, final, and breakdown speeds, number of data acquisition points, and time per points can be defined by user. At the beginning of test, rheometer starts to rotate with the defined initial speed. According to the inputs, rheometer increases the speed step by step. First 5 seconds of each speed, no data are acquired to stabilize the torque. Data are obtained by averaging the values received at rest of the time after the 5 seconds of each step. This software is based on Bingham model which is also used for AASC [17]. At the end of the test, yield stress is determined according to the data obtained during the test and it is calculated by the following formula:

$$\tau = \tau_o + \mu\dot{\gamma}$$

where $\tau$ is shear stress in Pa, $\tau_o$ is yield stress in Pa, $\mu$ is plastic viscosity in Pa.s, and $\dot{\gamma}$ is shear rate in 1/s. A vertical pan mixer which has 50-liter capacity is used to mix the concrete.

### C. Test Procedure

Prepared aggregate, water, slag, and water glass solution are waited in an oven for 48 hours to obtain materials in the desired temperature (18, 30, and 40°C). Solid materials including aggregate and slag are mixed in pan mixer for 2 minutes. Then, water and water glass solution are added to mixer and are mixed for 5 minutes. For all of the tests, end of the mixing process is considered as 0th minute of rheology test. At 0th minute, fresh concrete is placed into rheometer container, and rheology test is conducted. After the test conducted at 0th minute, fresh concretes used in the tests are placed into the pan mixer. Before performing rheology test at 15th and 30th, the pan mixer was started for 2 minutes again, and then AASC was transfer to rheometer. Same procedure as in 0th minute was applied again, and measurements were taken under the SC (1.85%, 3.00%, 4.15%, and 5.30%), WT (0th, 15th, and 30th), and CT (18 °C, 30 °C, and 40 °C) studied.

## III. Results And Discussions

### A. Yield Stress

The variation of yield stress values of the AASC depending on the SC, WT, and CT is presented in Fig. 3. According to the test results, higher yield stress values were obtained as time passed in all mixes; however, as seen in Fig. 3, in some rheology tests, data acquisition is terminated due to the sudden hardening. For the mixtures which have 1.85% SC, during the tests conducted at 18 °C and 40 °C, 240 and 279 Pa yield stress values were obtained at 0th minute, respectively. At 15th minute, yield stress rose significantly and reached to 710 and 680 Pa. However, before reaching to 30th minute, both were set, and the yield stress measurement might not be performed. On the other hand, for the test conducted at 30 °C, any hardening and/or dramatic increase in yield stress was not monitored. Latter readings at 15th and 30th minutes were 304 and 510 Pa, respectively.

In the rheological measurements of mixture which is produced with 3.00% SC at 30 °C and 40 °C, a gradual increase in yield stress was monitored. However, higher yield stress increment was observed at CT of 18 °C and WT of 0th and 15th minutes. At the further tests, AASC lost its fluidity completely, and any data could not be obtained in the measurement of 30th minute.

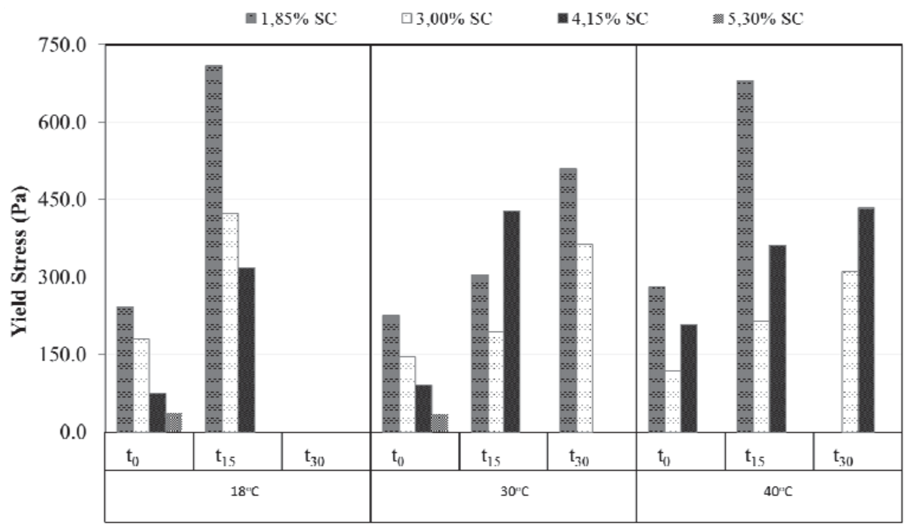


Fig. 3 Combined effect of temperature and WT on yield stress

For the AASC produced with 4.15% SC, yield stress was 208 Pa at the CT of 40 °C and WT of 0th minute. A slight increase was observed with the increase of WT to 15th and 30th minutes, and yield stress reached to 362 Pa and 433 Pa, respectively. This increase in yield stress is more prominent than those yield stresses measured at 18 °C and 30 °C. However, it remarks that yield stress values from 0th minute to 15th minute increased from 74 Pa to 417 Pa in measurements at 18 °C and from 90 Pa to 427 Pa in measurements at 30 °C. After these dramatic increases, concrete is hardened, and no yield stress value could be obtained.

During the rheology tests of the mixture which is produced with 5.30% SC, concrete was quite fluid at the beginning of the tests, and yield stress values are 34 Pa and 36 Pa at 18 °C and 30 °C, respectively. However, after first measurements, concrete in pan mixer is hardened instantly.

According to the rheology test results, AASC produced with 1.85% SC lost their workability in 15 minutes at CT of

18 ºC and 40 ºC. It can be clearly seen in Fig. 3 that there is a significant increase in yield stresses with the increase of WT. AASCs produced with 5.30% SC have quite low yield stresses at CT of 18 ºC and 30 ºC at the WT of 0th. However, at 15th minute, AASC was hardened instantaneously. On the other hand, during the test of AASC produced with 5.30% SC performed at CT of 40 ºC, instantaneous hardening is observed at the WT of 0th minute. This is the case which is also observed in the literature. It was mentioned in the literature that the shortest initial setting time of AASC might be monitored when it was produced with the silicate modulus between 1 and 2 [21]-[23].

AASC is too stiff and it is hard to get any rheological measurement, most probably due to the initial hydration [24]. Behavior of AASC is completely different from the conventional concretes and it has early hydration mechanism [25]. As seen in the test results, at the beginning of the tests, yield stresses of the mixtures which include 1.85% SC to 5.30% SC decreased. At 15th minute, yield stresses of 1.85% SC, 3.00% SC, and 4.15% SC increased by 294%, 236%, and 425% respectively. At this WT, 5.30% SC is hardened. But, at 30th minute, all of the mixes are hardened. It is seen that the mix having the highest SC is hardened first, and then the others hardened. At 30 ºC, yield stresses of 1.85% SC to 5.30% SC decreased as in the tests at 18 ºC. Yield stresses of 1.85% SC and 3.00% SC increased through all the tests, and setting was not seen. However, yield stress of 4.15% SC increased by 475% at 15th minute, hardened instantly before reaching 30th minute. 5.30% SC quite fluid at the beginning of test. However, it hardened before 15th minute. At 40 ºC, 5.30% SC hardened at the beginning of the test. Yield stresses of 3.00% SC and 4.15% SC increased while the WT was increasing, but interestingly 1.85% SC hardened before 30th minute.

All the results show that, when the SC increases, lower yield stress values are obtained after first minutes of mixing. But, it is seen that the higher concentration mixes reach the higher yield stresses and hardened before the lower concentration mixes. This behavior is associated with the results of [5] which used water glass solution as activator at a constant shear rate. It is stated that after the contacting of slag grains and water glass solution, decreasing shear stresses were obtained. However, after about 7 minutes, a rapid increase in shear stress is observed. After the peaking, shear stress follows a decreasing trend. Cause of this behavior is considered as the initial C-S-H formation. The Initial C-S-H gels are formed by the reaction of sodium silicate (water glass) and Ca from slag. With the mixing of sodium silicate and slag particles, C-S-H layers were occurred around the slag grains. The slag pellets surrounded by C-S-H gel layer are bounded by Van der Waals bounds. The bounded structure increases the yield stress of concrete. However, maintaining the mixing causes the breakage of initial C-S-H gel structure. Because of the breakage, yield stress follows a decreasing trend [25]-[27].

In literature, it is seen that yield stress is increased by increasing the SC, and yield stress growth is interrupted after a peak which is occurring about 15th minute [27]. However, in the current study, according to the results at 18 ºC and 30 ºC, yield stress is decreased by increasing the SC, and the growth in yields stress maintains the increasing as contrary to the finding of literature. It is thought that positive increments in yield stress for all the experiments in the current study and the yield stress behavior in literature are different from each other due to methodology of the tests. The rheology test in literature is continued along 30 minutes without pausing the mixing of concrete. However, in this study, mixing is stopped at each 15 minutes. This case caused to accelerate the initial C-S-H formation and setting.

### B. Plastic Viscosity

According to the rheology test results, time-dependent plastic viscosity behavior shows difference according to SC (Fig. 4). Plastic viscosity AASC which is produced with 1.85% SC concentration increased gradually with the increase of WT at 18 °C; however, it remained almost constant at 30 °C and followed by a decreasing trend at 40 °C. Data could not be collected at 30th minutes of 18 °C and 40 °C because of the sudden hardening. 3.00% SC showed a slight decrease at 18 °C. At 30 °C, plastic viscosity increased until 15th minute of test; however, it decreased at 30th minute. At 40 °C, plastic viscosity showed a small decrease until 15th minute, but it increased at 30th minute. Plastic viscosity of 4.15% SC decreased from beginning of test to 15th minute at 18 °C. At 30 °C, it showed a slight increase between 0th and 15th minutes. At 30th minute, no data could be collected by reason of sudden hardening. Plastic viscosity decreased between 0th and 15th minute at 40 °C, but it increased at 30th minute. Because of sudden hardening, experimental data could not be collected at 0th minutes of 18 °C and 30 °C rheology tests of 5.30% SC. According to overall plastic viscosity results of the experiments, it could be clearly seen that plastic viscosity decreases as the CT increases. It could be interpreted that dissolution of slag and reactions are faster at 18 °C and 30 °C. As seen in Fig. 4, as the SC increased, higher plastic viscosity values were obtained exceptionally; 5.30% SC at 0th minute and the values at 15th minute at 18 °C. However, those mixes were hardened before 30th minute of test.

## IV. Conclusions

In this research, the effect of three main parameters, namely, SC, WT after production, and CT on the rheological properties of AASC were investigated by keeping constant the slag content and (water + activator solution)/slag ratio. Yield stress and plastic viscosity values of AASC were defined due to the variation of SC, WT after production and CT concurrently by using the ICAR rheometer. Following conclusions might be drawn from the findings of the current research.

- Test results showed that there is no linear relation between the SC and rheological behavior of AASC. Increasing the SC generally decreased the yield stress values of the AASC when measured just after production; however, passing the time after production turned this behavior opposite: mixtures produced with higher SC set significantly earlier.

- Rheological measurement demonstrates that this kind of concretes are more sensitive to the WT after production, WT after production remarkably influenced the yield stress and plastic viscosity, passing time after production increased the rheological values. After 30 minutes' production time, almost all of the mixtures turned to set irrespective of the SC and CT.
- Evaluating of the effect of CT parameter on the rheological values of AASC illustrates that rheological behavior and CT are not proportional, so it might not be clearly mentioned that increasing the CT affected the rheological values positively or negatively.

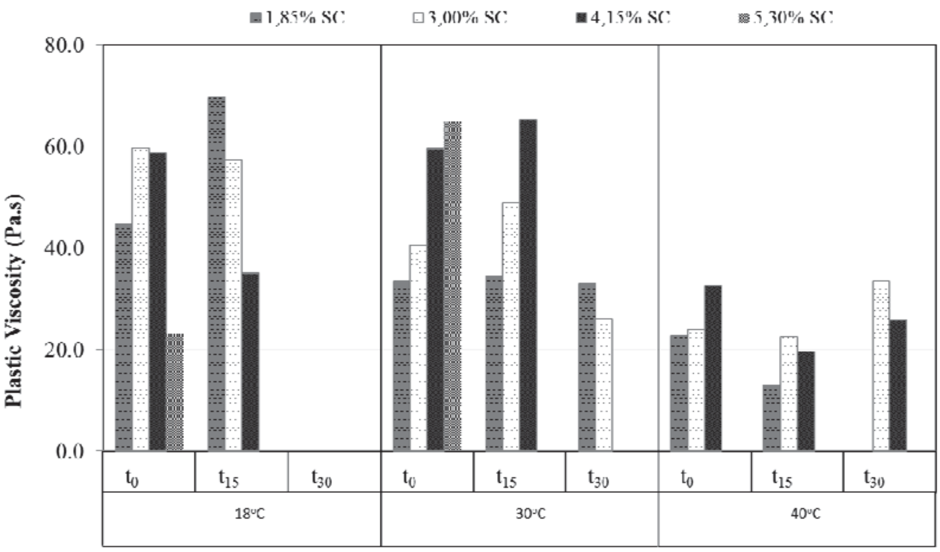


Fig. 4 Combined effect of temperature and WT on plastic viscosity

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