# EARLY ASSESSMENT OF ALKALI-SILICA REACTION IN ALKALI-ACTIVATED FLY ASH CONCRETE SUBJECTED TO THE CONCRETE PRISM TEST (ASTM C1293)

Juliana Neves<sup>1\*</sup>, Stephen Salwocki<sup>1</sup>, Farshad Rajabipour<sup>1</sup>

<sup>1</sup>The Pennsylvania State University, University Park, PA, USA

### Abstract

This research investigated the susceptibility of class F alkali activated fly ash (AAFA) concrete to ASR, through ASTM C 1293 concrete prism test. At 7 months of test, the results suggested a satisfactory response of AAFA concrete in the presence of highly reactive aggregates. Conversely, OPC samples exceeded the expansion threshold at the age of 2 months. To explore the reasons for the lack of deleterious ASR expansions in AAFA concrete, SEM, pore solution analysis, and mercury porosimetry (MIP) were performed. It was found that despite using very high alkaline activators needed to trigger fly ash reaction, OH<sup>-</sup> ions in the AAFA pore solution were rapidly consumed in reaction with fly ash and became significantly less available to attack aggregates and generate ASR. Also, AAFA pore solution contained elevated levels of dissolved Al, which helps in mitigating aggregate dissolution and as such, ASR. In comparison with OPC paste, AAFA pastes had similar or larger porosity and average pore size, despite their significantly lower ASR activity. This rules out a hypothesis that ASR is mitigated in AAFA concrete because of its low mass transport kinetics (e.g., permeability).

KEYWORDS: alkali-silica reaction, alkali-activated fly ash, pore solution analysis

# 1. INTRODUCTION

The objective of this research was to investigate the risks and early signs of alkali-silica reaction (ASR) in class F alkali-activated fly ash (AAFA) concretes. ASR is a leading cause of deterioration in concrete structures and involves a series of deleterious reactions between meta-stable silica (found in many natural aggregates) and the highly alkaline pore solution of concrete [1]. AAFA is a new generation of concrete materials where the binder phase is fully produced using pulverized coal fly ash and does not contain any portland cement. As such, AAFA provides significant environmental benefits (e.g., reduced CO2 emission and reduced energy use) in comparison to conventional portland cement concrete [2]. However, highly alkaline activators (with pH nominally above 14) are needed to promote fly ash reactivity and produce AAFA concrete with desirable strength [3]. As such, the risk of ASR in AAFA concrete is of concern.

A few publications exist where class F AAFA-based mortars were tested using ASTM C 1260 accelerated mortar bar test (AMBT) [4]–[8]. In these studies, AAFA binders showed consistently better response to reactive aggregates compared to OPC binders. However, no clear explanation for the lack of ASR was provided. It should be noted that AMBT is especially unsuitable for evaluating ASR in AAFA materials since the 1M NaOH bath used in the test has lower alkalinity than AAFA pore solution and as such, dilutes the OH<sup>-</sup> concentrations provided by the alkaline activator. To overcome this deficiency, the present work utilized ASTM C 1293 concrete prism test [9].

If it is proven that class F AAFA concretes are truly less prone to ASR, despite their initially high alkalinity, the following hypotheses can be proposed and evaluated in this research to explain the lack of ASR:

- Despite an initially high alkalinity, [OH-] and alkali concentrations in AAFA pore solution could decrease rapidly over time as a result of fly ash geopolymerization.
- AAFA pore solution may contain elevated levels of aluminum, and the presence of dissolved Al has been shown [10]–[12] to mitigate dissolution of aggregates, even at high pH.

Correspondence to: juliana.nevves@gmail.com

- The solid and dissolved calcium concentrations in class F AAFA binders are expected to be low and this might prevent gelation of dissolved silica. Even if ASR gel is formed, it may have low yield strength and viscosity [13] due to low Ca content, and as such, it may be non-deleterious.
- Mass transport and water permeability of AAFA binder may be low due to its lower volumetric liquid-to-solid ratio (L/S)<sub>binder</sub> compared to OPC paste, resulting in smaller porosity and pore sizes. This could slow down swelling of ASR gel.

The research outcomes could provide a step towards better industry acceptance of AAFA concretes as the knowledge gained could lead to development of guidelines and test methods for durable AAFA concrete mixtures.

# 2. MATERIALS AND METHODS

# 2.1 General

The ASR risk in AAFA concrete was assessed using ASTM C 1293 – Concrete Prism Test (CPT). To explore the ASR mechanisms, electron microscopy and mercury porosimetry of AAFA concretes and pastes, respectively, was performed. Additionally, pore solution of AAFA pastes was extracted and analyzed at different ages.

## 2.2 Materials and mix designs

Two AAFA concretes and their corresponding pastes (FA1 and FA2) were used in this study along with a control OPC concrete (Table 1). Each AAFA concrete and its corresponding paste consisted of the same binder phase, while the paste excluded aggregates. Some of the properties of the two mixtures are provided in Table 2. Our earlier research [3] demonstrated that FA1 and FA2 developed 1-day compressive strength of 18.0 MPa and 24.0 MPa and 28-day compressive strength of 32.9 MPa and 41.0 MPa, respectively. As such, both mixtures can be used as structural grade concrete, which is the reason why they were compared with a w/c=0.45 portland cement concrete (control mixture). The AAFA mixtures were proportioned based on the previous study of Kazemian et al. [3], where they were referred as G1 and G4 mixtures, respectively.

ASTM C150 [14] type I Portland cement was used for the preparation of the control mixture. Its specific gravity and oxide composition is shown in Table 3. The  $Na_2O_{eq}$  (percent of  $Na_2O + 0.658$ times percent of K<sub>2</sub>O) was raised to 1.25%, as required by ASTM C 1293, through direct addition of NaOH to the mix water. Class F fly ash was used as the binder phase of AAFA pastes and concrete. Its oxide composition and specific gravity is shown in Table 3. Distilled water was used in this research to avoid the impurities from tap water, specifically Ca2+ that can interact with the AAFA mixtures and affects their properties. The activator solutions used in AAFA were a mixture of sodium hydroxide pallets, sodium silicate solution and distilled water. Analytical grade NaOH pallets were dissolved in distilled water to prepare 12M solutions. A commercially available sodium silicate solution (PQ corporation) was used, which was composed of 28.6% of SiO<sub>2</sub>, 8.9% Na<sub>2</sub>O, 62.5% water, by mass, and had pH=11.3, modulus (SiO<sub>2</sub>/Na<sub>2</sub>O by weight) n=3.22, and specific gravity of 1.39 at 20 °C. Natural sand (Oley, PA), meeting ASTM C 33 [15] was used as fine non-reactive aggregate in the concrete mixtures. It has oven dry specific gravity 2.70, absorption capacity of 0.46% and AMBT expansion of 0.075% at 14 days. The coarse aggregate used was Spratt, a well-known highly reactive siliceous limestone, supplied by the Ministry of Transportation, Ontario, Canada. Spratt has OD specific gravity is 2.64, absorption capacity of 0.74% and AMBT expansion of 0.33% at 14 days.

As noted in Table 1, the amount of reactive aggregate was maintained constant in the three concrete mixes. While the OPC concrete required 1.97 kg of NaOH per cubic meter of concrete to boost its alkalinity to 1.25%, FA1 and FA2 required respectively 58.08 and 36.89 kg of NaOH per cubic meter of concrete to achieve the target activator pH, showed in Table 2. ASTM C 1293 requires w/cm between 0.42 and 0.45 by mass, which corresponds to liquid-to-solid (L/S)<sub>binder</sub> by volume in the range of 1.32 to 1.42. However, to produce AAFA concretes with sufficient strength and workability, the liquid-to-solid ratio (L/S)<sub>binder</sub> had to be decreased to 0.80 (by volume). Master Pozzolith 322 water-reducer admixture was used in FA1 and FA2 at a proportion of 2.5ml per kilogram of fly ash, also to increase workability of AAFA concrete and avoid a false set of the binders.

#### 2.3 Methods for assessment and analysis

ASTM C 1293 – Concrete Prism Test

AAFA and OPC prisms were prepared according to standard specifications. A pan mixer was used for mixing the concretes. After mixing, the AAFA concrete was poured in molds and cured at 23 °C, at 100% relative humidity for 24 hours. Next, the AAFA prisms were steam cured at 60 °C

for further 24 hours to allow proper strength development. Subsequently, the AAFA concrete prisms were demolded and stored in buckets at 38 °C at 100% relative humidity. The control (OPC) specimens were cured at 23 °C and 100% relative humidity for 24 hours and then demolded and stored in buckets at 38 °C at 100% relative humidity, as specified by ASTM C 1293. 24 hours before each periodic length measurement, the buckets containing the specimens were removed from the 38 °C room and cooled down at room temperature. Prism expansions and mass changes were monitored using a digital comparator with accuracy 0.0025 mm and a balance with accuracy 0.01 g.

### **SEM**

The specimens tested under CPT were also analyzed through scanning electron microscope (SEM). One 7-month-old concrete prism of each AAFA concrete mixture, FA1 and FA2, was sacrificed and imaged. A slice of approximately 2 cm was cut from the prism and then sawed to obtain small rectangular pieces. Afterwards, the pieces went through a solvent exchange process (isopropanol alcohol bath) for 7 days, which stopped the geopolymerization reaction and allows drying of the specimens with minimal microstructural damage. The specimens were then dried under vacuum at room temperature for 2 additional days. After the 48-hours drying, each AAFA concrete specimen was epoxy-impregnated, polished down to 0.05  $\mu$ m, and coated with carbon for SEM analysis. The backscattered images, which show variance in composition, were captured under high vacuum, spot sizes of 4 for FA1, and spot size 6.8 for FA2.

#### Pore Solution Analysis

Pore solution analysis was used to quantify the composition of pore solution of each AAFA mixture over time. It was performed on pastes to exclude the effects of alkali leaching and ASR on pore solutions. Also, it was determined to be easier to extract pore solution from AAFA pastes at later ages in comparison with AAFA concrete. The pastes were prepared according to the proportions shown in Tables 1 and 2. A Hobart mixer was used to prepare the pastes according to ASTM C305-14 [16]. After mixing, the AAFA pastes were poured into cylindrical plastic containers, and sealed cured at 23 °C and 100% relative humidity for 24 hours. Next, the sealed paste containers were steam cured at 60 °C for further 24 hours. Subsequently, the sealed paste containers were stored in buckets at 38 °C and 100% RH until the samples reach the age for pore solution extraction. The curing procedure of AAFA paste was exactly the same as that for AAFA concretes in ASTM C1293 test.

At the specified ages, 7, 30, 90, and 225 days, the pastes were taken from the storage buckets and broken into small pieces. The crushed paste was then placed inside a steel die specially designed for pore solution extraction, placed inside a compression-testing machine and pressurized up to 1,800 kN (about 400,000 lbs). This process was replicated until sufficient solution was collected for chemical analysis (ICP-AES) and for acid titration to be performed. Before analysis, pore solution was filtered through  $0.2\mu$ m filter to remove remaining particles.

#### Mercury Intrusion Porosimetry (MIP)

Micromeritics AutoPore V 9620 MIP device was used to assess the porosity and pore size distribution of AAFA and OPC pastes. The goal was to evaluate whether the mass transport is likely to be slower in AAFA binder compared to OPC, which would help slow down ASR. The pore sizes of FA1 and FA2 pastes were assessed and compared to OPC paste with w/c ratio of 0.45. The pastes were maintained at the same conditions as their corresponding concrete prism until they reach the age of 100 days, when MIP was performed. Before the test, the samples went through a solvent exchange process (isopropanol alcohol bath) for 7 days, which stopped the geopolymerization reaction and allows drying of the specimens with minimal microstructural damage. The specimens were then dried under vacuum at room temperature for additional 48 hours.

#### 3. RESULTS

#### ASTM C 1293 – Concrete Prism Test

Figure 1 shows the ASR expansion of the control (OPC) and AAFA concrete prisms over time. The data shows the OPC (control) specimen, exceed the 0.04% expansion threshold within two months from start of the test and reached approximately 0.20% expansion in one year. In contrast, FA1 and FA2 expanded no more than 0.01% and 0.018%, respectively, in roughly 300 days of test. FA1 and FA2 expansions correspond to 20 and 11 times smaller than the expansion of the control, and 4 and 2 times smaller than the standard threshold. It should also be noted that the expansion curve of the two AAFA concretes does not show large variation along the test.

#### SEM

The 7 months old concrete mixture FA1, which expanded less than 0.01% up to 335 days, did not show any signs of aggregate deterioration nor ASR gel formation under SEM, as shown in Figure 2.

FA2 concrete, also 7 months old (expansion<0.018% at 293 days) experienced some deterioration in a few of its coarse aggregates, as shown in Figure 3. The deterioration path happened both inside aggregates and at the interfacial transition zone (ITZ) on the aggregate surface. However, the desiccation cracks typical of ASR were absent. It should be highlighted, however, that deterioration was observed only at the minority of the reactive aggregates, while most of them looked undamaged.

# Pore Solution Analysis

Figure 4 (A to F) shows the composition of AAFA paste pore solution over time for up to 225 days of hydration. The initial and final concentrations are also shown on each curve. All day-0 concentrations ([OH-], [Na], and [Si]) were calculated from the activator composition at the time of mixing and before activator came into contact with fly ash. The other elements (Al, Ca, K) were not present in the pore solution at day-0, as the activator did not supply them. Since the pore solution analysis was performed on AAFA pastes, aggregates and ASR effects on pore solution composition were not considered.

### Mercury Intrusion Porosimetry (MIP)

The average pore diameter, continuous pore diameter (i.e., the mean diameter where maximum percolation occurs), threshold pore diameter (i.e., where significant intrusion starts to occur), and the total porosity of each paste were determined and are summarized in Table 4. The threshold diameter is considered to be the widest diameter of interconnected pores that would allow percolation of fluids in a paste [17]. It should be noted that pore volumes were reported in mL per g of dry paste, which can be used along with the skeletal density of each paste to calculate the total volumetric porosity of each paste (Table 4).

### 4. **DISCUSSION**

### ASTM C 1293 – Concrete Prism Test (CPT)

The results of concrete prism test (Figure 1) showed little ASR expansion of the AAFA concretes tested in this study. This was in contrast to the large expansions observed for the OPC concrete containing the same quantity of reactive aggregates. As such, other experiments in this study attempted to provide a better description about the reactivity of aggregates in the two AAFA concrete mixes. The test will continue up to one year, for full assessment on expansion of the AAFA concrete prisms.

#### SEM/EDS

The SEM results of AAFA concretes indicated possible ASR activity only in FA2. It was observed that a lower density product was generated, which is indicated by the darker gray (Fig. 4). Spratt aggregate was likely attacked by the alkaline pore solution, but still some undissolved particles are left. Despite this finding, neither cracks nor ASR gel were found, which consistently agrees with the results of the CPT. It should be highlighted that this deterioration pattern differs from the typical aggregated deterioration, well characterized by formation of cracks [1]. Further investigation is on going to track the evolution of aggregate deterioration throughout the duration of the concrete prism test (ASTM C 1293), if any.

#### Pore Solution Analysis

The pore solution analysis results (Figure 2-A) show a rapid decrease in OH<sup>-</sup> concentration (and reduced pH) in the pore solution of AAFA concrete over time. This is due to fly ash dissolution and geopolymerization. After day-90, the [OH<sup>-</sup>]=0.13M, which is already below a typically assumed minimum required threshold to sustain ASR (approximately 0.25M to 0.30M [18]). In concrete FA1, the [OH<sup>-</sup>] dropped significantly with time, and then plateau around 0.95M. This is however above the minimum [OH<sup>-</sup>] threshold to sustain ASR, suggesting that other factors beyond pH reduction contributed to ASR mitigation in AAFA concretes. It should be noted that the [OH<sup>-</sup>] values in pore solution of concrete prisms undergoing ASTM C 1293 test (CPT) are likely to be lower that the values obtained from sealed pastes, due to the effect of alkali leaching.

In contrast to AAFA, the [OH] in OPC paste often increases with age [19], [20]. In a previous study [21], a cement paste with similar cement composition (i.e. alkali content) and w/c=0.47 showed [OH]=0.78M immediately after mixing, which increased to [OH]=1.10M at 220 days under sealed curing condition.

In addition to [OH-], the pore solution concentrations of Al and Ca (Figures 2-D and 2-E) could be of significance with respect to ASR mitigation. Both, Al and Ca, originate from dissolution of fly ash, have their concentrations risen in the pore solution paste, likely over the first 7 days. Afterwards, their concentrations decline due to reaction with dissolved silica to form N-(C)-A-S-H binder products. For paste FA1, the long-term (225 days) Al concentration is 3.87mM. Past research [10] demonstrated that [Al]>3.9mM in the pore solution of concrete is sufficient to significant decrease aggregate deterioration due to OH- attack. This matches well with ASR expansion results for concrete FA1, as well as SEM images showing absence of aggregate deterioration in this concrete. In FA2, [Al] was considerably lower, possibly due to a lower pH, which reduces the solubility of Al in water. It is interesting to note that in concrete FA2, despite a lower pH, signs of aggregate deterioration (per SEM) were observed. This could underline the significance of dissolved Al in mitigating ASR.

Ca concentration in AAFA pastes spiked at early ages for FA1, due to dissolution of fly ash. However, [Ca] plateaued over time to low values in both FA1 and FA2 pastes. In OPC pastes, due to elevated availability of solid Ca[OH]<sub>2</sub>, calcium concentration in pore solution tends to remains near (and in fact slightly above) the Ca[OH]<sub>2</sub> solubility limit. In contrast to OPC, no solid Ca[OH]<sub>2</sub> is generated in AAFA pastes, as reported by previous studies [22]. Instead, the main AAFA reaction product (N-A-S-H gel) tends to bind all Ca from the pore solution and generate N(C)-A-S-H [22].

The concentration of other elements (Na, K, Si) in AAFA paste pore solutions, all showed a declining trend with age, due to fly ash geopolymerization reactions. The formation of N-A-S-H products consumes and binds all major elements from the pore solution. In effect, fly ash reactions (dissolution and geopolymerization) compete with ASR in consuming OH, Ca, Al, and alkali ions from the pore solution. And the higher surface area of fly ash, compared to that of aggregates, provides fly ash with a significant advantage in such competition.

#### Mercury Intrusion Porosimetry (MIP)

The MIP results (Table 4) show that paste FA1 had the highest total porosity and average pore size among the three pastes. In comparison with OPC paste, FA2 had a similar average pore size, a slightly smaller porosity, but a considerably larger threshold pore diameter. Comparison of these results with the expansion results (Figure 1) may suggest that mass transport (facilitated by larger porosity and pore size) was likely not a significant factor in determining the ASR activity of the concrete mixtures studied here. Concrete FA1 probably allows the fastest mass transport and permeability, but it showed the lowest ASR expansion and aggregate damage. However, it should be noted that FA1, in comparison to the other two mixtures, may be more susceptible to alkali leaching during ASTM C 1293 test, due to its larger porosity and pore size distribution.

# 5. CONCLUSIONS

The following conclusions can be drawn:

- Despite containing a highly reactive coarse aggregate, AAFA concretes studied in this research did not show ASR expansions exceeding the 0.04% threshold of the concrete prism test (ASTM C 1293).
- A likely significant contributor to ASR mitigation in AAFA concretes is the rapid decline of pore solution alkalinity over time as a result of reactions with fly ash. Despite an initially very high alkalinity of the AAFA mix water ([OH<sup>-</sup>]=10M or 6.3M for alkali activators in FA1 and FA2, respectively), [OH<sup>-</sup>] dropped rapidly to 0.95M for FA1 and 0.13M for FA2 in 90 days.
- Another major contributor to ASR mitigation was the presence of dissolved Al in the pore solution of mixture FA1, which may drastically reduce aggregate dissolution.
- SEM results suggest that concrete FA2, which had lower pH but also lower [Al], experienced some aggregate deterioration.
- Based on the results of porosity and pore size distribution measurements, mass transport (e.g., water permeability) was likely not a significant factor in determining the ASR reactivity of the three concrete mixtures studied here.

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TABLE 1: Mixture proportions of AAFA and OPC concretes.

Mix ID	NaOH (kg/m³)	Water (kg/m³)	Na2SiO3 (kg/m <sup>3</sup> )	Fly ash (kg/m³)	OPC (Kg/m³)	Oley sand (Kg/m³)	Spratt coarse agg. (Kg/m³)
FA1	58.06**	93.68**	34.08**	479.22	-	659.2*	1047.28*
FA2	36.89**	59.52**	96.1**	479.22	-	659.2*	1047.28*
Control (OPC)	1.97**	199.99**	-	-	420	703.42*	1047.28*
*Values expressed in OD condition							
**Values adjusted to account for absorption by aggregates							

TABLE 2: Properties of AAFA and OPC mixtures.

Mix ID	I/B (volumetric)	Activator solution composition					
	L/D (volumetic)	рН	n	Water (%)			
FA1	0.8	15	0.21	61.8			
FA2	0.8	14.8	0.76	62.1			
Control (OPC)	1.42*	Neutral	0	100			
* Equivalent to $w/c=0.45$ by mass.							

TABLE 3: Oxide composition (mass %) and properties of OPC and fly ash.

Oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO3	LOI	Specific Gravity	% Passing 45μm
OPC	19.90	5.44	2.26	62.50	2.31	0.30	0.89	4.93	2.51	3.15	81.0
Fly ash	46.69	22.44	19.43	4.99	1.04	0.58	1.77	0.76	2.00	2.64	98.4

TABLE 4: Pore characteristics of AAFA and OPC pastes

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	Continuous pore	Threshold	Average pore	Total Porosity					
	diameter (nm)	diameter (nm)	diameter (nm)	(% Vol)					
FA1	678.59	929.18	35.92	26.07					
FA2	110.95	204.22	13.46	22.28					
OPC	21.67	40.22	12.73	23.13					



FIGURE 1: Expansion curve of AAFA and OPC concretes (ASTM C1293).



FIGURE 2: Unreacted aggregate in FA1 concrete.



FIGURE 3: Sign of aggregate deterioration in FA2.



FIGURE 4: Pore solution composition of AAFA pastes sealed and stored at 38 °C and 100% RH: A) [OH<sup>-</sup>]; B) [Na]; C) [Si]; D) [Al]; E) [Ca]; F)[K].