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Assessment of alkali-silica reaction related expansion and damage in alkali-activated slag/fly ash concretes

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Abstract

Alkali-activated concretes involve the use of highly alkaline chemical activators like sodium silicate and sodium hydroxide which raises questions about their durability when alkali-silica reactive aggregates are used. Therefore, as part of a wider study dedicated to alkali-activated slag/fly ash concretes, mixtures were prepared with non-reactive (High purity – HP), moderately reactive (Sudbury – Su), highly reactive (Spratt – Sp) and extremely reactive (New Mexico – NM) aggregates. Mixtures with fly ash contents ranging from 20 to 40% were tested for two years using an approach inspired by CSA Standard Practice A23.2-28A. At the completion of the two-year testing period, the condition of the test specimens with expansion values exceeding the 0.040% limit was assessed by petrographic examination using the *Damage Rating Index* method (DRI).

Increasing fly ash replacement levels in the tested alkali-activated concretes resulted in reduced expansion and damage levels for all investigated reactive coarse aggregates. After two years of testing, only specimens from the mixtures with 20% of fly ash and either NM or Sp aggregates exceeded the 0.040% expansion limit. For these two concrete mixtures, DRI values of 364 and 235 were recorded, respectively, thus classifying them as slightly to moderately deteriorated. In comparison, the DRI values recorded for the 40% fly ash concretes with NM and Sp reactive aggregates reach 203 and 86, respectively (non to slightly deteriorated), whereas the 20% fly ash concretes with the non-reactive aggregate did not show any significant deterioration with a DRI value of 70; the latter was mainly associated with (pre-existing) cracks in the non-reactive aggregate related to aggregate processing operations.

Keywords: alkali-activated concrete; alkali-silica reaction; cracking; expansion; slag/fly ash

1. INTRODUCTION

Alkali-activated concretes incorporating aluminosilicate-type precursors require highly alkaline solutions for paste formation and hardening to occur. This justifies the need to investigate the behaviour of these systems towards ASR. It is well known that three conditions are essential for ASR to develop which are, high alkalinity, sufficient humidity (over 80%) and the presence of reactive siliceous phases in the aggregate particles [1]. Previous work on alkali-activated slag-based [2-4] and fly ash-based [5-7] mortars all showed lower expansions than equivalent OPC specimens although higher alkali concentrations in the pore solution are measured in alkali-activated systems [8]. The lower ASR related expansions in alkali-activated slag-based mortars when compared to OPC systems is believed to result from the lower [OH-] of these systems attributed to the binding of the alkalis in the binder products and the absence of portlandite [9]. The higher aluminum contents of slag and fly ash precursors can also explain the lower expansions values of these systems. Chappex and Scrivener [10] showed that aluminum is incorporated into the framework of reactive silica thus reducing its dissolution rate.

Although much work was done on different alkali-activated mortars, limited research exists regarding the development of ASR in alkali-activated concretes. Therefore, the main objective of this work is to evaluate the level of expansion reached in alkali-activated blended slag/fly ash concretes with different reactive aggregates and quantify their associated damage using the *Damage Rating Index* (DRI) method.

2. MATERIALS AND METHODS

2.1 Materials

A grade 80 ground granulated blast furnace slag (GGBFS) and a class F fly ash (FA) were used as precursors (Table 1). The GGBFS and the FA have a Blaine fineness of 479 and 334 m²/kg, respectively.

Oxides, wt.%	SiO ₂	Al ₂ O ₃	CaO	MgO	Fe ₂ O ₃	Na ₂ O	K ₂ O	TiO ₂	LOI
Slag Gr. 80	37.74	10.75	36.20	12.62	0.50	0.41	0.49	0.91	-0.8
Class F Fly Ash	56.72	24.07	9.29	1.05	3.14	2.50	0.64	0.65	1.2

Table 2.1: Chemical composition of precursors in major oxides %.

A mixture of sodium silicate solution (28.7% SiO₂, 9% Na₂O, 62.3% H₂O) combined with an 8 M sodium hydroxide solution at a sodium silicate-to-sodium hydroxide ratio of 0.5 was used as activator. The activator was prepared 24 hours before mixing to avoid heating of the fresh concrete while mixing. Table 2.2 presents the different aggregates used for testing and their main properties.

Aggregates	Bulk density, kg/m³	Absorption, %	Туре	Lithologies	ASR reactivity level
High purity limestone (HP)	2716	0.19	Crushed aggregate	Limestone	Non-reactive
Spratt (Sp)	2675	0.43	Crushed aggregate	Siliceous limestone with traces of chert	High
New Mexico (NM)	2534	1.52	Natural gravel	Andesite and rhyolite	Extreme
Sudbury (Su)	2687	0.50	Natural gravel	Siltstone, granite, claystone, greywacke, quartzite sandstone	Moderate
Non-reactive sand	2670	0.64	Natural sand	Derived from granite	Non-reactive

Table 2.2: Physical properties and characteristics of the coarse and fine aggregates.

2.2 Concrete mixtures

Table 2.3 presents the tested concrete mixtures with the proportions of coarse aggregate adjusted to an equivalent volume. Fly ash contents of 20, 30 and 40% of total precursor were used in replacement of GGBFS. Table 2.4 presents the reference 100% OPC concretes incorporating the Su and Sp aggregates from Lafrenière (2017) [11].

Aggregate	Aggregate Proportions, kg/m ³				Precursor.	Sand.	Added	NaOH	Na ₂ SiO ₃
S	14-20 mm	10-14 mm	5-10 mm	10 Total kg/m ³		kg/m³	water, kg/m³	8M, kg/m³	, kg/m³
HP	425	319	319	1063		670	56	94	47
Sp	419	314	314	1047	400				
NM	397	298	298	992	400				
Su	421	316	316	1052					

Table 2.3: Mixture proportions of tested alkali-activated concretes.

Aggregates	Aggregate Proportions, kg/m ³				Cement.	Water.	Sand,	
	14-20 mm	10-14 mm	5-10 mm	Total	W/B	kg/m³	kg/m³	kg/m³
Su	356	356	356	1068	0.43	422	181	718
Sp	367	356	356	1079	0.43	422	181	720

Table 2.4: Mixture proportions of reference OPC concretes with reactive Su and Sp aggregates [11].

2.3 Testing

2.3.1 Compressive strength

The compressive strength at 7 and 28 days was evaluated for all concrete mixtures in accordance with CSA A23.2-9C on three concrete specimens, 100 x 200 mm in size, per mixture.

2.3.2 Expansion related to ASR

The ASR testing was performed in accordance with the CSA A23.3-28A Standard Practice with some minor adjustments. Instead of the required 420 kg/m³ binder content, a 400 kg/m³ fixed content was used to maintain engineering properties of the tested mixtures. Moreover, no additional alkalis were added to the concrete mixtures due to the already high alkali contents of the activators. All concrete mixtures were made in a 40-L pan mixer. Test specimens (three per mixture), 75 x 75 x 300mm in size, were cured for 24 hours at 23°C and 100% R.H. before demoulding. During testing, the specimens were kept in hermetic plastic containers at 38° C and 100% R.H. and expansion readings were made at selected ages over a two-year period. Prior to length change measurements, the plastic containers were removed from the high-temperature room and stored for 16 \pm 4 hours at 23°C. After 2 years of testing, expansion values greater than 0.040% indicate a reactive system.

2.3.3 Damage rating index

Petrographic examination was performed on ASR affected concrete specimens following the method of the Damage Rating Index (DRI) adapted from Villeneuve *et al.* [12]. The selected specimens were analysed after completion of the CSA A23.2-28A standard procedure (i.e. after two years of expansion monitoring). Therefore, one concrete prism from each selected alkali-activated mixture was cut in two halves along its length and one of the two resulting surfaces was polished using a portable polishing device with different diamond-impregnated disks (no. 50 (coarse), 100, 400, 800 to 3000 (very fine)). After polishing, a grid composed of squares of 1 cm² was drawn on each polished section. Under a stereomicroscope, damage features are then counted for each square of the grid. The observed quantities of each features were then multiplied by their respective weighing factor and added up to obtain the DRI number, which is normalized to a 100 cm² area. Table 2.5 presents all the damage features and their weighting factors. A *Closed cracks in cement paste (CCCP)* feature was added and a weighing factor of 1 was attributed to it because of the probable low impact of this feature on the overall mechanical properties of the concretes. These cracks (CCCP) are very thin and already present at an early-age (<28 days) before significant initiation of ASR. The overall DRI values can be compared to the classification presented in table 2.6.

Petrographic features	Weighing factors
Closed cracks in coarse aggregate particle (CCA)	0.25
Opened cracks or network cracks in coarse aggregate particle (OCA)	2
Cracks or network cracks with reaction product in coarse aggregate particle (CA + RP)	2
Closed cracks in cement paste (CCCP)	1
Cracks in cement paste (CCP)	3
Cracks with reaction product in cement paste (CCP + RP)	3
Coarse aggregate debonded (Debon)	3
Disaggregated/corroded aggregate particle (RAP)	2

Table 2.5: Petrographic features and their weighing factors for the revised DRI method [12].

Damage classification	Reference expansion level, %	DRI (lab specimen)	Compressive strength loss, %		
Negligible	0.00-0.03	100-155	-		
Marginal	0.04 ± 0.01	210-400	(-) 10-15		
Moderate	0.11 ± 0.01	330-500	0-20		
High	0.20 ± 0.01	500-765	13-25		
Very high	0.30 ± 0.01	600-925	20-35		

Table 2.6: ASR damage classification for the DRI adapted from Fournier et al. [13] and Sanchez et al. [14].

2.4 Statistical analysis and errors

Compressive strength and expansion test results were analysed in terms of statistical error using the student distribution with a 95 % confidence interval. A margin of error related to the operator experimental precision was calculated for the DRI values using a technique developed by Champagne (2020) (to be published).

3. RESULTS AND DISCUSSION

3.1 Compressive strength

Table 3.1 presents the 7 and 28-day compressive strengths of all tested alkali-activated concrete mixtures. All the tested specimens were kept at 23°C and 100% R.H. until due for testing. For a given fly ash content, the 7-day compressive strengths are statistically identical with average values of 40.6, 38.6 and 36.3 MPa for the 20, 30 and 40% fly ash concretes, respectively. The 28-day compressive strengths of concretes incorporating the HP, Su and Sp aggregates are similar for any given fly ash content or aggregate with an average value of 50 MPa. The alkali-activated NM concretes with 20, 30 and 40% fly ash show slightly lower compressive strengths of 45.8, 43.5 and 39.0 MPa, respectively.

Table 3.1: Compressive strength after 7 and 28 days of moist curing of all ASR-tested alkali-activated	d
slag/fly ash concretes.	

Concr	ete mixture	Compressive	strength
Aggregate	Slag/Fly ash, % of binder	7-day, MPa	28-day, MPa
HP	80/20	39.9 ± 5.2	48.8 ± 8.5
Su	80/20	40.2 ± 2.2	52.4 ± 1.7
Su	70/30	38.4 ± 0.4	51.1 ± 4.3
Su	60/40	34.6 ± 5.6	50.3 ± 3.5
Sp	80/20	41.9 ± 1.1	48.0 ± 6.5
Sp	70/30	38.8 ± 1.4	52.2 ± 5.2
Sp	60/40	38.3 ± 1.3	47.4 ± 6.1
NM	80/20	40.5 ± 1.5	45.8 ± 11.3
NM	70/30	38.6 ± 3.6	43.5 ± 1.5
NM	60/40	35.9 ± 2.2	39.0 ± 3.0

3.2 Expansion and damage related to ASR

Figure 3.1 illustrates the expansion as a function of time for the various series of specimens tested. In all cases, increasing fly ash contents resulted in decreasing two-year expansions and for any given reactive aggregate. The only alkali-activated slag/FA concretes to exceed the 0.04% limit are the ones with 20% FA and the NM and Sp aggregates after 60 and 70 weeks, respectively. In comparison, OPC concrete prisms with Sp aggregates show an average expansion of 0.189% after one year of testing [11]. The NM 20% FA and Sp 20% FA showed two-year expansions of 0.054% and 0.046%, respectively. The alkali-activated concretes incorporating the Su aggregate showed two-year expansions of 0.011%, 0.007%, and -0.002% for FA contents of 20%, 30%, and 40%, respectively. In comparison, OPC concrete prisms with Su aggregates show an average expansion of 0.143% after one year of testing [11]. The non-reactive HP 20% FA mixture showed a two-year expansion of 0.006%. In all cases, the alkali-activated slag/FA concretes show much lower two-year expansions than OPC concretes incorporating the same reactive aggregates.

Table 3.2 presents the average two-year expansions for all tested alkali-activated concretes and, for specimens exceeding the two-year 0.04% expansion limit and the non-reactive (HP) specimens, their related DRI values. The damage observed in the tested specimens is, at most, marginal and the DRI values are all consistent with their respective average two-year expansions. The highest expansions and DRI values were observed in concretes with extremely reactive aggregates (NM) with a 0.054% expansion and 364 DRI for the 20% fly ash content. Moreover, when compared to OPC systems incorporating the same reactive aggregates, alkali-activated slag/fly ash concretes show much lesser damage. For example, OPC concretes with NM aggregates show DRI values over 600 for expansions of 0.25% [15]. Such damage is considered to be high to very high (Table 2.5).



Figure 3.1: Two-year concrete prism expansion of alkali-activated slag/FA mixtures incorporating reactive (NM, Sp, Su) and non-reactive (HP) aggregates compared to 100% OPC concretes [11] incorporating reactive (Sp, Su) aggregates.

Table 3.2: Average two-year expansions and DRI values for alkali-activated slag/fly ash concretes with
non-reactive (HP), moderately reactive (Su), highly reactive (Sp) and extremely reactive (NM)
aggregates. The margin of error for the DRI number is based on Champagne (2020).

Mixture	Average two-year expansion, %	DRI	Two-year expansion of specimen tested for DRI, %	Damage classification
HP 20%FA	0.006 ± 0.003	70 ± 19	0.004	Negligible
Su 20%FA	0.011 ± 0.008	-	-	-
Su 30%FA	0.007 ± 0.008	-	-	-
Su 40%FA	-0.002 ± 0.019	-	-	-
Sp 20%FA	0.046 ± 0.017	235 ± 31	0.035	Marginal
Sp 30%FA	0.023 ± 0.006	-	-	-
Sp 40%FA	0.017 ± 0.013	86 ± 20	0.013	Negligible
NM 20%FA	0.054 ± 0.004	364 ± 39	0.053	Marginal
NM 30%FA	0.032 ± 0.017	-	-	-
NM 40%FA	0.031 ± 0.004	201 ± 28	0.029	Negligible to marginal

Figure 3.2 presents the average condition of the polished sections from the alkali-activated specimens that were examined for DRI determination. Secondary reaction products are present in all the specimens with NM (Figure 3.2A and B) and Sp (Figure 3.2C and D) reactive aggregates. Figure 3.2A (NM aggregate) shows secondary products at the paste-aggregate interface (*CCP+RP and Debon*) whereas Figure 3.2B shows cracks within an NM aggregate particle with secondary products (*CA + RP*). Figure 3.2C shows the widespread cracking present in specimen Sp 20% fly ash that spreads across the aggregate particles and in the paste. Much of these cracks are filled with secondary reaction products. As for the Sp 40% FA (Figure 3.2D) specimen, most of the cracks with secondary reaction products are located within the aggregate particles (*CA + RP*). Finally, the HP 20% FA (Figure 3.2E-F) concrete specimen shows no secondary reaction products and the cracking is limited to closed cracks within the aggregate particles (CCA) which likely result from quarrying operations. Rodrigue *et al.* (2020) [16] confirm, after examination under the scanning electron microscope of specimens similar to those illustrated in figure 3.2, that the secondary products within the reactive aggregate particles are of similar nature to those generally associated with ASR.

Table 3.3 presents the detailed DRI results of the tested specimens. Increasing the fly ash content results in decreasing features of Cracks in the aggregates with reactive products (CA + RP) and Cracks in the paste with reactive products (CCP + RP). In concrete specimens incorporating the NM aggregate, increasing the fly ash content from 20 to 40% results in a decrease of the counts of CA + RP and CCP + RP features from 31 to 9 and from 35 to 10, respectively. In concrete specimens incorporating the Sp aggregate, counts for the CA + RP and CCP + RP features decreased from 12 to 6 and from 24 to 1 with increasing fly ash content (20 to 40%), respectively. In all cases, increasing the fly ash content from 20 to 40% results in decreasing counts of closed cracks in the paste (CCCP) from 60 to 33 and 75 to 21 for the NM and Sp aggregate mixtures, respectively. Previous work done on these concretes has shown that this feature is likely related to early-age autogenous shrinkage [17]. This early-age cracking could represent an issue in terms of water ingress through the paste. Ongoing work on these concrete mixtures will help clarify the importance of this damage feature when compared to other potential issues like alkali content in the pore solution. Rodrigue et al. [17] has also demonstrated that increasing fly ash contents results in increasing initial and final setting times on alkali-activated paste specimens. These observations imply lower early-age paste stiffness with higher fly ash contents that could more easily accommodate the early-age volume variations thus reducing the early-age cracking.

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Figure 3.2: Micrographs of polished concrete sections of the NM 20% FA (A) (0.053% expansion), NM 40%FA (B) (0.029% expansion), Sp 20%FA (C) (0.035% expansion), Sp 40%FA (D) (0.013% expansion) and HP control (E and F) (0.004% expansion) specimens. (See Table 2.5 for damage feature classifications).

Table 3.3: Detailed DRI results for the alkali-activated slag/fly ash concrete specimens with NM (20 and 40%FA), Sp (20 and 40%FA) and HP (20%FA) aggregates. The margin of error for the DRI number is based on Champagne (2020).

Specimen		CCA	OCA	CA + RP	Debon	RAP	ССР	CCP + RP	CCCP	DRI
	Sum of counts / 100 cm ²	111	40	31	3	1	6	35	60	
NIVI 20 %FA	DRI weight	28	80	61	9	2	19	105	60	504 ± 59
NM 40%EA	Sum of counts / 100 cm ²	79	28	9	2	0	13	10	33	201 + 28
NM 40%FA	DRI weight	20	56	17	6	0	40	29	33	201±28
	Sum of counts / 100 cm ²	186	2	12	2	0	4	24	75	235 ± 31
Зр 20 <i>%</i> ГА	DRI weight	46	4	23	5	0	11	72	75	
Sp 40% EA	Sum of counts / 100 cm ²	127	3	6	0	0	3	1	21	86 ± 20
Sp 40%FA	DRI weight	32	7	12	0	0	10	4	21	
HP 20%FA	Sum of counts / 100 cm ²	175	1	0	1	0	1	2	11	70 ± 19
	DRI weight	44	3	0	3	0	3	6	11	

4. CONCLUSION

Damage related to ASR can occur in alkali-activated slag/fly ash systems when reactive aggregates are used. In alkali-activated slag/fly ash concretes with a 20 % fly ash content, average two-year expansions of 0.006 (DRI = 70), 0.011, 0.046 (DRI = 235) and 0.054 % (DRI = 364) were recorded when incorporating the non-reactive, moderately reactive, highly reactive and extremely reactive aggregates selected for this study, respectively. However, even when extremely reactive aggregates are used, the overall damage classification can only be described as negligible to marginal after two years of testing under the standard conditions generally used for evaluating the preventive measures against ASR (Standard Practice CSA A23.2-28A). Increasing the fly ash contents of alkali-activated slag/fly ash concretes results in decreasing two-year expansions for all the tested aggregates and decreasing related DRI values. Two-year expansions of -0.002, 0.017 (DRI = 86) and 0.031 % (DRI = 201) were recorded for alkali-activated slag/fly ash concretes with a 40 % fly ash content and incorporating the moderately, highly and extremely reactive aggregates, respectively. Moreover, this work has allowed the introduction of an additional feature of deterioration in the DRI method/procedure: the *closed cracks in the paste (CCCP)* feature. More work on the influence of this damage feature is needed but it could

potentially represent an issue regarding water availability for ASR development. Increasing the fly ash content (20 to 40% of binder content) results in decreasing values of CCCP, cracks in the aggregates with reaction products (CA +RP) and cracks in the paste with reaction products (CCP + RP) for all the tested aggregates.

Further investigations will be needed to assess if a good correlation exists between laboratory results and the behaviour of these systems in practice.

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