Better Interpretation Techniques for the Alkali-silica Reaction of Aggregates

Namshik Ahn¹, Khoa Tan Nguyen², Chan Hong Nguyen³, Jaehong Lee⁴ and Young Hak Lee*⁵

¹Professor, Free Form Architecture Institute, Department of Architectural Engineering, Sejong University, Korea
²Ph.D. Candidate, Free Form Architecture Institute, Department of Architectural Engineering, Sejong University, Korea
³M.S. Candidate, Free Form Architecture Institute, Department of Architectural Engineering, Sejong University, Korea
⁴Professor, Free Form Architecture Institute, Department of Architectural Engineering, Sejong University, Korea
⁵Associate Professor, Department of Architectural Engineering, Kyung Hee University, Korea

Abstract
In the construction industry today, there is a need for improved testing procedures capable of accurately predicting the susceptibility of aggregates to the alkali-silica reaction (ASR). These new procedures will permit the use of some aggregates that have been excluded in the past on the basis of being classified as reactive. Effective mitigation methods will permit the economic use of reactive aggregates that would normally be excluded. Increasing the quality of the aggregate sources the target of this research, the results of which will help manufacturers sell more products while cutting prices for consumers. By modifying ASTM C1260, we expanded the length of testing from 14 to 56 days, changed the water content of the mixing proportion with the water to cement ratio controlled at 0.47, the molarity of the curing solution, and used different interpretation methods, specifically Avrami's model and the polynomial fit procedures. If the results of this research enabled even 25 percent of these materials to be used, the increased volume available for use would increase substantially.

Keywords: ASTM C1260; alkali-silica reaction; aggregates; expansions

1. Introduction
The alkali-silica reaction (ASR) is one of the most recognized deleterious phenomena in concrete. ASR is a chemical reaction between the reactive silica contained in the aggregates and the alkalis (Na₂O and K₂O) within cement paste¹⁻³. The result is an alkali-silicate gel that absorbs water and increases in volume. If the gel is confined by the cement paste, it builds up pressure as it grows causing internal stresses that eventually could crack the concrete. Identifying the susceptibility of an aggregate to the alkali-silica reaction (ASR) before using it in concrete is one of the most efficient practices to prevent damage. Several tests have been developed to identify aggregates subject to ASR such as ASTM C295, C227, C1260 or C1293, but each has its limitations. For example, ASTM C1260 is capable of detecting slowly reactive aggregates. The test has considerable value in providing a rapid means of detecting potential ASR aggregates. Recently, certain aggregates have been identified as innocuous when using C1260, but they have reacted deleteriously in the field with low alkali cements. There are also some aggregates that have been identified as reactive when using C1260 but have had over 30 years of good service records⁴. As a result, aggregate expansion results from this test are recommended to be combined with the results of a service record investigation for the aggregate in question or a long-term concrete testing program.

In summary, the overall objective of this study is to closely examine the alkali-silica reaction expansion potential in Portland cement concrete containing different aggregate sources in the United States. Further, this research relates the results of the laboratory testing performed on these aggregates to their reported field performance.

The results of this study will increase the aggregate sources that can be used in concrete, resulting in increased sales for producers and reduced costs for users. No accurate estimates exist regarding the annual tonnage of aggregates that has been eliminated because of the alleged susceptibility of ASR, but if the results of this research enabled even 25 percent of these materials to be used, the increased volume available for use would increase substantially.

*Contact Author: Young Hak Lee, Associate Professor, Department of Architectural Engineering, Kyung Hee University, Korea
Tel: +82-10-7183-2862 Fax: +82-31-204-3815
E-mail: leeyh@khu.ac.kr
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2. Materials and Test Methods

2.1 Materials

a. Aggregates

After contacting knowledgeable researchers and industry representatives, 200 aggregates were compiled from which the selections were made for the testing program. The aggregates possessed different physical and chemical properties, represented different regions of the US, had different field performances regarding ASR performance, and had different laboratory performances when tested for ASR. Aggregates were selected to cover the complete spectrum of ASR. They were also representative of most regions of the US.

Identifications of each aggregate tested were used throughout the study to determine the aggregate source. The first letter indicates the category to which the aggregate belongs. The next number indicates the number of that aggregate in its specified category. An odd number indicates a coarse aggregate while an even number indicates a fine aggregate. The next two letters indicate the state from which the aggregate was sent. For example, A1-WY is a Category A coarse aggregate from Wyoming and E4-NV is a Category E fine aggregate from Nevada.

Table 1. Aggregates and Sources Selected for the Study

| Category A: Poor field performance / tested reactive | Category B: Poor field performance / tested inconclusive |
|---------------------------------------------------|--------------------------------------------------------|
| A1-WY, A2-WY, A4-ID, A6-NM, A7-NC, A9-NE, A10-PA | B2-MD, B4-VA                                            |
| Category C: Poor field performance / tested nonreactive (slow reactive) | Category D: Good field performance / Tested nonreactive |
| C2-SD                                             | D2-IL                                                  |
| Category E: Good field performance / tested reactive |                                                        |
| E2-1a, E4-NV, E6-IN, E8-NM                                                                      |

b. Cement

Two types of cement with an average alkali content of 0.60 and 1.14% were used throughout the study.

2.2 Methods

The laboratory testing-program was divided into two stages:

• Stage 1 consisted of testing each aggregate to determine the physical properties required for concrete proportioning. Aggregates were also prepared for mortar and concrete batching.

ASTM C1260 requires that all aggregates be graded as per Table 2. As a result, all aggregates had to be separated into the required sieve sizes, washed over a #100 sieve, and then combined using the specified quantities for each sieve.

• Stage 2 consisted of testing all aggregates using ASTM C1260. Modifications to these tests were also investigated during this stage.

Aggregates were also used to cast 1-in. x 1-in. x 11-in. mortar bars. Three mortar bars were cast using each of the aggregates. Three bars were used because of the accuracy of the test. All mixtures were also tested for flow, unit weight, and air content in accordance with the respective requirements of ASTM C109 and C138. After 24 hours of moist curing, bars were stored in water at 80°C for 24 hours after which the initial readings were recorded. Subsequent measures consisted of storing the bars in a 1N NaOH solution at 80°C and recording expansion readings at 4, 7, 11, 14, 21, and 28 days. A 14-day expansion greater than 0.20% indicated a potentially reactive aggregate in the field, while a 14-day expansion smaller than 0.10% indicated a non-reactive (innocuous) aggregate. Recent specifications have mentioned that 14-day expansions higher than 0.10% should be used to classify aggregates as reactive.

Several research programs and papers have documented the effectiveness of C1260 for predicting the potential alkali-reactivity of aggregates and for investigating the effectiveness of mitigation alternatives such as the use of air entrainment, mineral admixtures, pozzolans, and chemical admixtures. In general, most researchers and users agreed that this test has good ASR prediction; however, it is too severe for some aggregates that have had good field performances (Category E aggregates). To overcome this obstacle, several modifications of the C1260 were investigated as follows:

1. Using different interpretation methods: Avrami's model and the polynomial fit procedures, both of which were discussed earlier.
2. Changing the molarity of the curing solution: 0.75N, 0.50N, and 0.25N.
3. Expanding the length of testing from 14 to 56 days.
4. Changing the water content of the mixing proportions so as to account for the absorption of the tested aggregate. The water-cement ratio is controlled at 0.47. This modification was not used to remedy the Category E problem but to improve the consistency of the mixing procedures.

This test was used to investigate its validity in predicting the alkali-silica reactivity of aggregates in concrete and to investigate potential ASR mitigation alternatives. ASTM C138 was used to measure the unit weight, yield, and air content, and ASTM C109 was used to measure the flow. The details of the mixture proportions for ASTM C1260 are graded aggregate 990 g (dry weight), cement 440 g, and water 207 g. The water-to-cement ratio is by mass 0.47. The unit weight γ is around 2054 to 2237 kg/m³, and the air content is about 0.26 to 2.88%. The mixture proportions for
absorption of aggregates are graded aggregate 1665 g (dry weight), cement 740 g, and water 353-443 g. The unit weight $\gamma$ is around 1988 to 2258 kg/m$^3$, and the air content is about 1.18 to 2.86%.

3. Results and Discussion

Aggregates are separated into specified sieve sizes, combined using specified amounts of each sieve size, and mixed with cement to make 1-in. x 1-in. x 11-in. mortar bars. After 24-hours of moist curing, the mortar bars are stored for an additional 24 hours in water maintained at 80°C, after which the bars are stored in a 1N NaOH solution maintained at 80°C. Expansion readings are taken at 4, 7, 11, and 14 days of storage in the NaOH solution. Fourteen-day expansions higher than 0.20% are considered reactive, 14-day expansions lower than 0.10% are considered innocuous, and 14-day expansions between 0.10% and 0.20% are considered inconclusive. Fig. 1 illustrates the expansions in graphical form.

The criteria proposed in the standard ASTM C1260 document to determine the reactivity of aggregates are as follows: 14-day expansions lower than 0.10% are considered innocuous, 14-day expansions between 0.10% and 0.20% are considered inconclusive, and 14-day expansions greater than 0.20% are considered reactive. Based on the results of the study conducted by Starks (1993)$^5$, it was concluded that aggregates showing 14-day expansions higher than 0.08% are considered reactive and 14-day expansions lower than 0.08% are considered innocuous. However, this conclusion was based on a limited amount of data and the 0.10% expansion criterion has been adequately justified for a voluminous amount of aggregates. As a result, the 0.10% criterion was adopted throughout this study for the interpretation of the ASTM C1260 results.

Aggregates listed in Category A are rapidly reactive and have shown 14-day expansions varying between 0.24 and 0.91%, all larger than 0.20%. Aggregates listed in Category B and C are slowly reactive and have shown 14-day expansions varying between 0.12 and 0.17%. Aggregates listed in Category D are innocuous and have shown 14-day expansions varying between 0.03 and 0.06%. Category E aggregates have shown 14-day expansions varying between 0.25 and 0.42% all larger than 0.20%, which implies that all Category E aggregates are reactive. Given these discrepancies, it can be concluded that the ASTM C1260 results were too severe for E2-IA and E6-IN.

Using the 14-day expansion criterion of 0.10%, efficiently detecting highly reactive aggregates that showed 14-day expansions greater than 0.20% was possible.

The test was also particularly useful in detecting slowly reactive aggregates showing expansions between 0.10% and 0.20%. Thus, 14-day expansions between 0.10% and 0.20% should not be considered inconclusive but rather as slowly reactive. The power of these procedures lies in being able to detect reactive aggregates, which was demonstrated by the correct classification of E4-NV and E8-NM. Even though the aggregates were erroneously listed as innocuous, the test allowed the correct assessment of their reactivity.

3.1 ASTM C1260 Performed by the National Aggregate Association (NAA)

In order to test the accuracy of the C1260 testing procedures, three aggregates, used in this study, were tested by NAA. The same cement used in this study was used by the NAA. The C1260 expansions for these
aggregates were reported as shown in Table 3. A comparison between the NAA results and those generated in this study is included in Table 3.

An inter-laboratory study evaluating the variation of C1260 resulted in the following conclusion: "For mortars giving average expansions after 14 days in a solution of more than 0.30%, the multi-laboratory coefficient of variation has been found to be 14.9%. Therefore, the results of two properly conducted tests in different laboratories on specimens of a sample of aggregate should not differ by more than 42% of the mean expansion" (Rogers 1996).

It can be seen from that table that the variations between the results of both labs are small and the results can be considered accurate. This was done to measure the accuracy of the testing being performed for this study and the results show that testing was conducted properly.

Table 3. ASTM C 1260 Performed by NAA

| Aggregate ID | Expansion, % |
|--------------|--------------|
|              | 3-day | 7-day | 11-day | 14-day |
| A2-WY        | 0.19   | 0.22  | 0.26   | 0.27   |
| A4-ID        | 0.43   | 0.58  | 0.68   | 0.73   |
| C2-SD        | 0.06   | 0.11  | 0.16   | 0.2    |

A comparison between the 14-day expansions generated for the 56-day procedures were compared to the 14-day expansions generated earlier using ASTM C1260 is shown in Fig.2. ASTM C1260 was essentially repeated, and the 14-day expansions of both sets of data should be comparable and should give an indication regarding the variation of the procedures when the same materials and procedures are used and when the tests are performed by the same laboratory operators.

Table 4. Differences between NAA Results and Results Generated in this Study

|            | A2-WY | A4-ID | C2-SD |
|------------|-------|-------|-------|
| 4-Day      | 5.00% | -5.00%| 17.00%|
| 7-Day      | 0.00% | 6.50% | 8.00% |
| 11-Day     | -6.00%| -9.00%| 19.00%|
| 14-Day     | -5.00%| -8.00%| 12.00%|

Positive = percentage, the NAA result is larger than the one generated in this study
Negative = percentage, the NAA result is smaller than the one generated in this study

3.2 Modified ASTM C1260

a. Expansions up to 56 days

Selected aggregates were retested using the C1260 procedures, but expansions were recorded over 56 days in order to investigate the possibility of a better interpretation of the results. Mixture proportions for these procedures were the same as before.

Roger (1993) suggested expanding the C1260 readings to 56 days instead of 14 and using the following criteria for assessing the reactivity of aggregates: 0.15% at 14 days, 0.33% at 28 days, and 0.48% at 56 days. Thus, for an aggregate to be classified as reactive, it should exceed all three criteria. A study conducted in Australia (Shayan 1992) concluded that these criteria were not effective in detecting the reactivity of several slowly reactive aggregates. These procedures were used to determine whether Category E aggregates could be accurately classified using these limits and whether these limits are effective with slowly reactive aggregates. That is why only two Category A aggregates were tested while all Category B, C, D, and E were investigated.

It can be seen from Fig.3. that the expansions are comparable with some differences that are comparable to the differences noted between the data generated for this study and the data examined by the NAA.

Using the criteria of 0.33% at 28 days and 0.48% at 56 days as proposed by Rogers (1996), the following observations were recorded:

1. Slowly reactive aggregates B4-VA and C2-SD showed 28-day expansions of 0.26%. The two aggregates also exhibited 56-day expansions of 0.46% and 0.48%, respectively. These two aggregates are slowly reactive aggregates as it was determined using ASTM C1260. Thus using the proposed criteria it was not possible to correctly characterize the reactivity of the slowly reactive aggregates tested.

2. Using the same line of reasoning, all Category E aggregates were characterized as reactive, which is true for E4-NV and E8-NM but not for E2-IA and E6-IN.

3. Using the proposed criteria for the 28-day and 56-day expansions, Category D aggregates were correctly characterized as innocuous.

In summary, the criteria were ineffective in detecting slowly reactive aggregates and incorrectly characterizing Category E aggregates.
b. Using a polynomial fitting procedure for interpretation of results

An attempt was made to use a polynomial fit procedure on the C1260 expansion test results versus time for each of the tested aggregates and then to plot the coefficients of these curves against each other. The results are shown in Fig.4. This process is discussed in greater detail by Johnston.

There was separation between reactive and innocuous aggregates as shown by the two lines in Fig.4. These lines were developed using the expansion readings over a duration of 14 days (specified in C1260). The more reactive aggregates were to the left of the graph, and the aggregate reactivity diminished from left to right. The slowly reactive aggregates (Categories B and C) fell in between the two lines. Most of the slowly reactive aggregates and innocuous aggregates were concentrated in the area where the lines meet. As a result, it was concluded that these procedures are not very accurate in assessing the reactivity of aggregates and interpreting the C1260 expansions.

c. Using Kolmogorv-Avrami-Mehl-Johnson’s model for interpretation of results

Johnston concluded that using the model allows the correct prediction of an aggregate reactivity with $\ln(k)$ equal to -6 being the separating value between reactive and innocuous aggregates. Aggregates with $\ln(k)$ greater than -6 are reactive, and aggregates with $\ln(k)$ lower than -6 are innocuous. The results are shown in Fig.5.

All Category D aggregates had $\ln(k)$ values smaller than -6 and $M$ values larger than 1. All Category A aggregates had $\ln(k)$ values larger than -6 and $M$ values smaller than 1. Category B and C aggregates had $\ln(k)$ values between -6 and -4 and $M$ values close to 1. Three of the Category E aggregates had $\ln(k)$ values larger than...
–6, while one had a value slightly smaller than –6. The K-A-M-J model is better than the polynomial fitting procedures. It provides better visualization and is more accurate. However, the model was unsuccessful in correctly predicting the reactivity of Category E aggregates.

This model resulted in the same conclusions as those generated using the 14-day expansion of 0.10% criterion. Thus, even though the model is a more realistic representation of the mortar bar expansions, it did not provide any new advantages.

d. Changing the molarity of the testing solution

In order to investigate the effect of the cement alkali content on ASR using C1260, the investigated aggregates were tested in solutions with varying molarities, namely, 1N (standard), 0.75N, 0.5N, and 0.25N. The results for this set of testing are illustrated in Figs. 6 and 7.

The varying levels of normality could be used to determine a cement alkali level below which the aggregates do not exhibit deleterious expansions. The different normality corresponds to cement alkali content defined by equation 1:

$$[OH^-] = 0.393 \frac{Na_2O}{w_c} + 0.022 \pm 0.06 \text{ moles/L}$$  \hspace{1cm} (1)

The following list of figures illustrates the generated expansion results using 1N, 0.75N, 0.50N, and 0.25N NaOH solutions. In each figure, the corresponding cement alkali content is indicated on a separate axis.

Starks was the first to investigate these procedures. He suggested that the test failure criteria must be adjusted progressively downward from 0.08% at 1.0N to a minimum of about 0.02% as solution normality decreases to about 0.6N. Thus, for the 0.5N (0.81% Na$_2$O equiv.) and 0.25N (0.46% Na$_2$O equiv.) NaOH solutions, the failure criterion should be 0.02% and for the 0.75N (1.15% Na$_2$O equiv.) NaOH solution the failure criterion should be 0.04%. These criteria were used to evaluate the results listed above.

The results indicated that as the solution normality decreased the expansions progressively decreased. The following observations were recorded:

1. The highly reactive aggregates A4-ID and A6-NM of Category A were reactive even when tested using the 0.25N solution (0.46% Na$_2$O equiv.).
2. The moderately reactive aggregates of Category A, namely A1-WY, A2-WY, A7-NC, A9-NE, and A10-PA showed 14-day expansions lower than 0.02% when tested using the 0.25N solution (0.46% Na$_2$O equiv.). When the 0.50N solution was used, all Category A aggregates showed expansions higher than the proposed limit of 0.04%.
3. A solution normality of 0.25N (0.46% Na$_2$O equiv.) was required to decrease the 14-day expansions of the slowly reactive aggregates of Categories B and C below 0.02%. Testing at a higher cement alkali content of 0.81% Na$_2$O equiv. (i.e. higher solution normality of 0.50N) resulted in 14-day expansions higher than the safe limit of 0.04%.
4. Category E aggregates had similar behavior to the slowly reactive aggregates. A solution normality of 0.25N (0.46% Na$_2$O equiv.) was required to decrease the 14-day expansions below 0.02%. Testing at a higher cement alkali content of 0.81% Na$_2$O equiv. (i.e. higher solution normality of 0.50N) resulted in 14-day expansions higher than the safe limit of 0.04%.

This contradicts the field performance reports of E2-IA, which indicate that the aggregate has been successfully used in field application with cement alkali content higher than 0.9%.
In summary, changing the solution molarity was not an effective modification of the ASTM C1260. However, changing the NaOH solution normality can be used to evaluate the effect of cement \( \text{Na}_2\text{O}_{\text{eqv.}} \) content on ASR. Using the proposed limit, it was found that a cement alkali content of about 0.5\% \( \text{Na}_2\text{O}_{\text{eqv.}} \) was effective in decreasing the 14-day expansions to safe levels for moderately and slowly reactive aggregates.

4. Conclusion

The following conclusions were generated on the use of ASTM C1260 in predicting the potential reactivity of aggregates:

ASTM C1260 is valuable in identifying aggregates with reactivity varying from innocuous to rapidly reactive. Aggregates with expansions lower than 0.10\% are considered innocuous. Aggregates with 14-day expansions between 0.10\% and 0.20\% are considered slowly reactive. Aggregates with 14-day expansions higher than 0.20\% are considered rapidly reactive.

Increasing the testing time from 14 days to 56 days and using the limits of 0.33\% at 28 days and 0.48\% at 56 days are ineffective in predicting the correct reactivity of aggregates.

Increasing the water content of the mortar bars to satisfy the absorption of aggregates and keeping a constant water-cement ratio of 0.47 resulted in the same aggregate classification as the standard C1260 procedures.

Using the polynomial fitting procedure to interpret the C1260 results is quite inaccurate. The Kolmogorov-Avrami-Mehl-Johnston model is more effective in representing the C1260 results.

Decreasing the normality of the testing solution can be used to determine the effect of lowering the alkali content of cement. These procedures can also be used to determine the effectiveness of mitigation alternatives at multiple alkali contents. However, it should be noted that these procedures represent worst-case scenarios and will give very conservative results.

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