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Autoclave test parameters for determining alkali-silica reactivity of concrete aggregates

Stephanie G. Wood [a,*], Eric R. Giannini [a,1], Monica A. Ramsey [b], Robert D. Moser [b]

[a] Department of Civil, Construction, and Environmental Engineering, The University of Alabama, Box 870205, Tuscaloosa, AL 35487-0205, United States
[b] US Army Engineer Research and Development Center, 3909 Halls Ferry Road, Vicksburg, MS 39180, United States

HIGHLIGHTS

- Autoclaving is used to determine alkali-silica reaction potential of aggregates.
- Appropriate ranges of autoclave test parameters are established for mortar bars.
- The same test parameters can be used for aggregates of various mineralogies.
- Rapid autoclave tests can be substituted for the accelerated mortar bar test.

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ABSTRACT

The objective of this study was to determine appropriate values for alkali loading, autoclaving temperature, and autoclaving duration for alkali-silica reaction testing of aggregates in mortar bars. Alkali loadings between 0.52 and 4.5%, autoclaving temperatures from 105 to 130 °C, and autoclaving durations between 5 and 48 h were used. A total of 72 tests involving eight aggregates of various mineralogies and reactivities were performed. Appropriate values for test parameters were found to be: alkali loadings between 3.0 and 3.5%, autoclaving temperatures between 120 and 130 °C, and autoclaving durations between 5 and 12 h for mortar bars, regardless of aggregate type.

1. Introduction

There has long been a need to quickly determine potential alkali-silica reactivity of concrete aggregates. The accelerated mortar bar test (ASTM C1260) provides final expansion results within 16 days of mixing, but the test has been known to produce both false positive and false negative test results [1,2]. Though considered to be a more reliable test method, the ASTM C1293 concrete prism test (CPT) requires one year to obtain results using ordinary portland cement concrete and is mainly used as a research tool. These two test methods comprise the majority of laboratory testing for alkali-silica reaction (ASR) in concrete aggregates.

ASR is a significant concrete durability issue in part because the reaction takes place between the constituents of the concrete itself. The reaction occurs when alkali hydroxides in the concrete pore solution attack reactive siliceous minerals present in some aggregates, dissolving the silica into the pore solution [3,4]. These alkalis are typically supplied by the cement but can also come from other sources such as deicing salts and even the aggregates themselves [5]. As a consequence of the reaction, a hydrophilic gel is formed. Absorption of moisture by the ASR gel results in cracking of the surrounding concrete, reducing its mechanical properties [6–8] and rendering the concrete susceptible to moisture ingress and other forms of attack.

The most important step in ASR is the dissolution of silica. Research has shown that silica dissolution is directly related to temperature, pore solution pH, and the amount of calcium present in the pore solution [9–13]. The solubility can also be suppressed by the presence of aluminum in the pore solution [14]. In conditions typically experienced by concrete structures in the field, amorphous and poorly crystalline silica minerals are more likely than quartz to be attacked and react [3,15,16]. In the laboratory, however, parameters that affect the reaction, such as internal

* Corresponding author at: US Army Research and Development Center, 3909 Halls Ferry Road, Vicksburg, MS 39180, United States.
E-mail address: Stephanie.G.Wood@usace.army.mil (S.G. Wood).
[1] RJ Lee Group, 350 Hochberg Road, Monroeville, PA 15146, United States.

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temperature of the concrete and the amount of alkalis present in the pore solution, can be made sufficiently severe to cause all forms of silica to become alkali-silica reactive, leading to false positive results. Likewise, if testing conditions are not sufficiently severe for rapid testing, the aggregate may be falsely identified as innocuous. Therefore, parameters of new test methods for ASR in concrete or mortar should be carefully considered so that false positive and false negative results are avoided.

To rapidly evaluate concrete aggregates, a number of autoclave test methods have been developed to determine the potential for ASR [17–22]. Some of these methods emphasized testing a large number of aggregates while others focused on evaluating the effects of several different test parameters on specimen expansions while testing a relatively small number of aggregates. Alkali loadings in these studies were between 1.5 and 3.5% by mass of cement, and autoclaving temperatures were between 111 and 150 °C. All of the test methods limited the autoclaving durations to between 2 and 6 h for mortar bars and between 4 and 24 h for concrete prisms.

The objective of this study was to determine appropriate parameters for alkali loading, autoclaving temperature, and autoclaving duration for silica dissolution in aggregates of different mineralogies and reactivity classifications in autoclave testing for ASR in mortar bars. Initially, 20 aggregates were tested in ASTM C1260 and the 5-h autoclaved mortar bar test described by Fournier et al. [20]. Eight of those aggregates plus one additional aggregate were tested in the autoclave using different test parameters, including alkali loadings between 0.52 and 4.5%, autoclaving temperatures ranging from 105 to 130 °C, and autoclaving durations between 5 and 48 h. These test parameters and their effects on specimen expansion expand upon what is documented in the literature. Recommendations for autoclaved mortar bar testing are provided based on expansion results in this study.

2. Materials and methods

2.1. Materials

Aggregate descriptions are given in Table 1. Reactivity classifications are based on guidance given in ASTM C1778, which is provided in the table, for interpreting 14-day expansions in ASTM C1260. Aggregates are listed from top to bottom in order of increasing mortar bar expansions. All aggregates were graded in accordance with ASTM C1260 prior to testing except for NR1 (ASTM C778 Graded Ottawa Sand), which was tested using the as-received gradation. Two aggregates, NR1 and HR3, were used more extensively as benchmark aggregates to better examine silica dissolution in the autoclave. Aggregate NR1 was not used in the 5-h autoclave test; it was selected as the non-reactive benchmark aggregate because of its performance in ASTM C1260 and common use in ASR studies as a non-reactive siliceous baseline aggregate [20,23,24]. Aggregate HR3 was selected as the highly reactive benchmark aggregate due to its well-studied performance in laboratory and simulated field exposure testing for ASR [25–27].

Two ASTM C150 Type I/II cements with similar equivalent alkali contents (Na_2O_{eq}) of 0.52 and 0.56% by mass of cement were used for all mixtures. Two cements were used in this study due to the limited supply of one of the cements. Previous work demonstrated the negligible effect of cement alkalinity on expansion in autoclaved concrete prism testing [28]. The cement oxide analyses are provided in Table 2.

2.2. Test procedure

The mixture proportions, aggregate gradations, w/cm (0.47), and specimen size (25 × 25 × 285 mm) matched those specified by ASTM C1260. Sodium hydroxide (NaOH) was added to the mixing water to boost the Na_2O_{eq} to the desired alkali loading. Four bars were cast for each mixture with metal studs embedded in the ends for length change measurements per ASTM C157. All mortar bars were cured for 48 h inside a moist curing room before they were demolded, measured, and autoclaved over a reservoir of deionized water in a Yamato SQ510C sterilizer. Once the autoclave temperature cooled to 90 °C, the bars were removed and cooled to 23 °C in a tap water bath over a period of approximately 15 min. The lengths of the mortar bars were measured once again to determine the expansion after autoclaving. Giannini et al. [22] provided details on the autoclaving procedure followed in this study.

The influence of three test parameters on the reactivities of the aggregates, indicated by the expansion of the mortar bars, were investigated – (1) total alkali loading, or Na_2O_{eq}, (2) autoclaving temperature, and (3) autoclaving duration. Test parameter variations included alkali loadings of 0.52, 0.56, 2.0, 2.5, 3.0, 3.5, and 4.5%, autoclaving temperatures of 105, 110, 115, 120, and 130 °C, and autoclaving durations of 5, 8, 12, 16, 24, and 48 h. Autoclaving duration is the amount of time the autoclave remained at peak temperature.

3. Results and discussion

Expansion results are broken into two sections. The first section involves mortar bars containing the six non-benchmark aggregates. These aggregates were selected from a larger pool of 20 aggregates used in a multi-laboratory study by Wood et al. [29]. Selection was based on aggregate mineralogy and sought to include a representative range of rock types. The second section focuses on the expansion results of mortar bars containing the two benchmark aggregates, NR1 and HR3. The concept behind the testing of these two aggregates was in part to identify extreme values for alkali loading, autoclaving temperature, and autoclaving duration and also to identify acceptable values of these parameters to be used in autoclave testing. As expansion results were obtained, new values of test parameters were selected to better isolate values that produced expansion results typical of the aggregates.

3.1. Non-benchmark aggregates

Average mortar bar expansions under the different test parameters for all aggregates except NR1 and HR3, the benchmark aggregates, are provided in Table 3, along with coefficients of variation (CVs), and are further illustrated in Figs. 1–3. The autoclave expansion results for the first six aggregates were compared to the ASTM C1260 expansions for these aggregates at 14 days. The average expansions and CVs were single-operator except where they were based on expansion data from two laboratories for the initial 5-h autoclave test. The expansion data obtained in both laboratories involved 8 mortar bars except for one set in which one mortar bar was broken during demolding. All other sets of tests were performed in only one laboratory and used 4 mortar bars. Tests using only 2 or 3 mortar bars because specimens occasionally broke during demolding are indicated in Table 3. In the figures, the horizontal dashed lines signify the expansion limits provided by Fournier et al. [20]. Because these limits were developed for autoclave testing using specific parameters (3.5% alkali loading at 130 °C for 5 h), they are used in this paper as merely a guide when different test parameters were applied. Expansions <0.150% suggest the aggregates are non-reactive (NR). Expansions equal to or greater than 0.150% indicate reactive (R) aggregates. Expansions greater than or equal to 0.250% denote highly reactive (HR) aggregates. The vertical dashed lines in the figures indicate the expansion limits recommended by ASTM C1778 based on 14-day expansions in ASTM