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A suggested screening test for ASR in cement-bound composites containing glass aggregate based on autoclaving

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Abstract

The alkali–silica reaction (ASR) in concrete and its effects were studied in great detail within the last half century. Although the phenomenon itself is not fully understood, its potentially severe consequences require extensive testing of mix recipes before service. A reliable yet rapid test to determine the reactivity of a specific mix is a necessity.

At Columbia University, research has been conducted to use post-consumer waste glass as concrete aggregate. One of the objectives was to develop mixes of relatively low reactivity containing glass, pozzolanic materials and various admixtures. With this large number of variables, the detection of unfavorable components prior to excessive long-term testing is essential. The work led to the development of a reliable and extremely rapid ASR test based on autoclave treatment, suitable for both, traditional concrete mixtures and multi-component systems. Test results can be obtained within only three days. Using relative comparison, the proposed autoclave test provides a valuable screening device to reduce the number of possible compositions during mix design.

Keywords: ASR; Autoclave test; Pozzolanic material; Multi-component systems; Glass

1. Introduction

For the last decades concrete producers have made wide use of waste materials, pozzolanic admixtures, organic and inorganic additives in order to ease disposal problems and to improve concrete properties. In the early 1970s, the use of recycled post-consumer waste glass as concrete aggregate was also considered yet failed to deliver promising results [1].

The alkali–silica reaction (ASR) in concrete and its potential for damage have been the subject of extensive research in the past few decades. Most of these studies were concerned primarily with identifying the reactivity of various aggregates and with developing methods to prevent crack-induced damage in concrete with reactive aggregate and high alkalinity. Recently, the basic factors that influence ASR in glass concrete were investigated in some depth [2–5]. For example, results of mortar bar tests according to ASTM C1260 [3] showed that expected expansions were influenced by several aggregate parameters, such as glass particle size, color and chemical composition.

It also was found that partial cement replacement by thermo-activated alumino-silicate clays could effectively counter ASR-induced expansions and damage. Mineral admixtures and superplasticizers can improve the workability of glass–cement–clay composites, thereby permitting reductions of the water–cement ratio. Such admixtures also affect the microstructure and mechanical properties of the composites [6,7].

Because of the complexity of the ASR phenomenon and its numerous influence factors, the development of a suitable test method to predict potential ASR-induced damage is a daunting task [8,9]. Details of ASR mechanisms are neither fully understood yet nor predictable. Some of the primary reasons for this are the exceedingly long time periods during which potentially reactive components can be activated and ASR take place. Modern scientific tools are capable of amassing a wealth of information. However, the most pressing problem is the need for a reliable accelerated test method capable of predicting expected ASR-induced damage in concrete [8].

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The test methods commonly used today, such as ASTM C1260 [10] or ASTM C227 and C1293 [11,12] focus on the expansion of mortar bars, for which the specimens are immersed in a bath of sodium-hydroxide solution of 80 ± 2 °C or above water at 38 °C, respectively. Whereas the ASTM C1260 test is relatively rapid, with a minimum duration of 16 days, the ASTM C227 and C1293 tests, lasting at least six months, clearly are not. Moreover, neither test is meant to evaluate combinations of cementitious materials and/or various admixtures with different aggregates. And neither test protocol can be considered to reflect service conditions of concrete structures in the field [8,13].

The proposed autoclave test, on the other hand, is suitable as a screening device prior to applying proven ASR tests in the search for optimized mix designs because of its extremely short duration. It may serve as a valuable tool in the development of new concrete products although in practical situations, the favorable mix is likely to be compared with other mixes, using also any one of the other ASR test methods with longer duration.

2. Testing of alkali–silica reactivity

It was the purpose of the study reported herein to develop a rapid test method for evaluating different kinds of concrete compositions involving systems of cementitious materials, pozzolans, and aggregates. The "ideal" test should satisfy the following criteria:

1. simulate service life conditions;
2. apply to traditional cement/aggregate as well as systems using uncommon components such as glass or various admixtures;
3. rely on measurements of mortar/concrete bar expansions or shrinkage;
4. yield results within no more than a few days;
5. require inexpensive and readily available equipment;
6. yield easily reproducible results.

Some compromises may have to be accepted in the development of a reliable test. In recent publications [14–16], the use of autoclave test methods has been proposed because such experiments have been conducted successfully in the past to study ASR in mortar systems. There is a fundamental difference between such tests and the existing standard procedures.

The ASTM C1260 test, e.g., is based on the assumption that a very high pH value of the pore solution initiates the reaction with potentially reactive aggregate. The intention was to create the most severe alkaline conditions as could be expected in the pore solution of mortar bars after hydrolysis, which is the interaction of alkalis and water. Therefore, test specimens are immersed in a hot and highly alkaline sodium-hydroxide solution (1 N = 1 M) to create a sufficiently aggressive environment for ASR to occur. Originally, the test was not designed to consider influences of other components of the mortar mix such as admixtures but solely to determine the reactivity of a given aggregate type [10].

The ASTM C227 and ASTM C1293 tests have a relatively long duration of at least six months. They optimize the conditions for expansive reactions involving alkali components of the cement and hydroxyl ions. In the ASTM C1293 test sodium-hydroxide solution may be added to obtain a Na_2O equivalent of 1.25% [12]. The high value of 1.25% "has been chosen to accelerate the process of expansion rather than to reproduce field conditions" [12, p. 655]. Free lime and free magnesia undergo hydration and carbonation while sulfate and sulfide anions cause a chemical attack upon the cement matrix. This process requires warm and humid conditions such as created during storage above water at 38 °C. The resulting expansion is determined and provides a measure for the reactivity of the tested mortar system.

The ASTM C151 test [17] was designed to rapidly determine the effects of thermally treated free lime (CaO) and free magnesia (MgO) in Portland cement with an autoclave. It utilizes high temperature (216 °C) and high pressure (2 MPa) for at least 3 h to activate lime and magnesia, which are readily available in sintered cement. However, high pressure and elevated temperature may change structure and properties of some compounds. The test environment is too harsh for multi-component mortars because the thermo-chemical characteristics of the individual materials differ greatly.

In the autoclave test proposed herein, steam with a temperature of 123 ± 2 °C and a steam pressure of 0.136–0.15 MPa maintains sufficient moisture to accelerate any potential ASR. The steam-curing regime differs greatly from the one specified in ASTM C151. All present mix components participate in the ensuing reaction process. However, no external components such as sodium-hydroxide disturb the chemical equilibrium, which could also be found in actual concrete during years of service. The steam curing is achieved solely by water, which itself does not contribute to any chemical alteration of a given composition. The pressure and temperature were selected for the suggested screening ASR test based on prior experience with autoclave methods. Tests had shown that temperatures around 123 °C prevent the condensation of water on the specimens' surfaces. The steam pressure of about 0.15 MPa is in the range of common home pressure cookers, which were used by some researchers to detect ASR [8].

3. Test preparations

3.1. Materials

The following materials were used in the experimental test program:

Cement: Portland cement Type I, with alkali content 0.88% Na_2O equivalent;
Pozzolans: kaolin and thermally treated kaolin (metakaolin);
Aggregate: non-reactive manufactured sand with grading as shown in Table 1; soda-lime glass from crushed clear bottles with grading of Table 1; unwashed soda-lime glass from crushed clear bottles, possibly containing leftover traces of sugar, juice soft drinks etc., with grading of Table 1; the chemical breakdown of the (washed) glass used is given in Table 2.
Admixtures: hydrophobic and hydrophilic siloxanes.

3.2. Experimental test program

The primary purpose of this investigation was the establishment of an autoclave test for alkali–silica reactivity as a screening device during the development of commercial glass concrete products. It should provide realistic results not only for reference samples with known reactivity but also for new compositions. Various mixes can be tested simultaneously without interaction between samples.

A second goal was to evaluate the new autoclave test in comparison with the ASTM C1260 test. The procedure of the latter was considered questionable when used to evaluate multi-component systems containing glass aggregate. Because sodium-hydroxide is an additional active component, the deleterious reaction on and with the glass surfaces is changed and reliable predictions cannot be made. Thirdly, the research sought to find an ASR test to evaluate the reactivity of mixes containing non-traditional concrete constituents that become more and more important: active pozzolans, active admixtures (either organic or inorganic), and passive organic compounds.

In both tests, length changes of test specimens were measured. The beneficial effect of pozzolanic material and the detrimental effect of reactive glass aggregate were determined, as well as their combined effects. In addition, the influence of the glass aggregate size and the significance of cleanliness of the post-consumer glass were assessed. Table 3 summarizes the various specimens that were tested.

Activated pozzolans and siloxanes were used to assess the effect of chemical attack by additional hydroxyl and sodium ions. Kaolin after thermal treatment contains

Table 1
Regular aggregate grading (% retained)

	US Standard Sieve #							
	4	8	16	30	50	100	200	Pan
Glass	11.4	27.5	25.8	17.5	9.4	6.2	1.7	0.5
Sand	0	10	25	25	25	15	0	0

Table 2
Main components of the glasses used (average % by weight)

Glass type	Oxide					
	SiO_2	Al_2O_3	K_2O, Na_2O	CaO, MgO	SO_3	Fe_2O_3
Clear soda-lime glass	73.4	1.8	13.8	10.7	0.22	0.05

Table 3
Experimental test program

Case no.	Binder	Aggregate	Washed	Admixtures (% of binder)
1	100% cement	Clear glass	Yes	–
2	80% cement, 20% metakaolin	Clear glass	Yes	–
3	80% cement, 20% kaolin	Clear glass	Yes	–
4	100% cement	Clear glass	Yes	Hydrophilic siloxane (0.025%)
5	100% cement	Clear glass	Yes	Hydrophobic siloxane (0.025%)
6	80% cement, 20% metakaolin	Clear glass	No	–
7	100% cement	Clear glass	No	–
8	100% cement	80% sand, 20% clear glass #16	Yes	–

reactive aluminum components (41.3% Al_2O_3), mostly in the form of alumino-silicates, which are capable of binding alkalis extremely fast during hydration [19] (the silicate content is not relevant in this matter). Hence, the risk of alkali–silica reaction is reduced. Siloxanes reorganize the aggregate surface and partially change surface charges. After contact with sodium-hydroxide solution an accelerated reaction occurs [20]. The speedy reactions with metakaolin and siloxanes prevent further chemical attacks on aggregate surface and transition zones.

The first group of specimens (cases 1, 2, 3) was tested to study the influence of pozzolans, i.e. to compare specimens with Portland cement only and those in which 20% of the cement was replaced by either kaolin or metakaolin. It was the purpose of the second group of samples (cases 4, 5) to study the effect of hydrophobic and hydrophilic siloxane admixtures. These can act as water-reducers, but are durable compared to other types of admixtures, which may disintegrate during testing. The third group of samples (cases 6, 7) was tested to evaluate the detrimental effects of unwashed reactive glass aggregate and the known beneficial effect of metakaolin. Test results reported in Refs. [2,5,18] identified a comparably reactive composition containing coarse clear glass as aggregate replacement (size #16). The mix proportions followed those of Ref. [2] (case 8).

3.3. Experimental test procedure

Specimens were prepared in accordance with ASTM C1260, i.e. the mortar bars had dimensions of 25.4 by 25.4 by 254 mm^3. For each test case four to six identical specimens were tested. Two to four of these were subjected to the standard or modified ASTM C1260 test, and the other two or three were tested in the autoclave, an AMSCO laboratory unit with a 1 by 0.48 by 0.48 m^3 chamber that allowed simultaneous testing of more than 20 specimens. The time of treatment varied from 3 to 72 h. The cooling process and method of length change measurement followed the procedure of ASTM C151. Surface cracking of the specimens was observed visually. A step-by-step procedure for the autoclave test is given in Section 3.5.

In order to highlight the influence of sodium-hydroxide solution as an active system component, two ASTM C1260 type tests were conducted in parallel. The first and standard procedure prescribes unmolding after 24 h, water curing for one day while increasing the temperature to 80 °C, and then immersion into a 1 M sodium-hydroxide solution with a temperature of 80 °C. In the second and slightly modified procedure the specimens are placed in the solution directly after unmolding, skipping the 24 h water storage. The pore solution changes extremely rapidly after contact with sodium-hydroxide, which is actively involved in the system's reaction. By exposing the cement composition immediately to the highly alkaline environment, products of the cement hydration could be different from those in the case if the composition had a 24 h period of water storage. This could render the test results less consistent.

3.4. Duration and accuracy of autoclave test

A preliminary test involving selected cases of Table 3 and mortar compositions with regular sand aggregate was conducted to determine the effect of test duration and any effect that the taking of intermittent measurements would have on the end results. Samples were subjected to autoclave treatment varying from 3 to 24 h between measurements. The maximum test duration was 72 h with five interruptions for taking measurements. Fig. 1 shows results of some of these preliminary tests with varying duration. The mix proportions were similar to those in Table 3.

The observations and results permit the conclusion that a 24 h test duration is sufficient to reveal the tendency of a test specimen to expand or shrink. Intermittent readings have barely an effect on the end results. A second 24 h period is only necessary if the measurement devices are not sensitive enough or readings are taken with insufficient care to capture the length changes accurately. Obviously, equipment and operators influence the accuracy of measurements but also the measuring conditions (temperature, pressure, humidity) effect the results.

The maximum length change after autoclave treatment was about 0.03%, the minimum −0.02%, leading to a range of 0.05%. Because of the small numbers it was crucial to evaluate the accuracy of the measurements. Therefore, readings were taken for three selected cases within 2 h. The specimens were of such age as to ensure no relative length change due to internal reactions. For repetitive measurements (8–22 repetitions), the maximum standard deviation (stdev) was found to be 0.0017.

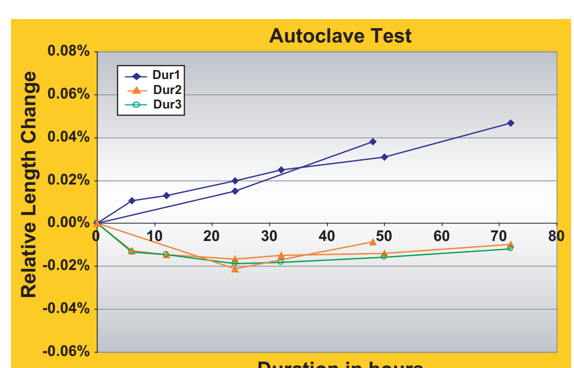

Fig. 1. Expansion as function of test duration (autoclave testing).

When three different operators took the six repetitive readings on the same specimens, stdev = 0.0010 was obtained. In both cases, the coefficient of variation was below 1%. Hence, the suggested procedure is very accurate and leads to reliable and reproducible test results, even for these comparably small numbers.

The assumption that environment and time do not affect the length measurements is true for the one-time only reading under stable conditions during the autoclave test but does not apply to the ASTM C1260 test. There the conditions change from measurement to measurement, and also the temperature of the specimen strongly depends on the speed of the operator. In the ASTM C1260 procedure [10], a period of 10 s is permitted to read a given value—enough time to lead to inconsistent readings caused by thermal contraction of the specimen. The accuracy is, at best, comparable to that of the proposed autoclave test, although the length changes are much larger.

In case of the ASTM C227 and C1293 tests, the measuring conditions vary also with the time period needed for reading, although the impact of thermal expansion/shrinkage should be much smaller than in the ASTM C1260 test. For all three ASTM tests, different lab personnel will inevitably obtain different results because drying-out and diffusion out of and into the specimens over time will lead to high variances. This might explain the wide scatter of results in a multi-laboratory test, with differences as large as 40% [13]. These observations agree with recent findings of other researchers, who showed that significant length changes take place in the first 12–30 h of treatment [21]. However, an absolute indicator of reactivity, if such exists, cannot be determined with the autoclave test.

3.5. Test procedure for the suggested ASR screening test based on autoclaving

(1) Prepare mortar bars with dimensions 25.4 by 25.4 by 254 mm^3 ($W * H * L$), following ASTM C1260.
(2) Store samples in climate chamber (immediately after casting).
(3) Unmold specimens 24 h after casting.
(4) Take length measurement, following ASTM C151.
(5) Place samples in autoclave and treat specimens in autoclave for 24 h at a temperature of 123 ± 2 °C and a steam pressure of 0.136–0.15 MPa (*OPTIONAL: duration of autoclave treatment may be increased to 48 h if greater length changes are desired*).
(6) Remove mortar bars from the autoclave 48 h after casting.
(7) Immerse samples into heated water bath and afterwards gradually cool them down to room temperature, following ASTM C151.
(8) Measure length of the specimens and calculate length change, following ASTM C151.
(9) Compare measured length change to reference results and evaluate whether further ASR testing of the mix design is required.

4. Test results

All test results are summarized in Table 4. In Section 4.1, the effect of modifying the ASTM C1260 test procedure is investigated. In Section 4.2, a comparison between the suggested autoclave test and the ASTM C1260 test is made. Section 4.3 analyzes the sample reactivity with respect to the different components.

4.1. Standard vs. modified ASTM C1260 test

In the modified ASTM C1260 test, specimens were placed in the 1 M sodium-hydroxide solution at 80 °C directly after unmolding. Expansions changed significantly compared with the standard ASTM C1260 procedure, confirming the fact that the sodium-hydroxide quickly changes the chemical activity of the pore solution and initiates reactions between outside alkalis and the various concrete components (see Table 4).

The early contact with the alkaline solution in the modified test activates the pozzolanic potential of kaolin and thus, the expansions of case 3 are reduced. Samples

Table 4
Test results

Case no.	Description[a]	Autoclave (24 h) length change, %	Modified ASTM C1260 length change, %	Standard ASTM C1260 length change, %
1	–	0.015	2.10	1.01
2	20% metakaolin	−0.020	0.04	0.03
3	20% kaolin	0.007	0.75	0.86
4	Hydrophilic	0.016	2.33	1.95
5	Hydrophobic	0.013	2.43	1.68
6	20% metakaolin, unwashed glass	−0.018	0.04	0.03
7	Unwashed glass	0.023	1.22	
8	Sand	0.029	1.10	0.62 [18]

[a] See Table 3 for details.

containing the extremely reactive metakaolin exhibit nearly no absolute changes of the very small expansions in the modified test, because the effect of the pozzolan is too strong (cases 2, 6). The activity of the siloxane admixture and its ability to alter the surface properties determine by how much the expansion increases. With the more sensitive hydrophilic siloxane (case 4) the change is less than with the hydrophobic siloxane (case 5), while the mix without admixtures (case 1) results in the most drastic increase of expansion when samples are exposed to the sodium-hydroxide solution after only 24 h. It is obvious that the ASTM C1260 test is very sensitive to changes in the test protocol.

4.2. Comparison between autoclave and ASTM C1260 test

The goal of any ASR test is to identify trends towards abnormal expansions of concrete caused by chemical reactions. One of the key problems is the search for suitable test conditions, especially if glass is used as aggregate. The reaction kinetics and alkali–silica reactivity depend on test conditions and therefore vary considerably. ASR attacks the aggregate surface areas and reactive aggregate undergoes phase transformations, which change the physical and chemical properties. Adding sodium-hydroxide solution into the system changes the chemical reactions involving glass particles and create conditions that are quite different from real life situations. In the autoclave the surrounding conditions are also altered but no active chemicals are added.

The ASTM C1260 test caused considerably larger expansions than the autoclave test (Table 4). Therefore, specimens tested according to ASTM C1260 exhibited surface cracking, while those tested in the autoclave did not. However, the surface color of the specimens tested according to ASTM C1260 was significantly darker, indicating a considerable change in the chemical composition (Fig. 2).

The ASTM C1260 test and the autoclave test led to relatively consistent results for different types of aggregate. However, when part of the cement was replaced by metakaolin, the results of the two tests did not agree. Whereas samples made with a combination of cement and metakaolin as binder and crushed glass as aggregate exhibited shrinkage in the autoclave test, the same material expanded by a small amount in the ASTM C1260 test (cases 2, 6). This effect is possibly caused by the comparably short duration of the autoclave test (compare Fig. 1).

Unwashed glass may contain a great variety of impurities, from caramelized sugar and mold to partially fermented beverage leftovers. It is expected that those pollutants have a detrimental effect on mortar samples. When comparing case 1 with case 7, expansions of bars with unwashed glass were larger after autoclaving but smaller after the ASTM C1260 test. A possible explanation is that the sodium-hydroxide solution might have a "washing" effect.

This effect is strikingly visible on the surface of specimens. Those made with unwashed glass exhibited surface stains and cracks after autoclaving, resulting from fermentation processes. During the ASTM C1260 test those effluents are washed out and no reaction products can be found on the specimen surfaces (Fig. 3). Even for the specimen that benefited from the metakaolin, surface cracks and stains were observed after autoclaving. Without the benefit of the metakaolin, the contaminants in the unwashed glass increased the autoclave expansions as one could expect (cases 1, 7). However, the reduction of the ASTM C1260 expansion is surprising but may be explained by the beneficial effect of extremely fine particles on the glass surface in combination with the rinsing effect during the repeated bathing in alkaline solution during the ASTM C1260 test.

The results of mortar bars containing siloxanes or coarse glass as partial sand substitute (cases 4, 5, 8) exhibit noteworthy differences. For samples with siloxane (cases 4, 5), the ASTM C1260 expansions were larger than for the reference case 1, while the autoclave test detected only moderate changes in expansions. Case 8 exhibited the largest expansion of all specimens during autoclave testing, while in the modified ASTM C1260 test the maximum length change (case 5) was more than twice as high as that of case 8.

(a)
(b)

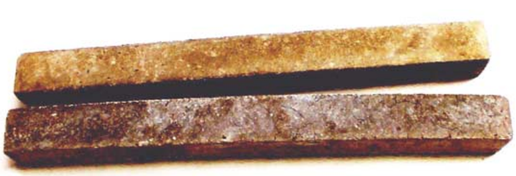

Fig. 2. Mortar specimens with hydrophilic siloxane admixture. (a) After 48 h of autoclave testing. (b) After ASTM C1260 testing (note in Figs. 2 and 3: different color, visibly larger expansion, bar distortions, and surface cracking after ASTM C1260 testing).

(a)
(b)

Fig. 3. Mortar specimens with unwashed glass aggregate. (a) After 48 h of autoclave testing (note the fermentation products on the surface). (b) After ASTM C1260 testing (note the different color and surface cracking).

4.3. Determination of aggregate reactivity

The autoclave test results reflect the relatively poor performance of kaolin during ASR testing of cement–alumino-silicate–glass–aggregate systems in contrast to the highly effective metakaolin (cases 2, 3). Still, kaolin reduces the expansion observed for mortar with only Portland cement as binder (case 1). The autoclave expansions of mixes with hydrophobic and hydrophilic siloxanes were not significantly different from the reference case (cases 1, 4, 5).

The change of chemical and physical properties in the interfacial transition zone caused by ASR can be observed particularly clearly if waste glass is used as aggregate. Aside from particle size and glass chemistry, these processes also depend on the glass "quality", i.e. whether the contaminants found in post-consumer glass are washed or not before it is used as an aggregate in concrete. Admixtures and impurities change the behavior of the aggregate–binder system. The autoclave test shows higher expansions if unwashed glass is used (cases 1, 7). Only specimens containing 20% metakaolin experienced negative length changes, i.e. shrinkage, regardless whether the glass aggregate was washed or not (cases 2, 6). Metakaolin has the ability to overcome the negative influence of organic impurities.

The autoclave test results confirmed the high reactivity caused by coarse glass particles as partial sand replacement in regular mortar bars, identifying this case as truly the worst-case scenario (case 8). They agree with prior findings [2,5] whereas the ASTM C1260 test failed to identify this suggested worst-case scenario.

5. Discussion of results

It is well known that autoclaving is a useful tool in experimental studies of ASR in concrete. For commercial manufacturers of (glass) concrete products an ultra-fast yet reliable screening device for ASR testing would be invaluable. The proposed autoclave test takes about 14 days less than the ASTM C1260 test and does not introduce an additional active component to the mix. Because the autoclave test uses only water steam, the role of components present in the concrete other than alkali becomes important. It seems that the ASTM C1260 test ignores the contributions of such components, e.g. impurities of unwashed glass because of the dominance of the sodium-hydroxide solution.

A duration of 24 h autoclaving is sufficient to exhibit distinct trends. Certain compositions showed abnormal expansions, which could be increased nearly proportionally after additional testing. The observed tendencies do not seem to require durations longer than 24 h, which is comparably rapid. However, for untrained personnel or insufficiently sensitive measuring devices the autoclave test duration might be extended to 48 h. The threshold values for reactive mortars are suggested to be 0.10% in the ASTM C1260 test [10] and 0.04% after one year in the ASTM C1293 test [12]. The comparably small expansions in the autoclave test do not lend themselves for recommendation of a specific limit to identify reactive compositions. Moreover, the autoclave test results do not necessarily permit the conclusion that a specific component is reactive or not but it can be used to determine which of a given number of mixes is the least or the most reactive composition.

The autoclave test results showed relatively low statistical scatter. The coefficient of variation of the relative length changes between the three specimens of each individual sample was on average 0.067, with a maximum value below 0.08. It was reported that for multi-laboratory tests (ASTM 1260, ASTM 1293) differences in the results varied from about 15% to as high as 40% [13]. During the autoclave test, measurements are taken under constant conditions after the specimens are carefully cooled. In contrast to the ASTM C1260 test procedure, the influences of temperature, humidity, pressure, and time are minimized. The new test proposed herein appears to offer a comparably greater reproducibility and reliability.

The ASTM C1260 test has proven its effectiveness in determining the reactivity of specific aggregates, yet its suitability for multi-component systems is questionable if irregular concrete components, such as siloxanes or glass, are used. While samples were literally "washed" during the ASTM C 1260 test, the suggested autoclave test did little harm to the samples and allowed for their components to react "naturally" (see Fig. 3). Unwashed glass is expected to cause higher expansions because additional potentially reactive components are introduced. After being exposed to the sodium-hydroxide solution samples with unwashed glass exhibit less expansions than the ones with washed glass (cases 1 and 7). We assume that pollutants washed out from the surface of the glass aggregate neutralize the pore solution and thus weaken the alkalinity and reactivity. In contrast to the ASTM C1260 test, the results of the autoclave test are consistent and confirm that unwashed glass is, as expected, more reactive. The new test seems to be superior and very useful for comparing the influences (reactivity) of various components. Thus, it can serve as a useful screening aid and reduce the number of long-term tests to be conducted in a multi-variable test setup.

The results of this study confirm the advantage of the autoclave tests reported by Nishibayashi [14], Tamura [15] and Fournier [16]. This method allows testing of concrete that contains not only regular components, such as water, cement, sand and gravel, but also additives, pozzolans, or irregular aggregates. The modification of existing autoclave tests led to a new rapid ASR test, which is capable of generating non-destructive test conditions to detect potential deterioration due to ASR. Mix components are not altered or even decomposed during testing and samples can be used for additional tests, e.g. microscope observations or petrography examination. It is recommended to use the results of autoclave testing for relative comparisons by calibrating them with reference samples containing regular aggregate of known reactivity.

6. Conclusions

1. In the past, ASR studies were successfully conducted using autoclave techniques. It is suggested to utilize these methods as a screening aid during the development of concrete products, especially when containing irregular components such as glass aggregate.
2. The proposed autoclave test method exhibits sufficient sensitivity and accuracy to expose the reactivity of a certain mix or composition. Although absolute values are not determined, relative expansions due to ASR can be detected in comparison to reference samples of known reactivity.
3. The suggested rapid ASR test can serve as a screening device to analyze multi-component systems with several variables. Its overall duration is comparably fast with less than three days including sample preparation, curing, autoclaving, and measuring. However, additional long-term ASR tests are needed to evaluate the behavior of promising compositions.
4. In the ASTM C1260 test, a new active component, sodium-hydroxide, is introduced, which interacts with specific admixtures or certain concrete constituents, thereby changing the behavior of the mortar bars. A modified procedure demonstrated this flaw of the ASTM C1260 test. Correlation between autoclave test and ASTM C1260 test results was observed only for mixes, which were not sensitive to contact with sodium-hydroxide solution. For compositions with glass aggregate the ASTM C1260 test seems not very reliable.
5. The introduction of unwashed glass as aggregate exhibits the risk of predicting a false reactivity of multi-component systems with the ASTM C1260 test. The pollutants on the glass surface are expected to increase the reactivity of tested systems. Changes of the pore solution after reaction with sodium-hydroxide solution cause a decrease in expansion when unwashed glass is used, which was not the case in the autoclave test.
6. The autoclave test corresponds well to the governing chemical principles during ASR occurrence. The length changes are caused solely by the original composition of specific mortars rather than by additional, external components as in other accelerated tests. Although the expansions are comparably small the measurements are sufficiently accurate to deliver conclusive results.
7. Further experiments are required to establish possible correlations between concrete behavior in field studies and under conditions of laboratory autoclave testing.

References

[1] Philips JC, Calm DC, Keller GW. Refuse glass aggregate in Portland cement concrete. In: Proceeding, 3rd Mineral Waste Utilization Symposium, U.S. Bureau of Mines and ITT Research Institute, Chicago, 1972.

[2] Meyer C, Baxter S. Use of recycled glass for concrete masonry blocks. New York State Energy Research and Development Authority, Albany, NY, Report No. 97-15, Nov. 1997.

[3] Meyer C. Development of glass concrete products. Empire State Development, Office of Recycling Market Development, Albany, NY, Final Report, 1999.

[4] Jin W, Meyer C, Baxter S. Glascrete—concrete with glass aggregate. ACI Mater J 2000;97(2):208–13.

[5] Jin W. Alkali–silica reaction in concrete with glass aggregate. A chemo-physomechanical approach. Ph.D. Thesis, Columbia University, New York, Apr. 1998.

[6] Fardis G et al. Effect of clay minerals on the hydration of cement. Adv Cem Based Mater 1994;1(6):243–7.

[7] Saad Morsy M, Abo El-Enein SA, Hanna BB. Microstructure and hydration characteristics of artificial pozzolana-cement pastes containing burn kaolinite clay. Cem Concr Res 1997;27(9):1307–12.

[8] Grattan-Bellew PE. A critical review of ultra-accelerated test for alkali–silica reactivity. Cem Concr Compos 1997;19(5–6):403–14.

[9] Corneille A, Bollotte B. Results of a round robin test program for the validation of the test method in the French recommendations for the prevention of AAR damage to concrete. In: American Concrete Institute, SP 145-38, Detroit, 1994. p. 725–40.

[10] ASTM C1260-94. Standard test method for potential alkali reactivity of aggregate (mortar-bar method). In: Annual book of ASTM standards, vol. 04.02. Philadelphia: ASTM; 1998. p. 126–30.

[11] ASTM C227-97A. Standard test method for potential alkali reactivity of cement–aggregate combination (mortar-bar method). In: Annual book of ASTM standards, vol. 04.02. Philadelphia: ASTM; 1998. p. 650–3.

[12] ASTM C1293-95. Standard test method for concrete aggregates by determination of length change of concrete due to alkali–silica reaction. In: Annual book of ASTM standards, vol. 04.02. Philadelphia: ASTM; 1998. p. 654–9.

[13] Fournier B, Bérubé M-A, Frenette J. Laboratory investigations for evaluating potential alkali-reactivity of aggregates and selecting preventive measures against alkali–aggregate reaction (AAR)—What do they really mean? In: Fournier B, Bérubé M-A, Durand B, editors. Alkali–aggregate reaction in concrete. Proceedings of the 11th International Conference on Alkali–Aggregate Reaction, Quebec City, QC, Canada, 2000. p. 287–96.

[14] Nishibayashi S, Yamura K, Matsushita H. A rapid method of determining the alkali–aggregate reaction in concrete by autoclave. In: Grattan-Bellew PE, editor. Concrete alkali–aggregate reactions. Park Ridge, NJ: Noyes Publications; 1987. p. 299–303.

[15] Tamura H. A test method on rapid identification of alkali reactivity aggregate (GBRC method). In: Grattan-Bellew PE, editor. Concrete alkali–aggregate reactions. Park Ridge, NJ: Noyes Publications; 1987. p. 304–8.

[16] Fournier B, Bérubé M-A, Bergeron G. A rapid autoclave mortar bar method to determine the potential alkali–silica reactivity of St. Lawrence lowlands carbonate aggregates. Cem Concr Aggregate 1991;13:58–71.

[17] ASTM C151-93A. Standard test method for autoclave expansion of Portland cement. In: Annual book of ASTM standards, vol. 04.01. Philadelphia: ASTM; 1998.

[18] Hudes PP. Ground waste glass as an alkali–silica reactivity inhibitor. In: Fournier B, Bérubé M-A, Durand B, editors. Alkali–aggregate reaction in concrete. Proceedings of the 11th International Conference on Alkali–Aggregate Reaction, Quebec City, QC, Canada, 2000. p. 663–72.
[19] Frias M et al. The effect that the pozzolanic reaction of metakaolin has on the heat evolution in metakaolin–cement mortars. Cem Concr Res 2000;30(2):209–16.
[20] Monteiro PJM et al. Influence of mineral admixtures on the alkali–silica reaction. Cem Concr Res 1997;27(12):1897–909.
[21] Xu Z et al. A new accelerated method for determining the potential alkali-carbonate reactivity. In: Fournier B, Bérubé M-A, Durand B, editors. Alkali–aggregate reaction in concrete. Proceedings of the 11th International Conference on Alkali–Aggregate Reaction, Quebec City, QC, Canada, 2000. p. 129–38.