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A Critical Review of Ultra-accelerated Tests for Alkali–silica Reactivity

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Abstract

A large number of ultra-accelerated test procedures, for determining the potential alkali reactivity of aggregates, have been developed, particularly in the past 15 years. An ultra-accelerated test method is defined as one which yields results within a few days or, at most, a few weeks. A number of ultra-accelerated test methods have been adopted as 'standard tests', but few have been adequately evaluated. The rapid globalization of the construction industry will require the harmonization of National Standard Test Methods. The major requirement of ultra-accelerated test methods is that they should correctly predict the potential reactivity of aggregates in greater than 95% of the cases. Due to the complexity and variability in the composition and grain size of aggregates, it is improbable that a single test method will be developed which would be appropriate for evaluating all types of aggregates. Another major requirement for ultra-accelerated test methods is that the inter-laboratory coefficient of variation should be low, preferably less than 12%. At present, only the NBRI accelerated mortar bar method has been subject to adequate inter-laboratory evaluation. However, a more limited inter-laboratory investigation showed that the autoclave mortar bar test also shows considerable potential, as a satisfactory ultra-accelerated test method. Further refinement of the NBRI and autoclave methods is required to improve their performance with a wide variety of aggregates. © 1997 Elsevier Science Ltd. All rights reserved.

Keywords: Alkali–aggregate reactivity (AAR), accelerated tests, evaluation.

INTRODUCTION

The frequent requirement, now, for large structures to have a life expectancy of up to 100 years, combined with the rapidity with which projects are conceived and constructed, has increased the need for rapid, reliable, test methods for ensuring that aggregates for use in concrete will not cause premature deterioration due to alkali–aggregate reaction. Many test methods have been developed, but little attention has been given to the inter-laboratory reproducibility of these tests. This is most important if costly litigation, due to incorrect diagnosis, is to be avoided.

Stanton (1943)[1] wrote: 'It is very desirable that some accelerated test procedure be developed to determine the potential expansion characteristics of any cement–aggregate combination'. The same statement could well be made today, 50 years later. It is possible that no single test will ever prove entirely satisfactory with all types of aggregates. It became evident, on looking back at the evolution of accelerated test methods, that many of the current standard tests were developed shortly after the discovery/identification of alkali–silica reactivity (ASR), in the late 1930s. By 1946 most of the test methods, in use today, were developed. These included the mortar bar test which later became standardized as ASTM C 227,[2] the gel pat test, the autoclave test, the chemical method, the dissolution weight loss method and the use of petrographic techniques to identify potentially reactive aggregates. By 1943 Stanton had also developed a further accelerating procedure for the mortar bar test by immersing the bars in 0.5 M NaOH @ 43°C. This test procedure was a forerunner of the NBRI test developed by Oberholster and Davies,[3] and subsequently adopted in many countries.

During the half century following the identification of ASR there have been significant advances in our understanding of the mechanism of ASR and the identification and understanding of the alkali–carbonate reaction (ACR), but with only a few exceptions, e.g. the chemical shrinkage test,[4] only some refinement in the basic test methods, e.g. the kinetic test, a modification of ASTM C 289.[5] In addition, a number of what may be termed petrographic/instrumental techniques have been proposed to aid in the identification of potentially reactive quartz (silica) minerals. These include the use of infrared (IR) spectroscopy, X-ray diffraction (XRD), thermal analysis (DSC), positron annihilation and transmission electron microscopy (TEM) for detecting the presence of defects in the quartz lattice which increase the free energy and, hence, solubility of quartz in an alkaline medium.[6–8] These instrumental methods are very useful for research purposes as they permit the evaluation of the potential reactivity of very small samples, consisting of only a few grains, separated out from rocks. They are also useful for the study of the reaction mechanisms. These techniques, therefore, make it possible to determine precisely which are the reactive components in multi-mineralic aggregates such as gneisses.[9] However, the development of ultra-rapid expansion tests (UAETs) which permit measurement of the expansion of mortar bars in a few days, or at most, a few weeks, have largely eliminated the need for instrumental methods for determining the potential expansivity of aggregates, or even determining which are the reactive components in a known expansive aggregate. The use of ultra accelerated expansion tests do not solve all the problems associated with the evaluation and acceptance of an aggregate for use in concrete. Due to the severity of these test methods, aggregates which have a satisfactory history of performance in concrete in the field may fail. For this reason, and for most projects, UAETs may be used to accept an aggregate, but not to reject it (CSA A23.1, 1994).[10] Exceptions might be concrete for use in dams which would be exposed to a continuously moist environment, or in nuclear generating stations, large bridges, or other structures with a 100 year life requirement. Even in the latter situations, a potentially reactive aggregate might be accepted if used in a mix in which a portion of the cement would be replaced by a supplementary cementing material in appropriate proportions.[11,12] Thus, there is a need for standard moderately accelerated tests, such as the ASTM C 227 mortar bar test, or the Canadian or French concrete prism tests to determine if an aggregate failing a UAET test would be acceptable for a particular job mix. The standard mortar bar and concrete prism test methods are well-documented and will not be further discussed. It is not the intention in this review to provide a catalogue of standard accelerated test methods as these are now well-understood and documented.[13] Instead, selected ultra-accelerated expansion tests which, in the authors opinion, are potential, viable and standard test methods, are discussed in this review.

CRITERIA FOR SELECTION OF AN ACCELERATED TEST PROCEDURE

Philosophy of testing

There are two main possibilities for selecting test procedures for aggregates. In the first, testing is undertaken to determine if the aggregate is, *a priori*, potentially expansive or not. If this alternative is adopted, testing should be done under severe conditions that will hopefully detect any potentially expansive aggregate. Of course, it carries the risk that some aggregates, with good field performance, will fail the test. The second alternative is to run a test under only moderate accelerating conditions that come closer to the conditions of the concrete in the field, this second choice eliminates UAETs. In theory the latter approach might be preferred, however, there is a real danger that such tests might not detect some of the more slowly expanding aggregates which might lead to the development of cracking in the structure, possibly, 10–20 years after construction. An example of this problem is the well-documented Sudbury gravel from Ontario, Canada (CSA A23.1-94, Appendix B).[10] When tested according to the, now superseded, CSA 1990 Standard, Appendix B, A23.1-14A,[14] concrete prism test with a specified cement content of 310 kg/m^3 and an alkali content of 3.875 kg/m^3, the aggregate appeared to be acceptable, yet concrete bridges made with this aggregate cracked, due to ASR, after a number of years. This problem was corrected in the 1994 CSA standard by increasing the specified cement content from 310 kg/m^3 (alkali content 3.875 kg/m^3) to 420 kg/m^3, for a total alkali content of 5.25 kg Na_2O eqv/m^3 concrete. As a result of this experience, it is my preference to test aggregates under sufficiently severe conditions to determine if the aggregate is potentially reactive with the alkalis in the cement. However, caution is needed to ensure that the test method is not so severe that non-reactive minerals, such as well crystalline quartz, do not start to react, as may happen if the temperature in an autoclave test is above about 150°C.[15] If an aggregate proves to be potentially expansive, then additional testing should be done to determine if it might be acceptable in concrete with a given mix design that would be exposed to specified environmental conditions. Sound engineering judgment and experience are necessary in selecting the test protocol to be used and in the interpretation of the results. There is probably no single test method which would be satisfactory with all types of aggregates. Blindly following standards may lead to acceptance of an aggregate which may be potentially expansive in a particular structure, or rejection of an aggregate which would perform satisfactorily in the field, leading to unnecessary expense of locating, and possibly trucking, an alternative aggregate. Probably, the best advice that can be offered, to someone needing an evaluation of the potential reactivity of an aggregate, is to send it to an experienced investigator rather than to the nearest testing laboratory.

Criteria required for an ultra accelerated test method

The following criteria are required for a satisfactory UAET method:

The test must be rapid with results obtained within a few days, or at most a few weeks.

The test should be relatively easy to run and the apparatus required should not be excessively expensive.

Ideally there should be good correlation between the test results and field experience with the same aggregates, and the test results should correctly differentiate between reactive and non-reactive aggregates in more than 90% of the cases.

The reaction products should be similar to those found in field concretes and in concretes tested in the concrete prism test run at 38°C.

The expansion limit should be greater than 0.05% to minimize errors due to the effect of temperature fluctuations or problems or errors in the measuring apparatus.

The test results must be reproducible. The coefficient of variation (CV) for repeat tests by one operator should preferably be less than 10%.

The CV between different laboratories should ideally be less than 12%.

There are now many tests that will give results in a few days or a few weeks, as a large number of modifications of the autoclave test, or of the NBRI test, have been developed during the past 15 years. The autoclave test, carried out in a modified cement autoclave, requires relatively expensive equipment. However, as cement autoclaves are already available in laboratories which undertake research and testing in the hydration of Portland cement, this may not be a major obstacle to the use of this test. Alternatively, an autoclave test may be carried out in a simple pressure cooker which may be purchased at low cost.[16] The Konometer apparatus,[4] for measuring chemical shrinkage of sands, containing flint, costs about $10,000 (U.S.). It is doubtful if this cost could be justified except, possibly, on some very large projects where there is a need for more or less continuous monitoring of the potential reactivity of the sand containing flint, chert or opal.

Obtaining good reproducibility between repeat tests within one laboratory generally does not pose a problem, so long as good experimental procedure is followed, and the measuring equipment and/or pressure are properly calibrated, and the storage temperature is adequately controlled. Obtaining a CV of less than 20% in inter-laboratory studies is difficult due to the large number of variables which need to be controlled. Tests done in Canada have shown that in an inter-laboratory study, once outliers have been removed, the highest and lowest expansions are often recorded by experienced laboratories. In a recent study of the concrete prism test it was found that when a standard mix was sent to each laboratory which then only needed to add water, mix and mold, the CV dropped to 14%, from the 24% obtained when each laboratory graded the coarse aggregate and used their own sand and cement.[17] This result indicates that the main problem is not in the measurement of the length change of the sample, but rather it is due to variations in the mixture proportions used by the different laboratories. Even though all the mixtures contained 5.25 kg of alkali per m^3 concrete, it is thought that variations in the alkali content of the cements used contributed significantly to the large CV. In the NBRI test, great care is needed to prevent shrinkage of the bars, due to cooling of the samples between the time when they are removed from the solution and when they are measured. Specification of a standard cement would probably improve the reproducibility of the NBRI or other tests. However, the provision of a standard cement, over a long period of time, poses storage and distribution problems.

The following example illustrates the effect of CVs of 12 and 25% on the results reported in an inter-laboratory study of the NBRI test: Suppose 10 laboratories evaluated four aggregates, a non-reactive, one marginally reactive, one moderately reactive and one very reactive, Fig. 1. Assume that the expansion limit is 0.15% at 14 days.

First examining the effect of a CV of 25% on the reported results. The non-reactive aggregate with a mean expansion of $0.04 \pm 0.10\%$ would be correctly predicted as non-reactive by all laboratories. The marginally expansive aggregate with a mean expansion of $0.13 \pm 0.033\%$ would be classed as expansive (0.163%) by some laboratories. The moderately expansive aggregate with a mean expansion of $0.18 \pm 0.022\%$ would be classified as innocuous (0.135%) by some laboratories. All laboratories would correctly predict the reactivity of the very reactive aggregate with an expansion of $0.26 \pm 0.065\%$.

Next examining the results when the inter-laboratory CV is $\pm 12\%$. All laboratories would correctly predict the reactivity of the non-reactive aggregate and the marginally reactive aggregate with an expansion of $0.13 \pm 0.016\%$. Some laboratories would report the moderately reactive aggregate with a mean expansion of 0.18%, as being only marginally reactive (0.158%). All laboratories would correctly predict the reactivity of the very reactive aggregate.

Summarizing the effect of variations in the CV on the diagnosis by different laboratories: when the CV is 25%, two of the aggregates would be incorrectly diagnosed by some labora-

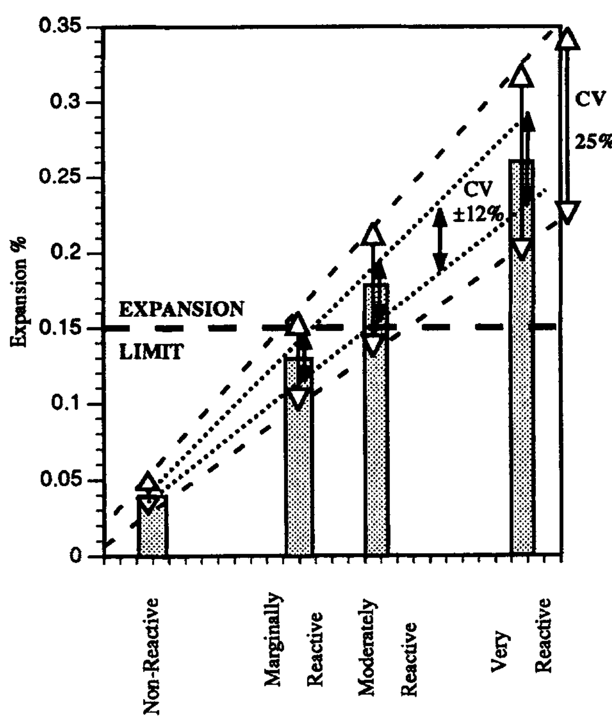

Fig. 1. Effect of inter-laboratory coefficients of variation (CVs) of 25 and 12% on the diagnosis of the potential reactivity of a non-reactive, a marginally reactive, a moderately reactive and a very reactive aggregate by a number of laboratories.

tories; when the CV is 12% only one aggregate would be incorrectly diagnosed by some laboratories. It is evident from the above discussion that the effect of a large CV on the results reported by a number of laboratories is most acute for aggregates with an expansion close to the expansion limit. Unfortunately, such marginally' reactive aggregates are very common and a erroneous diagnosis of such aggregates can lead to costly litigation.

It is very important to refine a test method to reduce the inter-laboratory CV to an acceptable level before approving, or standardizing it. A review of the literature revealed that only the NBRI test has been subjected to a rigorous inter-laboratory evaluation involving 37 laboratories, in six countries. A more limited inter-laboratory investigation is reported from France in which a maximum of 10 laboratories participated in the evaluation of two autoclave test methods, the chemical test ASTM C 289[18] and an ultra accelerated concrete prism test.[19] In principle it should be possible to obtain a satisfactory CV with autoclave tests, provided the participating laboratories all use the same calibrated autoclaves, because in this test, the large amount of alkali which is added to the mortar should override variations in the alkali content of the cement. This appears to be borne out by the results of the French evaluation of the GBRC autoclave test with a mean CVs of 15%, which was the lowest CV amongst the six tests which were evaluated.[19]

Only test methods, for which the proposed expansion limit is relatively large, possibly greater than 0.05%, should be selected, because tests with lower expansion limits, e.g. the 0.02% proposed for the 60°C concrete prism test in France,[20] the effect of fluctuations in the temperature and instrumental errors can significantly affect the results. Furthermore, significantly higher CVs have been observed with aggregates showing low expansions.

PROPOSED ULTRA ACCELERATED TEST METHODS

Chemical test (ASTM C 289-94)

The chemical test cannot be recommended as an acceptance test for aggregates. However, because of its widespread use in the past, some discussion of the problems with this test are warranted. Aside from the need for a skilled analyst to determine the amount of dissolved silica and the reduction in alkalinity or the SiO_2/Na_2O ratio, as suggested by Dent Glasser and Kataoka[21] and, subsequently, adopted by Sorentino *et al.*,[5] there are fundamental problems with the chemical process. Dent Glasser and Kataoka,[22] showed that the end point in the titration of the solution with HCl obtained using phenolphthalein, as specified in the standard, was indefinite. They suggested using methyl orange. Dent Glasser and Kataoka[21] showed that the amount of dissolved silica increased when the sample was stirred during dissolution. There is no provision for stirring the sample in the standard. The determination of the amount of silica in solution is further complicated by the observation of Dent Glasser and Kataoka[21] that the amount of dissolved silica passes through a maximum at a particular SiO_2/Na_2O ratio and then decreases. The particle size of the aggregate used in the dissolution test can also effect the results. This problem was discussed by Sorentino *et al.*[5] who opted for a range of particle sizes from 0 to 300 μm. It is evident that very careful analytical technique is required to obtain the optimum results with this test method and even then it only gives an indication of the potential reactivity of the aggregate and not a measure of its expansivity in concrete which is required for any satisfactory acceptance test.

NBRI test

The NBRI test is the most widely used method. Use of this test and various modifications of it have been reported from Australia, Argentina, Canada, France, Italy, Hong Kong, Japan, Norway, South Africa and U.S.A.[23,24,3,25–28] All the modifications of the NBRI test follow the experimental procedure specified by Oberholster and Davies. They differ in the length of the test and in the expansion limits, Table 1. The Canadian Standards version of the NBRI test has also been the subject of several thorough investigations.[29–31] The coefficient of variation, reported by Jiang and Rogers,[30] amongst 38 laboratories on the 14th day of the test was 13.94%, an acceptable value. The CV reported for repeat determinations by one operator in one laboratory is 6% which is excellent.[31] At present this test is the one most widely used and most thoroughly investigated and must,

Table 1. Comparison of storage times in NaOH solution limits in several modifications of the NBRI test

Author	*Country*	*Storage time, days in NaOH @ 80°C*	*Expansion limits %*
Oberholster & Davies[3]	South Africa	12	> 0.11[1]
Berra *et al.*[26]	Italy	12	> 0.10
Shayan *et al.*[28]	Australia	10–22	> 0.10 > 0.10[2]
Hooton & Rogers[29]	Canada CSA A23.2-25A	14	< 0.15[3]
–	USA ASTM C 1260-94	14	< 0.10[4]
Wigum & Lindgard[25]	Norway	14	> 0.15[3]
Batic *et al.*[27]	Argentina	28	no limit proposed

therefore, be the first choice. However, like any test method, care is needed in interpreting the results.

Marginally reactive aggregates might be accepted as innocuous when evaluated by the CSA criteria (< 0.15%), but would be classed as deleterious by the ASTM expansion limit (> 0.10%). The adoption of particular limits depends on the philosophy of testing and on local politics'. However, with the globalization of the construction industry, there is a need for harmonization of National Standards.

Cement fineness

Berra *et al.*[26] showed that expansion, in their modification of the NBRI test, was affected by the fineness of the cement. Two batches of mortar bars were made, one with the cement as received, the second with the cement ground to 37% higher Blaine. Mortar bars containing the cement with the higher Blaine expanded proportionately more. Additional research is needed to evaluate the effects of cement type and fineness on expansion in the NBRI test.

In some test procedures e.g. CSA A23.2-25A, the use of a Type 10 (ASTM Type 1) cement with a specified alkali content is required and as a further precaution against the use of cements, or other components, which may cause anomalous expansions of mortar bars in this test, the use of reference mortar bars, made with an aggregate with known expansion characteristics, is also specified. In ASTM C 1260, neither the type, fineness, nor the alkali content of the cement is specified, and there is no requirement to test reference mortar bars, with known expansion characteristics, along with the aggregate being evaluated

The pessimum

Aggregates which exhibit the pessimum effect in the mortar bar test ASTM C 227 also show it in the NBRI test. If petrographic examination indicates that the aggregate under investigation may exhibit the pessimum effect, it should be mixed and tested in various proportions with a non-reactive sand.[32] Limited results reported for the modified chemical test[5] indicate that this is a rapid method for determining which aggregates exhibit the pessimum. In this test the ASTM C 289 procedure is followed except that the solution is sampled and analyzed at intervals of up to 70 h (Fig. 2). Aggregates exhibiting the pessimum effect plot in the upper left part of the diagram.

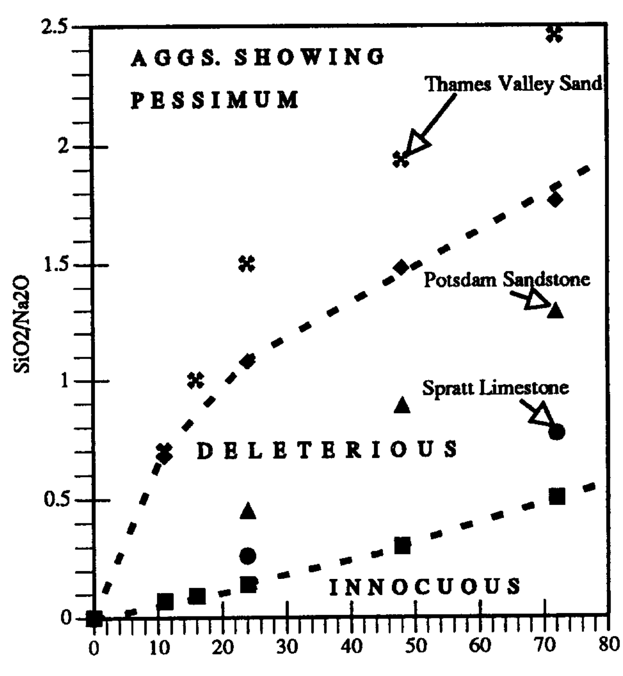

Fig. 2. Diagram of the kinetic chemical test, after Sorentino *et al.*,[5] showing aggregates exhibiting the pessimum plotting in the upper left-hand part of the diagram.

NBRI test results not in agreement with field performance

Hooton and Rogers[29] reported that some deleteriously reactive granites and gneisses of Grenville age from Maryland and Virginia, U.S.A., containing microcrystalline quartz, expanded less than 0.15% at 14 days. They state that this test may not be suitable for these types of aggregates. However, unpublished results of tests of gneiss from the Sanvel quarry in Massachusetts, U.S.A., in which microcrystalline quartz is the reactive component, by the author showed that the aggregate expanded by more than 0.265% in the NBRI test.[33]

Shayan[32] found that a non-vesicular basalt, classed as innocuous by the chemical test ASTM C 289 and the Standard mortar bar test, expanded, in the NBRI test, by 0.355% in 10 days. This aggregate was classified as reactive in the Australian concrete prism test with an expansion of 0.032%. (In this test the alkali content of the concrete is 6.9 kg Na_2O eqv/m^3 and the proposed limit is 0.030%.) However, this aggregate would be classified as innocuous in the CSA concrete prism test in which the alkali content of the concrete is 5.25 kg/m^3 and the limit is 0.04%.

The NBRI test may be too severe for some limestones and lithic sands from Quebec and New Brunswick, Canada which test as expansive, but have a satisfactory history of performance in the field.[34] Similar results have been reported from parts of the U.S.A., Fournier and Bérubé suggested that some of the expansion observed with non-reactive limestones may be due to the expansion of swelling clay minerals at 80°C in this test.[31] Hooton and Rogers[29] have suggested continuing the NBRI test for up to 56 days to help differentiate between marginally reactive, reactive and non-reactive aggregates (Fig. 3). A number of authors have compared the reaction products produced in the NBRI test with those in concrete affected by ASR in the field and in concrete prisms. They concluded that the reaction products were essentially the same.[35,31]

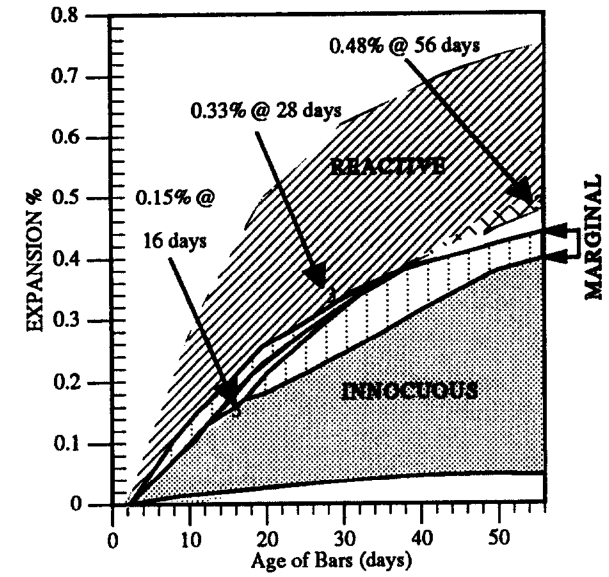

Fig. 3. Proposed expansion limits at 28 and 56 days for the NBRI mortar bar test, after Hooton and Rogers.[29]

AUTOCLAVE TESTS

Chinese autoclave test

Miniature mortar bars 1 × 1 × 5 cm are used in this test. The bars are made following ASTM C 227. They are cured in a fog room for 24 h before being remolded. The bars were then steam cured for 4 h. Finally, the bars were immersed in 1 M NaOH and autoclaved at 140°C for 6 h. This test method has also been standardized in France as AFNOR P18-588 where an inter-laboratory investigation was made.[19] The CV was found to vary between 15 and 30% amongst the four samples tested, with a mean CV of 21.5% The main disadvantage of this test is the difficulty of obtaining a representative sample due very small sample size, < 10 g. This test method is best suited for determining which is the reactive component in a gravel or sand by evaluating individual fragments. It has also been used for evaluating the potential reactivity of individual minerals separated from rocks.[9] It is probable that aggregates exhibiting a pessimum proportion in the mortar bars test would also show it in this autoclave test.

Japanese and canadian autoclave tests

Several modifications to the autoclave test have been proposed in Japan and in Canada.[36,16,37] In the autoclave methods, as in the NBRI method, care is needed in proportioning the mortar when aggregates suspected of exhibiting the pessimum are being evaluated, to insure that the aggregate is tested at, or close to, its pessimum proportion.

The gbrc autoclave method

The GBRC autoclave method[36] has been evaluated by Corneille and Bollotte[19] and Shayan *et al.*[38] In this method mortar bars 40 × 40 × 160 mm are used. The alkali content of the cement is adjusted to 2.5% by the addition of NaOH, although the table of mix designs, shown by Corneille and Bollotte,[19] indicate that the alkali content of the cement was adjusted to 4%. Both the above pairs of authors used a temperature of 127°C, although a temperature of 110°C is given by Tamura.[36] In his original paper, Tamura used visual observation of cracking and changes in the ultrasonic pulse velocity to determine the reactivity of an aggregate, but both of the other pairs of authors used length change. According to Corneille and Bollotte[19] the GBRC autoclave method gave the lowest CV (12–16%) of the five methods evaluated. Shayan *et al.*,[32] comparing expansions in the Australian modification of the NBRI (AUS. NBRI) and the autoclave tests, reported generally good correlation between the results of the two methods, except for some slowly expanding aggregates including some basalt, dacite, granite, metadolerite and quartzite all of which expanded less than the proposed 0.1% limit in the autoclave test, but more than 0.1% at 22 days in the AUS. NBRI test. They suggest that the autoclave test for slowly reactive aggregates might be improved by alterations to the mix design; further investigation is needed.

Laval autoclave method

Fournier *et al.*[39] developed an autoclave method broadly similar to the GBRC method, but using bars made according to the ASTM C 227 specifications (25 × 25 × 285 mm). They followed the ASTM C 151-93a[40] autoclave test procedure. The alkali content of the cement was raised to 3.5% Na_2O by the addition of NaOH to the mix. The operating temperature was 130°C. They determined the degree of reactivity my measuring length change of the bars. Fournier

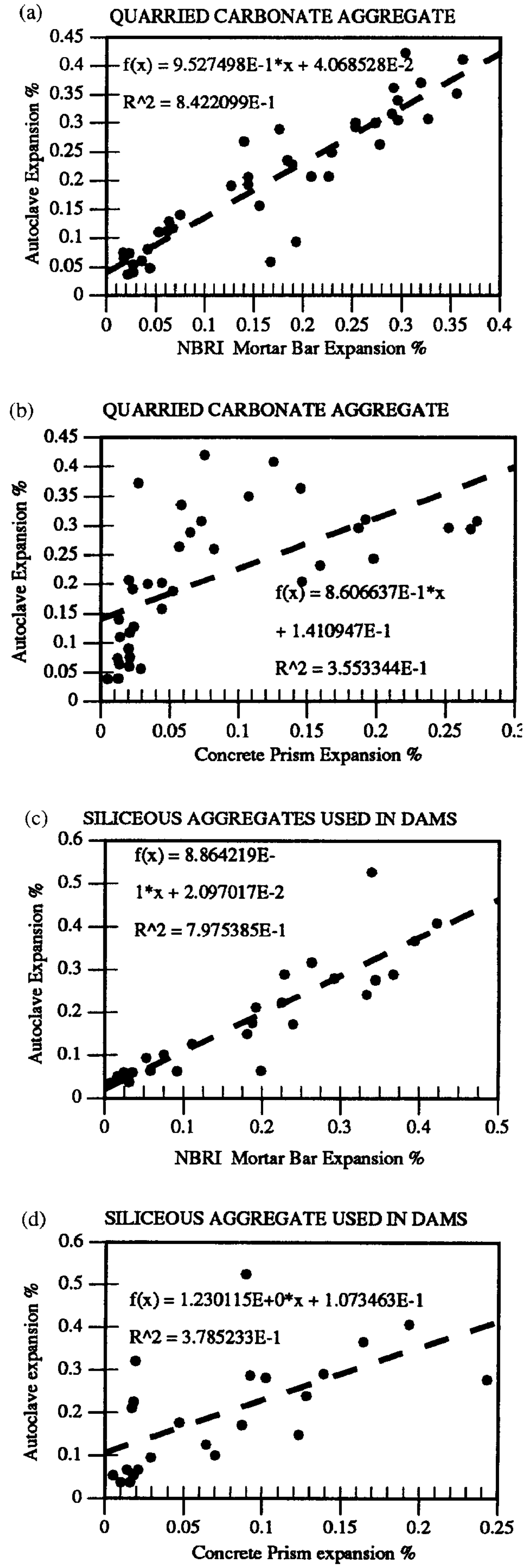

Fig. 4. (a) Comparison of expansions of quarried carbonate aggregate in the autoclave and NBRI tests; (b) comparison of expansions of quarried carbonate aggregate in the autoclave and concrete prism tests; (c) comparison of expansions of siliceous aggregates used in dams in the autoclave and NBRI tests; (d) comparison of expansions of siliceous aggregates used in dams in the autoclave and concrete prism tests.

et al., reported that the products of ASR were broadly similar to those found in field concretes except that the rosette-like gel observed in old structures was not found. They found good correlation between the results of the autoclave test, the NBRI test and ASTM C 227 tests, but only moderate correlation with the results of the concrete prism method Fig. 4. The lack of correlation with the results of the concrete prism test may be due to the use of concrete prisms containing only 350 kg cement/m^3 concrete and not the 420 kg specified in the 1994 CSA test protocol A23.2-14A. It was found that some more slowly expanding siliceous aggregates, evaluated as innocuous in the concrete prism test using 310 kg alkali/m^3 concrete, as specified in the 1990 CSA standard A23.2-14A, caused cracking in bridges.

In a more recent publication, Bérubé *et al.*[37] observed that the autoclave method was better than the NBRI method for differentiating between reactive and non-reactive aggregates, particularly sands and quarried siliceous aggregates. However, it remains to be demonstrated if this method is capable of correctly predicting the reactivity of the sands from New Brunswick which have a good field performance, but fail the NBRI test. However, as reported above, correlation between expansions in the autoclave and concrete prism tests, for both quarried carbonate aggregates and siliceous aggregates used in dams, were not good, possibly due to the use of only 350 kg of cement per m^3 (4.375 kg of alkali per m^3) concrete in the latter test. Rogers[41] showed that with some siliceous aggregates, significant expansion in the concrete prism test was only observed when the alkali content was > 5 kg/m^3 (Fig. 5). Both types of aggregates with expansions of 0.3% in the autoclave test had a range of expansions in the concrete prism test varying from 0.02% to over 0.24% (Fig. 4). Clearly more research is required to confirm that the autoclave test can be used to correctly predict the reactivity of a variety of aggregates. No inter-laboratory evaluation of the Laval autoclave test has yet been reported.

Nishibayashi method

In the autoclave method proposed by Nishibayashi *et al.*,[16] the protocol is broadly similar to that of Tamura,[36] except that a pressure cooking pot was used in place of a more expen-

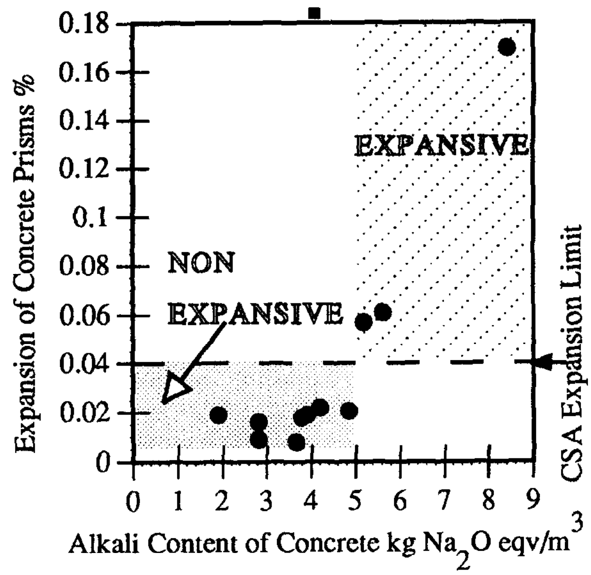

Fig. 5. Correlation between expansion and the alkali content of concrete prisms, after Rogers.[41]

sive industrial autoclave. The optimum pressure in the cooker was 0.15 MPa. The alkali content of the cement used in the prisms was boosted to 1.5% by the addition of NaOH. The autoclave time was 5 h. The mortar bars are made following the JIS specifications (40 × 40 × 160 mm). Equivalent expansions to those obtained in 6 months in the Japanese concrete prism test were obtained in 5 h with the autoclave. The reactivity of the aggregate is assessed by measuring the expansion of the mortar bars. No inter-laboratory evaluations, and only limited test results, have been reported to date. However, because of the low cost and ready availability of the equipment, this method appears to have considerable potential as a standard test method.

Discussion of autoclave methods

Although autoclave methods have been used successfully to differentiate between expansive and non-expansive aggregates there are potential problems that need to be recognized. Recently, Xiaofang[42] has shown that coarse grained calcite and dolomite, with a grain size > 1.25 mm, can cause expansion in the autoclave due to differential thermal expansion between the *a* and *c* crystallographic axes of these minerals. It is evident that it is essential to make a petrographic examination of aggregates, before running them in an autoclave, so that erroneous-diagnosis of the potential reactivity of the aggregate can be avoided. The possibility that expansion of coarse grained calcite and dolomite might also occur in the NBRI test needs to be investigated.

The Chinese autoclave test, although useful for research purposes, should probably not be considered as an acceptance test for concrete aggregates due to the extremely small sample size and, hence, small mass of aggregate (<10 g) evaluated. Inevitably, there must be some concern about the very high alkali contents in the mortar bars used in the GBRC and Laval autoclave mortar bars, 2.5% in the former and 3.5% in the latter, which are needed to obtain expansion even with well-documented reactive aggregates. There is less concern with the Nishibayashi method in which the alkali content of the cement in the mortar is only boosted to 1.5%, which is not that much higher than that used in the NBRI test or in the Canadian concrete prism test in which the alkali content of the cement is boosted to 1.25%. Caution should be exercised in the interpretation of autoclave test results until more experience has been gained with these methods.

CONCLUDING COMMENTS

The short lead time for many construction projects has lead to the need for a rapid test method which can be used to reliably predict the potential expansivity of an aggregate within a few weeks. Currently, the only method which has been thoroughly evaluated, and which satisfies this requirement is the NBRI accelerated mortar bar test.[23,24]

Care is needed in interpreting the results of the NBRI test, because, despite the generally satisfactory results which are reported with this method, it is very severe. A number of cases have been reported in which aggregates with good field performance fail the NBRI test. It has also been reported that some granites, with poor field performance, pass this test. For these reasons this test should always be carried out in conjunction with a thorough petrographic assessment of the aggregate.

Another advantage of the NBRI test is that it is relatively simple to carry out as no elaborate equipment is required. However, this can also create problems as inexperienced laboratories can readily set up to run the test without knowing how their test results compare with those from other laboratories. There is, undoubtedly, a need for some type of qualification program for laboratories and, or, operators, so that clients have some assurance that the test results obtained are satisfactory. This would also provide some protection for the laboratory, if litigation is brought against them, based on what the client perceives to be an incorrect diagnosis of their aggregate.

Autoclave test methods on mortar bars developed by Nishibayashi,[16] Tamura[36] and Fournier *et al.*[39] show considerable promise and may have a lower inter-laboratory coefficient of variation than the NBRI test. Good correlation has been reported between expansions obtained in autoclave and NBRI tests. The only disadvantage of the methods proposed by Tamura and Fournier *et al.*, is the high cost of the modified cement autoclaves. In contrast, the autoclave used by Nishibayashi consists of a relatively cheap pressure cooker. However, up to the present it has not been widely used and more experience with it will be needed before a final conclusion can be reached on it appropriateness as an ultra accelerated test method.

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