A CRITICAL REVIEW OF ACCELERATED ASR TESTS

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Abstract

A completely satisfactory accelerated test method for determining the potential expansivity of aggregates in concrete, due to alkali-silica reactivity, has still not been developed, despite 60 years of research. However, the NBRI accelerated mortar bar method, and several proposed autoclave methods, approach the ideal test, although some further refinement of these procedures is still required. The criteria for a satisfactory accelerated test procedure are that the results must be obtained within a few weeks, the test must be easy to run and the apparatus should not be too expensive. There must be good correlation between the test results and the performance of the aggregate in concrete in the field. The reaction products should be similar to those found in field concretes affected by alkali-silica reactivity, and finally the interlaboratory coefficient of variation must be low, <15%. Petrographic evaluation should be conducted in parallel with accelerated tests, because a number of cases have been reported in which the test results are not in agreement with the field performance of the aggregates in concrete. A number of rapid petrographic/ instrumental techniques which have been developed for determining the potential reactivity of aggregates are referred to, but as they do not yield a measure of the expansivity of the aggregates, they are only appropriate for research purposes, and are not discussed in detail.
Key Words: ASR, Accelerated tests, autoclave, NBRI, selection criteria.

Introduction

"It is very desirable that some accelerated test procedure be developed to determine the potential expansion characteristics of any cement-aggregate combination." This was written by Stanton 1943. Apparently little has changed in 50 years, and an accelerated test to solve alkali-silica reactivity (ASR) problems still alludes us. It became evident, on looking back at the evolution of accelerated test methods, that most of the current standard tests were developed shortly after the discovery/identification of alkali-silica reactivity (ASR), or "the handwriting on the walls" as it was referred to by Blanks, 1941. The first decade of the ASR era, 1936 to 1946 was a very productive one, during which most of the test methods, in use to day, were developed. These included the mortar bar test which later became standardized as ASTM C 227, 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, 1986.

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 (Knudsen 1987), only some refinement in the basic test methods, e.g. the Kinetic Test, a modification of ASTM C 289 (Sorentino et al 1992). 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, (Baronio et al., 1987), (Tang et al., 1989), (Thomson et al., 1994). 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. These techniques therefore make it possible to determine precisely which are the reactive components in multi mineralic aggregates such as gneisses, (Thomson & Grattan-Bellew 1993). However, the development of ultra rapid expansion tests (UAET's) which permit measurement of the expansion of mortar bars in a few days or at most two 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 (UAET's) 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 may fail which have a satisfactory history of performance in concrete in the field. For this reason, for most jobs, UAET's may be used to accept an aggregate for a particular job, but not to reject it, (CSA A23.1 1994). Exceptions might be concrete for use in dams which would be exposed to a continuously moist environment and where a guaranteed life of at least 100 years is required, or in nuclear generating stations. Even in the latter situations, 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, (Rogers 1993), (Thomas et al., 1992). 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 method as this has been, recently, most ably done by Bérubé and Fournier 1994. Due to space limitations, only selected ultra accelerated expansion tests are discussed in this review of accelerated test methods.

Criteria for Selection of an Accelerated Test Procedure

Philosophy of testing

The selection of a test procedure for evaluating aggregate for a particular job depends, to some extent, at least, on the philosophy adopted. There are two main alternatives. 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. 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 UAET's. 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, ten to twenty years after construction. An example of this problem is the well documented Sudbury gravel from Ontario, Canada, (CSA A23.1-94 Appendix B). When tested according to the, now superseded, CSA 1990 Standard Appendix B A23.1-14A, concrete prism test with a specified cement content of 310 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 to 420 kg/m^3, for a total of 5.25 kg Na_2O eqv/m^3 concrete. As a result of this experience, it is my philosophy 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 insure 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, (Tang et al., 1983). 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 is 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 with finding and possibly trucking an alternative aggregate.

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 >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 38C.
- The expansion limit should be>~0.05% to minimize errors due to temperature effects and the measuring error.
- The test results must be reproducible. The coefficient of variation (CV) for repeat tests by one operator should preferable be <10%.
- The CV between different laboratories should ideally be <15%.

There is no problem with finding a test 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. For a catalogue and description of accelerated test methods, see Bérubé & Fournier 1994.

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, (Nishibayashi et al., 1987). The Konometer apparatus, (Knudsen 1987,) for measuring chemical shrinkage of sands, containing flint, costs about $10,000 (US) which makes this test too expensive, unless there is a requirement for repeated long term testing for a major construction project.

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 interlaboratory 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, (Fournier 1994). This result indicates that the main problem is not in the measurement of the length change of the sample. However, 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.

It is very important to refine a test method to reduce the interlaboratory 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 interlaboratory evaluation involving 37 laboratories, in 6 countries. A more limited interlaboratory 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 and an ultra accelerated concrete prism test, (Corneille & Bollotte 1994). 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 born out by the results of the French evaluation of the GBRC autoclave test with a mean CV's of 15%, which was the lowest CV amongst the six tests which were evaluated, (Corneille & Bollotte 1994).

Only test methods, for which the proposed expansion limit is relatively large, possibly >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, (Criaud & Defossé 1995), the effect of fluctuations in the temperature and instrumental errors can significantly affect the results.

Proposed ultra accelerated test methods

NBRI test:

Most experience has been obtained with various modifications of the NBRI test. This test, and various modifications have been used in Australia, Argentina, Canada, France, Italy, Hong Kong, Japan, Norway, South Africa and USA, (ASTM C 1260,) (CSA A23.2-25A), (Oberholster & Davis 1986), (Wigum & Lindgard, 1994), (Berra et al., 1994), (Batic et al., 1994), and (Shayan et al.,1988). 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, (Hooton & Rogers 1993), (Jiang Rogers 1994) & (Fournier Bérubé, 1991). The coefficient of variation, reported by Jiang & Rogers 1994, 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, (Fournier Bérubé, 1991). At present this test is the one most widely used and most thoroughly investigated and must 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.

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

Author	Country	storage time, days in NaOH @ 80°C	Expansion limits %
Oberholster & Davies 1986	South Africa	12	>0.11*1
Berra et al., 1994	Italy	12	>0.10
Shayan et al., 1988	Australia	10 22	>0.10 >0.10*2
Hooton & Rogers, 1993	Canada CSA A23.2-25A	14	<0.15*3
-	USA ASTM C 1260-94	14	<0.10*4
Wigum & Lindgard, 1994	Norway	14	>0.15*3
Batic et al., 1994	Argentina	28	no limit proposed

*1 *Expansion >0.11% indicates potentially deleterious expansion in concrete.*
*2 *Expansion <0.01 @ 22 days indicates innocuous aggregate.*
*3 *Expansion <0.15% indicates innocuous aggregate.*
*4 *Expansion <0.01 indicates innocuous aggregate.*

Cement Fineness

Berra et al., 1994 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, (Shayan, 1992).

NBRI test results not in agreement with field performance

Hooton & Rogers 1992 reported that some deleteriously reactive granites and gneisses of Grenville age from Maryland and Virginia, USA, 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, USA, in which microcrystalline quartz is the reactive component, by the author showed that all the aggregates expanded by more than 0.265% in the NBRI test. See also Kerrick & Hooton 1992.

Shayan, 1992 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 siliceous aggregates in Quebec and New Brunswick, Canada which test as expansive but have a satisfactory history of performance in the field, (Bérubé & Fournier 1992). Fournier & 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, (Fournier Bérubé 1991). Hooton & Rogers 1993 have suggested continuing the NBRI test for up to 56 days to help differentiate between marginally reactive, reactive and non-reactive aggregates, Figure 1.

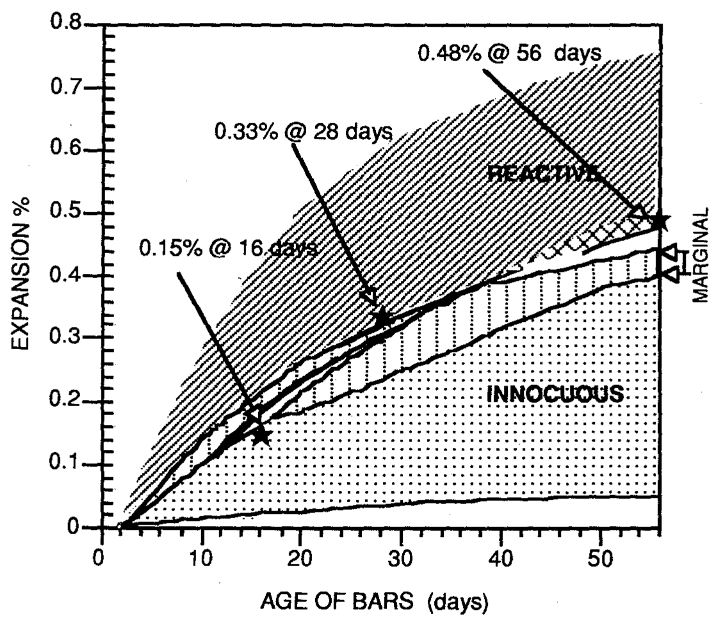

Figure 1. Proposed expansion limits at 28 and 56 days for the NBRI mortar bar test. After Hooton & Rogers 1993.

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, (Shayan Quick 1989), (Bérubé Fournier 1991).

Chinese autoclave test:

This test method has also been standardized in France as AFNOR P18-588 where an interlaboratory investigation was made, (Corneille & Bollotte 1994). 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, (Thomson & Grattan-Bellew 1993).

Japanese Autoclave tests

Several modifications to the autoclave test have been proposed in Japan and in Canada, (Tamura 1987, Bérubé et al., 1992, Nishibayashi et al., 1987).

The GBRC autoclave method

The GBRC autoclave method (Tamura 1987) has been evaluated by Corneille & Bollotte 1994 and Shayan et al., 1992. In this method mortar bars 40 x 40 x 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 & Bollotte ibid, 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 1987. 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 ibid the GBRC autoclave method gave the lowest CV (12 to 16 %) of the 5 methods evaluated. Shayan et al., 1992 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, Bérubé & Bergeron 1991 developed an autoclave method broadly similar to the GBRC method, but using bars made according to the ASTM C 227 specifications (25 x 25 x 285 mm). They followed the ASTM C 151-93a. 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 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 Figure 2. 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.

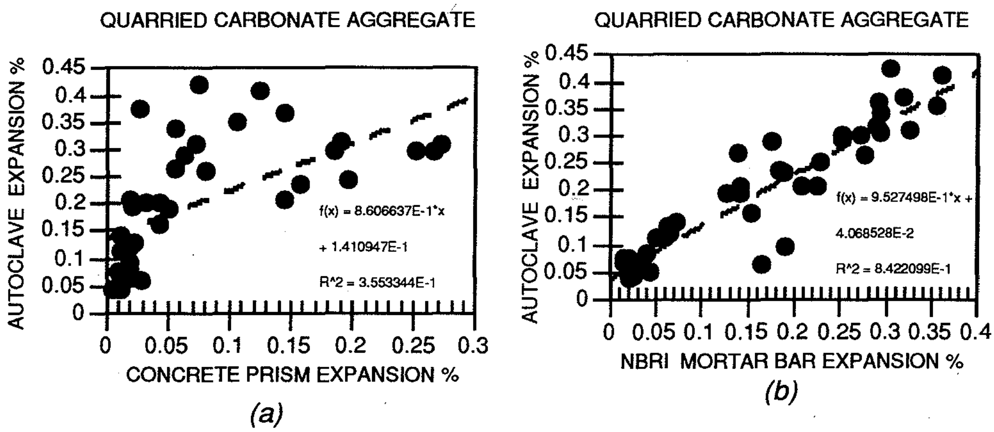

Figure 2, a & b. Comparison of expansions of quarried carbonate aggregates in the autoclave, the NBRI and the CSA concrete prism tests. After Fournier et al 1992

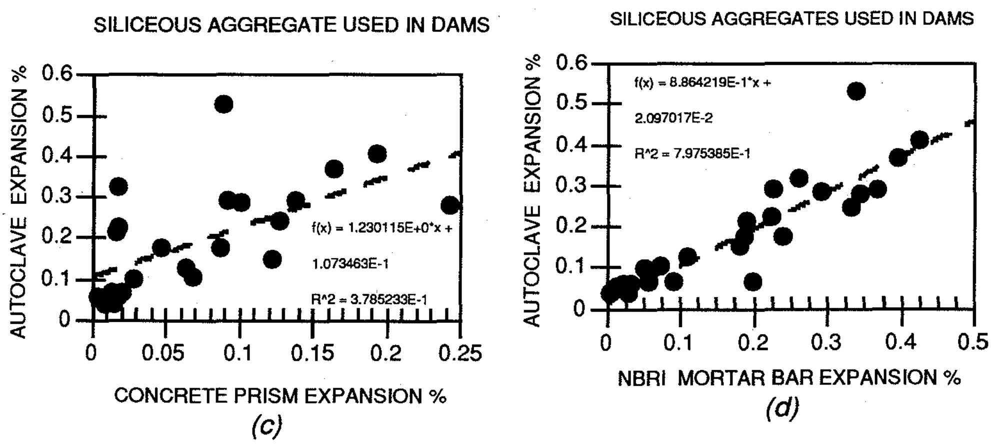

Figure 2, c& d Comparison of expansions of siliceous aggregates in the autoclave, the NBRI and the CSA concrete prism tests. After Fournier et al 1992

In a more recent publication, Bérubé et al., 1992 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, 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 cement/m^3 (4.375 kg alkali/m^3) concrete in the latter test. Rogers 1990 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, Figure 2a. 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%, Figure 3. Clearly more research in required to confirm that the autoclave test can be used to correctly predict the reactivity of a variety of aggregates. No interlaboratory evaluation of the Laval autoclave test has yet been reported.

In the autoclave method, 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, it's pessimum proportion.

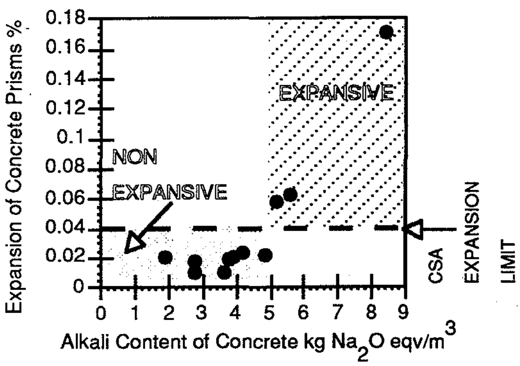

Figure 3. Correlation between expansion and the alkali content of concrete prisms. After Rogers 1990.

Nishibayashi method

In the autoclave method proposed by Nishibayashi et al., 1987, the protocol is broadly similar to that of Tamura 1987, except that a pressure cooking pot was used in place of a more expensive 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 hours. The mortar bars are made following the JIS specifications (40 x 40 x 160 mm). Equivalent expansions to those obtained in 6 months in the Japanese concrete prism test were obtained in 5 hours with the autoclave. The reactivity of the aggregate is assessed by measuring the expansion of the mortar bars. No interlaboratory 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.

CONCLUSIONS

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, (ASTM C 1260-94), (CSA A23.2-25A 1994).

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.

Autoclave test methods on mortar bars developed by Nishibayashi 1987, Tamura 1987 and Fournier et al., 1991 show considerable promise and may have a lower interlaboratory 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.

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