Multi-Laboratory Study of the Accelerated Mortar Bar Test for Alkali-Silica Reaction

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ABSTRACT

The accelerated mortar bar test, developed by NBRI in South Africa, has been adopted in North America for the rapid identification of potentially alkali-silica reactive aggregates. The purpose of the study, involving 46 laboratories, was to obtain data to develop a multi-laboratory precision statement for the test. The aggregate used in this study was Spratt siliceous limestone from a quarry in Ottawa, Ontario. At an age in solution of 14 days, the mean expansion was found to be 0.364% (coefficient of variation 13%) when laboratories used the same cement, and 0.417% (coefficient of variation 15%) when laboratories used their own cement. Autoclave expansion, and the alkali, sulphate and MgO contents of cement were not related to mortar bar expansion. A relationship was found between MgO content and autoclave expansion.

Keywords: mortar bar test, alkali-silica reaction, precision, cement, chemistry.

INTRODUCTION

Alkali-silica reactions between silica from aggregates and alkalies from cement can cause damaging expansion and cracking of concrete. These reactions need three things to occur: concrete that is sufficiently moist, reactive aggregate and sufficient alkalies in the concrete to start and support the reaction.

One of the most important tests to detect alkali-silica reactive aggregates is the mortar bar expansion test. This test was developed in the early 1940's by Stanton [1943] in California and was adopted by ASTM as a tentative test method in the early 1950's (ASTM C227). The test involves making cement mortar bars with a suspect aggregate and storing the bars at 38°C over water for a year or more. Excessive expansion of the bars indicates a potentially deleterious, expansive aggregate cement combination.

The test has often been found to be impractical because of the length of time before results are obtained. Concrete is often placed and in service before results are available showing compliance of the aggregates with the specification. In the late 1980's, it was also found that wide variations in the test could be caused by leaching of the alkalies out of the mortar bars during storage [Rogers and Hooton 1991]. This was traced to the type of container used for storing the mortar bars. Different storage containers, meeting the specification, could make a test furnish results either meeting or failing the specification requirements.

In the early 1980's, Oberholster and Davies [1986], at NBRI, developed a means of accelerating the expansion of the mortar bars by storage in hot NaOH solution. It is worth noting however, that Stanton [1943] was the first to experiment with storage of bars in hot sodium hydroxide solution. In the NBRI test, the bars are de-moulded after one day and placed in water which is then heated to 80°C. After 24 hours, the bars are measured for length while at 80°C and immediately placed in a 1N NaOH solution preheated to 80°C. They are stored at 80°C in the solution and expansion is measured by quickly removing the bars from the solution, measuring length and returning the bars to the solution. Normally, expansion after 14 days storage in solution has been found to be a convenient time period to determine if the aggregate is deleteriously expansive. In some cases, other times (12 days, 28 days) have been adopted for setting specification limits.

Draft test procedures for this test were adopted by CSA in 1988 and by ASTM in 1989. This test was formally adopted by both CSA and ASTM in 1994 (CSA A23.2-

25A, ASTM C1260). The history of the adoption in North America will be found in a paper by Hooton and Rogers [1992].

A requirement of any satisfactory engineering materials test is that the test have a known and low multi-laboratory variation. Two multi-laboratory studies of the variability of the test have been conducted. Davies and Oberholster [1987], in a study within one laboratory with three different technicians, found a coefficient of variation of 10.4% (mean expansion 0.34%). Hooton [1990], in a study using the Spratt aggregate (used in this study) and nine laboratories, found an average multi-laboratory coefficient of variation of 22.5%. When he excluded three inexperienced labs, the variation was reduced to 9.4% (mean expansion 0.42%). These studies have been done with limited numbers of laboratories, and so do not give a true picture of likely multi-laboratory variation. Generally, to get stable precision estimates, the number of laboratories should be at least 30. A large number of laboratories are now doing this test in North America, and the CSA and ASTM sub-committees responsible for the test procedure decided to conduct a multi-laboratory study. Another reason for doing the study was to qualify a stockpile of alkali-silica reactive aggregate that could be used in the future as a reference material for control purposes within individual laboratories.

LABORATORY TESTING

Experimental Design

To ensure the maximum number of laboratories participated, it was decided to keep the study as simple as possible while still collecting data on multi-laboratory variation. Normally, multi-laboratory studies should be conducted on at least three different materials and two replicates of each material should be tested [ASTM C802]. It was decided not to attempt such an ambitious program. The test procedure used by the participants was that published by CSA. This procedure is identical in all technical respects to ASTM C 1260 except that the w/c ratio specified by CSA and used in this study was 0.50 rather than 0.47 specified by ASTM and there are different cement requirements. The design called for each laboratory to receive 6 kg of aggregate, enough to do at least two mortar mixes. In addition, each laboratory received 1 kg of cement from the organizing laboratory. The program called for each laboratory to make two mortar mixes, one with the standard cement and one with the cement normally used by that laboratory. This design would allow the estimation of multi-laboratory variation but would not allow estimates of within laboratory variation. Such a design would also show how cement properties affected test variability. Previous ruggedness testing had shown that cement properties, within broad limits, did not significantly influence expansion in the test. This data had been obtained with small numbers of different cements and there was speculation that more rigorously specifying the cement or the use of a standard cement would significantly reduce between laboratory variation.

Aggregate

The aggregate selected for the study was a siliceous limestone from the Spratt quarry near Ottawa, Ontario. This horizontally bedded limestone contained 3-4% microscopic chalcedony and black chert. The rock consisted of calcite and small amounts of dolomite with an insoluble residue of 10% consisting of silica, illite and pyrite. The principal reactive component is finely disseminated silica not visible with normal optical methods. The aggregate, one of the most expansive alkali-silica aggregates in Canada, has been used in previous studies of alkali-reaction test methods [Rogers and Hooton 1991]. For the purposes of this study, a new 100 tonne stockpile of 5-20 mm stone was established in Toronto. On delivery, Four samples (40kg each) were taken at random from the stockpile and tested in the accelerated mortar bar test. The results showed that the materials gave a range of expansion after 14 days in solution of 0.335-0.344% (average 0.339%). Following this testing, a sample of about 400 kg, representative of the stockpile, was oven dried and sieved. Each sieve fraction was placed in separate sample containers. Sixty samples of 6 kg each were made by combining aggregate of each sieve size according to the gradation of the material in the stockpile. These samples were stored in canvas bags.

Cement

The coordinating laboratory had a stockpile of about 2 tonnes of high alkali cement. Specimens of this cement were prepared by sequentially placing about 200 g of cement taken from one plastic barrel with a small scoop in each of 50 plastic bags with a self sealing closure. Additional 200 g aliquots were added until each bag contained about 1 kg of cement. The bags were sealed and placed inside another self sealing plastic bag.

Testing

Each laboratory received a specimen of cement, a sample of aggregate, a copy of the test procedure and forms for recording expansion data, equipment details and laboratory conditions. Each laboratory was asked to ship a 1 kg sample of the cement used to the coordinating laboratory. The coordinating laboratory tested each sample in the autoclave expansion test (ASTM C151) together with 11 specimens of a laboratory control cement. The Lafarge Canada, Montreal laboratory tested chemical composition using XRF (ASTM C114). The laboratories were asked to record mortar bar expansion at ages of 7, 13, 14, 15, 21 and 28 days in NaOH solution. They were asked to record average (3 bars) expansion to 0.0001%. This is more accurate than required by the test procedure, but was requested in order to avoid rounding errors influencing the estimate of precision.

RESULTS AND DISCUSSION

Statistical analysis

Expansion data was received from 46 laboratories. Data from five laboratories was excluded from the final analysis. Laboratory 10, which gave the highest expansion, was rejected because they noted that they mixed the mortar by hand. This probably resulted in inadequate mixing and poor consistency of the paste. Laboratory 19, which gave relatively low expansion, was rejected because they were not using mortar bars of the correct length (210 mm instead of 285 mm). Laboratory 27 was rejected because they reported very high expansion at 7 days and very little subsequent expansion between 7 and 28 days. The laboratory gave no reason and did not reply to enquiries about the problem. Laboratory 35 was rejected because they had not previously conducted the test and reported problems with their measuring device. Laboratory 36 was rejected because they used a RILEM mortar bar (40 x 40 x 160 mm) and obtained relatively low expansion. These were believed to be sound reasons to reject data. With the exception of Laboratory 27, no data was rejected just because 'it looked wrong' or because it exceeded three standard deviations of the mean.

Following the removal of outlying data, standard deviation and coefficient of variation was calculated at the various times of storage. Figure 1 shows the coefficient of variation at various ages before and after removal of outliers. Figure 2 shows the variation in the form of a scatter diagram. The scatter diagram shows that there were a number of laboratories that tended to get either consistently high results or consistently low results, in addition to those already rejected. These laboratories are ones which show some bias in their testing. There was however no obvious physical reason to reject their data and it has been included in the analysis.

It is possible to prepare a precision statement as follows: For mortars giving average expansions after 14 days in solution of more than 0.3%, the multi-laboratory coefficient of variation (1s% of ASTM C 670) has been found to be 14.9%. Therefore, the results of two properly conducted tests in different laboratories on specimens of a sample of aggregate should not differ by more than 42% (d2s% of ASTM C670) of the mean expansion.

Table 1. Summary of multi-laboratory test information before and after removal of outlying data.

standard cement, all data included						n = 46
age	7 day, %	13 day, %	14 day, %	15 day, %	21 day, %	28 day, %
mean exp.	0.2326	0.3477	0.3653	0.3860	0.4943	0.6533
max exp.	0.5563	0.5652	0.5663	0.5827	0.8233	0.9767
min exp.	0.1455	0.2155	0.2310	0.2435	0.3406	0.4570
Std. Dev.	0.0624	0.0663	0.0658	0.0714	0.0899	0.1096
C. of V.	26.8%	19.1%	18.0%	18.5%	18.2%	16.8%
standard cement, five laboratories excluded						**n = 41**
age	7 day, %	13 day, %	14 day, %	15 day, %	21 day, %	28 day, %
mean exp.	0.2290	0.3459	0.3641	0.3850	0.4919	0.6549
max. exp.	0.3493	0.4887	0.5003	0.5190	0.6837	0.8804
min. exp.	0.1455	0.2673	0.2936	0.3088	0.3693	0.4752
Std. Dev.	0.0369	0.0477	0.0483	0.0526	0.0725	0.0968
C. of V.	16.1%	13.8%	13.3%	13.7%	14.7%	14.8%
individual laboratories cement, all data included						**n = 46**
age	7 day, %	13 day, %	14 day, %	15 day, %	21 day, %	28 day, %
mean exp.	0.2703	0.3990	0.4166	0.4367	0.5368	0.6830
max exp.	0.5646	0.5990	0.6367	0.7027	0.9647	1.2970
min exp.	0.1413	0.2426	0.2546	0.2768	0.3810	0.4736
Std. Dev.	0.0698	0.0763	0.0779	0.0844	0.1070	0.1471
C. of V.	25.8%	19.1%	18.7%	19.3%	19.9%	21.6%
individual laboratories cement, five laboratories excluded						**n = 41**
age	7 day, %	13 day, %	14 day, %	15 day, %	21 day, %	28 day, %
mean exp.	0.2676	0.3981	0.4159	0.4356	0.5318	0.6756
max. exp.	0.3787	0.5360	0.5493	0.5740	0.7783	0.9773
min. exp.	0.1663	0.2912	0.3037	0.3166	0.3810	0.4736
Std. Dev.	0.0495	0.0602	0.0621	0.0660	0.0862	0.1138
C. of V.	18.5%	15.1%	14.9%	15.2%	16.2%	16.9%

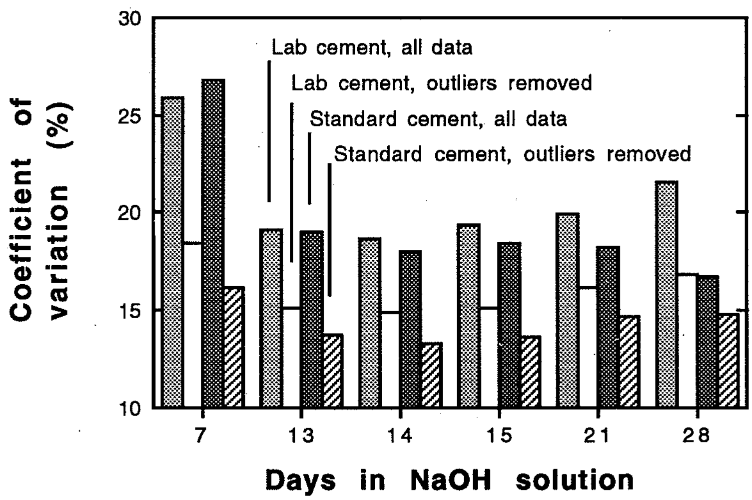

Fig. 1 Multi-laboratory coefficient of variation for accelerated mortar bar test at various ages.

Cement composition

To what extent does the composition of the cement effect the expansion measured by a laboratory? The data shows that with the cements studied, the chemistry of the cement

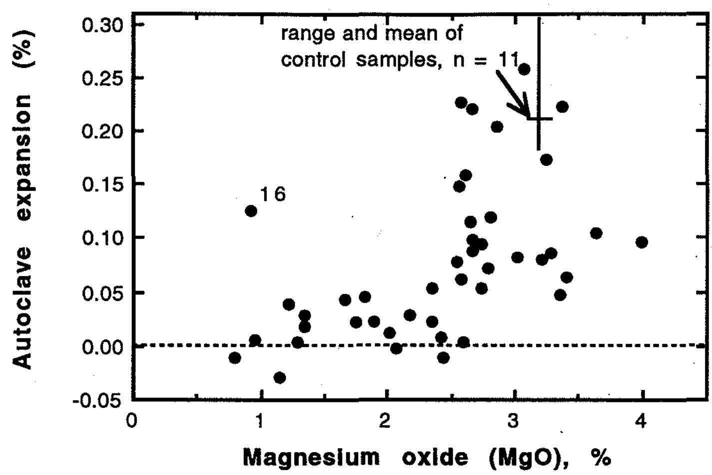

Fig. 4 Cement autoclave expansion, ASTM C151, compared with MgO content of the cement.

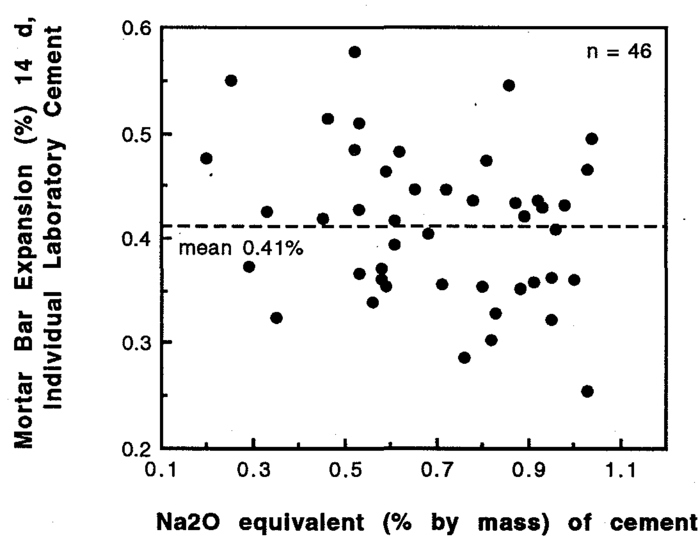

Fig. 5 Mortar bar expansion at 14 days in solution compared with alkali content of the cement.

Figure 4 does show that there is a relationship between MgO content and autoclave expansion in the ASTM C151 test. This is logical. With the exception of cement used by Laboratory 16, there is a threshold of MgO content at about 2.5% above which autoclave expansion may be significant and below which it is negligible. Lea [1971] noted that cement could accommodate about 1.5-2% MgO both in the glass phase of clinker and in solid solution with other cement minerals, and not cause a problem. Above this level of MgO, periclase would form and depending on crystal size, determined by cooling rate, cause unsoundness.

Figure 5 shows that there is no consistent relationship between alkali content of the cement and expansion. This confirms the observations of Davies and Oberholster [1987] and Hooton [1990]. Figure 6 shows that the sulphate in the cement (measured as total SO_3) is not related to expansion of the mortar bars. It can be concluded that the

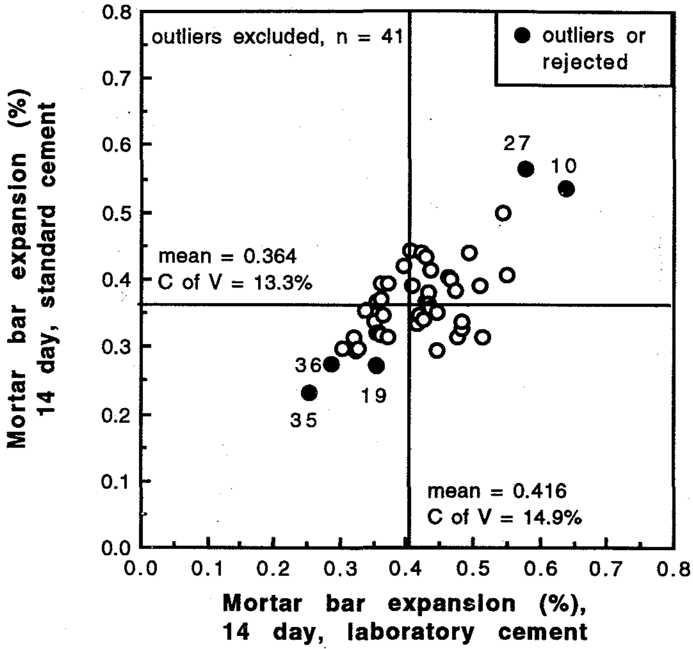

Fig. 2 Scatter diagram showing multi-laboratory variation of accelerated mortar bar test made with individual laboratories cement and standard cement after 14 days in solution.

had little systematic effect on expansion. The standard cement gave average expansion results of 0.364% at 14 days that were about 12% less than the average expansion when different cements were used. The variation was also slightly, but consistently, lower using the same cement compared with different cements as shown in Figure 1.

Chemical analysis of the cements used by the individual laboratories was compared with mortar bar expansion. Figure 3 shows the relationship between MgO content and expansion of mortar bars at 28 days. No obvious correlation is seen. It had been suspected that cements with high periclase content, measured by MgO might contribute to expansion, especially in view of the high temperatures of storage in the test (80°C).

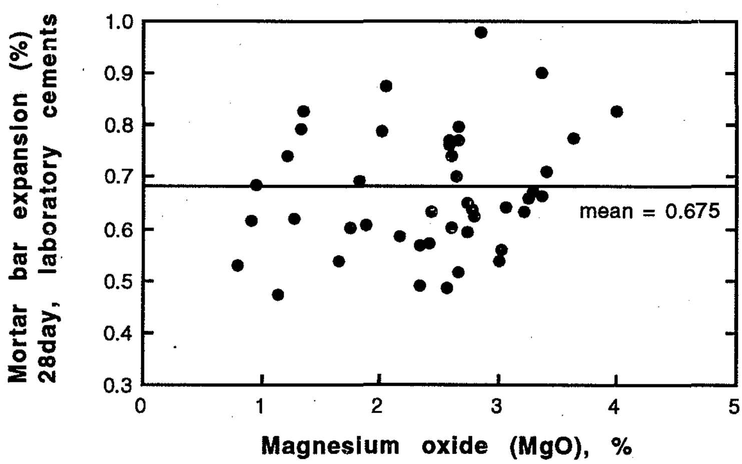

Fig. 3 Mortar bar expansion at 28 days in solution compared with MgO content of the cement.

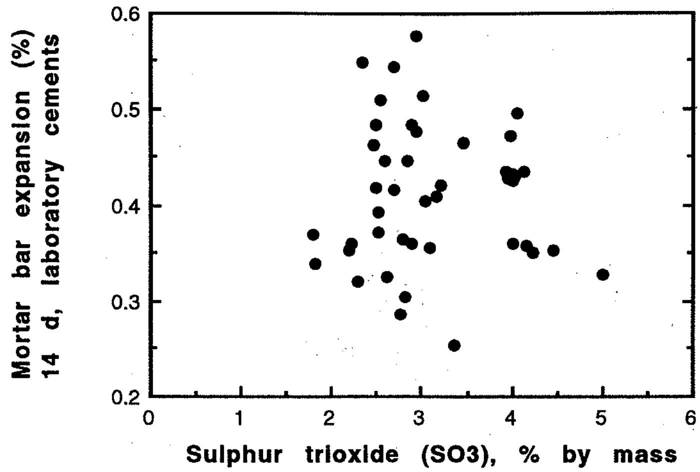

Fig. 6 Mortar bar expansion at 14 days in solution compared with the SO_3 content of the cement.

chemistry of the cements used in this study did not have a major influence on expansion of the mortar bars. It is possible that variation caused by cement was masked by test variability. A specific cement may have an influence on the expansion in this test. In the future it would be good practice to qualify a cement with the aggregate used in this study, before embarking on an aggregate testing program.

CONCLUSIONS AND RECOMMENDATIONS

In a study involving 46 laboratories, it was found that the multi-laboratory coefficient of variation after 14 days in solution was 13.3% when the same cement was used and 14.9% when each laboratory chose a different cement. Precision can be stated as follows: For mortars giving average expansions after 14 days in solution of more than 0.3%, the multi-laboratory coefficient of variation (1s% of ASTM C670) has been found to be 14.9%. Therefore, the results of two properly conducted tests in different laboratories on specimens of a sample of aggregate should not differ by more than 42% (d2s% of ASTM C670) of the mean expansion.

The use of a standard cement did reduce multi-laboratory variation by a small amount (11%). It is probably not worthwhile to establish a standard cement to ensure slightly better precision. At present the CSA version of the test method (A23.2-25A, 1994) calls for a cement with a total alkali content of 0.9 ± 0.10%. The ASTM version (C1260-94) calls for a cement with a autoclave expansion of less than 0.20%. In future, consideration should be given to allowing the use of any normal Portland cement provided it can be qualified using the aggregate used in this study. Mortar bars would have to give expansions between 0.329% and 0.504% which is the d2s% applied to the mean expansion of 0.417% at 14 days found when various cements were used.

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REFERENCES

Davies, G., and R.E. Oberholster 1987, 'An interlaboratory test programme on the NBRI accelerated test to determine the alkali-reactivity of aggregates', National Building Research Institute, CSIRO, Special Report BOU 92-1987, Pretoria, 16p.

Hooton, R.D. 1990, 'Interlaboratory study of the NBRI rapid test method and CSA Standardization process', In *Canadian Developments in Testing Concrete Aggregates for Alkali-Aggregate Reactivity*, Ontario Ministry of Transportation, Engineering Materials Report EM-92, 225-240.

Hooton, R.D., and C.A. Rogers 1992, 'Development of the NBRI rapid mortar bar test leading to its use in North America,' Proc. 9th International Conference on Alkali-Aggregate Reaction in Concrete, Concrete Society, London, 1, 461-467.

Lea, F.M. 1971, '*The chemistry of cement and concrete*', Third Edition, Chemical Publishing Company, N.Y.

Oberholster, R.E., and G. Davies 1986, 'An accelerated method for testing the potential alkali reactivity of siliceous aggregates', *Cement and Concrete Research*, 16, 181-189.

Rogers, C.A., and R.D. Hooton 1991, 'Reduction in mortar and concrete expansion with reactive aggregates due to alkali leaching', *Cement Concrete and Aggregates*, CCAGDP, 13, 42-49.

Stanton, T.E. 1943, 'Studies to develop an accelerated test procedure for the detection of adversely reactive cement-aggregate combinations', ASTM Proceedings, 43, 875-904.