

INTER-LABORATORY STUDY ON THE CSA A23.2-14A CONCRETE PRISM TEST FOR ALKALI-SILICA REACTIVITY IN CONCRETE

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ABSTRACT

Two alkali-silica reactive aggregates were used in this study in which 24 laboratories from Canada, one from the U.S.A. and two from France participated. Each participant was asked to make three concrete mixtures according to the CSA A23.2-14A-M94 Concrete Prism Test method, to cast a number of test prisms from each one of these mixtures and to test them for expansion in different storage conditions.

The results obtained in this study showed that the use of well-controlled testing conditions or parameters in the concrete prism test procedure, such as a reference sand, a reference cement, a standard storage container and fixed concrete mixture proportioning, can greatly contribute in reducing the between-laboratory variability of the test results. For both aggregates investigated, the between-laboratory standard deviation and the coefficient of variation were significantly lower for the test prisms in the NaOH solution than for those subjected to control storage condition.

Key words: Aggregate, Alkali-aggregate reaction, Concrete prism test, Interlaboratory study, Test methods.

INTRODUCTION

In the late 1980's and early 1990's, concern had been raised that the potential alkali-reactivity of a number of concrete aggregates used in Eastern Canada, particularly some marginally or slowly reactive quartz-bearing rocks, was not correctly appraised by the CSA A23.2-14A-M90 Concrete Prism Test. Consequently, a series of changes were adopted for the 1994 edition of the above Standard, which consisted mainly of: 1), increasing the cement content in the concrete mixture from the 310 kg/m³ to 420 ± 10 kg/m³, 2) raising the total alkali content in the mixture to 1.25% Na₂O equivalent, by mass of cement, i.e. total of 5.25 kg/m³ expressed as Na₂O equivalent, 3), adopting more precise mixture proportioning parameters, and 4), using a "standard" storage container. An interlaboratory study was then undertaken by CANMET to investigate the between-laboratory variation of the modified test procedure.

INVESTIGATION PROGRAM

A total of 27 laboratories, 24 from Canada, one from the U.S.A. and two from France, participated in this study. Each participant received materials, supplies, and instructions to make three concrete mixtures according to CSA A23.2-14A-M94 Concrete Prism Test Method. Table 1 gives the details on the various concrete mixtures made in this study (Fournier *et al.* 1995). For mixture 1, specific quantities of the Spratt limestone, CANMET control non-reactive fine aggregate, ASTM Type I high-alkali control cement, and NaOH pellets were prepared and preweighed in CANMET laboratories; these were supplied to the participants for mixing without any further preparation using a specified amount of tap water (Table 1). For mixtures 2 and 3, 100-kg of unscreened Spratt limestone (Mixture 2) and Sudbury Gravel (Mixture 3) aggregates were provided to the participants for use in concrete mixtures according to the CSA A23.2-14A-M94 test procedure; each participant was required to use a local fine aggregate and normal Portland cement meeting the requirements of the test procedure (Table 1). From each of the above three concrete mixtures, test cylinders for compressive strength determination at 28 days, and test prisms for length change determination under specified high temperature and humidity conditions, were to be made.

Table 1: Scope of the study

| Mixture designation | Aggregates ² | | Cement | | Water Content | Alkalies in the mix | Concrete Specimens to be cast | Storage conditions ⁴ |
|-------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------|-----------------------------------------|--------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| | Coarse | Fine | Type | Content | | | | |
| 1 "Ready to use" | Spratt Limestone batch: 57.3 kg | CANMET (control) (non-reactive) ³ batch: 38.2 kg | CSA Type 10 (ASTM Type I) (CANMET control) 0.85% Na ₂ O equivalent. | 420 kg/m ³ batch: 22.3 kg | Effective w/c: 0.42 batch: 9.6 kg | 1.25% (Na ₂ Oe) by cement mass i.e. total alkali content of 5.25 kg/m ³ In the batch: 115 g of NaOH | <ul style="list-style-type: none"> Six + three (optional) prisms, 75 by 75 by 275 to 405 mm in size, for AAR expansion testing. Two cylinders, 100 by 200 mm in size, for compressive strength determination at 28 days. | <ul style="list-style-type: none"> A) Prisms directly in plastic pails at 38°C and R.H. > 95%. B) Prisms wrapped in damp wipers, placed in plastic bags and then in a plastic pail; pail stored at 38°C. C) <u>Optional</u>: storage at 38°C but using containers currently being used by the participants. |
| 2 According to CSA test procedure A23.2-14A ¹ | Spratt Limestone 100 kg of < 25 mm particles provided by CANMET to be graded by the participant | Non-reactive M.F. of 2.70 ± 0.2 <u>local source</u> | CSA Type 10 (ASTM Type I) Alkali content: 0.9% ± 0.1 Na ₂ O equiv. <u>local source</u> | 420 ± 10 kg/m ³ | Effective w/c: 0.42 to 0.45 | 1.25% (Na ₂ Oe) by cement mass i.e. total alkali content of 5.25 kg/m ³ NaOH pellets to be added: <u>local source</u> | <ul style="list-style-type: none"> Three + three (optional) prisms, 75 by 75 by 275 to 405 mm in size, for AAR expansion testing. Two cylinders, 100 by 200 mm in size, for compressive strength determination at 28 days. | <ul style="list-style-type: none"> B) Prisms wrapped in damp wipers, placed in plastic bags and then in a plastic pail; pail stored at 38°C. C) <u>Optional</u>: storage at 38°C but using containers currently being used by the participants. |
| 3 According to CSA test procedure A23.2-14A | Sudbury Gravel 100 kg of < 25 mm particles provided by CANMET to be graded by the participant | Non-reactive M.F. of 2.70 ± 0.2 <u>local source</u> | CSA Type 10 (ASTM Type I) Alkali content: 0.9% ± 0.1 Na ₂ O equiv. <u>local source</u> | 420 ± 10 kg/m ³ | Effective w/c: 0.42 to 0.45 | 1.25% (Na ₂ Oe) by cement mass i.e. total alkali content of 5.25 kg/m ³ NaOH pellets to be added: <u>local source</u> | <ul style="list-style-type: none"> Six + three (optional) prisms, 75 by 75 by 275 to 405 mm in size, for AAR expansion testing. Two cylinders, 100 by 200 mm in size, for compressive strength determination at 28 days. | <ul style="list-style-type: none"> A) Prisms directly in plastic pails at 38°C and R.H. > 95%. B) Prisms wrapped in damp wipers, placed in plastic bags and then in a plastic pail; pail stored at 38°C. C) <u>Optional</u>: storage at 38°C but using containers currently being used by the participants. |

1 CSA Test procedure: CSA-A23.2-14A. Potential Expansivity of Aggregates (Procedure for Length Change Due to Alkali-Aggregate Reaction in Concrete Prisms). CSA A23.2-M94: Methods of Test for Concrete, Canadian Standards Association, ISSN 0317-5669, Rexdale, Ontario, pp. 205-214.

2 Coarse to fine aggregate ratio of 60 : 40 by mass.

3 Non-reactive fine aggregate, i.e. producing expansion < 0.10% at 14 days in the following test methods: ASTM Designation C 1260-94. Standard Practice for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method). Annual Book of ASTM Standards Vol. 04.02, Concrete and Mineral Aggregates, American Society for Testing and Materials, Philadelphia, U.S.A. or CSA-A23.2-25A. Test Method for Detection of Alkali-Silica Reactive Aggregate by Accelerated Expansion of Mortar Bars. CSA A23.2-M94: Methods of Test for Concrete, Canadian Standards Association, ISSN 0317-5669, Rexdale, Ontario, pp. 236-242.

4 A total of five 20 to 25-litre plastic pails were provided by CANMET to each participant to store concrete prisms of mixtures 1 to 3 under conditions (A) and (B).

CASTING, STORAGE AND TESTING OF SPECIMENS

Specimens for AAR Testing

A total of fifteen concrete prisms, i.e. six each for mixtures 1 and 3, and three for mixture 2, were to be made by the participants (Table 1). Enough material was sent to the participants so that those interested in casting additional sets of test prisms to evaluate their own storage containers may do so. The test prisms had dimensions of not less than 75 by 75 by 275 mm and not more than 75 by 75 by 405 mm.

Storage of test specimens

Each participant was provided with five storage containers. These consisted of 22-litre plastic pails for the participants using 300 to 360 mm-long concrete prisms, and of 25-litre plastic pails for the participants using 360 to 400 mm-long concrete prisms. Two of these pails were to be used in each one of the mixtures 1 and 3 and one pail for mixture 2 (Table 1). Each plastic pail was lined with an absorbant terry-cloth material in order to assure proper humidity conditions in the storage container during the test. Each participant also received a roll of 180-mm wide polyethylene tubing and sheets of an absorbant wiper to be used for the new proposed storage condition B (Table 1).

The participants were asked to store the concrete prisms in the storage conditions described in Table 1. In addition, four participants made an extra set of test prisms from each one of the mixtures 1 and 3, and stored them in a 1N NaOH solution at 38°C.

Testing of specimens

The changes in length of the concrete prisms were determined at various ages and the data reported by the participants to CANMET.

Various types of measuring devices were used by the participants in this study. These may be grouped as follows:

- a) Prisms in a vertical position during measurement:
 - Mortar bar type measuring device using a dial gauge or a micrometer.
 - Swenson type measuring device using a micrometer (CSA A23.2-14A M94).
- b) Prisms in an horizontal position during measurement.

AAR TEST RESULTS

Effect of the Storage conditions on the expansion test results

Figure 1 compares the curves of between-laboratory average expansion values versus time for the various series of test prisms from concrete mixtures 1 to 3, and subjected to the different storage conditions investigated. Table 2 gives the between-laboratory average 52- and 104-week expansion values for each one of the above mixtures.

Table 2: Summary of the 52- and 104-week between-laboratory expansion data with associated statistical information

| Mix no. | Storage Conditions | 52 weeks | | | 104 weeks | | |
|---------|-----------------------------|----------|------------|----------|-----------|------------|----------|
| | | Average | Std. Dev.* | C. of V. | Average | Std. Dev.* | C. of V. |
| 1 | A: Plastic pails (control) | 0.17006 | 0.02077 | 12.2 | 0.19346 | 0.02516 | 13.0 |
| | B: Plastic sleeves in pails | 0.15013 | 0.02086 | 13.9 | 0.16746 | 0.02686 | 16.0 |
| | C: Other containers | 0.16582 | 0.02736 | 16.5 | 0.18948 | 0.03469 | 18.3 |
| 2 | B: Plastic sleeves in pails | 0.16196 | 0.03915 | 24.2 | 0.17612 | 0.05072 | 28.8 |
| | C: Other containers | 0.17643 | 0.04518 | 25.6 | 0.19524 | 0.05216 | 26.7 |
| 3 | A: Plastic pails (control) | 0.09333 | 0.03698 | 39.6 | 0.13906 | 0.06741 | 48.5 |
| | B: Plastic sleeves in pails | 0.08681 | 0.03317 | 38.2 | 0.11860 | 0.05571 | 47.0 |
| | C: Other containers | 0.09054 | 0.05293 | 58.5 | 0.12583 | 0.06342 | 50.4 |

* Std. Dev.: Reproducibility Standard Deviation ("S_R" as defined in ASTM E691). This value takes into account the repeatability (or within-laboratory) standard deviation ("S_r" in ASTM E691).

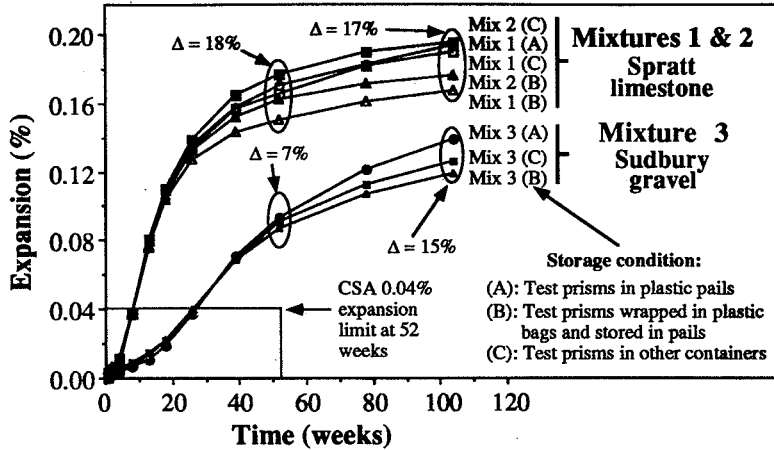


Fig. 1: Between-laboratory average expansion curves for series of test prisms cast from concrete mixtures 1 & 2 (Spratt limestone) and mixture 3 (Sudbury gravel).

Highly-reactive Spratt limestone (Mixtures 1 and 2)

- Between-laboratory expansion curves for the test prisms incorporating the Spratt limestone are all characterized by a rapid onset of expansion followed by the main expansion phase between the 4- and 18-weeks (Fig. 1). During this period, the average expansion values were basically the same for all the test prisms investigated. This is followed by a reduction in the expansion rate causing the expansion curve to level off progressively up to the end of the testing program.
- The differences between the highest and the lowest between-laboratory average expansion values obtained for the series of test prisms cast from concrete mixtures 1 and 2 and subjected to the various storage conditions investigated were about 18% after one year and about 17% after two years (Fig. 1); however, the differences between the individual test results obtained by the participants showed much greater variation. For example, the difference between the individual expansion values obtained for companion test prisms cast from mixture 1 and subjected to storage conditions A and B ranged from -6% (i.e. expansion in storage condition A being 6% less than the expansion in storage condition B) to about +34% at one year (Fig. 2A); the difference ranged from about -14% to + 32% for companion test prisms subjected storage conditions A and C (Fournier *et al.* 1995). For mixture 2, the difference in expansion for companion test prisms subjected to storage conditions B and C ranged from "-29%" to about "+23%" at one year (Fournier *et al.* 1995).

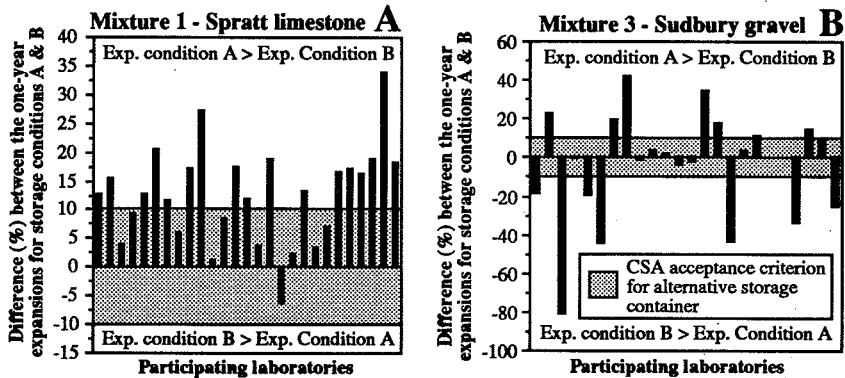


Fig. 2: Difference between the one-year expansion values obtained for companion series of test prisms cast from mixtures 1 and 3 and subjected to the various storage conditions investigated

- According to CSA A23.2-14A-M94, it is possible to use an alternative storage container to that specified in the standard (Condition A of this study); however, in order to be acceptable for use, the following two conditions must be specified: (1), the efficiency of the alternative must be evaluated with a standard alkali-reactive aggregate, and (2), the expansion at 52 weeks must be within 10% of that obtained using the specified container. The difference between the 52-week expansion values obtained for the companion series of test prisms cast from concrete mixture 1 and tested under conditions A and B was > 10% in 63% of the cases (Fig. 2A); the difference was > 10% in 27% of the cases for prisms tested under conditions A and C. For concrete mixture 2 and when using condition B as the control, the difference in expansion was > 10% in 55% of the cases for test prisms tested under conditions B and C (Fournier *et al.* 1995).

Moderately-reactive Sudbury gravel (mixture 3)

- Between-laboratory expansion curves for the test prisms incorporating the Sudbury gravel are all characterized by a relatively slow onset of expansion up to about 8 weeks, and then increases at a fairly constant rate up to 39 weeks. This is followed by a slow down in the expansion rate with the change in the slope being relatively more evident after the end of the first year (Fig. 1).
- The between-laboratory average expansion values obtained for the different series of concrete prisms subjected to the three storage conditions investigated ranged from about 0.087 to 0.093% at one year and from 0.119 to 0.139% at two years (Table 2). The test prisms placed directly in the plastic pails showed average expansion values of more than 7% at one year and 15% at two years than those stored in the plastic sleeves (Table 2). The differences between the individual expansion values obtained by the participants showed a wide variation. For example, the difference in expansion for companion test prisms subjected to storage conditions A and B ranged from "-81%" to about "+42%" at one year (Fig. 2B).
- As mentioned before, the criterion for an alternative storage condition to be acceptable for use in the Concrete Prism Test is that the expansion at one year with the proposed condition should be within 10% of that obtained using the specified container. The difference between the 52-week expansion values obtained for the companion series of test prisms tested under conditions A and B was > 10% in 58% of the cases (Fig. 2B).

As illustrated on Figs. 3A and 3B, for companion series of test prisms cast from the same mixture but tested under different storage conditions, the participants were generally consistent in getting systematically high or low expansion values compared with the between-laboratory average.

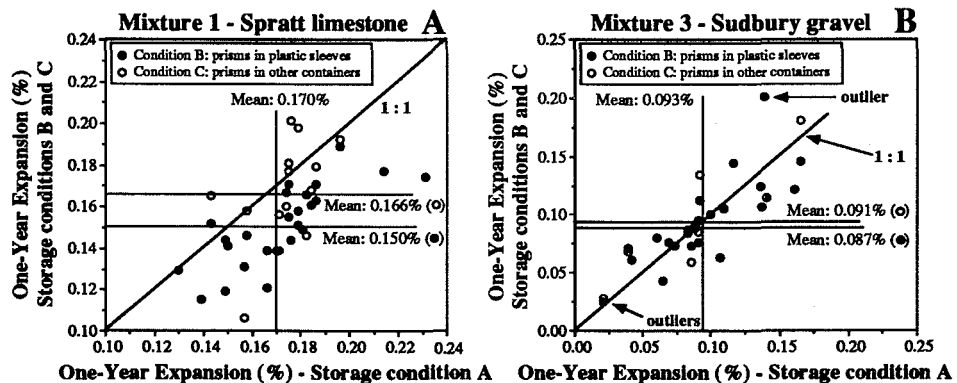


Fig. 3: Comparison between the expansion values of companion series of test prisms cast from concrete mixture 1 (A) and 3 (B) but subjected to different storage conditions

Between-Laboratory Variability of Test Results

Highly-reactive Spratt limestone (Mixtures 1 and 2)

For all the three storage conditions investigated, the between-laboratory C.V. for the various series of test prisms cast from mixtures 1 and 2 were found to decrease rapidly with time and become generally stable after about 18 to 26 weeks of testing (Fig. 4A). The best value of between-laboratory C.V. of 12.2% (at 52 weeks) was obtained for the series of test prisms cast from concrete mixture 1 and tested under storage condition A; the between-laboratory C.V. for the test prisms cast from concrete mixture 2 and tested under storage condition B increased to 24.2% (Table 2).

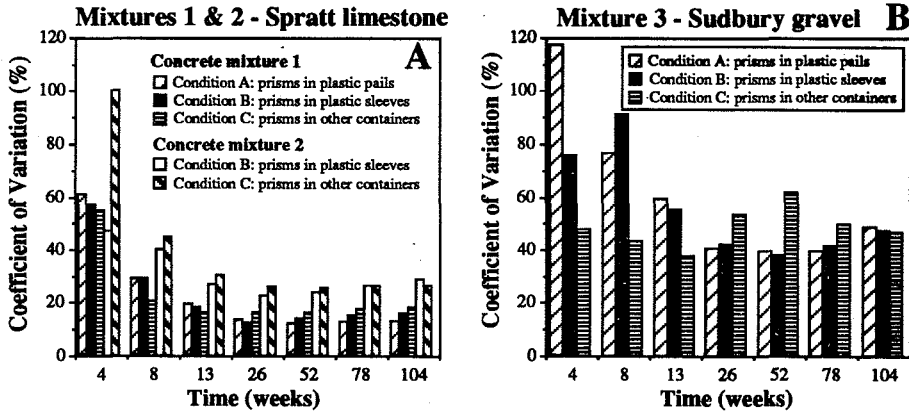


Fig. 4: Plot of the coefficient of variation against time for series of test prisms cast from concrete mixtures 1 and 2 (A), and 3 (B) and subjected to different storage conditions

Moderately-Reactive Sudbury Gravel (concrete mixture 3)

For all the three storage conditions investigated, the between-laboratory C.V. was found to decrease slowly but progressively with time, but remaining fairly variable up to the end of the two-year testing period (Fig. 4B). The lowest value of the C.V. of 38.2% at one year, was obtained for the test prisms subjected to the storage condition B, while the highest C.V. of 58.5% at one year was obtained for the test prisms subjected to the storage conditions C (Table 2).

Discussion on the between-laboratory variability of test results

The expansion results obtained by the participants for the series of test prisms cast from concrete mixture 1 suggest that a reasonably low between-laboratory variation can be achieved by using a number of well-controlled testing conditions, i.e. a reference sand, a reference cement, a standard storage container and a fixed concrete mixture proportion. The between-laboratory variability then depends primarily on the process of concrete mixing, specimen preparation, the reliability of the 38°C storage facilities, of the measuring device and of the personnel in charge of taking the measurements. In fact, the value of the between-laboratory C.V. of about 12.2% obtained for the above series of test prisms is close to the 11.3% for the 28-day compressive strength values reported by the participants for mixture 1 (Fournier *et al.* 1995). This suggests that the between-laboratory variability due to the above parameters is not unduly large.

The much higher between-laboratory values of the Reproducibility S.D. (SR according to ASTM E691) and the C.V. obtained for mixtures 2 and 3 of the program suggest that an additional and important part of the variability of the test method is related to the permissible range of mixture proportioning parameters allowed in the standard procedure, e.g. the use of local sources of cement and fine aggregates, and the permissible range in the w/c. The use of different storage containers also influences the expansion test results, but to a limited extent. As can be seen in Table 2, a between-laboratory C.V. of 13.9% was obtained for the series of test prisms cast from mixture 1 and stored in plastic sleeves. The use of different storage containers for the companion

series of test prisms cast from the same mixture resulted in an increase in the C.V. of about 4% (vs condition A). A small difference was observed for the C.V. obtained for the companion series of test prisms cast from mixture 2, i.e. 24.2% for test prisms under storage condition B versus 25.6% for test prisms under storage condition C.

Effect of Other Parameters on the Expansion Test Results

Cement composition

As mentioned before, participants were asked to make concrete mixtures 2 and 3 using a local normal portland cement having an alkali content of $0.90 \pm 0.10\%$. The total alkali content of the concrete mixture was then to be raised at 1.25% Na_2O equivalent by adding NaOH to the mixture water.

Forty-six per cent of the participants have used a cement with an alkali content falling outside of the range prescribed in the standard test procedure; however, as can be seen in Fig. 5A, the alkali content of the cement used by the participants for the above mixtures did not seem to have a significant influence on the expansion values obtained in the concrete prism test.

Reactivity of the fine aggregate

The participants in this study were also asked to make concrete mixtures 2 and 3 using a fine aggregate from a local source. This sand was to be non-reactive, producing an expansion $< 0.10\%$ at 14 days in the Accelerated Mortar Bar (AMB) Test (CSA A23.2-25A, ASTM C 1260). Six out of the twenty-four sands used by the participants gave AMB expansions higher than the above limit; however, as can be seen in Fig. 5B, no correlation was observed between the one-year expansion values of concrete prisms cast from mixtures 2 and 3 (storage condition B) and the 14-day AMB expansion values obtained for the fine aggregates used by the participants for the above mixtures.

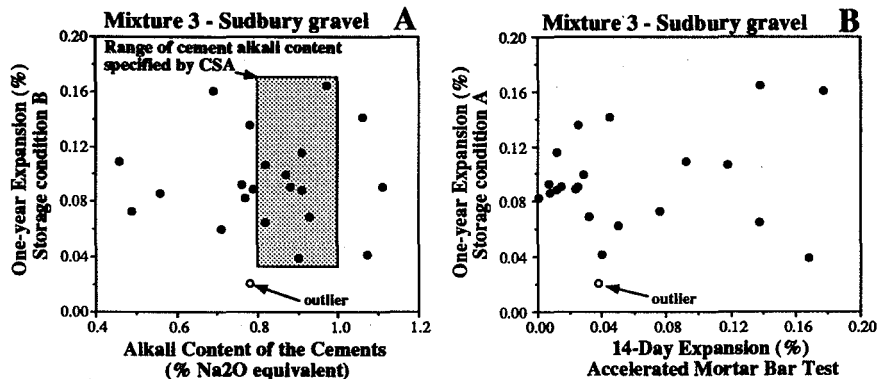


Fig. 5: Effect of the alkali content of the cement (A) and of the reactivity of the fine aggregate (B) used by the participants on the expansion values measured in the concrete prism test.

Alternative testing condition

Figure 6A shows the average expansion values plotted against time for the different series of test prisms cast from concrete mixture 1 and subjected to the various storage conditions investigated. At one year, the test prisms stored in the 1N NaOH and those subjected to storage condition A showed similar expansions of 0.180%; however, at two years, the test prisms in the NaOH solution showed 15% more expansion than those subjected to the storage condition A (Table 3).

The immersion testing showed some interesting results for the moderately reactive Sudbury gravel. Test prisms immersed in the 1N NaOH showed a more rapid onset of expansion than the companion series of test prisms subjected to storage conditions A and B (Fig. 6B). The test prisms immersed in the 1N NaOH solution showed 26 and 58% more expansion at the end of the one- and two-year periods, respectively, than the prisms subjected to control storage condition A (Table 3).

For both aggregates, the use of the immersion testing in the NaOH solution resulted in significant reductions in the values of the S.D. and the C.V. compared with the values obtained for the companion series of test prisms subjected to storage conditions A and B (Table 3).

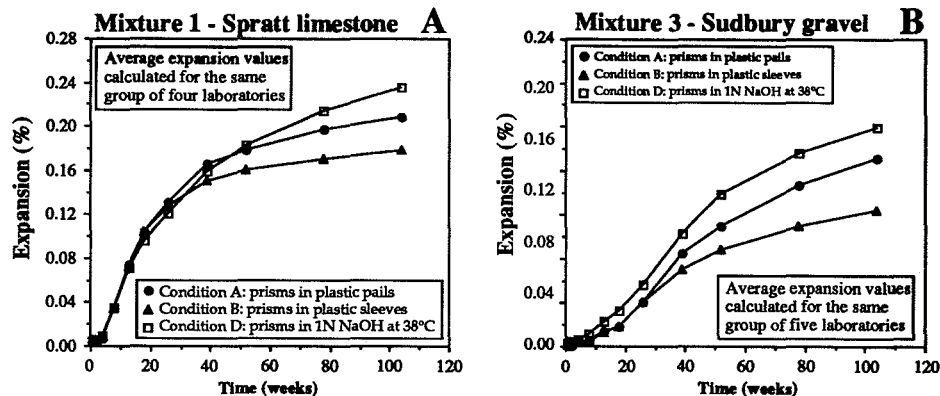


Fig. 6: Multi-laboratory average expansion curves for companion series of test prisms cast from concrete mixture 1 (A) and mixture 3 (B) and subjected to the different storage conditions.

Table 26: 52-week and 104-week Expansion Data with Associated Statistical Information for the Same Group of Laboratories that Have Performed the Immersion Testing in 1N NaOH at 38°C

| Mix no. | Storage | Data at 52 weeks | | | Data at 104 weeks | | |
|---------|-----------------------------|------------------|-----------|----------|-------------------|-----------|----------|
| | Conditions | Average | Std. Dev. | C. of V. | Average | Std. Dev. | C. of V. |
| 1 | A: Plastic pails | 0.17808 | 0.03649 | 20.5 | 0.20742 | 0.04688 | 22.6 |
| | B: Plastic sleeves in pails | 0.15933 | 0.02970 | 18.6 | 0.17750 | 0.03348 | 18.9 |
| | D: 1N NaOH at 38°C | 0.18200 | 0.02549 | 14.0 | 0.23533 | 0.03189 | 13.6 |
| 3 | A: Plastic pails | 0.11047 | 0.04332 | 39.2 | 0.17147 | 0.10571 | 61.7 |
| | B: Plastic sleeves in pails | 0.08820 | 0.04394 | 49.8 | 0.12407 | 0.07234 | 58.3 |
| | D: 1N NaOH at 38°C | 0.13747 | 0.03349 | 24.4 | 0.19953 | 0.04358 | 21.8 |

CONCLUSION

The results obtained in this study showed that the use of well-controlled testing conditions or parameters in the concrete prism test procedure, such as a reference sand, a reference cement, a standard storage container and fixed concrete mixture proportioning, can greatly contribute in reducing the between-laboratory variability of the test results. Parameters such as the cement composition (alkali and the SO₃ contents), the potential alkali-reactivity of the fine aggregate and the size of the test prisms did not seem to have a significant influence on the test results when taken individually; however, when taken all together, these parameters probably contribute in increasing the between-laboratory variability of the test method.

For both aggregates investigated, the between-laboratory standard deviation and the coefficient of variation were significantly lower for the test prisms in the NaOH solution than for those subjected to control storage condition. This method appears particularly promising for testing marginally to moderately reactive aggregates.

REFERENCES

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