

# MODIFICATIONS OF THE CONCRETE PRISM TEST WHEN TESTING LOW-ALKALI CEMENT WITH SCM SYSTEMS FOR MITIGATION OF ALKALI-SILICA REACTION

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## Abstract

When carrying out laboratory tests for ASR, the currently used ASTM C1260 and C1293 methods cannot be used for evaluating the benefits of using low-alkali cement, with or without supplementary cementitious materials (SCM). This has been found by the concrete industry to be a severe limitation on assessment of ASR mitigation options in some regions of North America (especially in the western parts of Canada and the US, where most cements are low-alkali). The effect of cement alkali content and SCM replacement on the expansion and leaching in the concrete prism test was investigated, as well as ways to minimize alkali leaching during the test. Approximately 45% of the alkalis were leaching from 100% PC concrete prisms during the two year test period, regardless of cement (high-, medium- or low-alkali cements). Use of SCM replacements decreased the alkali leaching to around 25%, likely due to a more impermeable pore structure. The most effective way to minimize the leaching was to cover each prism with a plastic bag, where the magnitude of alkali leaching was reduced to only 10%. Also, by modifying the procedure for boosting the level of alkalis in the C1293 concrete prism test and with the use of improved storage conditions to minimize alkali leaching from the samples, it is possible to use this test for evaluating the benefits of concrete mixtures made with low-alkali cements and combinations with SCMs.

**Keywords:** Alkali-Silica Reaction, low-alkali cement, test methods, preventive measures, guidelines

## 1 INTRODUCTION

There is a general consensus that the most accurate laboratory test method for alkali-silica reaction (ASR) is the concrete prism test (CPT). However, this method is not without limitations. Due to leaching of alkalis during the test period, it is impossible to evaluate the effectiveness of low-alkali cementing systems with or without supplementary cementing materials (SCMs). To counteract this leaching, in ASTM C1293, a high-alkali cement ( $\text{Na}_2\text{O}_{\text{eq}} = 0.9\% \pm 0.1\%$ ) is required [1] and the alkali loading of the mixture is raised to 1.25% by dissolving NaOH in the mix water. This is seen as a severe limitation by the industry in some regions of North America (especially in the western parts of Canada and the US) where low-alkali cements are predominately used and often in combination with SCMs. Currently, work is in progress to create a performance test, where one of the main requirements is to test all cementing systems and SCMs [2]. This objective cannot be obtained if it is not possible to include cementing systems with cements with  $\text{Na}_2\text{O}_{\text{eq}}$  less than 0.9%.

The objective of the project is to adapt current standardized test methods so they are able to detect the beneficial effects of the low alkali cementing systems. In order to do so, the limitations of each method must be known and changes made to eliminate or minimize their effects. This paper focuses on evaluating the amount of leaching from different cementing systems as well as to test out methods to decrease the extent of leaching. Previous research has shown that the alkali leaching during the test period can be 22-45% within the first year [3] and is affected by storage conditions, binder type and temperature. With the very high relative humidity inside the containers, even the allowable  $\pm 2^\circ\text{C}$  temperature fluctuations can result in condensation and consequently leaching.

## 2 EXPERIMENTAL PROGRAM

### 2.1 General

The modified tests were evaluated using a known reactive aggregate as a coarse aggregate. Testing according to ASTM C1293 requires casting 3 prisms from a mixture with set portions of aggregate and cement. The w/cm of the mixture should be in the range of 0.42-0.45 and the alkali

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content of the concrete mix is raised to 1.25% by dissolving NaOH pellets in the mix water [1]. The specimens are then stored at  $38 \pm 2^\circ\text{C}$  in a sealed container with a small water reservoir in the bottom and measured regularly over the test duration.

Different methods were evaluated for minimizing alkali leaching from the concrete prisms, either by changing the storage conditions or by changing the sample geometry. ASTM C1293 uses a prism with a cross-section of 75 x 75 mm and 285 mm in length. A concrete test specimen with a bigger volume to surface area would be expected to reduce the rate of leaching [2]. Concrete structures or exposure blocks in the field do not leach as much as the smaller laboratory prisms. Changing the sample geometry may also lead to a decrease in leaching and/or required test duration. Two concrete mixtures, one made with high-alkali cement and the other with low-alkali cement, were cast into three different size cylindrical samples with 75 mm, 100 mm, and 150 mm diameter in addition to the standard 75 x 75 mm prisms as well as prisms with cross-section of 100 x 100 mm. The length of the samples was kept constant at 285 mm to fit into the length change comparator.

In an attempt to minimize alkali leaching from the samples, four different measures were tested. To protect the samples from condensing water, (a) the prisms were covered with either a commercially available, convex plastic disc (i.e. a Frisbee) covering all three prisms or (b) the top half of each prism was covered by an inverted plastic bag, (c) the ends as well as  $\sim 1$  cm up the length of one set of prisms were sealed with epoxy to reduce alkali leaching, or (d) two sets of prisms were kept and measured at  $38^\circ\text{C}$ , eliminating the need for cooling prior to measurement.

In addition, changes were made to the alkali boosting procedure used in the ASTM C1293 standard. The concrete prism tests used mixtures with different cementitious binders. All mixtures were boosted to 40% above the  $\text{Na}_2\text{O}_{\text{eq}}$  of the cement used to counteract leaching. This was chosen based on data provided in the literature [3] and based on the fact that the boosting from 0.9% to 1.25% as required by the standard is 40%. Using this approach, all mixtures cast with cements of higher alkali content than 0.9% will have higher alkali loading than required in the standard test method.

## 2.2 Materials and mix designs

### *Aggregates*

The primary aggregate used was Sudbury gravel, containing reactive greywacke and argillite from Sudbury, Ontario [4]. The Sudbury gravel is a slowly reactive aggregate but has shown satisfactory results in field structures made with low-alkali cement and low cement content [5]. Similar aggregate has shown satisfactory performance over 40 years with fly ash and low cement contents [6]. Some concretes were also cast using a highly reactive siliceous limestone, Spratt #3 [7], from near Ottawa, Ontario. The Spratt aggregate is, on the other hand, highly reactive and can show deleterious expansion even with low-alkali cement, i.e. the alkali loading of the concrete must be much lower than for the Sudbury gravel. It has been shown that the Spratt aggregate can cause expansion and cracking in field samples with alkali loading as low as  $1.9 \text{ kg/m}^3$  [8]. Both aggregates were obtained from stockpiles kept by the Ontario Ministry of Transportation.

The concrete mixtures did not all use the same non-reactive fine aggregate. This occurred because they were cast over the span of few years. Both of the sand sources used are commonly used concrete sand sources in Toronto and do not show any ASR issues in the field. The initial sand source showed a 14 day expansion in the AMBT of 0.10%. When that source was no longer available, the sand source chosen was the one showing the lowest expansion in the AMBT. The second sand had an expansion of 0.07% at 14 days. As the samples were all cast in sets, most of the time the samples being compared in this paper were cast with the same sand source.

### *Cementitious materials*

The high-alkali cement is CSA A3001 Type GU cement from Holcim's (now CRH) Mississauga Ontario plant, with an alkali content of 1.01%  $\text{Na}_2\text{O}_{\text{eq}}$ . The low-alkali cement is from Lafarge's, Alpena Michigan plant and has an alkali content of 0.57%  $\text{Na}_2\text{O}_{\text{eq}}$ . When deciding on the materials, the goal was to obtain a medium-alkali cement with an alkali content around 0.8% but chemical analysis showed that the alkali content of the medium-alkali cement (GU cement from St. Constant, Quebec) was 0.89%, i.e. a bit higher than preferred. Due to this, a sample was prepared with a 50/50 mixture of the Alpena cement and the St. Constant cement, creating a cement with 0.70%  $\text{Na}_2\text{O}_{\text{eq}}$ . Chemical analysis of the cements is shown in Table 1.

To look at the effect of different cement alkali contents and SCMs on the leaching of alkalis during the CPT, samples were cast with high-, medium-, and low-alkali cement. The high-alkali and low-alkali cements were also used with a class F fly ash from SRMG, Cholla Plant, in NM, USA at 15,

25, and 35% replacement levels and slag from Holcim, Mississauga, at 25%, 35% and 50% replacement levels. The medium-alkali cement was only used with slag replacement. All of the mixtures were cast with the Sudbury aggregate.

#### *Other materials*

Sodium hydroxide pellets were dissolved in mix water to increase the alkali loading of the concrete mixtures. The pellets were of reagent grade and >97% pure. In all calculations and measurements, no adjustments were made and it was assumed they were 100% NaOH.

#### *Mix designs*

The proportions of aggregates, cement and water in the mixtures was according to ASTM C1293. The only change made to the mixtures was with regard to the boosting of the alkalis. The standard calls for boosting to 1.25% from an average  $\text{Na}_2\text{O}_{\text{eq}}$  of  $0.90 \pm 0.1\%$ . Instead of the standardized alkali boosting, the alkalis in the mixtures were boosted to 40% above the alkali content of the cement used. Only the alkalis in the cement were considered when calculating the level of alkali boosting, i.e. a mixture with low-alkali cement and fly ash replacement was not boosted to the same alkali loading as the mixture made with low-alkali cement alone.

### **2.3 Methods for assessment and analysis**

In addition to measuring length change, the samples were weighed and a sample of the water in the bottom of each storage container was collected.

The water samples were analysed as soon as possible: in most cases only few hours after sampling. The hydroxyl ion concentration was measured using potentiometric titration with 0.05M  $\text{H}_2\text{SO}_4$  and the  $\text{Na}^+$  and  $\text{K}^+$  concentrations were measured with a flame photometer. Only results based on changes in the hydroxyl ion concentrations are presented.

## **3 RESULTS**

#### *Effect of cement alkali and SCM replacement*

The expansion at 2 years is shown in Figure 1. As expected the expansion decreases with SCM replacement but for some reason the mix with medium-alkali cement is showing more expansion than the one with high-alkali cement. The low-alkali mixture made with 100% cement did not show significant expansion (the limit at 2 years is 0.04% [1]).

The use of SCMs did reduce leaching significantly but the replacement level does not seem to have much influence on the leaching from the samples, as shown in Figure 2. The leaching from samples with slag or fly ash replacement is only 20-30% of their initial alkali content. It is also noticeable that the leaching from high- and low-alkali mixtures without SCMs is the same (close to 50%), while the medium-alkali mixture exhibited much less leaching after 2 years (only 25%). This mixture also showed higher expansion than the high-alkali mixture.

#### *Specimen geometry*

Figure 3 shows the expansion over 2 years for those samples. As can be seen, the cylindrical samples made with the high-alkali cement mixture, had higher expansion than the prisms but there was no difference in expansion between cylinders with different diameters even though the leaching after 2 years from 75 mm cylinders is higher than the leaching from the larger cylinders (Figure 4).

The bigger prisms do show higher expansion than the smaller prisms, and leaching from the bigger 100 x 100 mm prisms is less than from the smaller 75 x 75 mm ones. The bigger prisms do not, however, show expansions as high as the cylindrical specimens. Specimens made with different geometries and sizes with low-alkali cement showed no difference in expansion. The 2-year expansion in all cases is only around 0.02%, as shown in Figure 3.

#### *Minimizing leaching during CPT*

By minimizing the leaching from the specimens during testing, less alkali boosting would be needed for the test. This is one of the major issues in the making of a reliable performance test [2]. Figure 5 shows the expansion from concrete specimens where various ways to minimize leaching were used. The only one of these methods that resulted in a significant decrease in leaching was when an individual plastic bag was inverted over each prism. Figure 6 shows that these plastic bags decreased the leaching from 45% of the initial alkali loading down to ~10-20%.

Lastly, length change measurements for two sets of prisms were made at 38°C to try to minimize the leaching by limiting the condensation that occurs on cooling: one set made with Sudbury

aggregate and another with Spratt aggregate. Both sets included the second sand source and used medium-alkali cement with alkali equivalent of 0.89%. The samples that weren't cooled down prior to measurements actually showed slightly less expansion than the other ones and they were more difficult to measure, as they tended to cool down fast and dry out the prism surfaces quickly while being measured. Care was taken to measure them as soon as possible after removal from the 38°C storage room and to cover them with a moist cloth after measurement before they were placed back in the container. The change in the measuring technique did not, however, have much effect on the alkali leaching from the prisms. As shown in Figure 6, the prisms made with Sudbury aggregate showed the same amount of leaching while the ones made with Spratt aggregate showed a decrease in leaching from ~35% to 25% of original alkali loading after 1 year.

#### *Verification of the proposed modification*

Since using low alkali loading with Sudbury aggregate has shown to be effective in the field, it was decided to test out the most promising modifications by using Spratt aggregate. This is due to the more expansive nature of Spratt. The results of those tests are shown in Figure 7. The alkali content of all of these samples were boosted by 40% of the alkali content of the cement used. The results from this suggest that with this aggregate, low-alkali cement would not be a sufficient mitigation option, but if used together with 25% slag or 15% fly ash, it should be satisfactory.

To verify that the combination of 40% alkali boosting and limiting leaching are suitable, additional tests were also run with Spratt aggregate and low-alkali cements, with and without plastic covers, and with different levels of alkali boosting. Those results are shown in Figure 8 and when Spratt aggregate is used in the CPT without boosting, it does not expand beyond the 0.04% limit. By minimizing the level of alkali leaching after 1 year down to ~20%, the sample does expand past the 0.04% limit at 1 year, but only slightly. Boosting the alkali content by 20% with NaOH does push it over the limit. When combined with the plastic bags over the tops of the prisms to minimize the leaching, the prisms are showing similar expansion prisms in standard storage conditions but boosted by 40%. The leaching of those samples is shown in Figure 9.

It is also believed that cement with an alkali content lower than 0.7% would be sufficient to reduce deleterious expansion caused by Sudbury aggregate [9]. To test the modified method, two sets of prisms were cast with Sudbury aggregate and a mixture of medium- and low-alkali cements and then boosted by 40% with NaOH. One set of prisms was tested in a standard container and the other was covered with plastic bags to minimize the leaching. Figure 8 shows that both mixtures expanded more than the 0.04% expansion limit.

## **4 DISCUSSION**

Bigger samples showed less leaching, but changing the sample geometry is not sufficient for testing all cement types. The leaching is decreased by almost half by the use of bigger samples and, due to their lower surface to volume ratio, cylinders show less leaching than prisms. Even though that is the case, the expansion does not change very much when tested with low-alkali cements. All of low-alkali cement samples showed expansion of 0.02%, regardless of shape or size. Moisture may have difficulty penetrating the bigger samples and therefore slowing down the alkali-silica reaction in addition to the decreased leaching. Lindgård et al. [10] showed that larger prism cross sections will lead to slightly lower internal RH as well as a larger gradient in RH from the outer surface to the prism core. Bigger samples also do increase problems in handling and storage.

The most effective way to decrease the leaching was to cover the top half of the prisms with plastic bags. One bag was used per prism and the length of the bag was set to cover the top half of the prism length. The idea was to sufficiently protect them from the condensing water but not to limit the access of humid air to the prisms needed to drive the ASR reaction. The inverted plastic bag covers were able to decrease the leaching down to only 10-15% while use of a Frisbee as a cover and the sealing of the prism ends with epoxy did not show any significant decrease in leaching. It is possible that the curvature of the Frisbee disc was not sufficient as it was noticed that water was condensing underneath it, just like on the underside of the container lids without the Frisbees. Not cooling the samples down before measurements was found to be problematic and did not solve the leaching problem. The leaching was not decreased and if the measurements are not carried out in a room kept at 38°C, the samples cool down rapidly in a few seconds and the surfaces can dry out as well. The extra effort needed to take the measurements without cooling the samples was not worth it as it did not provide better results.

Increasing the alkali loading of the concrete mixture based on the alkali content of the cement being used showed promising results but it would be even more useful if the need to boost the alkalis

could be completely avoided. The tests with Spratt aggregate do show that even though alkali leaching is decreased to 10% or 15% of the original alkali loading, it is not enough to obtain expansions above 0.04% in a concrete made with a highly reactive aggregate. Boosting the alkali loading by 20% and using plastic bags to decrease the leaching resulted in a similar expansion to boosting the alkali loading by 40% of the cement alkali content and using the ASTM C1293 standard storage method. However, this was only tested using two reactive aggregates and needs to be confirmed with wider range of aggregates of known reactivity in the field.

## 5 CONCLUSIONS

The CPT may be useful to identify deleteriously expansive concrete mixtures using cements with a wider range of alkali contents than is currently allowed in ASTM C1293. Boosting the alkali level to a fixed percentage of the alkali content of the cement to be used provides a way to test cements of different alkali contents. If the alkali level of the cementing system is low, the leaching in the test will have a bigger effect on the expansion measurements with the risk of a mixture showing innocuous behaviour in the laboratory test but deleterious expansion in the field. The leaching from the prisms in ASTM C1293 can be decreased by loosely covering the upper half of each prism with a plastic bag, leaving the bottom half of the prism open to the moisture in the storage container. Using both of these measures, it is possible to create a modified ASTM C1293 test method that can distinguish between reactive and innocuous mixes, even when low-alkali cement is used.

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TABLE 1: Chemical analysis of the cements used.

	High-alkali cement (Hol)	Medium-alkali cement (Con)	Low-alkali cement (Alp)
SiO <sub>2</sub> (%)	19.47	19.72	19.99
Al <sub>2</sub> O <sub>3</sub> (%)	5.12	4.27	4.75
TiO <sub>2</sub> (%)	0.26	0.20	0.24
P <sub>2</sub> O <sub>5</sub> (%)	0.13	0.24	0.10
Fe <sub>2</sub> O <sub>3</sub> (%)	2.31	2.66	2.81
CaO (%)	62.03	61.97	63.20
MgO (%)	2.47	2.52	2.79
Na <sub>2</sub> O (%)	0.25	0.25	0.21
K <sub>2</sub> O (%)	1.16	0.98	0.54
Mn <sub>2</sub> O <sub>3</sub> (%)	0.08	0.09	0.17
SrO (%)	0.09	0.22	0.06
SO <sub>3</sub> (%)	3.98	3.91	2.65
LOI (%)	2.24	2.73	2.20
BaO (%)	-	0.00	0.00
ZnO (%)	0.01	0.03	0.06
Cr <sub>2</sub> O <sub>3</sub> (%)	0.01	0.02	0.02
Total (%)	99.64	99.79	99.80
Na <sub>2</sub> O <sub>eq</sub> (%)	1.01	0.89	0.57

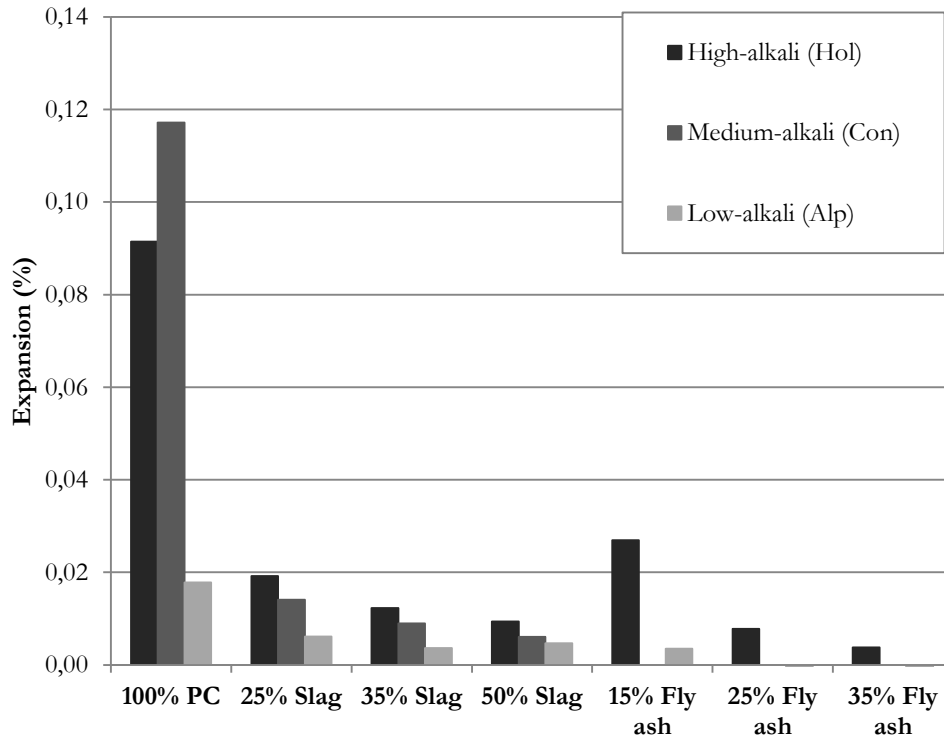


FIGURE 1: Effect of cement type and SCM replacement on 2 year expansion in the CPT. All samples were cast with Sudbury aggregate and boosted by 40% of the alkali content of the cement used.

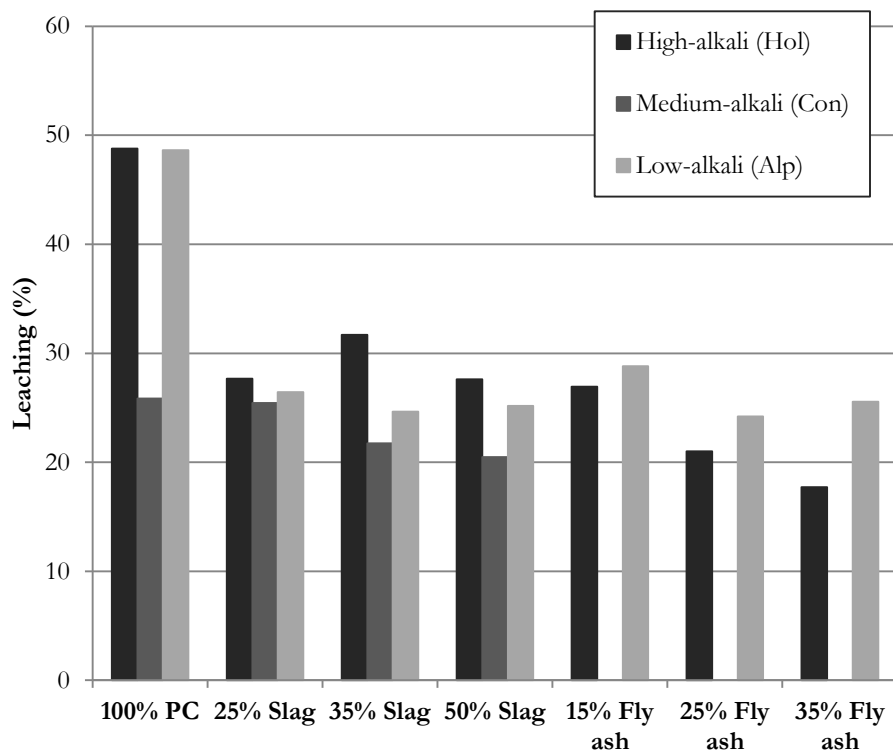


FIGURE 2: Effect of cement type and SCM replacement on leaching after 2 years in the CPT. All samples were cast with Sudbury aggregate and boosted by 40% of the alkali content of the cement used.

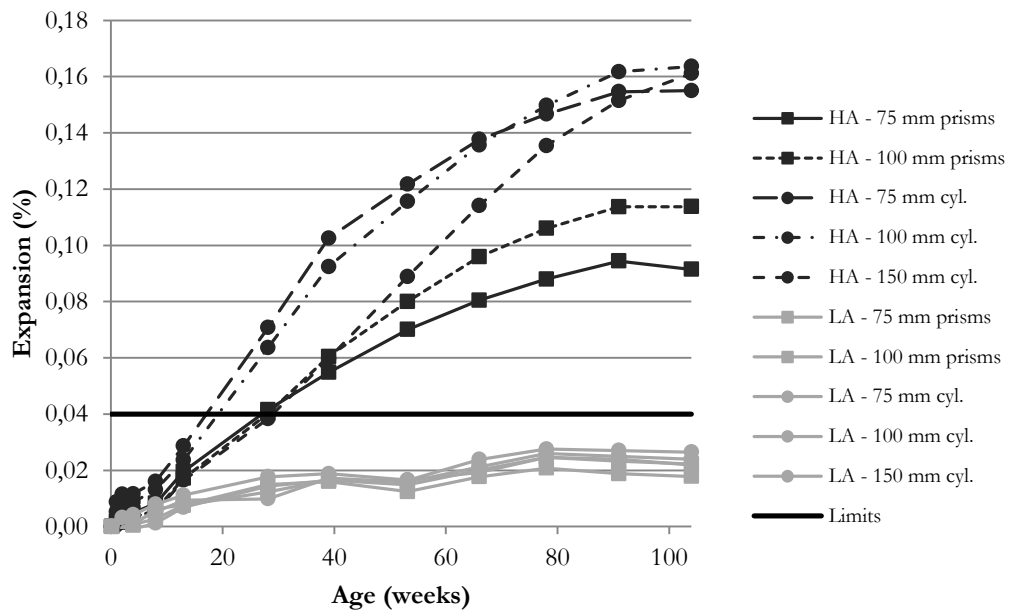


FIGURE 3: Effect of sample geometry (shape and size) on expansion in the CPT. All samples were cast with Sudbury aggregate and boosted by 40% of the alkali content of the cement used.

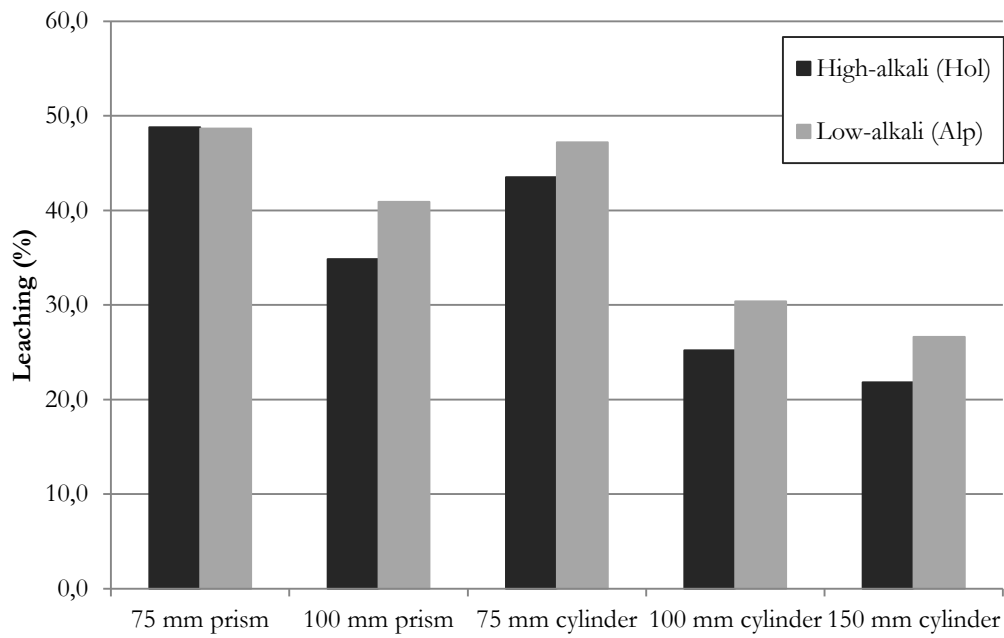


FIGURE 4: Effect of sample geometry (shape and size) on leaching at 2 years in the CPT. All samples were cast with Sudbury aggregate and boosted by 40% of the alkali content of the cement used.

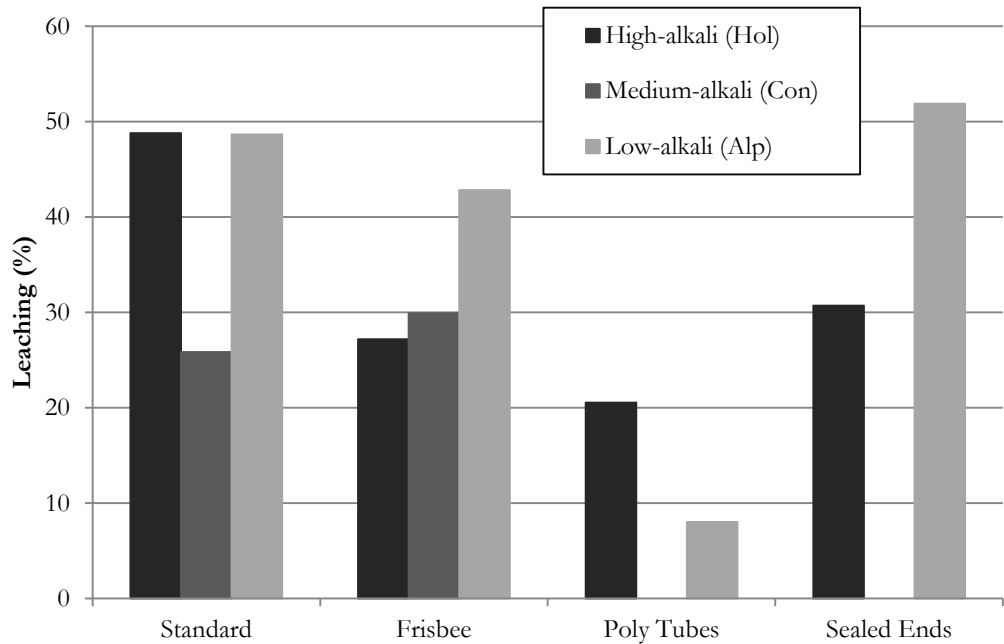


FIGURE 5: Methods in attempt to minimize leaching at 2 years from the CPT. All samples were cast with Sudbury aggregate and boosted by 40% of the alkali content of the cement used. The medium-alkali cement was only tested in a standard container and with Frisbee.



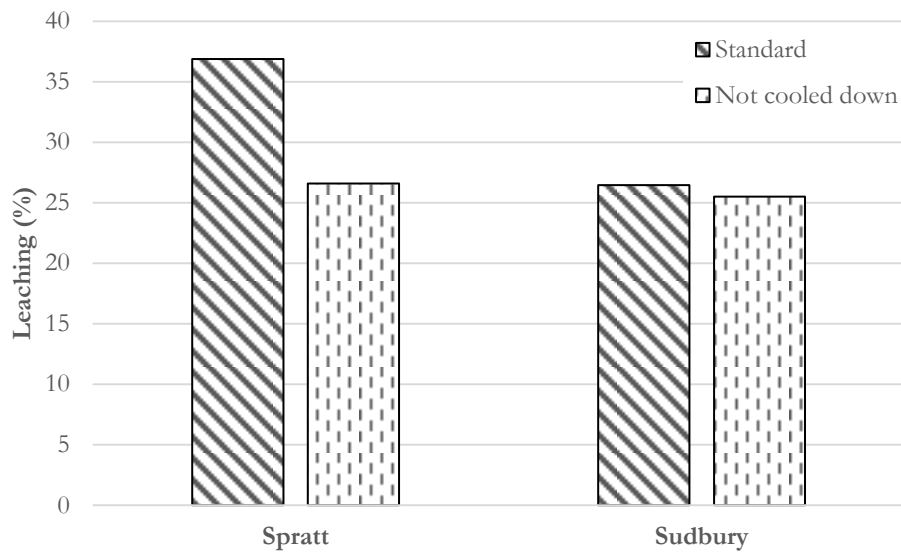


FIGURE 6: Leaching at 1 year in the CPT from prisms made with Sudbury or Spratt aggregate and medium-alkali cement. Samples were either tested according to ASTM C1293 (cooled down to 23°C before measurements) or measurements were made at 38°C.

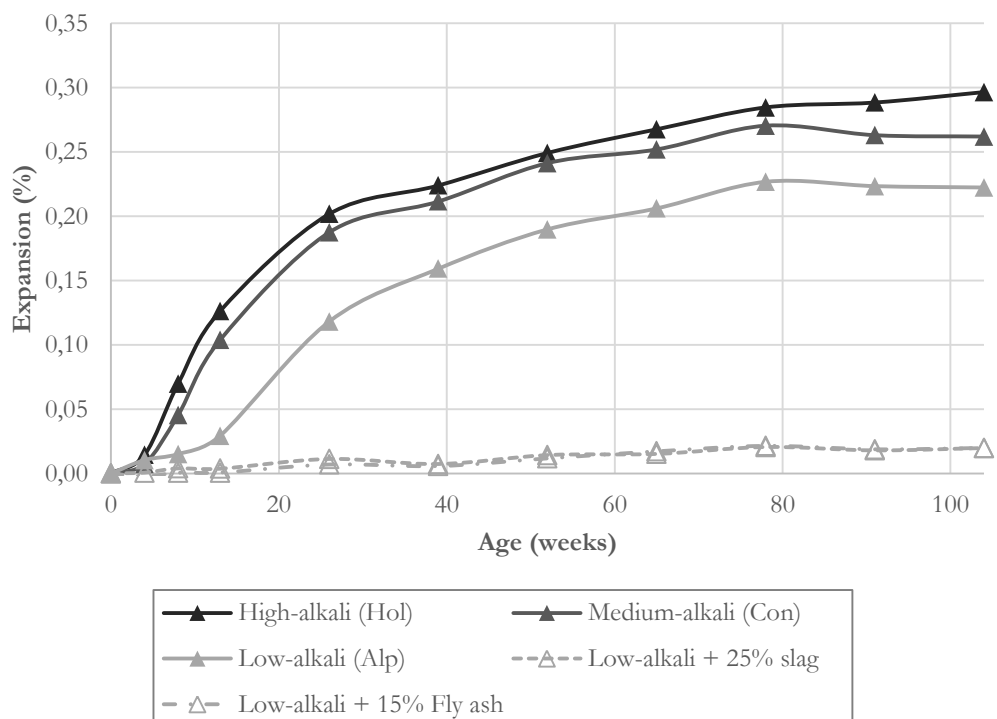


FIGURE 7: Expansion of Spratt aggregate in the CPT. All mixes were boosted by 40% of the alkali content of the cement used.

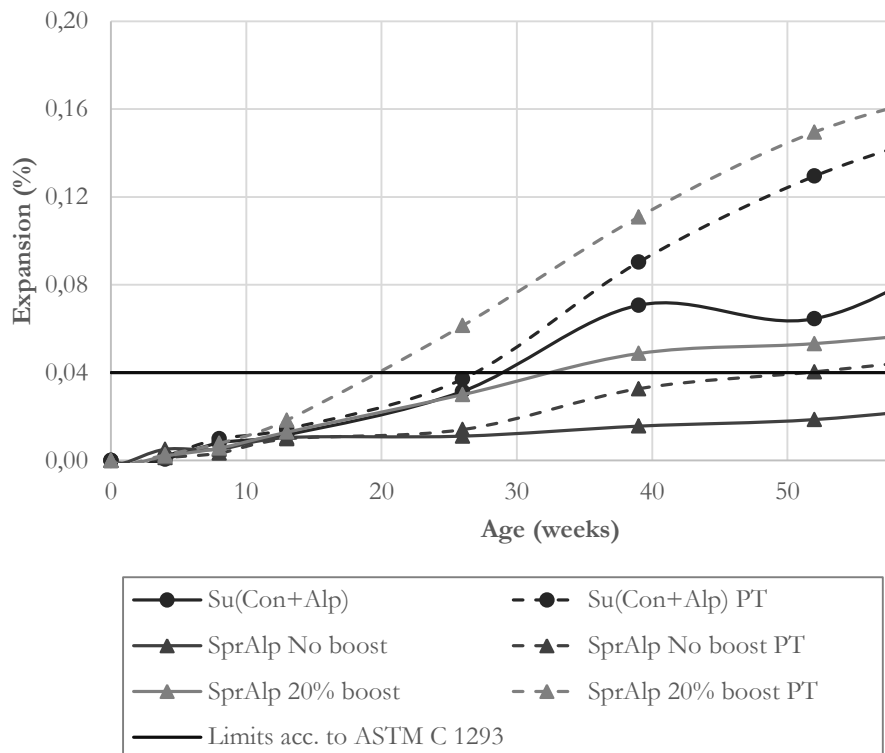


FIGURE 8: CPT Expansions. Two sets of prisms are made with Sudbury aggregate (Su(Con+Alp) and Su(Con+Alp) PT) and the others with Spratt aggregate. The prisms were either tested in standard containers or loosely covered with plastic bags inside the containers to minimize leaching (PT).

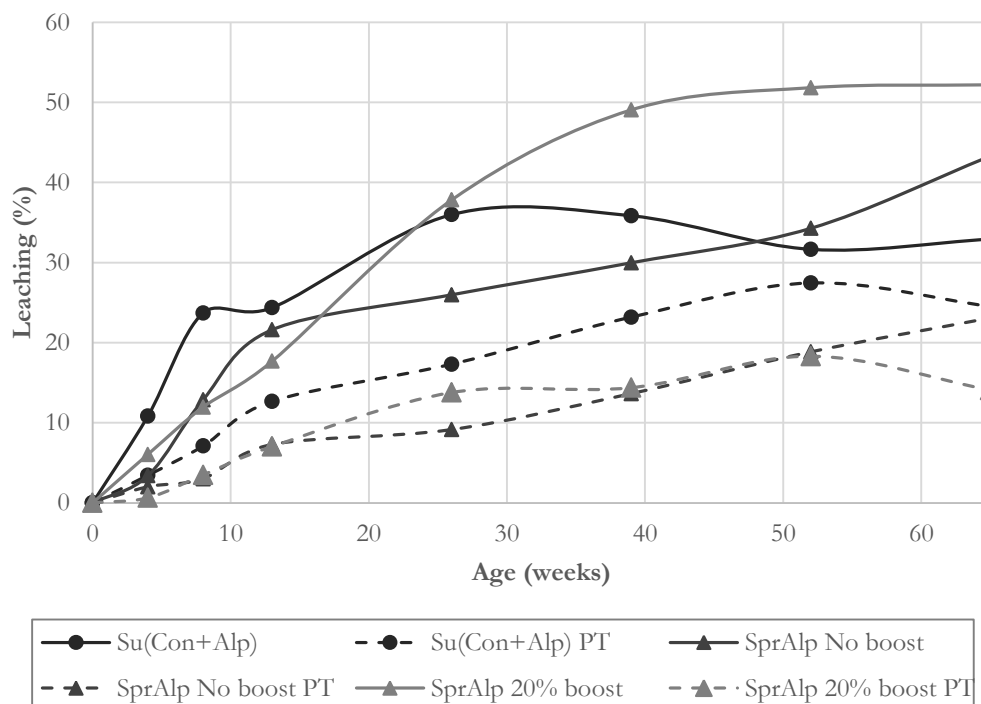


FIGURE 9: Alkali leaching measured during the CPT. Two sets of prisms were made with Sudbury aggregate (Su(Con+Alp) and Su(Con+Alp) PT) and the others with Spratt aggregate. The prisms were either tested in standard containers or loosely covered with plastic bags inside the containers to minimize leaching (PT).