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LEACHING OF ALKALIES IN ALKALI–AGGREGATE REACTION TESTING

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ABSTRACT

Mortar bar expansion tests for evaluating alkali-silica reactive aggregates are not always reliable. Containers with efficient wick systems may cause excessive leaching of alkalies out of mortar bars, thus reducing the expansion. Similar effects are found with concrete prisms made with alkali-carbonate reactive aggregate. The use of Pyrex glass is not satisfactory for calibrating storage conditions because it contributes alkalies to the reaction.

INTRODUCTION

Concrete and mortar bar expansion tests have been used for over thirty years for selecting appropriate aggregates or aggregate-cement combinations that will not cause damage due to alkali-aggregate reactivity. In North America, the concrete prism test (CSA, 1986) has usually been used for evaluating alkali-carbonate reactivity. The mortar bar expansion test (ASTM C 227) has been successfully used for evaluating alkali-silica reactivity.

A study reported at the last conference (Rogers, 1987) found that the multilaboratory coefficient of variation for concrete prism expansions greater than 0.014% was 23%. This wide variation was attributed, in part, to variable leaching of alkalies out of the concrete in various humidity conditions. The mortar bar expansion test has also been thought to suffer from the same problem. However, a study by Hooton (1987) using Pyrex glass found that the greater the amount of leaching of alkalies out of the mortar bars, the greater the expansion. In an effort to resolve these conflicting observations, further experiments were conducted.

LABORATORY TESTING

Mortar Bar Expansion Test

A number of identical mortar bars were made using an alkali-silica reactive limestone. The bars were made according to ASTM C 227 with a blended laboratory cement of 1.17% Na_2O equivalent alkalies. The water/cement ratio was 0.50, giving a flow of about 107. The reactive aggregate chosen for this study was a quarried limestone containing 4% microscopic chalcedony from Spratt's Quarry near Ottawa, Ontario. Fortunately, this aggregate has a limited field performance history. Concrete made with this aggregate showed expansion and cracking at an age of nine years. Petrographic examination showed alkali-silica gel filling and lining air voids and filling cracks in the coarse aggregate.

Following curing, the mortar bars were assigned randomly to storage in one of ten storage containers. The storage containers were described by Hooton (1987) and are listed in Table 1. The containers were stored at 38°C, and the bars measured periodically up to an age of one year. One bar from each container was removed at an age of thirteen months and pulverized to pass a 75 µm sieve for chemical analysis. Water-soluble alkalies were determined as follows: 1 g of powder was added to 100 ml of distilled water, boiled for ten minutes, and allowed to stand overnight. The suspension was filtered, and the resulting solution made up to 100 ml by a further addition of distilled water. Sodium and potassium were determined by flame photometry. The acid-soluble alkalies were determined following digestion in hydrochloric acid. Results are presented in Table 1. Petrographic examination was conducted on the retained end pieces of each bar. The air voids were inspected using a stereoscopic microscope at a magnification of between 20 and 40. The number of air voids either lined or filled with alkali-silica gel are also reported in Table 1.

Concrete Prism Expansion Test

A number of identical concrete prisms were made according to the Canadian Standards Association Procedure (CSA, 1986). The cement was a normal Portland cement of 0.9% Na_2O equivalent alkalies. The cement content was 313 kg/m^3. The alkalies were increased to 1.25% Na_2O by weight of cement (3.9 kg/m^3 of Na_2O) by dissolving an appropriate quantity of NaOH in the mixing water. The coarse aggregate was a quarried dolomitic limestone from the Pittsburg Quarry near Kingston, Ontario. This rock is alkali-carbonate reactive and causes rapid expansion and cracking of concrete. The fine aggregate was a non-reactive natural sand.

Following curing, the prisms were assigned randomly to one of four storage conditions: 1. Normal humid room curing at 23°C (100% humidity). 2. Same as condition 1, but each individual prism was sealed in a plastic bag containing 100 ml of water. (The water was replaced and the bag resealed after every reading.) 3. Immersed in a closed container containing a 5% sodium chloride solution at 23°C, or 4. In a sealed box, over water with filter paper wicks at 38°C. The prisms were periodically measured for change in length.

At an age of 130 weeks, a prism from each storage condition was removed and air dried. One half of each prism was pulverized, and the water-soluble alkalies remaining in the concrete were determined in the same manner as for the mortar bars. The remaining half of each prism was cored with a diamond drill. Parts of the outside layer and core were pulverized, and alkalies measured to determine the variation with depth.

RESULTS AND DISCUSSION

Mortar Bar Expansion Test

The average mortar bar expansions obtained in the various containers are shown in Figure 1. It can be seen that all mortar bars stored in containers with wicks gave expansions that met the ASTM six-month requirement of 0.1%. The container which resulted in the least expansion was the new (1987) standard ASTM C 227 container. When the same container was used both with and without wicks, as in the case of Containers 4 and 5, removing the wicks resulted in a 400% increase in expansion.

Figure 2 shows that the greater the residual alkali content, the greater the expansion of the mortar bars. This is well correlated with the observable alkali-silica gel. Those bars showing the least expansion also exhibited low residual alkali levels and small amounts of alkali-silica gel. The only exception to this general rule is the results obtained with Container 9. In this case, after three months, instead of adding 10 ml of water to each plastic sleeve, 200 ml was inadvertently added. This resulted in excessive leaching of alkalies and a cessation of expansion after twenty weeks. This bar contained high amounts of visible alkali-silica gel, suggesting that gel formation in voids takes place at a relatively early age in this test. In the case of an identical container (10), where only 10 ml of water was used, expansion continued up to an age of one year.

Do other natural aggregates show the same effect? At the Ministry of Transportation laboratories, over one hundred mortar bar expansion tests have been conducted on natural aggregates with one bar in a plastic bag and the other in an unwrapped condition in the same container with filter paper wicks. Aggregates which have a good field performance, and are judged non-reactive, show little or no difference in expansion. Aggregates with poor field performance due to alkali-silica reaction invariably show a substantial (at least 200%) increase in expansion when stored in a plastic bag.

Why are the observations made here opposite to those previously reported by Hooton (1987)? He found, using the same cement with Pyrex glass aggregate, that mortar bars stored in Containers 1, 2, 3, 4, 7, and 8 all showed over 0.3% expansion in two weeks. Much less expansion was found in Containers 5, 6, and 10 which, in this study, promoted the most expansion. Pyrex contains significant amounts of alkalies which are released in the highly alkaline environment of a mortar bar. While in the efficiently wicked containers, alkalies are leached into the water at the bottom of the container, but the Pyrex contributes more alkalies to continue the reaction.

Pyrex is not a typical alkali-reactive aggregate. Aggregates rarely release alkalies to concrete; if they did so, the use of lower alkali contents and cement contents would not be universally helpful in controlling alkali-aggregate reactions. The use of mortar bar containers with efficient wicks is successful in promoting expansion in Pyrex glass mortars. However, these containers and wick systems are not successful in promoting expansion with the majority of alkali-silica reactive aggregates. It appears that successful testing requires removing the wicks from containers or sealing the bars in plastic bags. To control laboratory variation, a reference aggregate should be used. In the past, Pyrex glass has been used but is clearly unsatisfactory due to the contribution of alkalies by the glass. In the future, it will be necessary to use an alkali-silica reactive aggregate that does not contain significant amounts of alkalies.

Concrete Prism Expansion Test

The concrete prism expansion results are shown in Figure 3. Storage in a sealed box over water at 38°C gave the most expansion. The prisms stored at 23°C clearly show the effects of alkali leaching on expansion. The prism stored in the humid room showed the least expansion, as well as the greatest amount of alkali leaching. Storage in a plastic bag resulted in less leaching of alkalies and more expansion. Analysis for alkalies in the prisms in the humid room and plastic bag showed 12 and 18% more alkali at their centres than in their outside layers, as expected. The prism stored in 5% sodium chloride solution showed an increase in alkalies due to penetration of sodium into the concrete, and the most expansion of all the prisms stored at 23°C. This prism had equal amounts of sodium in the outside layer and centre, but showed an increased loss of potassium from the outside, as expected. The sodium ion is very mobile and had easily penetrated the concrete. A separate analysis for chloride ions showed 35% more in the outside layer than the centre due to adsorption of the Cl by the cement. In concrete used in construction, leaching of alkalies will take place depending on the environment. Which of the expansion curves shown in Figure 3 best represents the real world? The Ministry of Transportation has built some experimental concrete sidewalks using this alkali-carbonate reactive coarse aggregate. After three years, prisms stored in 5% sodium chloride solution showed the best correlation with measured expansion of the sidewalks.

The solution to control conditions of storage of concrete prisms is the same as that recommended for the mortar bar expansion test - the use of a reference aggregate of known performance. A 110-tonne stockpile of the aggregate used in this study has been established by the Ministry of Transportation. When unknown aggregates are being tested in the concrete prism expansion test, the reference aggregate should also be tested. If the expansion with the reference aggregate is not within certain limits, the test results obtained with the unknown aggregate would be invalid or at least suspect.

CONCLUSIONS

1. Mortar bars, made with a known alkali-silica reactive aggregate and stored in containers with wicks as mandated in ASTM C 227-87, do not show significant expansion. If the mortar bars are stored in containers without wicks or are sealed in plastic bags, significant expansion takes place. The amount of expansion is significantly correlated with the amount of alkalies remaining in the mortar bars after one year.

2. Concrete prisms, made with a known alkali-carbonate reactive aggregate, give different expansion, depending on the condition of storage. The amount of expansion at 23°C is related to the amount of alkalies remaining in the prisms after 2.5 years.

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TABLE 1: Alkalies and Alkali-Silica Gel in Mortar Bars After One Year

Container Number	Type of Container	Number of Bars Tested	ALKALIES Na_2O EQUIV. % Water Soluble	ALKALIES Na_2O EQUIV. % Acid Soluble	No. of Voids with A.S.G. 10 cm^2 Surface	Figure No. in Hooton (1987)
1	New ASTM 6 Bar, Wicks	4	0.047	0.061	5	1
2	Photo Tank 4 Bar, Wicks	4	0.064	0.076	1	4
3	Old ASTM 25 Bar, Wicks	8	0.076	0.083	5	5
4	Ontario Hydro 2 Bar, Wicks	4	0.106	0.112	13	3
5	Ontario Hydro 4 Bar, No Wicks	4	0.258	0.258	22	3
6	Laval Pail No Wick	4	0.245	0.215	19	7
7	Ontario Hydro Pail Wicks	8	0.071	0.076	7	8
8	MTO 18 Bar, Wicks	10	0.081	0.094	3	6
9	MTO Plastic Bag Inside #8 With 200 ml Water	4	0.077	0.093	22	6
10	MTO Plastic Bag Inside #8 With 10 ml Water	2	0.235	0.210	24	6

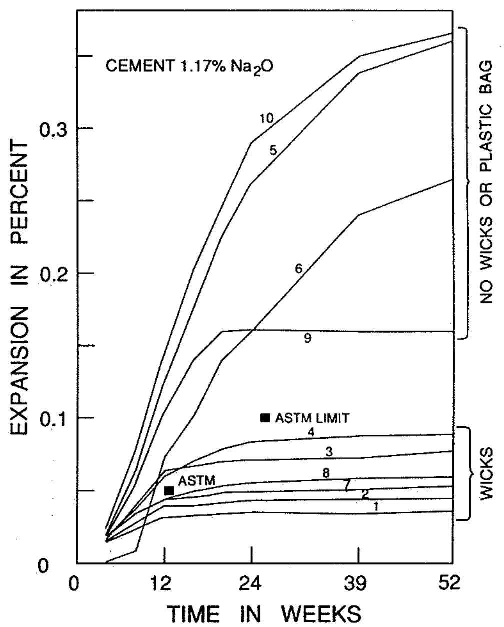

Figure 1: Expansion of mortar bars in various containers depends on presence or absence of wicks.

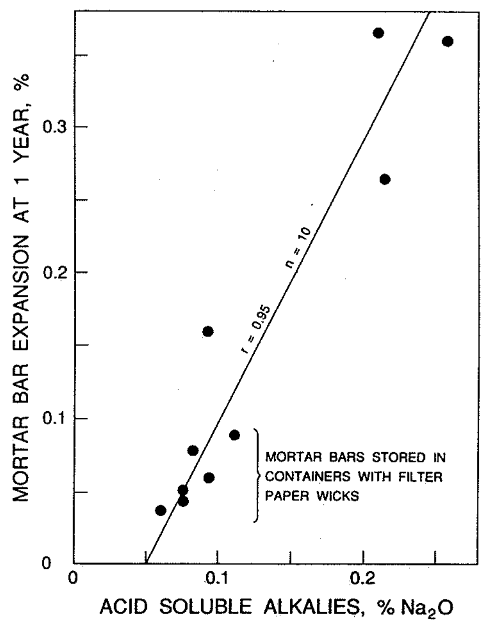

Figure 2: Relationship between mortar bar expansion and acid-soluble alkalies remaining in the bars after 1 year.

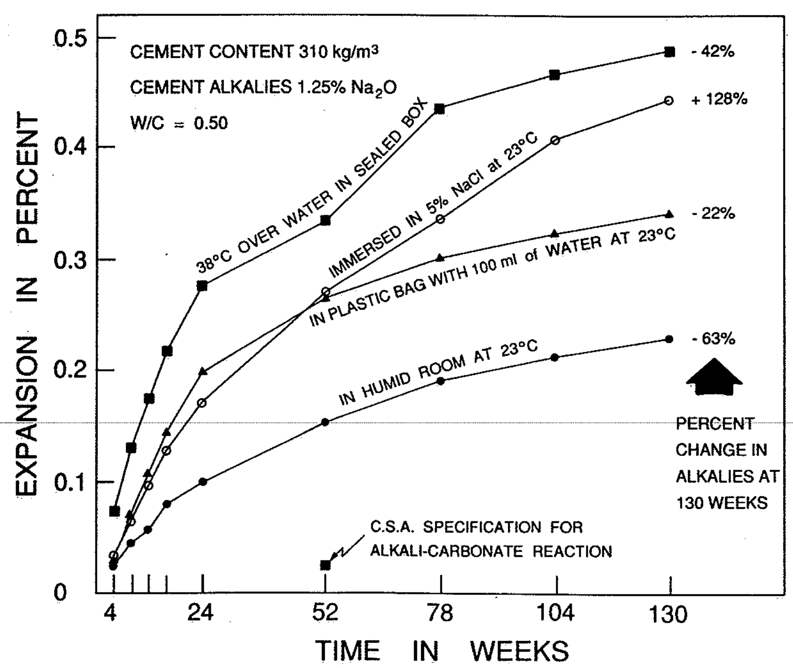

Figure 3: Relationship between concrete prism expansion, type of storage condition and water-soluble alkalies remaining in the concrete after $2\frac{1}{2}$ years.