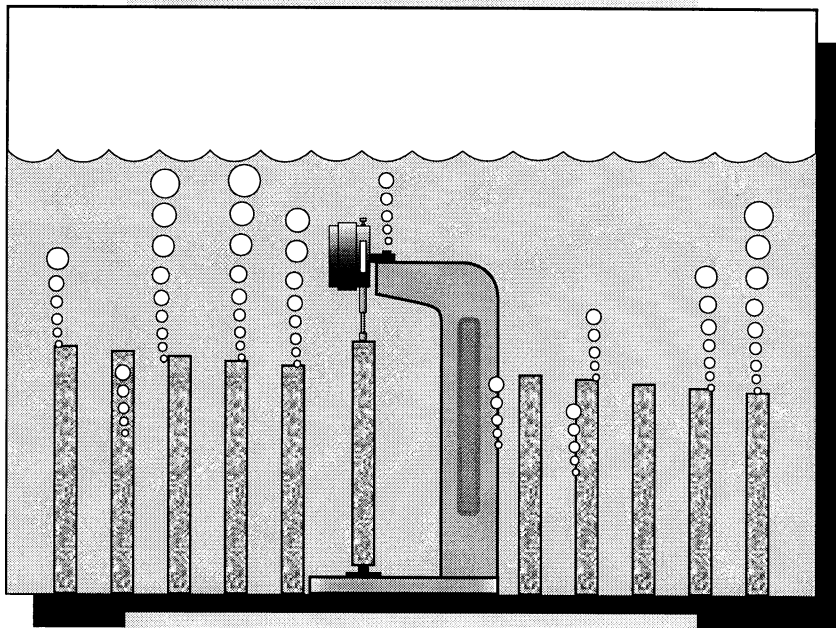FINAL REPORT

USE OF THE RAPID IMMERSION TEST TO EVALUATE THE EFFICACY OF ADMIXTURES TO MITIGATE ALKALI-SILICA REACTIVITY

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16. Abstract

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(The opinions, findings, and conclusions expressed in this report are those of the author and not necessarily those of the sponsoring agencies)

ABSTRACT

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INTRODUCTION

In implementing the products of the Strategic Highway Research Program SHRP C-343 report (Stark et al, 1993) under Concrete and Structures ASR Test and Evaluation Project 34, the Federal Highway Administration (FHWA) Office of Technology Applications (OTA) sponsored an alkali-silica reactivity (ASR) Interlaboratory Testing Study to validate results of the SHRP C-343 report. The testing program was developed by the Expert Task Group (ETG) on ASR and focused on two findings of the SHRP research:

- That the rapid immersion test for aggregate reactivity (NBRI test, ASTM P 214-90) (Oberholster and Davies, 1986) could be used to evaluate the effectiveness of mineral admixtures in preventing ASR.

- That lithium salts could mitigate expansion of concrete resulting from ASR.

This report presents the results and findings of Virginia Transportation Research Council (VTRC) participation in the ASR Interlaboratory Testing study. The results and findings presented herein are based only on the work performed by the VTRC and constitute only a portion of the FHWA-OTA study. A summary report incorporating the results and findings of all participating laboratories will be issued by the FHWA.

Background

Mineral admixtures such as fly ash and slag (ground granulated blast-furnace slag) have been shown to be effective in preventing ASR in laboratory tests when used in appropriate amounts (Pepper and Mather, 1959; Hogan and Meusel, 1981; Farbiarz et al, 1989). The traditional method used to evaluate the efficacy of mineral admixtures in preventing ASR is ASTM C 441, Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction. In C 441, mortar bars are fabricated using the cementitious materials to be tested and extremely alkali-reactive crushed Pyrex glass. Following one day of moist curing, the bars are stored over water in a container at 38°C for a period of 14 days and then measured for expansion. The 14 day test period of C 441

allows it to meet one of the critical elements always looked for in materials tests, that of rapid results. However, recent research has suggested that a test period of 56 days is needed to allow the pH of the pore solution in the bars to reach equilibrium with the alkali content of the mixture with respect to expansion (Lane and Ozyildirim, 1995).

In C441, the short test period is a function of the extreme reactivity of the pyrex glass. This has led to criticism because the behavior of Pyrex glass differs from that of natural aggregates (Stark, Morgan, Okamoto, and Diamond, 1993). Furthermore, C 441 data exhibit rather high variability, probably resulting from variations in the reactivity of the glass as well as other undetermined factors. This variability must be addressed in data analysis (Lane and Ozyildirim, 1995). On the positive side, the C 441 method permits the direct and realistic comparison of different cements and mineral admixtures because it achieves the accelerating effect without directly altering the chemistry of the cementitious system or using extreme temperatures.

The rapid immersion test was originally developed as a method to quickly identify reactive aggregates. The conditions used in this test are immersion in 1N NaOH solution maintained at 80°C, thus achieving acceleration by altering pore solution chemistry at extreme temperature. The developers investigated its potential for evaluating the ability of mineral admixtures to inhibit alkali-silica reactivity (Davies and Oberholster, 1987). They reported success, but indicated that because of the severity of the test conditions (immersion in 1N NaOH at 80°C), the test tends to underestimate the effectiveness of mineral admixtures. Consequently, they suggest that the amount of a particular admixture which is effective in the accelerated immersion test would be the maximum needed in an actual service situation. Stark et al (1993) reported that the amounts of various mineral admixtures necessary to inhibit ASR expansion of aggregates in the rapid immersion tests are in accord with general expectations for those admixtures.

While the results of these and other research programs have been promising, concern exists about this use of the rapid immersion test because of uncertainties regarding the mechanism by which expansion is suppressed. In general, these concerns center around the relative roles of alkali reduction through the pozzolanic reactions and reduced paste permeability, the primary means by which mineral admixtures are believed to suppress expansion under ordinary conditions. A recent study suggests that alkali reduction is the primary mechanism by which mineral admixtures inhibit expansion in the rapid immersion test (Berube, Duchesne, and Chouinard, 1995). Berube et al also suggest that the alkali content of the test mortar be controlled at a high level (1.25% Na_2O equivalent by mass of cementitious material) when testing mineral admixtures to negate the influence of reduced permeability on the results during the short test period.

The SHRP report (Stark et al, 1993) also generated renewed interest in the use of lithium to inhibit expansion resulting from ASR. Although much remains to be learned about the role of lithium in ASR, it appears that it incorporates into alkali-silica reaction products, dramatically

reducing the swelling potential. At present, further research is needed to develop a method suitable for determining the effective dosage of lithium to prevent ASR-related expansion.

PURPOSE AND SCOPE

The purpose of this study was to develop information on the use of the rapid immersion test for evaluating the effectiveness of fly ash and lithium hydroxide in preventing expansion caused by ASR. A total of 31 mortar batches were mixed and tested in the laboratory using a variety of material combinations and immersion solutions. This report covers only the results and findings of the VTRC.

METHODOLOGY

Mortars were mixed and three 25 x 25 x 285 mm bars were molded from each batch according to AASHTO TP14. A water-cement ratio of 0.50 was used for all batches. After molding, the bars were cured in a moist room for 24 hours, after which the three bars from each batch were demolded, placed in a storage container and covered with water. The storage containers with the specimens submerged in water were placed in an environmental chamber maintained at a temperature of 80°C for an additional 24 hour period. At the end of this period the storage containers were removed, one at a time, and the individual bars measured immediately on removal from the container with a length comparator. This measurement on Day 0 was used as the initial or zero reading on which all expansion data was based.

Immediately following the initial measurement, the bars were placed in a different storage container which already contained sufficient immersion solution at 80°C to completely cover the bars. The storage container was then returned to the environmental chamber. On Days 3, 7, and 11 the storage containers were removed, one at a time, from the environmental chamber and the bars measured for expansion. Immediately following the measurement, the bars were replaced in the storage container and the container returned to the environmental chamber. On Day 14, following the final expansion measurement, the test was ended.

Equipment

The environmental chamber was a Despatch 1600 Series model. The storage containers were Rubbermaid No. 3863-87, microwavable, 7.8 liter capacity rectangular containers measuring 120 x 235 x 350 mm with self-sealing lids. Two Plexiglas strips measuring approximately 3 x 12 x 200 mm were placed in each storage container to support the test specimens above the bottom of the container. A length comparator with a digital gage was used to measure the bars.

Materials

Cements and Mineral Admixtures

The cementitious materials used included a Type I portland cement from Kentucky and a Type IP cement from Nevada. Two mineral admixtures, a Class C and a Class F fly ash, both produced in Illinois, were also included. The chemical composition of the cements and fly ashes are given in Table 1.

Table 1. Chemical Composition of Cement and Fly Ashes

Compound	Type I cement	Type IP cement	Class C fly ash	Class F fly ash
SiO_2	20.28	30.69	33.00	48.26
Al_2O_3	5.17	4.40	18.90	25.34
Fe_2O_3	2.38	3.22	5.97	19.78
SAF (sum)	---	---	57.87	93.38
CaO	63.41	54.72	27.00	1.07
MgO	2.16	1.03	5.28	0.70
SO_3	3.4	2.55	2.60	0.10
LOI	1.76	---	0.29	0.47
Insol. Res.	0.25	---	---	---
Na_2O	0.12	0.88	1.99	0.17
K_2O	0.08	0.75	.039	2.05
Total alkali[1]	0.18	1.38	2.25	1.52
C 311 avail. alk.[1]	---	---	0.86	0.51
C_3S	53	---	---	---
C_2S	18	---	---	---
C_3A	11	---	---	---
C_4AF	7	---	---	---
Sp.G.	---	---	2.76	2.45

[1] Expressed as Na_2O equivalent ($Na_2O\% + (0.685) K_2O\%$).

Chemical Admixture

Lithium hydroxide monohydrate (LiOH·H$_2$O) was used as a chemical admixture in seven batches.

Aggregates

Three aggregates were used in these tests. A non-reactive aggregate labeled "Control" and an alkali-silica reactive aggregate labeled "Reactive" were supplied by Construction Technology Laboratories, Inc. (CTL) to each participating laboratory. The third aggregate was selected individually by each participating laboratory from their locally available sources and is labeled "Local" in this report.

The Control non-reactive aggregate was a quarried limestone from Rock Island, Illinois. It is reported to have good field performance and yield a 14 day expansion of 0.03% in the rapid immersion test (Stark, et al, 1993).

The Reactive aggregate was a natural sand and gravel from Albuquerque, New Mexico, containing volcanics ranging from rhyolite to andesite in composition. It is reported to be rapidly reactive in the field and yield a 14 day expansion of 0.87% in the rapid immersion test. The reactive constituents in this aggregate are glassy to cryptocrystalline volcanics (Stark et al, 1993).

The Local aggregate is a quarried metarhyolite (Hylas) from Rockville, Virginia. It has exhibited deleterious reactivity in a section of continuously reinforced concrete pavement constructed around 1980 as a part of I-295 north of Richmond (Lane, 1994). Examples of typical distress associated with the Hylas aggregate are shown in Figures 1 and 2. It has yielded a 14 day expansion of 0.39% in previous rapid immersion tests (ASTM P214). The reactive constituent is believed to be microcrystalline quartz (Lane, 1994).
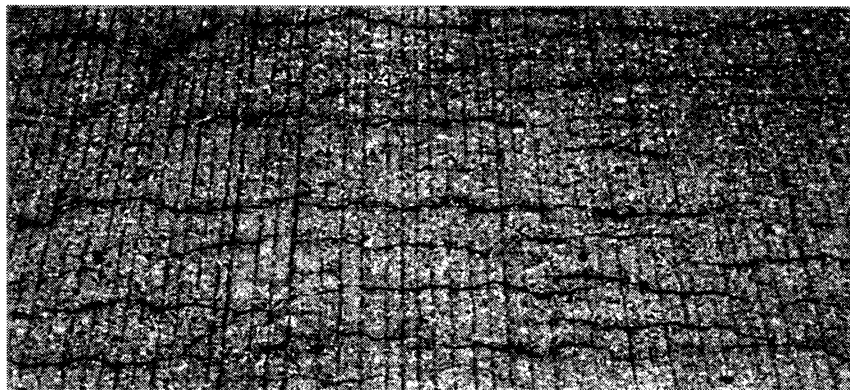

Figure 1. Pavement damage on I-295 associated with Local (Hylas) aggregate.
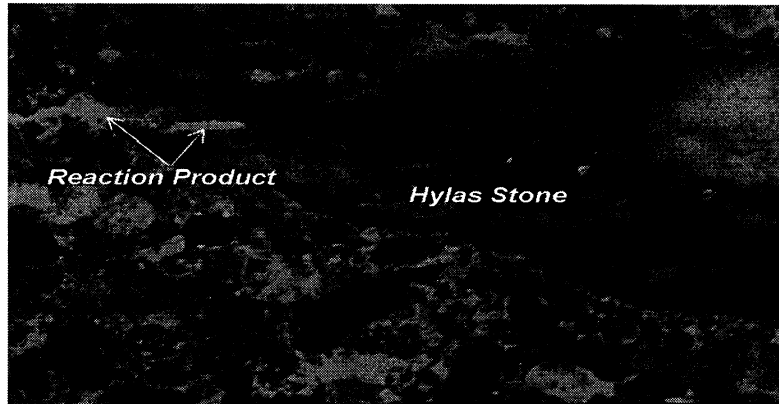
Figure 2. Polished slab from core of damaged I-295 pavement.

The Local (Hylas) aggregate has also been subjected to tests modeled after the Canadian concrete prism test (CSA-A23.2-14A), a version of which was recently adopted by ASTM as C 1293. In the tests conducted by the VTRC, 75 x 75 x 285 mm prisms were molded from air-entrained concretes with a nominal cement content of 377 kg/m^3 (the CSA and ASTM methods use a cement content of 420 kg/m^3) and a w/c of 0.45 using a Type I portland cement with an alkali content of 0.92% Na$_2$O equivalent. Sodium hydroxide (1N NaOH) was added to the mixing water in the amount necessary to raise the alkali content of the concrete to that which would have been present had the cement had an alkali content of 1.25% Na$_2$O equivalent. The concretes were produced using Hylas stone as the coarse aggregate. The coarse aggregate content was 1015 kg/m^3. The fine aggregate was a natural siliceous sand from Richmond similar to that used in the concrete pavement. This fine aggregate contains reactive chert and quartzite and has been associated with ASR-related deterioration (Lane, 1994). It yields 14 day rapid immersion test expansion of 0.19%. The fine aggregate content in the straight portland cement mixture was 684 kg/m^3. In addition to the straight portland cement concrete batch, batches were mixed in which 15%, 25%, and 35% by mass of the portland cement was replaced with a Class F fly ash (Carbo). Adjustments were made in the fine aggregate content to maintain a constant volume between mixtures. The fine aggregate contents in the fly ash concrete were 668 kg/m^3, 656 kg/m^3, and 645 kg/m^3 respectively. Table 2 gives the relevant chemical parameters of this fly ash.

After moist curing for 14 days, the prisms were measured for initial length. They were then placed over water in airtight containers which were stored at 38° C for a period of 336 days. The prisms were measured periodically for expansion and the results are shown in Table 3. Based on Canadian experience, expansion in excess of 0.04% at one year are considered excessive. Damage associated with the high expansion of the straight portland cement concrete batch is shown in Figure 3.

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Table 2. Oxide Analysis of Carbo Fly Ash

Oxide	
SiO_2	54.40%
Al_2O_3	25.14%
Fe_2O_3	7.73%
SAF	87.27%
CaO	5.61%
Na_2O equivalent (total alkali)	2.49

Table 3. Expansion of Concrete Prisms Made with Local
Aggregate (Hylas Stone) and Carbo Class F Fly Ash

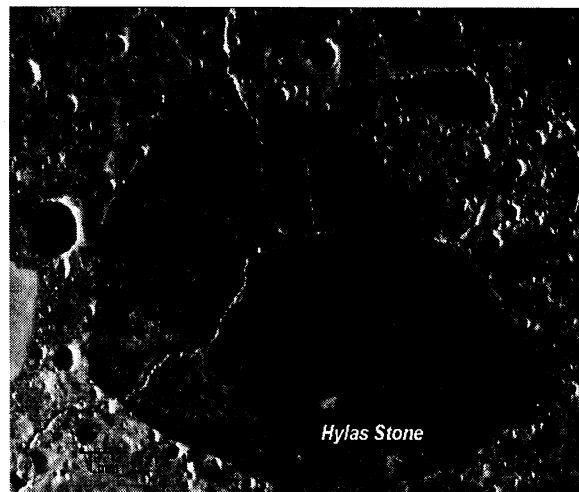|	% Expansion @ Age (days)		
Concrete	84	168	336
100% Portland	0.02	0.06	0.17
15% Class F	0.00	0.01	0.02
25% Class F	-0.01	0.00	0.01
35% Class F	0.00	0.00	0.01

Figure 3. Damage associated with high expansion of straight portland cement concrete containing Local (Hylas) aggregate.

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Mortar Batches

Mortars were mixed according to AASHTO TP14 using 990 g of aggregate graded as indicated in the standard. The total mass of cementitious material (portland cement plus mineral admixture) in each batch was 440 g, with a constant water-cement ratio of 0.50.

Mineral Admixtures

A total of 16 batches, eight with the Class C fly ash and eight with the Class F fly ash were mixed. In each case the mineral admixture was used to replace 20% of the portland cement by mass.

Chemical Admixture

Seven batches contained $LiOH\cdot H_2O$ as a chemical admixture. The admixture dosage was based on the chemistry of the immersion solution through a relationship which exists between immersion solution normality (concentration) and the expected OH^- concentration (pH) of the pore solution in concrete (Stark et al, 1993). This relationship is dependent on the alkali content of the cement and w/c. A 1N NaOH solution has a pH equivalent to the pore solution of a concrete made with portland cement with 1.4% Na_2O equivalent alkali content and a w/c of 0.50. Since the mortar batches contained 440 g of cement, 6.16 g (440g x 0.014) $LiOH\cdot H_2O$ was added. The $LiOH\cdot H_2O$ addition was accomplished by dissolving it in 214 ml of mixing water to achieve a mass of 220 g of mixing water plus admixture for these batches. Consequently, the batches containing the $LiOH\cdot H_2O$ had a slightly lower water content than the other batches.

Immersion Solutions

Three different immersion solutions were used in these tests. The standard immersion solution, 1N NaOH, was used with 19 of the batches including all control batches. The two experimental solutions both contained $LiOH\cdot H_2O$ in addition to NaOH and were used with six batches each. One experimental solution was 1N, the other 0.6N. Both solutions were mixed to maintain a Na:Li of 1:0.667. The formulation of each immersion solution is shown in Table 4. The storage container for each batch was filled with approximately 2250 ml of immersion solution.

Table 4. Chemical Formulation of Immersion Solutions

Solution	NaOH per liter	LiOH·H₂O per liter
Standard 1N NaOH	40.00g (1 mole x 40g/mole)	None
Experimental 1N(Na,0.667Li)OH	24.00g (0.6 moles x 40g/mole)	16.80g (0.4 moles x 42g/mole
Experimental 0.6N (Na,0.667Li)OH	14.40g (0.36 moles x 40g/mole)	10.08g 0.24 moles x 42g/mole)

Table 5 presents an outline of the material combinations used in each batch. A total of 31 batches were mixed on four separate days. A control batch containing the non-reactive aggregate was included in each day's mixing and is identified by mix group. In order to evaluate the potential for Lithium to leach from the bars, samples of the soaking water after the initial 24 h soaking period and samples of the immersion solution at days 0 and 14 were taken and sent to CTL for analysis. The findings of these analyses are not included in this report.

Table 5. Tabulation of Batch Mixtures and Immersion Solutions

Mix Group	Batch	Cement	Aggregate	Mineral Admixture	Chemical Admixture	Immersion Solution
A	1	Type I	Control	---	---	1N NaOH
A	2	Type I	Reactive	---	---	1N NaOH
A	3	Type I	Local	---	---	1N NaOH
A	4	Type I	Reactive	Class C 20%	---	1N NaOH
A	5	Type I	Local	Class C 20%	---	1N NaOH
B	6	Type I	Reactive	---	LiOH·H₂O	1N NaOH
B	7	Type I	Local	none	LiOH·H₂O	1N NaOH
D	8	Type I	Reactive	---	---	Exp 0.6N(Na,Li)OH
C	9	Type I	Reactive	---	---	Exp 1N(Na,Li)OH
D	10	Type I	Local	---	---	Exp 0.6N(Na,Li)OH
						Cont'd

Table 5 (Cont'd)

Mix Group	Batch	Cement	Aggregate	Mineral Admixture	Chemical Admixture	Immersion Solution
C	11	Type I	Local	---	---	Exp 1N(Na,Li)OH
A	12	Type I	Reactive	Class F 20%	---	1N NaOH
A	13	Type I	Local	Class F 20%	---	1N NaOH
B	14	Type I	Reactive	Class C 20%	LiOH·H$_2$O	1N NaOH
B	15	Type I	Local	Class C 20%	LiOH·H$_2$O	1N NaOH
B	16	Type I	Reactive	Class F 20%	LiOH·H$_2$O	1N NaOH
B	17	Type I	Local	Class F 20%	LiOH·H$_2$O	1N NaOH
D	18	Type I	Reactive	Class C 20%	---	Exp 0.6N(Na,Li)OH
C	19	Type I	Reactive	Class C 20%	---	Exp 1N(Na,Li)OH
D	20	Type I	Local	Class C 20%	---	Exp 0.6N(Na,Li)OH
C	21	Type I	Local	Class C 20%	---	Exp 1N(Na,Li)OH
D	22	Type I	Reactive	Class F 20%	---	Exp 0.6N(Na,Li)OH
C	23	Type I	Reactive	Class F 20%	---	Exp 1N(Na,Li)OH
D	24	Type I	Local	Class F 20%	---	Exp 0.6N(Na,Li)OH
C	25	Type I	Local	Class F 20%	---	Exp 1N(Na,Li)OH
B	26	Type I	Control	---	LiOH·H$_2$O	1N NaOH
C	27	Type IP	Reactive	---	---	1N NaOH
D	28	Type IP	Local	---	---	1N NaOH
B	1B	Type I	Control	---	---	1N NaOH
C	1C	Type I	Control	---	---	1N NaOH
D	1D	Type I	Control	---	---	1N NaOH

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RESULTS AND DISCUSSION

The primary mechanism by which ASR is activated in the rapid immersion test is through the chemistry (pH) of the immersion solution. In these tests, immersion solutions with three different chemical formulations were used: the standard 1N NaOH solution, which has demonstrated effectiveness in activating ASR; and two experimental solutions which contained $LiOH \cdot H_2O$ in addition to NaOH at a molar ratio of 1:0.667, Na:Li. The 1:0.667, Na:Li ratio was selected because earlier work suggested it was the minimum Na:Li ratio at which Li was effective in suppressing ASR expansion (Stark et al, 1993).

The experimental solutions were made at two concentrations, 0.6N and 1N. At a w/c of 0.50, a concrete produced with a cement having an alkali content of 0.8% Na_2O equivalent would have a pore solution concentration of 0.6N, while a 1.4% Na_2O equivalent cement would produce a pore solution with a 1N concentration (Stark et al, 1993).

The results of the rapid immersion tests are presented in Table 6. They are grouped according to immersion solution type. Although various expansion maxima have been suggested as a criterion for innocuous behavior (CAN/CSA 23.1 Appendix B; ASTM C 1260; Stark et al, 1993; Lane, 1994), 0.08% expansion at 14 days as suggested by Stark et al will be used for purposes of discussion.

Standard Immersion Solution

A total of 19 batches were tested in the standard 1N NaOH immersion solution. Four batches containing the Control aggregate without admixtures were tested, one for each group of batches. These control batches all yielded 14 day expansions less than 0.02%. When tested without admixtures, the Reactive aggregate yielded a 14 day expansion of 0.66% compared to 0.87% reported in the SHRP study (Stark et al, 1993). The Local aggregate yielded an expansion of 0.61%, although earlier tests at VTRC on this aggregate produced an expansion of 0.39% (Lane, 1994). The expansions of the Reactive and Local aggregates are well in excess of the 0.08% limit.

Both the Reactive aggregate and the Local aggregate were tested individually with the Class C fly ash, the Class F fly ash, the IP cement, and the $LiOH \cdot H_2O$ admixture. Each aggregate was also tested with a combination of the Class C fly ash and the $LiOH \cdot H_2O$ admixture as well as the Class F fly ash and the $LiOH \cdot H_2O$ admixture. The Control aggregate was also tested with the $LiOH \cdot H_2O$ admixture.

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Table 6. Results of Rapid Immersion Tests

Batch	Agg[1]	Admixtures FA[2]	LiOH[3]	3 Day	Expansion (Percent) 7 Day	11 Day	14 Day
1N NaOH Immersion Solution							
2	R	--	---	0.1603	0.4903	0.6020	0.6633
6	R	--	Y	0.0177	0.1010	0.2020	0.2523
4	R	C	---	0.1823	0.3677	0.4227	0.4473
14	R	C	Y	0.0427	0.0940	0.1380	0.1667
12	R	F	---	0.0147	0.0393	0.0900	0.1180
16	R	F	Y	0.0800	0.0230	0.0310	0.0365
27	R	IP	---	0.0550	0.1633	0.2263	0.2663
7	L	--	Y	0.0067	0.1713	0.3230	0.3917
3	L	--	---	0.0327	0.2723	0.4830	0.6093
5	L	C	---	0.0583	0.2490	0.3673	0.4343
15	L	C	Y	0.0647	0.1830	0.2667	0.3090
17	L	F	Y	0.0087	0.0233	0.0347	0.0503
13	L	F	---	0.0043	0.0253	0.0567	0.0793
28	L	IP	---	-0.0020	0.0053	0.0157	0.0323
26	C	--	Y	0.0063	0.0053	0.0070	0.0093
1	C	--	---	0.0103	0.0067	0.0147	0.0180
C2	C	--	---	0.0007	0.0027	0.0023	0.0027
C3	C	--	---	-0.0007	0.0010	0.0007	0.0067
C4	C	--	---	-0.0013	-0.0000	0.0020	0.0057
0.6N (Na+,0.667Li+)OH Immersion Solution							
8	R	--	---	0.0087	0.0030	0.0080	0.0097
10	L	--	---	0.0093	0.0077	0.0107	0.0130
18	R	C	---	0.0050	0.0050	0.0063	0.0100
20	L	C	---	0.0077	0.0063	0.0097	0.0127
22	R	F	---	0.0063	0.0043	0.0060	0.0063
24	L	F	---	0.0123	0.0107	0.0127	0.0157
C4*	C	--	---	-0.0013	-0.0000	0.0020	0.0057
1N (Na+,0.667Li+)OH Immersion Solution							
9	R	--	---	0.0063	0.0090	0.0117	0.0100
11	L	--	---	0.0123	0.0167	0.0183	0.0210
19	R	C	---	0.0047	0.0117	0.0123	0.0173
21	L	C	---	0.0050	0.0137	0.0167	0.0313
23	R	F	---	0.0025	0.0080	0.0035	0.0095
25	L	F	---	0.0083	0.0117	0.0097	0.0103
C3*	C	--	---	-0.0007	0.0010	0.0007	0.0067

[1]Aggregate: R=Reactive; L=Local; C=Control
[2]Fly Ash used in batch; C=Class C; F=Class F; IP=Blended cement used in batch
[3]LiOH used as admixture in batch
* Control batch, tested in standard 1N NaOH immersion solution.

Class C Fly Ash

Expansions of the bars containing the Reactive and the Local aggregate with the Class C fly ash were 0.45% and 0.43% respectively at 14 days. These expansions are lower than were achieved with the straight portland cement mixture, but still well in excess of the 0.08% limit.

In the SHRP research (Stark et al, 1993), this Class C fly ash was tested in the rapid immersion test with three different aggregates (including the Reactive aggregate used in these tests) at replacements of 30 to 60%. Although progressively higher replacements resulted in a reduction in expansion with each aggregate, in all cases expansion remained in excess of the 0.08% limit.

Berube et al (1995) tested a Class C fly ash at a 40% replacement in the rapid immersion test, and expansion was roughly the same as their control mixture at 14 days (~0.35%). They extended their test through 48 days and the Class C fly ash did provide some reduction at later ages although expansion was still excessive.

Kakodkar et al (1994) reported testing five different Class C fly ashes in the rapid immersion test (ASTM P 214) using various replacement levels of 10 to 30%. In their tests, four of the five fly ashes progressively reduced expansion of the reactive aggregate as the fly ash percentage was increased. Satisfactory reduction was achieved with one of the fly ashes at the 25% replacement level and two of the others at 30%. Expansion with the fourth fly ash only slightly exceeded 0.08%. With the fifth fly ash, expansion at all replacement levels exceeded the expansion of the control batch.

Other researchers (Carrasquillo and Snow, 1987; Farbiarz et al, 1989) tested Class C fly ashes under ASTM C 227 conditions (mortar bars over water at 38°C), and reported findings similar to those of Kakodkar et al: that at replacement levels of 30% or more, some Class C fly ashes can be effective in suppressing expansion related to ASR in laboratory tests.

Class F Fly Ash

Rapid immersion test expansions of the Reactive and the Local aggregate with the Class F fly ash at 14 days were 0.12% and 0.08% respectively. These expansion are significantly lower than those obtained without the fly ash. The Local aggregate meets the 0.08% expansion maximum, indicating 20% replacement using this fly ash (48.26% SiO_2, 93.38% SAF) will effectively suppress ASR expansion. These results compare favorably with the results of concrete prism tests on this aggregate using a different Class F fly ash (54.40% SiO_2, 87.23 SAF). In those tests, a 15% replacement was effective in concretes with an alkali content of 1.25% Na_2O equivalent by mass of cement. The rapid immersion test with 1N NaOH simulates the pore solution of a 0.50 w/c concrete with 1.4% Na_2O equivalent by mass of cement.

In the SHRP study (Stark et al, 1993), the Reactive aggregate was tested with two different Class F fly ashes. One had a SAF equal to 86.00% and the other 83.95%, while the SAF for the Class F fly ash in this study was 93.38%. In the SHRP study, three replacement levels were used: 10%, 20%, and 30%. With both fly ashes, increasing replacement levels resulted in decreasing expansions. The fly ash with 86.00% SAF (52.4% SiO_2) held expansion below 0.08% at 20% and 30% replacement levels. The fly ash with 83.95% SAF (48.71 SiO_2) was only effective at the 30% replacement level with the Reactive aggregate. The results in this study with the 93.38% SAF (48.26 SiO_2) fly ash were similar to those of the SHRP tests with the 83.95% SAF fly ash. Here, expansion with the Reactive aggregate exceeded the 0.08% limit with 20% replacement, and it seems likely that satisfactory suppression of expansion would be achieved at slightly higher replacements.

In comparing the results obtained with the two Class F fly ashes in the SHRP study with the result obtained in this study, the effectiveness of a fly ash in preventing ASR expansion seems more related to simple SiO_2 content than SAF.

These rapid immersion test results with the Class F fly ash, taken at face value, can be interpreted to suggest that 20 to 30% replacement of cement with similar fly ashes would be effective in preventing excessive expansion of highly reactive aggregates using portland cements having total alkali contents up to 1.4% Na_2O equivalent. This contrasts with the results of C 441-type tests which suggested that >35% replacements with Class F fly ashes would be needed to adequately suppress expansion with cements having alkali contents in excess of 0.9% Na_2O equivalent (Lane and Ozyildirim, 1995). This raises certain issues for consideration.

In this study, the alkali content of the pore solution in the test specimens was initially quite low (the alkali content of the portland cement used was 0.18% Na_2O equivalent). Diffusion of alkalis from the immersion solution into the bars is necessary to raise the alkali content to that assumed from the normality of the immersion solution. Because pozzolanic reactions reduce capillary pore space and disrupt their continuity, diffusion rates are reduced. Consequently, it is uncertain what equivalent alkalinity the pore solution in the test specimen attained during the test period. For this reason, Berube et al (1995) suggested that the mortar be made with an initially high (1.25% Na_2O equivalent) alkali content when testing mineral admixtures.

With regard to the C 441 type tests, the point can be made that the high replacement levels suggested are a function of the extreme reactivity of the pyrex glass. Thus, they are overestimates of the amount of fly ash necessary to adequately suppress expansion. However, in the tests described by Lane and Ozyildirim (1995), the test specimens had ceased expanding and thus evaluations were based on a system which was stable with respect to reactivity. Consequently, although perhaps conservative, they can be viewed with a large measure of confidence. In the immersion tests conducted in this study, evaluations are made at a point when expansion and reactivity are continuing. Because the system is still in a dynamic state, the establishment of the test period and expansion limits is critical and should be carefully considered.

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Blended Cement

The IP cement was tested with the Reactive and Local aggregates. With the Reactive aggregate, an expansion of 0.27% was obtained compared to the aggregate's 0.66% expansion without mineral admixture. When used with the Local aggregate, the IP cement reduced expansion from 0.61% to 0.03%, well below the 0.08% criterion for effectiveness. The difference in effectiveness with the Reactive and Local aggregates may be related to differences in the reactive constituents in the two aggregates, which were glassy to cryptocrystalline volcanics and microcrystalline quartz, respectively; or may be related to some aspect of the specific pozzolan used in the blended cement.

LiOH·H$_2$O Admixture

LiOH·H$_2$O was added as an admixture to mortars made with the Control, Reactive, and Local aggregate and also to mortars containing the Reactive and Local aggregates with both Class C and Class F fly ashes. In mortars without mineral admixture, the LiOH·H$_2$O had no effect on the expansion with the control aggregate.

With both the Reactive and Local aggregates, with and without Class C and Class F fly ash, expansions were substantially reduced. With both aggregates, when tested without mineral admixture or with the Class C fly ash, expansion still remained well in excess of 0.08%. When combined with the Class F fly ash, expansion of both aggregates remained below 0.08%. However, the LiOH·H$_2$O was clearly more effective with the Reactive aggregate, reducing expansion 62% without mineral admixture, 62% with Class C fly ash and 67% with Class F fly ash, while with the Local aggregate, expansions were only reduced 36%, 28% and 38% respectively. The difference in effectiveness of LiOH·H$_2$O with these two aggregates may reflect differences in the reactive constituents in the aggregates and/or the reaction products formed from them. The reactive constituents in the Reactive aggregate are volcanic glasses, while that of the Local aggregate is microcrystalline quartz.

In evaluating the results obtained when the LiOH·H$_2$O was used as an admixture, recall that although the dosage rate was based on an assumed pore solution alkali content of 1.4% Na$_2$O equivalent to provide a 1:1 molar ratio Na$^+$:Li$^+$, the actual dosage was computed from the mass of cement batched into the mortar. However, the volume of immersion solution used in these tests, nominally four times the volume of the bars tested, presents a relatively limitless supply of Na$^+$ relative to Li$^+$.

Experimental Immersion Solutions

Six duplicate batches, three each with the Reactive and Local aggregates, were tested with the experimental immersion solutions (0.6N and 1N, (Na$^+$,0.667Li$^+$)OH). With both

immersion solutions, each aggregate was tested without mineral admixture, with 20% Class C fly ash, and with 20% Class F fly ash. In all of these cases, 14 day expansions were 0.03% or less. For comparison, expansions of the Reactive and Local aggregates tested in the standard immersion solution (1N NaOH) were 0.66% and 0.61% respectively (batches 2 and 3). However, it should be considered that the effective NaOH normalities of the 0.6N and 1N $(Na^+,0.667Li^+)OH$ solutions are 0.36 and 0.6 respectively. The SHRP study showed that reducing the normality of NaOH immersion solutions resulted in reductions in expansion (Stark et al, 1993) and suggested an alternate expansion limit for NaOH solutions below 1N. For solutions up to 0.6N NaOH the suggested limit is 0.02% expansion. The Local aggregate expansion exceeded 0.02% in the 1N $(Na^+,0.667Li^+)OH$ solution in mortars without mineral admixture and with the Class C fly ash.

The manner in which $LiOH·H_2O$ admixtures are proposed to be used serves to increase the pH of the pore solution of the concrete above that which would have resulted without the $LiOH·H_2O$. Consequently, it appears that a more appropriate comparison for these batches would be to add the $LiOH·H_2O$ to a 1N NaOH solution, or to test the aggregates in 0.36N and 0.6N NaOH solutions for control purposes.

Batches made with the Control aggregate were tested at the same time as the experimental immersion solution batches but in the standard (1N NaOH) immersion solution. In each case the expansion of the Control aggregate batch was lower than any of the experimental batches, but only slightly so. In general, expansion of bars tested in the 0.6N solution was slightly lower than for the duplicate bars tested in the 1N solution. With both experimental solutions, expansion of bars made with the Local aggregate was slightly higher than expansion of the equivalent (cementitious materials) Reactive aggregate bars. When tested in the standard 1N NaOH solution, bars with Reactive aggregate yielded higher expansion than Local aggregate bars. This difference in response of the two aggregates on exposure to $LiOH·H_2O$ is similar, although not as pronounced, to that which occurred when it was used in the bars as an admixture.

CONCLUSIONS

These conclusions are based on the results obtained by the VTRC during participation in an interlaboratory testing program. As such, they are provided to help focus attention and discussion during the analysis of the complete set of data from all participating laboratories.

1. Rapid immersion tests of the Control, Reactive, and Local aggregates using the standard 1N NaOH solution yielded expansions which agreed with expectations based on reported field performance and previous laboratory tests.

2. Expansion of the Reactive and Local aggregates in 1N NaOH was reduced when mortars were mixed with 20% replacement of cement with a Class C fly ash but remained well in excess of the 0.08% limit indicating effective suppression. However, Kakodkar et al (1994) reported that

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some Class C fly ashes were effective in suppressing expansion in rapid immersion tests when used at replacement levels of 25% or 30%, depending on the oxide chemistry.

3. Expansion of the Reactive and Local aggregates in 1N NaOH was significantly reduced when mortars were mixed with 20% replacement of cement with a Class F fly ash. Expansion of the Reactive aggregate remained slightly above the 0.08% expansion limit, but increasing the fly ash amount would likely provide effective suppression. The 20% replacement provided effective suppression with the Local aggregate, and these results agreed with the results of concrete prism tests (over water at 38°C) containing a different Class F fly ash. However, because expansion of test specimens and consequently reactivity continued throughout the 14 day test period, the establishment of suitable expansion limits for any given test period must be considered carefully and critically.

4. Because pozzolanic reactions reduce the permeability of mortars and concrete, it cannot be assumed that the alkalinity of the pore solution of test specimens containing mineral admixtures reaches equilibrium with that of the immersion solution during the 14 day test period.

5. Tests conducted using an IP cement with the Reactive and Local aggregates provided contrasting results. Although effective suppression was obtained with the Local aggregate (0.03%), expansion of the Reactive aggregate (0.27%) greatly exceeded the 0.08% limit. This result is puzzling because of similarities in results for the two aggregates when tested with both Class C and Class F fly ashes. A possible explanation lies in differences in the reactive constituents in the two aggregates or the pozzolan used in the IP cement.

6. The addition of $LiOH \cdot H_2O$ to mortars containing the Reactive and Local aggregates reduced the expansion of bars stored in the 1N NaOH but expansion still remained well above 0.08%. The $LiOH \cdot H_2O$ was much more effective in suppressing expansion of the Reactive aggregate than the Local aggregate. Differences between the reactive constituents in the two aggregates, or the reaction products formed from them may provide an explanation. Expansion of the Control (non-reactive) aggregate was not affected by the $LiOH \cdot H_2O$.

7. The addition of $LiOH \cdot H_2O$ reduced the expansion of bars containing Class C and Class F fly ash with both the Reactive and the Local aggregate. Effective suppression (<0.08%) was achieved with both aggregates and the Class F fly ash while the Class C mixtures remained well in excess of the limit.

8. The ineffectiveness of the admixed $LiOH \cdot H_2O$ may result from an imbalance of $Na^+:Li^+$ due to the large volume of immersion solution relative to the amount of admixed $LiOH \cdot H_2O$.

9. When $LiOH \cdot H_2O$ was used in 0.6N and 1N immersion solutions at a molar ratio of 1:0.667 $Na^+:Li^+$, minimal expansions of the Reactive and Local aggregates were achieved with and without mineral admixtures. These solutions are effectively equivalent to 0.36N and 0.6N NaOH solutions based on Na^+ content, and lower expansion would be expected in such solutions

relative to a 1N NaOH solution. Using the SHRP recommended limit of 0.02% expansion for testing in reduced normality solutions, excessive expansions were exhibited by the Local aggregate when tested without mineral admixture and with Class C fly ash in the 1N $(Na^+, 0.667Li^+)OH$ solution.

10. The $LiOH \cdot H_2O$ was more effective in suppressing the expansion of the Reactive aggregate containing glassy and cryptocrystalline volcanics than the Local aggregate, which contained microcrystalline quartz as the reactive constituent. This differential effectiveness was noted both when the $LiOH \cdot H_2O$ was used as an admixture in the mortar and when it composed part of the immersion solution.

RECOMMENDATIONS

1. Alternative methods such as ASTM C 1293 should be considered to evaluate the effectiveness of lithium salts in suppressing expansion resulting from ASR.

2. The Virginia Department of Transportation (VDOT) should continue its current policies regarding the use of Class F fly ash, slag, silica fume, and other pozzolans to inhibit the potential effects of alkali-silica reactivity.

3. The VTRC should conduct further laboratory research into the effects of lithium salts on concrete properties and the development of a test method suitable for evaluating lithium dosage rates. Field trials of lithium-bearing admixtures should be considered.

4. The VTRC should conduct further research of test methods for evaluating the effectiveness of pozzolans and slag to mitigate ASR.

ACKNOWLEDGMENTS

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