Evaluating ASR Mitigation Potential of Supplementary Cementing Materials and Lithium Admixture in the Presence of Potassium Acetate Deicer – Revised EB-70 Test Method

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ABSTRACT

Recently a deicer-modified mortar bar test method, revised EB-70 test method, was developed to evaluate aggregate reactivity in the presence of potassium acetate deicer. This test employs a soak solution with a composition of 3M KAc and 1N NaOH, wherein the mortar bars with the suspect aggregates are exposed over a period of 28 days, and the mortar bar expansion is periodically recorded. Previous research has shown that this test captures the interactions that occur between a concentrated deicer solution and a highly alkaline environment within the pore solution of concrete. Results from investigation of over 30 aggregates in this test method yielded positive correlation with the field performance of the aggregate. In this paper the applicability of this test method to evaluate effectiveness of typical ASR mitigation measures such as fly ashes, slag and lithium admixtures, was investigated. Findings from these studies suggest that the revised EB-70 test method can be employed to evaluate the effectiveness of ASR mitigation measures in the presence of potassium acetate deicer. Factors such as the chemical composition of the SCM and its dosage rate in the mixture appear to play a significant role in effectively mitigating ASR in the presence of deicers.

1. INTRODUCTION

Recent laboratory investigations on the impact of airfield deicing chemicals on concrete durability have shown that deicers such as potassium acetate (KAc) are capable of inducing deleterious alkali-silica reaction (ASR) in concrete (1-3). Evidence from forensic field investigations suggested that while the penetration of potassium acetate into sound concrete was minimal (only to an extent of 10-15 mm from the pavement surface), its penetration into concrete with pre-existing cracks was found to be significant, particularly along the length of the crack (4-5). It is therefore conceivable that when deicers do penetrate concrete, its potential to inflict ASR distress can be significant. In order to investigate the susceptibility of aggregates to under ASR in the presence of potassium acetate deicer, a deicer-modified mortar bar test method (EB-70 test method and revised EB-70 test method) was recently developed (1,6-9). A detailed description of this test method and its comparison with the standard ASTM C 1260 test method is presented elsewhere (7). The principal difference between the deicer-modified test and the standard ASTM C1260 test is the composition of the soak solution employed in the test method. In the deicer-modified test method, a 3M KAc + 1N NaOH solution is employed as soak solution instead of the standard 1N NaOH solution. The reason for using the 3M KAc + 1N NaOH soak solution is to capture the pH jump that is observed in deicer solutions when blended with alkali hydroxide solutions. It was shown that the increase in the pH observed in the proposed soak solution (i.e. 3M KAc + 1N NaOH) was not just based on the concentration of the hydroxyl ions, but due to an increase in the activity coefficient of the hydroxyl ions (7, 10). It should be noted that in a previous version of the deicer-modified test method (EB-70 test method), where 6.4 M KAc was employed as a soak solution, the high pH observed in the soak solution was entirely
due to interaction of the KAc deicer with Ca(OH)$_2$ (from the hydration of portland cement) \((10,11)\). The use of 6.4M KAc deicer soak solution in the test method resulted in a high pH of the soak solution (owing to the increase in the activity of the hydroxyl ions contributed by Ca(OH)$_2$) however, owing to the limited solubility of Ca(OH)$_2$ in water, the concentration of hydroxyl ions was low.

The proposed soak solution 3M KAc + 1N NaOH not only provides for a high pH resulting from the increased activity coefficient of hydroxyl ions, but also maintains a high enough hydroxyl ion concentration over the course of the test method of 28 days. The details of the chemistry involved in the interactions between the deicer and alkaline solutions are presented elsewhere \((10)\). The revised EB-70 test method was validated by conducting tests on 32 aggregates of various lithologies and of known reactivity and comparing the results with those obtained from the standard ASTM C 1260 tests. Figure 1 shows the 14-day mortar bar expansion of aggregates in the standard ASTM C 1260 and revised EB-70 test method. Findings from this study showed that the deicer-modified mortar bar test method was not only able to identify aggregates that were alkali-silica reactive in nature, with similar accuracy as the standard ASTM C 1260 test method, but also was able to screen aggregates that were sensitive to KAc deicer compared to 1N NaOH alone.

As an extension in the development of the revised EB-70 test method, in this paper the effectiveness of typical supplementary cementing materials (SCMs) such as Class F fly ash, Class C fly ash, slag and lithium admixtures, in mitigating ASR was evaluated in the presence of KAc deicer using the deicer-modified mortar bar test method. The performance of these SCMs and lithium admixture in the deicer-modified mortar bar test is compared with the results from the standard ASTM C 1567 test method. In this investigation, six fly ashes of different chemical composition, one slag, and a 30% solution of lithium nitrate were evaluated in combination with twenty aggregates of different levels of reactivity.

2. OBJECTIVES

The principal objectives of this research study were:

1. To evaluate the effectiveness of selected SCMs and lithium admixture in mitigating ASR in the presence of deicing chemicals in the revised EB-70 test method.
2. Compare the performance of the SCMs and lithium admixture in the revised EB-70 test method with their performance in the standard ASTM C 1567 test method.
3. Determine the influence of chemical composition of fly ashes on their ability to mitigate ASR in the revised EB-70 test method and the standard ASTM C 1567 test method.
3. EXPERIMENTAL PROGRAM

3.1. Materials

3.1.1. Aggregates

Table 1 shows the aggregates used in this study that have a known historical performance. The field performance of the aggregates was based on assessment of several highway and airfield pavement structures by the authors and/or by respective DOT personnel from where a specific aggregate was obtained.

3.1.2. Cement

High alkali cement (Type I) with a Na₂O equivalent of 0.82% (Na₂Oeq) and an autoclave expansion of 0.12% was used for this study. The chemical composition of this cement is provided in Table 2.

3.1.3. Fly Ash

In this study, six different fly ashes were used as supplementary cementitious material (SCM). The chemical composition of the fly ashes is provided in Table 2. In this study, all fly ashes were used at a dosage of 25% by mass replacement of cement.

3.1.3. Slag

A grade 120 ground granulated blast furnace slag (GGBFS) was used as a supplementary cementitious material (SCM) at a dosage of 40% by mass replacement of cement, in this study. The chemical composition of the slag is provided in Table 2.

3.1.4. Lithium Nitrate

The lithium nitrate (LiNO₃) used in this study was a 30% wt. solution in water. The properties as described by the manufacturer are as follows: density @25°C (77°F) is 1.20 g/cm³ (10.0 lb/gal), pH (1:6 dilution) at 25°C ranging from 7 – 10, freezing point (incipient crystallization) -8°C (18°F), boiling point 110°C (230°F). In this study, lithium admixture was evaluated at a single dosage of 100%, as represented by the amount of LiNO₃ needed to achieve a Li/Na molar ratio of 0.74 in the mortar bar, where the Na content is based only on the cement alkali content. Only a 50% dosage (i.e. a Li/Na molar ratio of 0.37) was employed in all the soak solutions. In revised EB-70 test method, the contribution of 3KAc to the alkalinity of the soak solution was minimal in comparison to the 1N NaOH, and therefore the lithium dosage in soak solution was also based on 1N NaOH.
3.1.5. Deicers and Reagents

In this study, Cryotech E-36, a commercial grade runway liquid deicer was used as the soak solution. This deicer is a 50% wt. solution of KAc (~6.4 molar concentration) with a pH of 10.85 at room temperature and a density of approximately 1.25-1.30 g/cc. The deicer contains a proprietary organic corrosion inhibitor and a dyeing agent. The deicer solution contains less than 200 ppm of sulfate as impurities. In addition, reagent grade NaOH pellets were used to prepare the 1N NaOH soak solutions for conducting the standard ASTM C 1260 and C 1567 tests. Soak solutions for all of the revised EB-70 test method (1N NaOH + 3M KAc) were prepared by using combination of reagent grade NaOH and commercial grade KAc deicer.

3.2 Test Methods

3.2.1. Standard ASTM C 1260 (1N NaOH Soak Solution)

In this test method, mortar bars (25mm X 25mm X 285 mm) with gage studs embedded at the ends were cast and moist cured for 24 hours in a curing room. After demolding, the bars were cured at 80°C for 24 hours in a water bath. After curing in the water bath, the bars were kept in 1N NaOH soak solution, which was preheated to 80°C for 24 hours. Periodic length change measurements were taken at regular intervals up to 28 days, and percent expansions were calculated. In this study, the expansions of mortar bars less than 0.10% at 14 days were considered to reflect the non-reactive nature aggregates, and expansions of mortar bars over 0.10% were considered to reflect the reactive nature of the aggregates.

3.2.2. Revised EB – 70 test method (1N NaOH + 3M KAc deicer Soak Solution)

The principal revision in the revised EB-70 test method was the use of a soak solution that has a concentration of 1N NaOH + 3M KAc, instead of the 6.4M KAc solution as used in EB-70 test method. The basis for using this combination of 1N NaOH and 3M KAc deicer was previously discussed and presented in depth elsewhere (7,10). One liter of the soak solution for the revised EB-70 test method was prepared by dissolving 40 g of NaOH in 460 ml of 6.4M KAc deicer solution and then diluting the combination to one liter with deionized water, to achieve a concentration of 1N NaOH + 3M KAc in the resulting solution.

3.3. Test Program

A comparative evaluation of the aggregate reactivity in the standard ASTM C 1260 test method and the revised EB-70 test methods were conducted on 32 aggregates (REFERENCE). These results serve as a reference point for evaluating the efficacy of ASR mitigation measures.
All the mitigation measures were evaluated in the standard ASTM C 1567 test and the revised EB-70 test method. To evaluate ASR mitigation measures such as fly ashes and slag, a portion of portland cement was replaced by the respective SCM.

In evaluating the lithium admixture, a modified version of CRD–C 662-10 method was employed. In this method, the 30% solution of lithium nitrate was added to the mix water at 100% dosage level (Li/Na molar ratio of 0.74), and to the soak solution at 50% dosage level (Li/Na molar ratio of 0.37) with no other changes to the soak solution composition.

In this study, six fly ashes were evaluated at a 25% dosage level using aggregates #1 through #4 and #32 in order to understand the impact of chemical composition of fly ash on their ability to mitigate ASR in presence of deicing chemical. A more extensive study was conducted with Ash-1 at 25% dosage using a range of aggregates, i.e. #17 through #31, to better understand how different aggregates behaved in the revised EB-70 test method in presence of a given ash. In investigations involving slag and lithium admixtures, aggregates #1 through #4 were employed. Using the revised EB-70 protocol, limited studies using aggregate #1 were conducted in which combinations of lithium admixture with 25% Ash-1 were evaluated.

4. RESULTS AND DISCUSSIONS

4.1. Evaluation of Effectiveness of Fly Ashes in Mitigating ASR in the Revised EB-70 Test Method

Figures 2 and 3 show a comparison of 14-day expansions in mortar bars with and without Ash-1 in 1N NaOH soak solution, and a combination of 3M KAc + 1N NaOH solutions, respectively. It is evident from these figures that the use of Ash-1 at 25% dosage level is highly effective in reducing the mortar bar expansions, regardless of the soak solution composition. Figure 4 shows a comparison of 14-day mortar bar expansions in the 1N NaOH solution and a combination of 3M KAc + 1N NaOH solution. These results clearly illustrate that Ash-1, which a low-lime Class F fly ash, is highly effective in both test methods in suppressing the mortar bar expansions with a wide range of aggregate types. With an exception of a couple of aggregates (Agg-1, Agg-3, and Agg-4), in all the other cases the 14-day mortar bar expansion were below 0.10% at 14 days in both the test methods.

Figure 5 shows the 14-day expansion behavior of mortar bars containing Ash-1 through Ash-6 in combinations with five different aggregates (Agg-1 through Agg-5). Ash-1 and Ash-2 represent low lime fly ashes, with an average lime content of 1.15%. Ash-3 and Ash-4 represent intermediate lime content with an average lime content of 17.26%. Ash-5 and Ash-6 represent high lime fly ashes with an average lime content of 27.05%. The results shown in Figure 5 illustrate that as the lime content of the fly ash increases, the 14-day mortar bar expansion in the revised EB-70 test method increase proportionately with all the aggregates. It can also be
observed that with majority of the reactive aggregates (3 out of 4 reactive aggregates), the high-lime fly ash does not offer any significant mitigation in expansion compared to the control mixtures without fly ash.

4.2. Evaluation of Effectiveness of Slag in Mitigating ASR in the Revised EB-70 Test Method

Figure 6 shows the 14-day mortar bar expansion behavior of test specimens prepared with and without slag in the revised EB-70 test method. It is evident from these results that while the presence of 40% slag in the mix significantly reduces the expansion compared to the control specimen, the reduction in expansion is not below 0.10 % for majority of the aggregates (3 out of 4 aggregates evaluated). Therefore, it appears that a slag dosage of 40% may not be adequate to effectively mitigate ASR in the presence of KAc deicer, and higher dosage levels would be required.

4.3. Evaluation of Effectiveness of Lithium Admixture in Mitigating ASR in the Revised EB-70 Test Method

Effectiveness of lithium admixture in mitigating ASR in the presence of KAc deicer was evaluated using a method similar to that used in the CRD-C 662-10 test procedure. Results from these tests are shown in Figure 7. These results illustrate that in the presence of potassium acetate deicer, lithium admixture used at nominal dosage level (based on Li/Na molar ratio of 0.74) of 100% was not adequate in mitigating deleterious levels of expansions in mortar bars. In case of all the four aggregates, the reduction in expansion of the mortar bars was minimal, and in the case of aggregate #3, the use of lithium resulted in slightly increased expansion. It should be noted that in these studies, the lithium dosage in the soak solution of the revised EB-70 test method (i.e. Li/Na molar ratio of 0.37) was based on 1N NaOH alone and not based on the 3M KAc + 1N NaOH. The reason for this approach lies in the fact that 3M KAc solution by itself does not contribute any significant amount of hydroxyl ions in the soak solution.

Additional investigation was conducted to evaluate the effectiveness of combination of fly ash with lithium admixture to mitigate mortar bar expansion of Agg-1 mixture in the revised EB-70 test method. These results are shown in Figure 8. Based on these findings, it appears that the combination of a class F fly ash (Ash-1) and lithium admixture produces a synergistic effect and the mortar bar expansion in the revised EB-70 test method is virtually eliminated.

5. CONCLUSIONS

Based on the tests conducted in this investigation, the following conclusions can be drawn:
1. The sensitivity of an aggregate to undergo alkali-silica reactivity in the presence of potassium acetate deicer can be ascertained using the proposed revised EB-70 test method.

2. The proposed EB-70 test method can be used to evaluate the effectiveness of typical supplementary cementing materials such as fly ashes and slags in mitigating alkali-silica reaction in the presence of potassium acetate deicer.

3. The chemical composition of fly ash, particularly its lime content, appears to bear a significant relationship to the ability of a fly ash to mitigate mortar bar expansion in the revised EB-70 test method. This behavior is identical to the performance of fly ashes in the presence of 1N NaOH solution, as observed in the standard ASTM C 1567 test method.

4. Fly ashes with lime content less than 15% appear to be effective in mitigating mortar bar expansions to below 0.10% for majority of the aggregates at a dosage rate of 25%. Higher lime content fly ashes are generally ineffective in mitigating ASR at the 25% dosage level in the presence of potassium acetate deicer.

5. Use of slag at 40% cement replacement level can significantly reduce mortar bar expansions in the presence of potassium acetate deicer, however, higher dosage levels may be needed to effectively mitigate mortar bar expansions to below 0.10% with majority of the reactive aggregates evaluated in this study.

6. In the presence of potassium acetate deicer, lithium admixture by itself does not appear to be as effective in controlling mortar bar expansions to below 0.10% at 14 days, however, in combination with a low-lime fly ash, lithium admixtures are highly effective in controlling ASR.

6. RECOMMENDATIONS

While the findings from this research study provide a rational basis to evaluate ASR mitigation measures in the presence of deicing chemicals such as potassium acetate, these results should be calibrated using larger specimens and field exposure conditions. In the interim, however, it is recommended that the revised EB-70 test method be employed as a screening protocol to evaluate aggregate sensitivity to deicers and also to evaluate effectiveness of SCMs and lithium admixtures in mitigating ASR in the presence of deicing chemicals.

7. ACKNOWLEDGEMENTS

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8. REFERENCES


9. ACPA R&T Update 11.01, “Mitigating Deicer Induced Distress Potential – A Revision of an Interim Procedure”, American Concrete Pavement Association, Skokie, IL, April 2011.


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Table 1 - Mineralogy and Field Performance of Aggregates*

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* Field performance of aggregate reactivity was based on assessment by respective DOT or Airfield personnel. “D” indicates field performance of aggregate under KAc deicer exposure.
### Table 2 - Chemical Composition of Cementitious Materials

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Figure 1 – Comparison of 14-day mortar bar expansions in the standard ASTM C 1260 test and revised EB-70 test method.
Figure 2 – Comparison of 14-day mortar bar expansions of test specimens with and without Ash-1 in 1N NaOH soak solution.
Figure 3 – Comparison of 14-day mortar bar expansions of test specimens with and without Ash-1 in 3M KAc + 1N NaOH soak solution.
Figure 4 – Comparison of 14-day mortar bar expansion of test specimens with and without Ash-1 in 1N NaOH solution (ASTM C 1567) and combination of 3M KAc + 1N NaOH solution (Revised EB-70).
Figure 5 – Influence of chemical composition (lime content) of fly ash on mortar bar expansion in revised EB-70 test method.
Figure 6 – 14 Day Mortar Bar Expansions of Mortar Bars Containing 0% and 40% Slag in the Revised EB-70 Test Method
Figure 7 – 14 Day Mortar Bar Expansions of Mortar Bars Containing 100% Lithium Dosage in CRD-C 662-10 Test and Revised EB-70 Test Methods
Figure 8 – Synergistic Effect of Combination of Class F Fly Ash (Ash-1) and Lithium Admixture in the Revised EB-70 Test Method Using Aggregate-1 as a Reference Reactive Aggregate