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Mitigation of ASR in Presence of Pavement Deicing Chemicals

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

EXECUTIVE SUMMARY

Findings from the IPRF 03-9 research study on the potential of airfield pavement deicing chemicals to cause alkali-silica reaction (ASR) distress had indicated that alkali-acetate and the alkali-formate deicers caused deleterious expansions and cracking in test specimens containing reactive aggregates. This report presents the results from a research study conducted to determine the effectiveness of ASR mitigation measures such as supplementary cementing materials and lithium admixtures in mitigating the effects of deicing chemicals. In this study potassium acetate deicer was used as a reference deicer solution due to its wide-spread use in airfield pavements across the United States.

In this investigation three types of ASR mitigation measures – fly ash, slag and lithium admixtures – were evaluated. Considering the wide variability in the composition of fly ashes and the influence of fly ash composition on its ability to mitigate ASR, fly ashes from three sources that represented a range of lime contents were selected. One slag and one lithium admixture were also evaluated. The effectiveness of each of the three fly ashes was studied at dosage levels of 15%, 25% and 35% cement replacement by mass. Slag was investigated at 40% and 50% cement replacement levels. Lithium admixture was evaluated at different dosage levels based on the Li/Na or Li/K molar ratios and the concentration of the soak solution.

In this investigation reactive aggregates from four sources were selected to represent a variety of aggregate mineralogy, degree and rate of reactivity. These aggregates include: a gravel containing highly reactive rhyolite, two crushed aggregates consisting of moderately reactive argillite and siliceous limestone, and another crushed aggregate consisting of a slowly reactive quartzite.

As part of this investigation, a series of mortar bar tests were conducted to evaluate the alkali-silica reactivity of aggregate and the effectiveness of supplementary cementing materials in mitigating the ASR induced by deicer solution. These tests were based on modifications to the standard ASTM C 1260 and ASTM C 1567 test methods in which potassium acetate deicer solution was used as the soak solution for the mortar bars. In addition to these tests standard ASTM C 1260 and standard ASTM C 1567 tests were conducted to evaluate the reactivity of all the aggregates and the ASR-mitigation effectiveness of supplementary cementing materials, respectively. The results from the standard tests were compared with the results from the modified tests to better understand the role of ASR mitigation measures in resisting the effects of deicer solutions.

Based on the findings from these tests it was established that fly ashes with lower lime contents are in general more effective in mitigating expansions in mortar bars exposed to potassium acetate deicer solution and 1N sodium hydroxide solution. However, the specific level of mitigation offered by the fly ashes depended on the chemical composition and dosage level of the fly ash and the reactivity of the aggregate. While 25% dosage level of low-lime and intermediate-lime fly ash was found to be adequate for aggregates with moderate and low-levels of reactivity, 35% dosage level was required to mitigate mortar bars containing highly reactive aggregate. High-lime fly ash was not effective at any of the three replacement levels considered in mitigating expansions in mortar bars exposed to either potassium acetate deicer solution or 1N sodium hydroxide solution. In fact, mortar bars containing high-lime fly ash expanded more than the control mortar bars when exposed to potassium acetate deicer solution. This evidence suggests possible negative interaction between mortars containing high-lime fly ash and potassium acetate deicer solution.

The use of slag in mortar bars at a cement replacement level of 50% was found to be effective in mitigating expansions in both potassium acetate deicer solution and 1N sodium hydroxide solution. However, at a 40% dosage level, slag was found to be ineffective in mitigating expansions in mortar bars with a majority of the aggregates.

The effectiveness of lithium nitrate admixture in mitigating expansions in mortar bars was determined in two series of experiments. In the first series of experiments lithium nitrate admixture was added to the mortar bars alone; no lithium was added to the potassium acetate deicer soak solution. In the second series of experiments, lithium nitrate was added only to the potassium acetate deicer soak solution and not to the mortar bars. Based on the results from these tests, it was observed that addition of lithium to potassium acetate deicer solution alone, even at low Li/K molar ratios, significantly mitigated the expansions in mortar bars. These results suggested that the addition of lithium to both mortar bars and potassium acetate deicer solution simultaneously would therefore not yield results that would be representative and hence was not pursued. The addition of lithium nitrate admixture to mortars alone was also found to reduce expansions in bars exposed to potassium acetate deicer solution. However, additional research is needed to ascertain the amount of lithium that needs to be added to the soak solution, to adequately capture the mitigation mechanism.

In summary, ASR mitigation measures such as low-lime and intermediate-lime fly ashes, slag and lithium admixtures are able to considerably reduce expansions in mortar bar specimens exposed to potassium acetate deicer solution, when used in adequate dosage levels. In particular, low-lime and intermediate-lime fly ash have been found to be effective in mitigating mortar-bar expansions with all the aggregates at dosage levels of 35% by mass replacement of cement, and with majority of the aggregates at dosage level of 25% by mass replacement of cement. High-lime fly ash has been found to be ineffective in mitigating mortar-bar expansions at all the dosage levels investigated. Slag has been found to be effective at dosage level of at least 50% replacement by mass of cement with all the aggregates evaluated in this study. While lithium nitrate admixture has also been found to significantly reduce expansions in mortar bars with all the aggregates, additional research is needed to develop an appropriate test protocol.

1. INTRODUCTION

1.1. PURPOSE

Research conducted as part of Innovative Pavement Research Foundation (IPRF) project 03-9 had shown that airfield pavement deicing chemicals based on alkali-acetates and alkali-formates can cause deleterious alkali-silica reaction (ASR) in mortar and concrete test specimens [1]. In an effort to find possible solutions to counter the effects of these deicing chemicals, IPRF project 04-8 was initiated. The purpose of this research study was to investigate the potential of selected supplementary cementing materials and lithium admixtures to mitigate ASR that is caused by the use of potassium acetate-based deicer on concrete. The supplementary cementing materials considered in this study include fly ash and ground granulated blast furnace slag. The lithium admixture considered in this study was a commercial grade lithium nitrate based admixture (30% by weight solution), widely used in the industry to mitigate ASR.

1.2. BACKGROUND

A majority of the airports across the United States that experience severe winter weather conditions use anti-icing and deicing chemicals on a routine basis for ice and snow removal operations. Over the last decade a new generation of deicing chemicals, such as potassium acetate, sodium acetate, and sodium formate, has replaced the traditional pavement deicers, such as urea, ethylene and propylene glycol-based formulations. These new deicing formulations were developed in response to growing environmental concerns resulting from the run-off from airfield pavements on which urea and glycol-based chemicals were used.

While the new generation of deicers had adequately addressed safety and environmental concerns, there is growing evidence to indicate the aggressive influence of these chemicals on certain concrete pavements.

Recent investigation into premature deterioration of concrete pavements in some airfields (for instance Colorado Springs Airport and Denver International Airport) raised concerns over the potential ability of these deicing chemicals to trigger ASR in concrete containing aggregate that is susceptible to ASR. Figure 1 shows typical distress pattern observed in airfield concrete pavement exposed to the new generation deicing chemicals.

In order to investigate the potential of these deicing chemicals to trigger ASR in concrete, IPRF project 03-9 – Potential for Acceleration of ASR in Presence of Pavement Deicing Chemicals – was carried out [1]. The principal objective of this study was to determine if selected deicing chemicals including potassium acetate, sodium acetate, sodium formate and potassium formate have a potential to cause or accelerate ASR in concretes containing reactive aggregates.

Results from this study revealed that all the deicers based on sodium and potassium salts of acetate and formate have a significant potential to cause deleterious ASR in mortar and concrete test specimens containing reactive aggregates. However, no ASR-related effects were observed in test specimens containing non-reactive aggregate [1].

FIGURE 1. TYPICAL DISTRESS OBSERVED IN CONCRETE PAVEMENT EXPOSED TO AIRFIELD DEICING CHEMICALS

While limiting the use of alkali-acetate and alkali-formate deicers is an ideal solution to minimize the distress induced by these deicers in concrete pavements, the availability of suitable alternate deicers is extremely limited at the present time. Therefore, continued use of these deicers on airfield pavements appears likely in order to maintain functional runways and taxiways in winter conditions.

The use of non-reactive aggregates is another strategy to minimize the potential for ASR in concrete. However, the availability of such aggregates is increasingly becoming scarce in certain parts of the country. In locations where non-reactive aggregates are not readily available, it is often cost-prohibitive to have such aggregates transported from other locations. Therefore, it is imperative that the use of suitable ASR mitigation measures in concrete be considered in construction of new airfield pavements that are likely to experience heavy deicer application.

ASR mitigation measures such as use of low-alkali cement and limiting the alkali content of concrete may not be completely effective against the effects of external sources of alkalis (Na, K) such as the alkali-acetate and alkali-formate deicers. Findings from the previous study had indicated that the use of low-alkali cement was not effective in mitigating ASR in mortar and concrete test specimens exposed to solutions of alkali-acetate and alkali-formate deicers [1-4]. It was observed in these studies that even though the pH of plain potassium acetate deicer solution was only about 11, its pH increased by over 3 orders of magnitude (~14) upon interacting with hydrated cement paste in mortar and concrete test specimens [1-4]. These high pH levels are believed to be critical in triggering deleterious ASR reactions in test specimens. Investigations on other deicers yielded similar findings. In these studies, the alkali content of the cement was not found to have any influence on the observed increase in pH of the deicer solutions. Therefore, it is believed that the use of low-alkali cement as an ASR mitigation measure may not completely address the distress induced by deicer solutions.

Although literature on the effectiveness of supplementary cementing materials (SCMs) and lithium compounds in mitigating ASR is widely available, much of this information pertains to situations in which the alkalis are contributed by either portland cement or from other internal sources in concrete [5-9]. In this regard, not much information is available in the literature on the effectiveness of SCMs and lithium compounds in mitigating ASR in concrete, induced by exposure to external alkali sources such as deicing chemicals, in particular alkali-acetate and alkali-formate deicers.

In light of the fact that the use of alkali-acetate and alkali-formate based deicers will likely continue to grow due to their deicing effectiveness and environmentally friendly nature, it is important to determine the effectiveness of ASR mitigation measures such as SCMs and lithium admixtures to resist the aggressive effects of these deicers on concrete.

This report presents the research study conducted to determine the effectiveness of selected SCMs and lithium admixtures in mitigating ASR induced by deicing chemicals. In this study, a potassium acetate based formulation was considered as a representative deicer, as this is the most widely used deicer for snow and ice removal in airports across the United States. Based on the findings in this study specific recommendations are provided to evaluate and identify suitable mitigation measures for use in new concrete pavement construction to resist the effects of deicing chemicals.

1.3. OBJECTIVES

The principal objectives of this research study are:

1. To determine the potential of selected supplementary cementing materials (fly ash and slag) to mitigate ASR induced by potassium acetate deicer, with particular emphasis on the effect of fly ash and slag dosage and chemical composition of fly ash.

2. To determine the potential of lithium nitrate admixture to mitigate ASR induced by potassium acetate deicer.

3. Develop recommendations on potential alternatives for mitigating ASR induced by potassium acetate deicers and suggest a test procedure to evaluate the efficacy of ASR mitigation measures in presence of the deicing chemicals.

1.4. SCOPE OF RESEARCH

In this study the effectiveness of three different fly ashes with significantly different lime contents, one slag and one lithium nitrate admixture were evaluated in their potential to mitigate ASR induced by potassium acetate deicer solution.

In this investigation, a series of modified ASTM C 1567 tests were conducted to evaluate the effectiveness of three different ASR mitigation measures in presence of potassium acetate deicer. Additional tests including standard ASTM C 1260 and standard ASTM C 1567 were conducted to establish certain baseline references to compare the behavior of aggregates and mitigation measures in potassium acetate deicer solution with 1 normal sodium hydroxide solution – i.e. the standard reference solution. In all of these tests, mortar bars prepared with different aggregates and different ASR mitigation measures were tested in presence of either potassium acetate deicer soak solution or 1 normal sodium hydroxide solution. In addition to length-change measurements of mortar bars over time, change in dynamic modulus of elasticity of test specimens was monitored. Scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analyses were conducted on selected samples to study the microstructure of test specimens affected by the deicer solution.

Figure 2 shows a matrix of different ASR mitigation measures evaluated in this study to mitigate ASR induced by potassium acetate deicer solution. Each of the three fly ashes was evaluated at 15%, 25% and 35% cement replacement levels. Slag was evaluated at 40% and 50% replacement levels. A 30% lithium nitrate solution was evaluated in two different modes:

(i) as an admixture in mortar bar only at four different Li/Na molar ratios
(ii) as an additive to soak solution only at two different Li/K molar ratios

The reason for evaluating effectiveness of lithium admixture as an additive to either mortar bar only or deicer soak solution only is discussed in section 3.3.4.

In tests with supplementary cementing materials, a 6.4 molar concentration solution of potassium acetate (50% wt. solution) was used as the soak solution. In tests with lithium admixture, depending on the specific mode of evaluation a range of deicer concentrations were used. In tests where lithium nitrate solution was evaluated as an admixture in mortar bars (i.e. added only to mortar bars) to mitigate ASR, a 6.4 molar solution of potassium acetate deicer was used as soak solution. In tests, where the lithium nitrate solution was evaluated as an additive to potassium acetate deicer solution to mitigate ASR, a range of potassium acetate deicer concentrations (1, 2, 3 and 6.4 molar solutions) in combination with two different lithium dosages (Li/K molar ratio = 0.19 and 0.74) were investigated.

The effectiveness of all the ASR mitigation measures was evaluated in combination with 4 different reactive aggregates, each with a different level of reactivity.

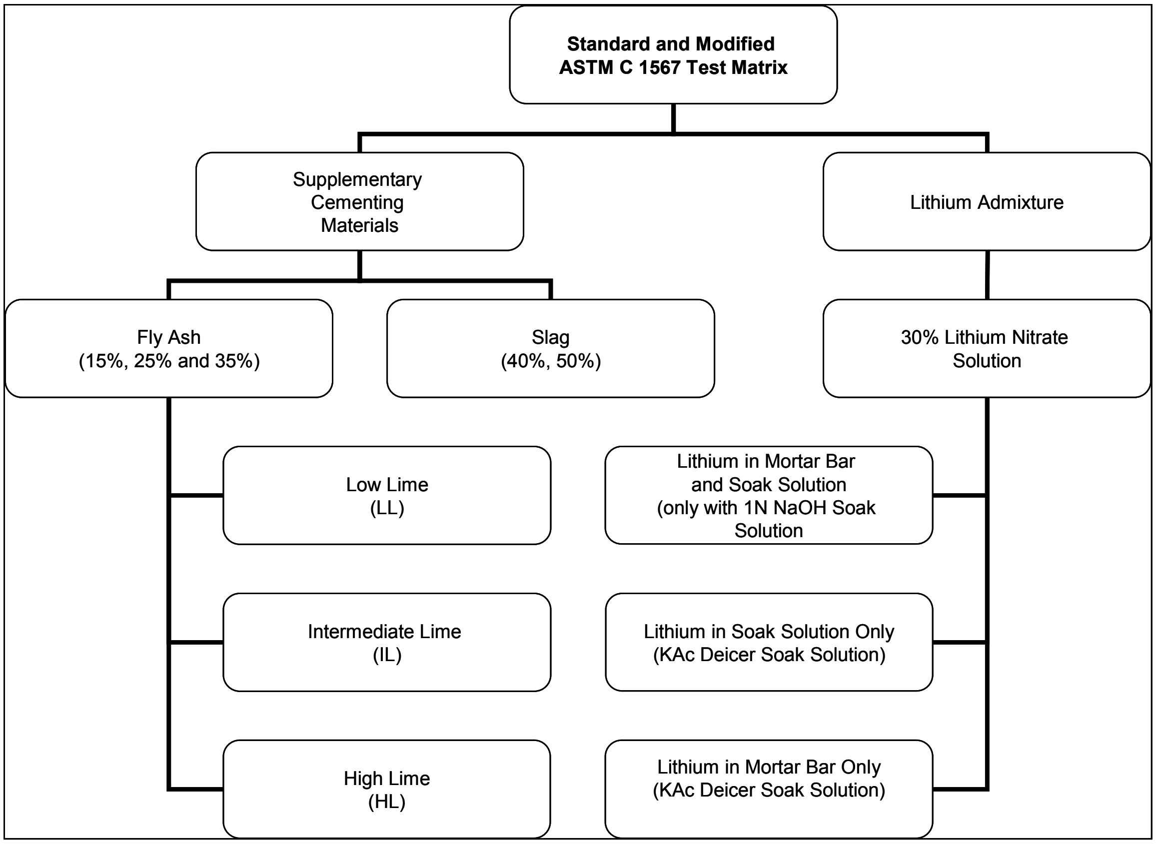


FIGURE 2. MATRIX OF DIFFERENT ASR MITIGATION MEASURES EVALUATED IN THIS STUDY

2. LITERATURE REVIEW

2.1. ALKALI-SILICA REACTION

ASR is a chemical reaction in concrete that occurs between certain reactive siliceous components in aggregates and typically the highly alkaline concrete pore solution. This reaction produces a hygroscopic reaction product, often referred to as ASR gel. The mechanism that leads to cracking and deterioration of concrete is usually not the production of the ASR gel itself, but its expansion in the presence of moisture, particularly in restricted spaces such as cracks in aggregates. The high pH environment required for the siliceous aggregates to react is typically generated from the dissolution of alkali sulfates and other alkali-bearing phases in portland cement. In some cases the alkalis may be supplied from other components of concrete such as supplementary cementing materials [10], admixtures or in some unique instances from within the aggregate itself [11-13]. External sources of alkalis, such as from marine exposure and from application of deicing salts, have also been found to trigger ASR [14-22].

2.2. INFLUENCE OF DEICERS ON ASR IN CONCRETE

Extensive research has been carried out on studying the influence of the most common deicer – sodium chloride (NaCl) – in initiating or aggravating ASR in concrete [14-22]. In these studies, it was observed that the degree of deterioration depended on such factors as the alkali and C_3A contents of the portland cement, the concentration of the NaCl solution, and the temperature at which testing was conducted. Several different hypotheses were presented in these studies to explain the aggressive influence of the NaCl solutions on ASR.

However, after nearly 65 years of research on ASR since its discovery in 1940 by Stanton [23] and after 12 International Conferences on the subject, and despite several thousand published papers and reports, investigations on the possible influence of other kinds of deicers on ASR, for example - alkali-acetate and alkali-formate deicers are virtually non-existent in published literature.

In light of the growing concerns associated with premature deterioration of concrete pavements in airfields and the widespread use of alkaline deicing chemicals, IPRF Project 03-9 was initiated to investigate the potential of the airfield deicing chemicals to cause or accelerate ASR in concrete. The intent of this investigation was to determine if alkali-acetate and alkali-formate deicers have a potential to initiate and/or accelerate ASR in concrete. Findings from this study are published elsewhere [1]. However, a brief summary of the research program and the principal findings are presented, and the need to investigate the effectiveness of ASR mitigation measures against deicer-induced ASR is highlighted.

2.3. SUMMARY OF IPRF PROJECT 03-9

2.3.1. Research Scope

In this study mortar bars and concrete prisms prepared with aggregates of known reactivity were exposed to alkali-acetate and alkali-formate deicer solutions. The condition of the mortar bars and concrete prisms was periodically monitored and the observed distress was characterized.

In this investigation aggregates from four sources well known for their ASR reactivity were selected. These included: a reactive gravel from Las Placitas pit (containing reactive rhyolite) from New Mexico, a crushed stone (containing reactive argillite) from Gold Hill quarry, North Carolina, a crushed stone (containing reactive siliceous limestone) from Spratt quarry, Ontario, Canada, and a crushed stone (made of quartzite) from L.G. Everist quarry, Sioux Falls, South Dakota. In addition, two sources of non-reactive aggregate – a graded Ottawa sand from Ottawa, Illinois (meeting ASTM C 778 requirements) and a dolomite from Thornton quarry, Illinois were selected. Test specimens were prepared with these aggregate using two cements, one with high alkali content (0.82% $Na_2O_{eq.}$) and the other with a low-alkali content (0.31% $Na_2O_{eq.}$). These specimens were subjected to standard and modified accelerated mortar bar tests (ASTM C 1260 test method) and to concrete prism tests (ASTM C 1293 test method). The specific deicers investigated in the study were two liquid deicers - 50% by weight, aqueous solutions of potassium acetate and of potassium formate, and two solid deicers – pellets of sodium acetate and sodium formate.

In the modified ASTM C 1260 procedure the mortar bars were subjected to a deicer soak solution instead of the standard 1N sodium hydroxide (NaOH) solution specified in the standard procedure. In these tests the liquid deicers were used as soak solutions at a concentration as supplied by the manufacturer. In tests involving solid deicers saturated aqueous solutions at room temperature were employed as the soak solution. Length changes taking place in the mortar bar specimens exposed to these soak solutions were monitored over a period of 28 days. In the standard and the modified ASTM C 1260 tests mortar bar expansions less than 0.1% at 16 days after casting (or at 14 days after soaking in deicer solution or 1N NaOH) were considered to indicate innocuous nature of the aggregate with respect to ASR.

In addition, scanning electron microscope (SEM) and energy dispersive x-ray (EDX) analysis were conducted after each of the tests to investigate the microstructure and composition of the reaction products. Dynamic modulus of elasticity of test specimens subjected to the deicer soak solutions were also determined so as to assess the loss in physical strength and stiffness. Periodic pH measurements were also made on soak solutions to monitor the changes in the deicer solutions.

2.3.2. Principal Findings

Results from this study established that all the alkali-acetate and alkali-formate airfield pavement deicers initiated and accelerated deleterious ASR reactions in test specimens containing reactive aggregates. The damage was characterized by excessive expansions in test specimens accompanied with severe cracking and deformation. Microstructural investigation using SEM and EDX analysis revealed extensive micro-cracking in aggregate, paste and paste-aggregate interface, with deposits of ASR gel in the affected test specimens. Specimens exposed to potassium acetate deicer solution, in particular, showed the presence of minor amounts of secondary reaction products composed of potassium sulfate phases in cracks and around aggregate grains. In case of all the test specimens that exhibited excessive expansions in deicer solutions significant decrease in dynamic modulus of elasticity was observed. In these studies it was observed that the use of low-alkali cement was not particularly effective in controlling the ultimate expansions in test specimens exposed to deicer solutions, although the rate of expansion of test specimens with low-alkali cement was somewhat lower at early ages. In comparison, test specimens containing non-reactive aggregates did not exhibit any distress upon exposure to any of the deicer solutions, regardless of the alkali content of the cement used. In a vast majority of the tests specimens soaked in deicer solutions showed either similar or higher levels of expansion compared to those soaked in 1 normal sodium hydroxide solution.

One of the principal findings of this study was that the pH of all alkali-acetate and alkali-formate deicing solutions was found to be very susceptible to significant increase upon interaction with calcium hydroxide – a common hydration product in portland cement concrete. The high level of pH caused by the interaction of deicer solutions with hydrated cement paste appeared to be the primary reason for the occurrence of ASR in test specimens containing reactive aggregates.

2.4. ASR MITIGATION MEASURES

The use of non-reactive aggregates in concrete is typically the most common strategy to eliminate the potential of ASR. However, the availability of non-reactive aggregates is increasingly becoming scarce or cost-prohibitive for use in concrete pavement in certain regions of the country.

Results from the previous study indicated that ASR mitigation strategies such as use of low-alkali cement or limiting the alkali content of the concrete are unlikely to be successful in controlling expansions in test specimens exposed to deicer solutions [1].

Although literature on the effectiveness of supplementary cementing materials (SCMs) and lithium compounds in mitigating ASR is widely available, much of this information pertains to situations in which the alkalis are contributed by cement or from other internal sources in concrete [5-9]. In this regard, not much information is available in literature on the effectiveness of SCMs mitigating ASR induced by external sources such as deicing chemicals, in particular alkali-acetate and alkali-formate deicers.

In light of the fact that the use of alkali-acetate and alkali-formate based deicers at airfield pavements and other facilities will only likely continue to grow, it is important to determine the effectiveness of ASR mitigation measures such as supplementary cementing materials and lithium admixtures to resist the effects of these deicers.

3. EXPERIMENTAL PROGRAM

3.1. RESEARCH APPROACH

The research approach adopted in this study can be summarized in the following steps:

1. Select representative ASR mitigation measures that are widely used in the industry.

2. Select representative aggregates that have an established history of being either reactive or non-reactive in nature, from an ASR standpoint.

3. Evaluate reactivity of aggregates (without any ASR mitigation measures) in presence of 1 normal sodium hydroxide solution and potassium acetate deicer solution using standard and modified ASTM C 1260 test methods. Results from this study will establish a baseline reference for measuring effectiveness of various ASR mitigation measures evaluated in this study.

4. Evaluate the ASR mitigation potential of selected measures in presence 1 normal sodium hydroxide solution and potassium acetate deicer solution using standard and modified ASTM C 1567 test methods. The results from these tests will be compared to the baseline reference measurements to determine the relative benefit offered by each of the ASR mitigation measures.

5. Determine the physical deterioration in the test specimens exposed to 1 normal sodium hydroxide and potassium acetate deicer solutions by measuring their dynamic modulus of elasticity during the course of testing.

6. Study the microstructure and composition of the reaction products in test specimens, using scanning electron microscope (SEM) and energy dispersive x-ray analysis (EDX).

7. Develop recommendations of suitable ASR mitigation measures to resist the effects of deicer solutions.

8. Establish a standard protocol to evaluate the effectiveness of ASR mitigating measures in presence of deicing chemicals.

In this investigation the ASR mitigation measures considered included fly ash, slag and lithium admixtures. These mitigation measures were selected as they represented the most commonly adopted strategies in the industry to minimize ASR-related distress in concrete pavements and are most likely to be the choices for mitigating ASR in construction of new airfield pavements.

It is well established that the ability of a fly ash to mitigate ASR is dependent on its chemical composition [7, 10]. In particular, the lime content of the fly ash has been shown to have a decisive influence on its ability to mitigate ASR [7].

In order to better understand the influence of fly ash composition on its ability to mitigate deicer-induced ASR, three fly ashes that differed significantly in their lime content were selected. Information pertaining to the source and the composition of fly ashes is given in the Experimental Materials section (see Section 3.2.4, Table 2). For other ASR mitigation measures evaluated in this study, one source of slag and one source of lithium admixture were considered.

Traditionally, standard test methods such as those described in ASTM C 1260 and ASTM C 1293 procedures are used to assess the alkali-silica reactivity potential of an aggregate source. Also, test methods such as ASTM C 441, ASTM C 1567 and ASTM C 1293 are used for evaluating the effectiveness of a supplementary cementing material or lithium admixture in mitigating ASR.

Findings from previous research study showed that the modified ASTM C 1260 and modified ASTM C 1293 test methods were effective in detecting the potential of a deicing chemical to trigger deleterious ASR in mortar and concrete test specimens [1]. In these test methods the specimens were soaked in a deicer solution, instead of the environment specified in the ASTM standard specification.

Based on the success in identifying the deleterious effects of deicers using the modified ASTM C 1260 and ASTM C 1293 tests in the previous research [1], a modified ASTM C 1567 test was proposed in this study. In the modified ASTM C 1567 test mortar bars prepared with a specific aggregate in combination with an ASR mitigation measure such as supplementary cementing material or lithium admixture were soaked in a potassium acetate deicer solution, instead of the standard 1 normal sodium hydroxide solution.

In evaluating the effectiveness of supplementary cementing material in standard or modified ASTM C 1567 test method, no changes to the composition of the soak solutions (whether 1 normal sodium hydroxide solution or potassium acetate deicer solution) were made. However, when lithium admixtures were evaluated two different approaches were taken. With 1 normal sodium hydroxide soak solutions, lithium was dosed not only in the mortar bars, but also in the soak solution to minimize the effect of leaching of lithium from mortar bars into soak solution on the observed mitigation. However, in tests with deicer soak solutions lithium was dosed only in the mortar bars, or only in the soak solution. The reason for this deviation is discussed more elaborately in Sections 3.3.3 and 3.3.4.

In all of the standard and modified ASTM tests conducted in this investigation the effectiveness of an ASR mitigation measure was evaluated based on the expansions observed in mortar bars upon exposure to different soak solutions. In this investigation, a mortar bar expansion in excess of 0.1% at 16 days was considered to indicate an ineffective ASR mitigation measure.

Detailed description of the standard and the modified ASTM C 1260, ASTM C 1567 test methods is presented in Section 3.3 of this report.

3.2. EXPERIMENTAL MATERIALS

3.2.1. Deicers and Reagents

In this study, a commercial grade potassium acetate deicer (KAc) with a concentration of 50% wt. solution (6.4 molar) was used as a soak solution in the modified ASTM C 1567 tests. The choice of deicer concentration for the soak solution in the modified ASTM C 1567 tests was based on practical and realistic considerations. In routine anti-icing operations (i.e. application of chemical before an ice or snow precipitation event), the potassium acetate deicer is applied on the bare pavement surface to prevent bonding of snow and ice to the pavement surface. Though the subsequent melting action of snow and ice may dilute the concentration of the deicers on the pavement surface, it is likely that with repeated applications of the deicers and through repeated freeze-thaw and wet-dry cycles the pore solution in the near-surface concrete would go through periodic cycles of dilution and concentration. It should be noted that the saturation concentration of potassium acetate in water at room temperature is 72.9% by weight. Therefore, the use of 50% wt. solution of potassium acetate deicer presents a realistic, yet severe exposure condition for investigating the influence of deicers.

A reagent grade sodium hydroxide (NaOH) in pellet form was used in this study to prepare the 1N sodium hydroxide soak solution for conducting the standard ASTM C 1260 and C1567 tests.

3.2.2. Aggregates

In this study, four types of reactive aggregates and a reference non-reactive aggregate were used. The non-reactive aggregate is a quarried dolomite stone from Illinois that had an established history of good field performance and had been used as reference non-reactive aggregate in previous laboratory studies [26]. The four reactive aggregates include:

- **Spratt Limestone** – This aggregate is obtained from Spratt quarry in Ontario Province of Canada. It primarily consists of calcite with minor amounts of dolomite and about 10% insoluble residue. The reactive component of the rock is reported to consist of 3% to 4% of microscopic chalcedony and black chert, which is finely dispersed in the matrix [24]. This aggregate has an established history of being alkali-silica reactive in field structures and has been used as a reference aggregate in many previous ASR studies.

- **NM Rhyolite** – A reactive gravel from Las Placitas Gravel Pit from Bernalillo County in New Mexico. This aggregate primarily consists of rhyolite that has shown very high levels of reactivity [25, 26].

- **NC Argillite** – This aggregate is a quarried material from the slate belt of North Carolina from Gold Hill Quarry in North Carolina. This aggregate primarily consists of reactive metatuff/argillite. This aggregate has an established history of poor field performance in several bridge structures in North Carolina [27].

- **SD Quartzite** – This aggregate is obtained from crushing quarried rock from Sioux Falls quarry, located in the southeastern South Dakota. This aggregate consists of strained quartz grains that are cemented with interstitial secondary quartz cement. In addition, the interstitial matrix also consists of microcrystalline quartz, hematite and kaolinite. This aggregate has an established history of being reactive in concrete pavements in Minnesota and South Dakota [28, 29]. Table 1 shows the basic physical properties of all the aggregates used in this study.

TABLE 1. PROPERTIES OF AGGREGATES

Aggregate Property	**Spratt, Limestone**	**SD, Quartzite**	**NC, Argillite**	**NM, Rhyolite**	**IL, Dolomite**
Water absorption,%	0.456	0.42	0.344	1.087	2.12
Bulk specific gravity (OD)	2.69	2.51	2.75	2.60	2.66
Bulk specific gravity (SSD)	2.706	2.52	2.76	2.63	2.71
Dry-Rodded Unit Weight, kg/m^3	1568.3	1557.62	1566	1585.25	1563.7

3.2.3. Cement

In this study a high-alkali cement (Type I) with a Na_2O equivalent of 0.82% and an autoclave expansion of 0.08% was used. The chemical composition of the cement is provided in Table 2.

3.2.4. Fly Ashes

In this study, three fly ashes differing significantly in their lime content were used. The low lime (LL), intermediate lime (IL) and high lime (HL) fly ashes contained 5.2%, 15.7% and 29.4% lime content. The effectiveness of each of the three fly ashes in mitigating ASR distress was investigated at 15%, 25% and 35% cement replacement levels by mass. A detailed oxide composition of the fly ashes is provided in Table 2.

3.2.5. GGBF Slag

A grade 120 was used for this study at 40% and 50% cement replacement levels by mass. The chemical composition of this slag is presented in Table 2.

TABLE 2. CHEMICAL COMPOSITION OF CEMENTITIOUS MATERIALS USED IN THE STANDARD AND MODIFIED ASTM C 1260 TESTS

Oxide, %	**High-Alkali Cement**	**Fly Ash**			**Slag**
		Low-Lime	**Intermediate Lime**	**High Lime**	
SiO2	19.74	57.83	49.18	33.20	38.17
Al2O3	4.98	21.26	15.27	18.72	7.31
Fe2O3	3.13	10.16	6.72	6.28	0.78
CaO	61.84	**5.19**	**15.74**	**29.37**	39.12
MgO	2.54	---	---	---	12.48
SO3	4.15	0.49	1.00	2.40	2.56
Available Alkali	---	0.39	1.32	1.29	---
LOI	1.90	0.03	0.10	0.59	---
$Na2O_{equivalent}$	0.82	---	---	---	---
K2O	---	---	---	---	0.34
Insoluble Residue	0.25	---	---	---	---
C3A	8.00	---	---	---	---
C3S	52.00	---	---	---	---
TiO2	---	---	---	---	0.78
Mn2O3	---	---	---	---	0.40

3.2.6. Lithium Nitrate Admixture

The lithium admixture used in this study was a commercial grade 30% wt. solution of lithium nitrate. It is odorless, white to yellow in color. The pH of the solution @ 25° C was 8.2. The specific gravity of the solution was 1.20 g/cc at 25° C.

In modified ASTM C 1567 tests, in which lithium nitrate was added to potassium acetate deicer solution to study the mitigation effects of lithium blended deicing solution, a reagent grade lithium nitrate salt was used.

3.3. EXPERIMENTAL METHODS

3.3.1. Standard ASTM C 1260 Test Procedure [30]

The standard ASTM C 1260 test known as “Accelerated Mortar Bar Test” is a method to assess the reactivity of aggregates. In this test, mortar bars (25 mm x 25 mm x 285 mm) with gage studs at ends are prepared at a water-to-cement ratio of 0.47. The aggregate-to-cement ratio by mass is maintained at 2.25. After 24 hours of curing in a moist cabinet, the mortar bars are demolded and transferred into a storage container with sufficient water to immerse all samples. The sealed container is placed in an oven at 80ºC for 24 hours. After 24 hours, the mortar bars are removed from the oven and a zero reading is taken. The mortar bars are subsequently transferred into a 1 N sodium hydroxide solution, which is preheated to 80ºC. Length change readings are taken thereafter at periodic intervals to determine the percent expansion. In this research, the length-change measurements were taken up to 56 days.

According to ASTM C 33 specification, mortar bar expansion of 0.1% or less at 16 days in the standard ASTM C 1260 test (i.e. 14 days of exposure to soak solution) is considered to indicate the innocuous nature of the aggregate. Mortar bar expansion greater than 0.2% at 16 days is considered to indicate the potentially reactive nature of the aggregate. Mortar bar expansion between 0.1% and 0.2% is considered to not conclusively indicate the nature of the aggregate. With these aggregates, additional evaluation using concrete prism test (ASTM C 1293) is recommended, when adequate field performance data is not available to ascertain the reactivity of the aggregate.

FAA Advisory Circular 150/5370-10B – Standard for Specifying Construction of Airports – considers aggregate to be reactive when expansions in mortar bars subjected to the standard ASTM C 1260 test exceed 0.1% at 16 days [31]. However, this limit is modified on a regional basis.

For instance, the revised AC 150/5370-10B specification issued by the Northwest Mountain Region Airports Division considers aggregate to be reactive if the expansion of mortar bars in the standard ASTM C 1260 test exceeds 0.1% at 30 days after casting (i.e. 28 days of exposure to soak solution). In addition, when the use of pavement deicing chemicals such as potassium acetate solutions is anticipated a modified ASTM C 1260 test is employed to assess the reactivity of the aggregate [32]. In the modified ASTM C 1260 test the mortar bars are soaked in a deicer solution after the initial curing regimen, instead of 1N sodium hydroxide solution. When deicer usage is anticipated, the aggregate is considered reactive if expansion of mortar bars in the modified ASTM C 1260 test exceeds 0.08% at 30 days after casting. The tighter specifications may be justified on airfield pavements, where concerns and consequences arising from Foreign Object Debris (FOD) are serious.

In the present investigation, an aggregate is considered reactive if the expansions of mortar bars exceed 0.1% at 16 days, in the standard or modified ASTM C 1260 test methods. However, the expansion results will also be evaluated against the more stringent expansion limits imposed by the revised AC 150/5370-10B standards specified by the Northwest Mountain Regions Airport Division, where applicable.

3.3.2. Modified ASTM C 1260 Test Procedure

In the modified ASTM C 1260 test method the mortar bars are soaked in a potassium acetate deicer solution, instead of the standard 1N sodium hydroxide solution. Other aspects of the testing are similar to that of the standard ASTM C 1260 test procedure. In this research study expansion of test specimens over 0.1% at 16 days was considered to represent an aggregate source that is unacceptable or an ineffective ASR mitigation strategy.

3.3.3. Standard ASTM C 1567 Test Procedure [33]

3.3.3.1. Test to Evaluate Supplementary Cementing Material

The standard ASTM C 1567 test procedure is identical to the standard ASTM C 1260 test method in all aspects of testing, with the exception that a portion of the portland cement in the mortar bars is replaced with a supplementary cementing material on an equivalent mass replacement basis. In this study fly ash was studied at three cement replacement levels of 15%, 25% and 35% by mass. Slag was investigated at two cement replacement levels including 40% and 50% by mass.

3.3.3.2. Test to Evaluate Lithium Admixture with Lithium in Mortar Bar and Soak Solution

Although the standard ASTM C 1567 test method does not allow for evaluation of lithium admixtures, a variant of the standard test method was used in this study to evaluate lithium admixtures for their potential to mitigate ASR. In this study, tests conducted to evaluate lithium admixture in the presence of 1N NaOH solution are referred to as “standard” ASTM C 1567 tests. Tests conducted to evaluate lithium in the presence of potassium acetate deicer solution are referred to as “modified” ASTM C 1567 tests.

In this test method the lithium admixture was dosed into the mortar bars at a specific Li/Na ion molar ratio in which the Na ion concentration is governed by the alkali content of the cement used. In addition, lithium admixture was also added to the 1N sodium hydroxide soak solution at a level that resulted in equivalent Li/Na molar ratio as present in the mortar bars. This procedure was adopted to minimize effects on mortar bar expansion that would result from leaching of lithium from mortar bars into soak solution.

In the present investigation four different Li/Na ion molar ratios – 0.23, 0.74, 1.0 and 1.25 – were considered. The range of lithium dosage considered in this study was selected to potentially mitigate even the most reactive aggregate used in this study – NM rhyolite.

3.3.3.3. Test to Evaluate Lithium Admixture with Lithium in Soak Solution Only

In order to study the potential effect of lithium addition to the soak solution on the observed mitigation, a series of mortar bar tests were conducted in which lithium was added only to the 1N sodium hydroxide soak solution. No lithium was added to the mortar bars. The molar ratio of Li/Na in the 1N sodium hydroxide solution was maintained at 0.23, 0.74, 1.0 and 1.25, such that a comparative assessment can be made between the effects of adding lithium to soak solution only versus adding lithium to both soak solution and mortar bars.

In these tests, the effectiveness of the ASR mitigation measure is evaluated based on the level of expansion observed in the mortar bars at 16 days. Mortar bar expansions less than 0.1% indicate the effectiveness of the specific ASR mitigation measure being evaluated.

3.3.4. Modified ASTM C 1567 Test Procedure

In this study, a modified ASTM C 1567 test method was used to assess the effectiveness of fly ash, slag and lithium admixture in mitigating ASR in presence of deicing chemicals.

3.3.4.1. Test to Evaluate Supplementary Cementing Material

In the modified ASTM C 1567 tests conducted to evaluate supplementary cementing materials, the mortar bars were soaked in potassium acetate deicer solution instead of the 1N sodium hydroxide solution. With the exception of the soak solution composition employed in the test method, the procedure to prepare mortar bar specimens and their subsequent storage regimen is identical to the procedure described in standard ASTM C 1567 test method.

3.3.4.2. Test to Evaluate Lithium Admixtures

When evaluating effectiveness of lithium admixtures in mitigating ASR induced by potassium acetate deicer, two modifications to the test procedure described under standard ASTM C 1567 test (in sections 3.3.3.2 and section 3.3.3.3) were employed. In one modification, the lithium admixture was added only to the mortar bar (without addition to the potassium acetate deicer solution). In this case, the lithium dosage in the mortar bar was based on the alkali content of the cement used. In these studies, four different Li/Na molar ratios – 0.25, 0.50, 0.75 and 1.0 were used. The soak solution consisted of the potassium acetate deicer solution.

In another modification, the lithium admixture was added only to the soak solution without addition to the mortar bar. In these tests, the lithium was dosed into the soak solution based on the concentration of the deicer solution. In these studies, two Li/K molar ratios of 0.19 and 0.74 were considered. Further, in case of tests with soak solution consisting of Li/K molar ratio = 0.19, four different concentrations of potassium acetate deicer soak solution were investigated including 1, 2, 3 and 6.4 molar concentrations. In case of tests with soak solutions of Li/K molar ratio = 0.74, three different molar concentrations of potassium acetate were investigated including 1, 2 and 3 molar concentrations. With a 6.4 molar concentration of potassium acetate, the solution is too concentrated to add significant lithium to achieve Li/K molar ratios.

Preliminary investigation into effectiveness of lithium admixture using the two modified ASTM C 1567 test methods (i.e. lithium in the bar only and lithium in the soak solution only) suggested that even when lithium admixture was added only to potassium acetate deicer soak solution, expansions in mortar bars were significantly reduced. Similar reductions in expansions were observed at 14 days when lithium was dosed in mortar bars alone and not in soak solution. Results from these tests are presented in Section 5. However, careful comparisons of the overall expansions from the mortar bar-only dose and the soak-only dose, reveals that there is a greater suppression of expansions with the soak-only dose (for similar Li/K ratios). This makes physical sense as well, as there is significantly more Li in the soak than there is in the mortar bars.

The implication of this finding was that the addition of lithium admixture to the potassium acetate deicer soak solution would completely mask the effect of lithium admixture in the mortar bar, if the two ways of including lithium admixture in this testing were done simultaneously, and with the same Li/K ratios in both modes of testing. Therefore, it was concluded from this work that adding lithium admixture to both the mortar bar and the soak solution at the same Li/K ratio in the case of potassium acetate deicer solution would not yield results that would be representative, and hence was not conducted in this investigation.

In this research study the mortar bars were stored in the soak solution for either 28 days or 56 days in the soak solution, instead of typical 14 days as required in the standard ASTM C 1260 and ASTM C 1567 test procedures. The extended testing was conducted to assess the effectiveness of mitigation measures in suppressing the effects of ASR at later ages (i.e. > 14 days of soaking). During the course of 56 days, periodic length-change measurements were taken at 0, 3, 7, 11, 14, 21, 28, 42 and 56 days. The results of all the standard and modified ASTM C 1567 tests discussed in this research study are based on an average of readings obtained from 4 mortar bars.

3.3.5. Dynamic Modulus of Elasticity

The dynamic modulus of elasticity (DME) of the mortar bars was measured at periodic intervals to quantify the physical distress occurring in the mortar bars subjected to the ASTM C 1260 and 1567 tests. The DME values were determined using the resonant frequency method based on impulse excitation technique based on ASTM E 1876-01 test procedure [33]. A GrindoSonic^{TM} instrument was used to determine the resonant frequencies of the mortar bars.

In this test the mass and the resonant frequency of the mortar bar specimens were determined soon after taking the length-change measurements. The dimensions of the mortar bars were assumed to be constant (i.e., 25mm x 25mm x 285mm) and the effects of the metal gage studs at the ends of the bars were neglected, as it was a common factor for all measurements. DME values of mortar bars were calculated for the same ages at which length-change measurements were made. Changes in DME values were correlated with expansion measurements to understand the progressive deterioration in stiffness of the mortar bars.

3.3.6. SEM and EDX Analyses

SEM and EDX examinations were conducted on polished sections of mortar bars using an ASPEX SEM Instrument. The instrument was operated at an accelerating voltage of 20KeV.

The samples for the SEM-EDX were retrieved from the mortar bars by slicing the test specimens using a slow-speed diamond saw. These samples were oven-dried at 50° C and then embedded using a Low Viscosity Epoxy. The epoxy embedded samples were then polished on a series of diamond embedded discs with progressively increasing fineness up to 1200 grit (mean particle size of 5 microns). The polishing of the samples culminated with a final polish using 0.25 micron diamond paste on a polishing cloth.

3.4. MATRIX OF STANDARD AND MODIFIED ASTM C 1260 AND 1567 TESTS

3.4.1 Standard and Modified ASTM C 1260 Tests

Table 3 shows the standard and the modified ASTM C 1260 tests conducted in this investigation. The results from these tests were used as reference to determine the effectiveness of different supplementary cementing materials and lithium admixtures in mitigating ASR. In each of these tests results from four mortar bars were averaged and used in further analysis.

TABLE 3. STANDARD AND MODIFIED ASTM C 1260 TESTS

Aggregate Type	**Standard ASTM C 1260 Test (1 normal sodium hydroxide solution)**	**Modified ASTM C 1260 Test (6.4 molar potassium acetate deicer solution)**
Spratt Limestone	X	X
NM Rhyolite	X	X
NC Argillite	X	X
SD Quartzite	X	X
Illinois Dolomite	X	X

3.4.2. Standard and Modified ASTM C 1567 Tests to Evaluate Supplementary Cementing Materials

Table 4 shows the standard and modified ASTM C 1567 tests conducted in this investigation to evaluate the effectiveness of supplementary cementing materials in mitigating ASR. In this study a total of fifty standard and modified ASTM C 1567 tests were conducted.

TABLE 4. STANDARD AND MODIFIED ASTM C 1567 TESTS TO EVALUATE SUPPLEMENTARY CEMENTING MATERIALS

Aggregate Type	**Standard ASTM C 1567 Test (1 normal sodium hydroxide soak solution)**					**Modified ASTM C 1567 Test (6.4 molar potassium acetate deicer soak solution)**				
	Fly Ash Dosage			**Slag Dosage**		**Fly Ash Dosage**			**Slag Dosage**	
	15%	**25%**	**35%**	**40%**	**50%**	**15%**	**25%**	**35%**	**40%**	**50%**
Spratt Limestone	X	X	X	X	X	X	X	X	X	X
NM Rhyolite	X	X	X	X	X	X	X	X	X	X
NC Argillite	X	X	X	X	X	X	X	X	X	X
SD Quartzite	X	X	X	X	X	X	X	X	X	X
Illinois Dolomite	X	X	X	X	X	X	X	X	X	X

3.4.3. Standard and Modified ASTM C 1567 Tests to Evaluate Lithium Admixture

Table 5 shows the matrix of standard ASTM C 1567 tests conducted in this investigation to evaluate the effectiveness of lithium admixture in presence of 1 normal sodium hydroxide solution. In this study two parallel series of tests were conducted. In one series, the lithium admixture was dosed into mortar bars as well as into the soak solution. In another series, the lithium admixture was added only to the soak solution.

In this investigation, the lithium admixture was studied at four dosage levels of Li/Na molar ratio – 0.23, 0.74, 1.00 and 1.25. The Li/Na molar ratio represents the amount of lithium added to the mortar bar relative to the amount of sodium equivalent alkalis present in the mortar bar. The Li/Na molar ratio also represents the lithium dosage in the soak solution based on a 1 normal sodium hydroxide concentration of a standard solution.

TABLE 5. STANDARD ASTM C 1567 TEST TO EVALUATE ASR MITIGATION POTETNIAL OF LITHIUM ADMIXTURE IN PRESENCE OF 1N SODIUM HYDROXIDE SOLUTION

Aggregate Type	**Standard ASTM C 1567 Test (1 normal sodium hydroxide solution)**			
	Li/Na Molar Ratio			
	0.23	**0.74**	**1.00**	**1.25**
Spratt Limestone	X	X	X	X
NM Rhyolite	X	X	X	X
NC Argillite	X	X	X	X
SD Quartzite	X	X	X	X

In evaluating lithium admixtures for their effectiveness in mitigating ASR induced by potassium acetate deicer solution two variations of modified ASTM C 1567 test were employed. Description of these two modified ASTM C 1567 test methods was presented in section 3.3.4. Table 6 shows the matrix of tests in which the lithium admixture was added only to the mortar bars, but not to the soak solution.

TABLE 6. MODIFIED ASTM C 1567 TEST METHOD TO EVALUATE ASR MITIGATION POTENTIAL OF LITHIUM ADMIXTURE IN PRESENCE OF POTASSIUM ACETATE DEICER SOLUTION (LIHTIUM ADMIXTURE ONLY IN BAR)

Aggregate Type	**Modified ASTM C 1567 Test (6.4 molar potassium acetate deicer solution) (Lithium in Bar Only)**			
	Li/Na			
	0.25	**0.50**	**0.75**	**1.00**
Spratt Limestone	X	X	X	X
NM Rhyolite	X	X	X	X
NC Argillite	X	X	X	X
SD Quartzite	X	X	X	X

The lithium admixture was dosed into mortar as a function of Li/Na ion molar ratio, where the Na ion represents the alkali content of the cement. In this investigation, the lithium admixture was studied at four dosage levels of Li/Na molar ratio – 0.25, 0.50, 0.74 and 1.00. In all of these tests, the soak solution consisted of a 6.4 molar potassium acetate deicer concentration.

Table 7 shows the matrix of tests in which the lithium admixture was added only to the soak solution, but not to the mortar bars. In order to better understand the ASR mitigation effects of lithium-blended potassium acetate deicer solutions, lower concentrations of potassium acetate solutions were proposed. As a result, the test matrix consisted of three concentrations of potassium acetate deicer solution at 1, 2 and 3 molar solutions. At each of these concentrations of potassium acetate deicer solution, lithium nitrate solution was added to achieve Li/K molar ratios of 0.19 and 0.74. Using this procedure, it was not possible to achieve a solution with Li/K molar ratio of either 0.19 or 0.74 at concentrations higher than 3 molar. To circumvent this situation, a lithium nitrate salt was added to potassium acetate deicer to achieve a Li/K molar ratio of 0.19 with a potassium acetate concentration of 6.15 molar solution.

It was observed in these investigations that it is physically not possible to blend either 30% lithium nitrate solution or lithium nitrate salt with 6.4 molar solution of potassium acetate to achieve a Li/K ratio of 0.74 at due to solubility limitations.

TABLE 7. MODIFIED ASTM C 1567 TEST METHOD TO EVALUATE ASR MITIGATION POTENTIAL OF LITHIUM-BLENDED POTASSIUM ACETATE DEICER SOLUTIONS (LIHTIUM NITRATE ONLY IN SOAK SOLUTION)

Aggregate Type	**Li/K molar ratio = 0.19**				**Li/K molar ratio = 0.74**		
	Potassium Acetate Deicer Solution Concentration (molar)				**Potassium Acetate Deicer Solution Concentration (molar)**		
	1	**2**	**3**	**6.15***	**1**	**2**	**3**
Spratt Limestone	X	X	X	X	X	X	X
NM Rhyolite	X	X	X	X	X	X	X
NC Argillite	X	X	X	X	X	X	X
SD Quartzite	X	X	X	X	X	X	X

* It is not possible to prepare a soak solution containing a Li/K molar ratio of 0.19, using a 50% potassium acetate deicer solution. Hence, the concentration of potassium acetate in the soak solution was diluted to 6.15 molar in these tests to achieve the desired Li/K molar concentration. Section 3.5.4 provides the explanation for this deviation.

3.5 MIXTURE PROPORTIONS OF MORTARS AND COMPOSITION OF SOAK SOLUTIONS

3.5.1. Mixture Proportions of Mortars Containing Supplementary Cementing Materials

Table 8 shows the mixture proportions of mortars used in preparing the test specimens for standard and modified ASTM C 1260 and ASTM C 1567 tests, in which supplementary cementing materials were evaluated.

TABLE 8. MATERIAL PROPORTIONS FOR PREPARING A BATCH OF FOUR MORTAR BARS CONTAINING SUPPLEMENTARY CEMENTING MATERIALS

Materials	**CONTROL**	**ASR MITIGATION MEASURE**				
		FLY ASH			**SLAG**	
		15%	**25%**	**35%**	**40%**	**50%**
Cement, g	500	425	375	325	300	250
Fly Ash, g	0	75	125	175	0	0
Slag, g	0	0	0	0	200	250
Water, g	235	235	235	235	235	235
Sand, g	1125	1125	1125	1125	1125	1125
Lithium, g	-	-	-	-	-	-
w/cm	0.47	0.47	0.47	0.47	0.47	0.47

3.5.2. Mixture Proportions of Mortars Containing Lithium Admixture in Standard ASTM C 1567 Test

Tables 9 and 10 show the mixture proportions used in preparing mortar specimens for the standard and modified ASTM C 1567 tests, respectively, in which lithium admixture was dosed in the mortar batch.

In tests where lithium admixture was added only to the potassium acetate deicer solution, the mortar bars were proportioned and prepared similar to control bars as indicated in Tables 9 and 10. The soak solutions in these tests consisted of two molar ratios of Li/K at 0.19 and 0.74, and four different concentrations of potassium acetate deicer solution at 1, 2, 3 and 6.15 molar solutions as described in Table 7.

Sample calculations to determine the amount of 30% lithium nitrate solution to be added to mix water to arrive at the desired Li/Na ratio in the mortar batch are shown in Section 3.5.4.

TABLE 9. MATERIAL PROPORTIONS FOR PREPARING FOUR MORTAR BARS CONTAINING LITHIUM ADMIXTURE FOR USE IN STANDARD ASTM C 1567 TEST

Materials	CONTROL	LITHIUM DOSAGE (Li/Na)			
		0.23	0.74	1.00	1.25
Cement, g	500	500	500	500	500
Fly Ash, g	0	0	0	0	0
Slag, g	0	0	0	0	0
Water*, g	235	230	219	213.7	208.4
Sand, g	1125	1125	1125	1125	1125
30% Lithium Nitrate Solution, g	0	6.99	22.51	30.41	38
w/cm	0.47	0.47	0.47	0.47	0.47

* Water contributed by lithium nitrate solution is considered in determining the actual water content of the batch, in order to maintain a constant w/cm ratio of 0.47 across all mixtures.

TABLE 10. MATERIAL PROPORTIONS FOR PREPARING FOUR MORTAR BARS CONTAINING LITHIUM ADMIXTURE FOR USE IN MODIFIED ASTM C 1567 TEST

Materials	CONTROL	LITHIUM DOSAGE (Li/Na)			
		0.25	0.50	0.75	1.00
Cement, g	500	500	500	500	500
Fly Ash, g	0	0	0	0	0
Slag, g	0	0	0	0	0
Water*, g	235	229.68	224.36	219.04	213.7
Sand, g	1125	1125	1125	1125	1125
30% Lithium Nitrate Solution, g	0	7.60	15.20	22.80	30.40
w/cm	0.47	0.47	0.47	0.47	0.47

* Water contributed by lithium nitrate solution is considered in determining the actual water content of the batch, in order to maintain a constant w/cm ratio of 0.47 across all mixtures.

3.5.3. Composition of Soak Solutions

Soak solutions for standard ASTM C 1260 and ASTM C 1567 tests consists of 1 normal sodium hydroxide soak solution. This solution was prepared by dissolving 40 grams of reagent grade sodium hydroxide in 900 ml deionized water. Subsequently, the solution was further diluted using sufficient deionized water to make 1000 ml solution.

In case of modified ASTM C 1260 and ASTM C 1567 tests involving supplementary cementing materials, a commercial grade potassium acetate deicer solution (50% wt. solution) was used as the soak solution.

In case of tests in which lithium admixture was evaluated, a defined volume of 30% lithium nitrate solution was added to 500 ml of 2N sodium hydroxide solution, which was subsequently diluted to 1000 ml to arrive at a defined Li/Na molar ratio. Sample calculations and the methodology used to arrive at the specific Li/Na molar ratios in the soak solution are presented in Section 3.5.4.

In case of modified ASTM C 1567 tests in which lithium nitrate solution was blended with potassium acetate deicer solution, a defined amount of 30% lithium nitrate solution was added to a defined amount of 50% solution of potassium acetate, and the blend was subsequently diluted to a 1000 ml volume to arrive at a specific Li/K molar ratio (i.e. 0.19 or 0.74). This methodology was used in preparing solutions with 1, 2 and 3 molar concentrations of potassium acetate. Sample calculations and methodology used are presented in Section 3.5.4.

In case of soak solution with Li/K molar ratio of 0.19 prepared with a 6.15 potassium acetate deicer solution, 965 ml of 6.4 molar potassium acetate deicer solution was added to a 1000 ml flask. 83.9 grams of dried lithium nitrate salt was introduced into the flask to make a solution of 1000 ml. The resulting solution had a Li/K molar ratio of 0.19, with a 6.15 molar potassium acetate concentration.

3.5.4. Sample Calculations

Sample Calculation to Determine the Dosage of 30% Lithium Nitrate Solution in Mortar Batch to Arrive at a Desired Li/Na Molar Ratio

Desired Li/ Na ion molar ratio = 0.74

Cement content per batch
of four mortar bars = 500 grams
Cement alkali content (%) = 0.82% $Na_2O_{eq.}$
Alkali Content per batch (grams) = 0.0082 X 500 grams = 4.1 grams
Molecular Weight of Na_2O = (2 x 23 + 16) = 62 grams/mole
Moles of Na_2O = Alkali Content/ 62 = 4.1/ 62 = 0.0661 moles
Moles of Na = 2 X 0.0661 = 0.1322 moles
Moles of Lithium / Moles of Na = 0.74
Moles of Lithium = 0.74 x 0.1322
Moles of Lithium = 0.0978 moles

1 Mole of $LiNO_3$ contains 1 Mole of Lithium ion

Molecular Weight of $LiNO_3$ = 69 grams/mole
Grams of $LiNO_3$ that contain

0.0978 moles of Li ion = 69 x 0.0978 = 6.753 grams

30% solution of $LiNO_3$ that
contains 6.753 grams of lithium = 6.753/0.30 = 22.510 grams (Add to mix water).

Adjustment to mix water content = 0.7 x 22.510 = 15.75 grams (removed from mix water)

Sample Calculation to Determine the Dosage of 30% Lithium Nitrate Solution Required to Arrive at a Desired Li/Na Molar Ratio in Soak Solution

Desired Li/ Na ion molar ratio in Soak Solution = 0.74

Molecular weight of $LiNO_3$ = 69 grams

Amount of $LiNO_3$ salt required = Li/Na ratio X Molecular Weight of $LiNO_3$
= 0.74 X 69
= 51.06 grams

Amount of 30% $LiNO_3$ Solution Required for 1 Liter of Soak Solution = 51.06/0.3 = 170.2 grams

Volume of 30% $LiNO_3$ solution needed for 1 Liter of soak solution = 170.2 / 1.20 = 141.8 ml

To prepare one liter of soak solution with a Li/Na molar ratio of 0.74, use the following procedure:

Step 1 - Prepare a stock solution of 2N NaOH by adding 80 grams of NaOH pellets to 900 ml of deionized water and further diluting to prepare one liter of 2N NaOH solution

Step 2 – Add 500 ml of 2N NaOH solution to a one-liter flask

Step 3 – Add 141.8 ml of 30% solution of $LiNO_3$ to flask in Step 2

Step 4 – Dilute contents of flask to one-liter solution by adding deionized water

Sample Calculation to Determine the Dosage of 30% Lithium Nitrate Solution Required to Arrive at a Desired Li/K Molar Ratio in Soak Solution

Desired Li/ K ion molar ratio in Soak Solution = 0.19

Molecular weight of $LiNO_3$ = 69 grams

Molecular Weight of Potassium Acetate (CH_3COOK) = 98.15 grams / mole

Concentration of
Potassium Acetate
Deicer Solution = 1.0 molar = 98.15 grams / liter of solution

Number of Moles of
K^+ ions in 1.0 molar CH_3COOK
Solution = 1.0 moles
Amount of $LiNO_3$ salt
Required for Li/K = 0.19 = Li/K molar ratio x Mol. Wt. of $LiNO_3$ x Molar Concentration of K^+

= 0.19 x 69 x 1.0 = 13.11 grams

Amount of 30% $LiNO_3$ Solution
Required for 1 Liter of Soak
Solution = 13.11 / 0.3 = 43.7 grams

Volume of 30% $LiNO_3$ solution
needed for 1 Liter of soak
solution = 43.7 / 1.20 = 36.4 ml

To prepare one liter of soak solution with a Li/K molar ratio of 0.19 with a 1.0 molar concentration of potassium acetate, use the following procedure:

Step 1 – Add 196.3 grams of 50% potassium acetate deicer solution (6.4 molar concentration) to an empty 1000 ml flask. This volume of solution contains 1 mole of CH_3COOK.

Step 2 – Add 109.25 ml of 30% $LiNO_3$ solution to the contents of the 1000 ml flask.

Step 3 – Dilute the contents of the flask by adding deionized water to make a solution of 1000 ml. This solution contains 1 mole of K^+ ions and 0.19 mole of Li^+ ions.

Table 11 shows the composition of different lithium nitrate blended potassium acetate deicer solution employed as soak solutions in the modified ASTM C 1567 tests.

TABLE 11 COMPOSITION OF LITHIUM NITRATE BLENDED POTASSIUM ACETATE DEICER SOLUTIONS

Li/K Molar Ratio	Mass of 30% Lithium Nitrate Solution and 50% Potassium Acetate Solution in One liter of Blended Soak Solution (Grams)					
	Potassium Acetate Deicer Solution Concentration					
	1 molar		2 molar		3 molar	
	$LiNO_3$ Solution (g)	KAc Deicer (g)	$LiNO_3$ Solution (g)	KAc Deicer (g)	$LiNO_3$ Solution (g)	KAc Deicer (g)
0.19	43.7	196.3	87.4	392.6	131.1	588.9
0.74	170.2	196.3	340.4	392.6	510.6	588.9

In addition to the soak solutions shown in Table 11, a more concentrated lithium nitrate blended potassium acetate soak solution was prepared by adding lithium nitrate salt to a 6.4 molar solution of potassium acetate such that a Li/K molar ratio of 0.19 is achieved. However, in this blend the maximum possible concentration of potassium acetate that is achievable was 6.15 molar. In preparing a soak solution with Li/K molar ratio of 0.19, 83.9 grams of lithium nitrate salt was mixed with 965 ml of 50% potassium acetate deicer solution to prepare a 1000 ml solution.

4. RESULTS FROM TESTS TO EVALUATE ASR MITIGATION POTENTIAL OF SUPPLEMENTARY CEMENTING MATERIALS

This section presents results from the following tests conducted on both reactive and non-reactive aggregates:

(i) Standard ASTM C 1260 test
(ii) Modified ASTM C 1260 test
(iii) Standard ASTM C 1567 test
(iv) Modified ASTM C 1567 test

The specific results presented from each of these tests include:

1. Length-change behavior of mortar bars
2. Dynamic modulus of elasticity of mortar bars
3. Microstructure of selected mortar bars

In these tests, mortar bar expansions over 0.1% at 14 days of exposure to 1N sodium hydroxide solution or potassium acetate deicer solution is considered to represent a deleterious level of expansion.

4.1. LENGTH-CHANGE IN MORTAR BARS

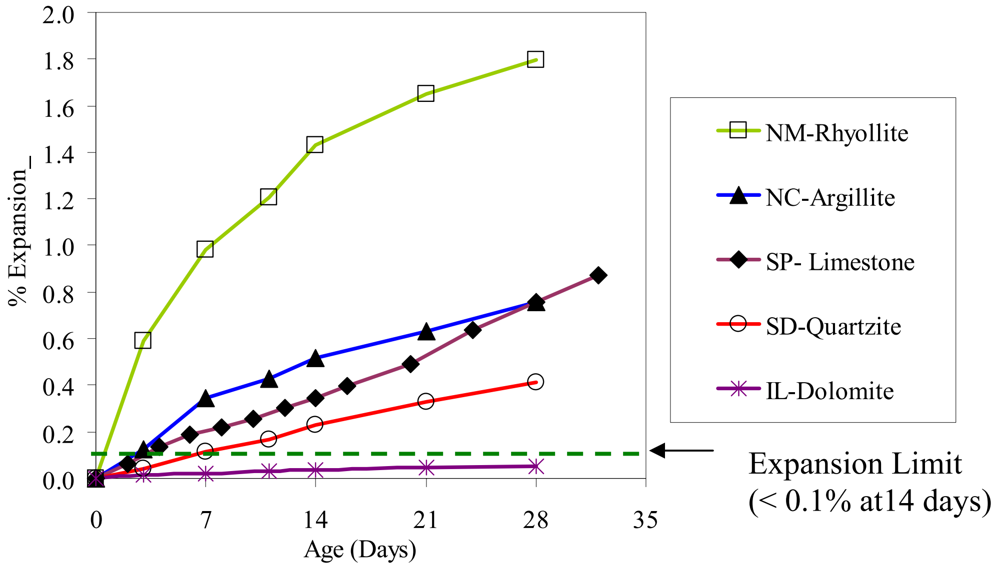
4.1.1. Standard ASTM C 1260 Test

Figure 3 shows expansion behavior of mortar bars containing reactive aggregates, including Spratt limestone, NC argillite, SD quartzite and NM rhyolite aggregates, and non-reactive IL dolomite in the standard ASTM C 1260 test.

From these results, it is apparent that mortar bars prepared with each of the four reactive aggregates showed expansion above 0.1% at 14 days of exposure to 1N sodium hydroxide solution, indicating highly reactive nature of the aggregates. In contrast, the non-reactive IL dolomite showed very little expansion. It was also observed from these results that mortar bars with the reactive aggregates showed continued expansion well beyond 14 days at a rate that is comparable to that observed before 14 days, while mortar bar with non-reactive IL dolomite showed no such tendency.

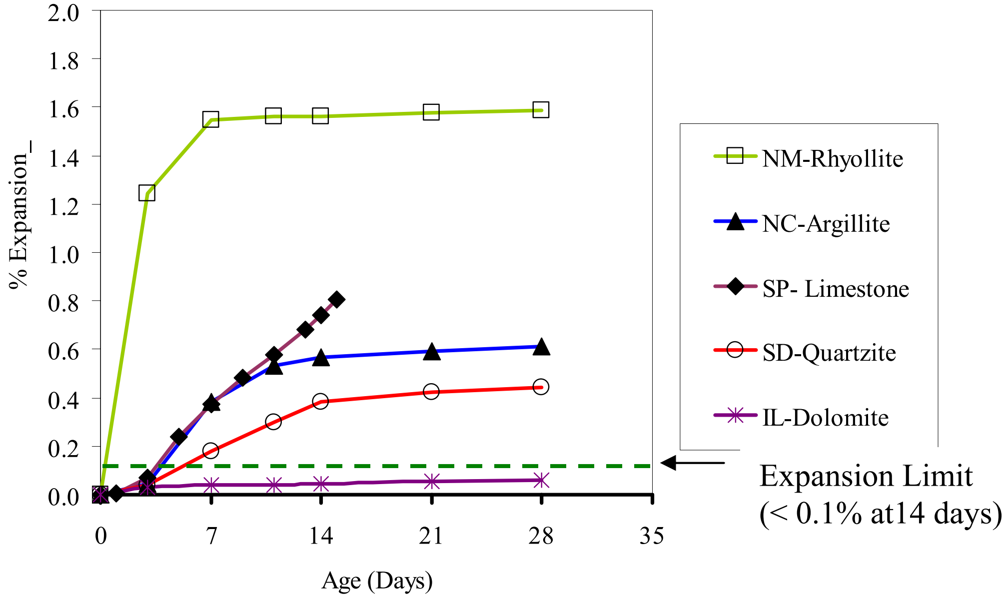
4.1.2. Modified ASTM C 1260 Test

Figure 4 shows the expansion behavior of mortar bars prepared with each of the reactive aggregates and the non-reactive aggregate in the modified ASTM C 1260 test. Results from this test reveal that all the mortar bars with reactive aggregates showed considerable expansion (> 0.1% at 14 days of exposure to soak solution) in potassium acetate deicer solution, while the non-reactive IL dolomite aggregate showed little expansion.



Expansion Limit
(< 0.1% at14 days)

FIGURE 3. EXPANSION OF MORTAR BARS IN THE STANDARD ASTM C 1260 TEST (1N NaOH SOAK SOLUTION)



Expansion Limit
(< 0.1% at14 days)

FIGURE 4. EXPANSION OF MORTAR BARS IN THE MODIFIED ASTM C 1260 TEST (KAc DEICER SOAK SOLUTION)

By comparing the results from the standard and the modified ASTM C 1260 tests in figures 3 and 4, it is evident that mortar bars with reactive aggregates showed higher levels of expansions in the presence of potassium acetate deicer solution than 1N sodium hydroxide solution at 14 days. This indicates that the aggregate reactivity as characterized by the standard ASTM C 1260 test may not be very representative in situations where exposure to potassium acetate deicer solutions is to be expected.

4.1.3 Standard and Modified ASTM C 1567 Tests to Investigate Effectiveness of Fly Ashes

In this section results from the standard and the modified ASTM C 1567 tests are presented for mixtures that contain low-lime, intermediate-lime and high-lime fly ashes at 15%, 25% and 35% cement replacement levels. The results from these tests are presented for each of the four reactive aggregates and one non-reactive aggregate evaluated in this study.

While expansion of mortar bars at 14 days of exposure to soak solution is typically used as a gauge in assessing aggregate reactivity or mitigation potential of a supplementary cementing material, in this investigation expansion data was gathered up to 56 days to observe if later age trends in the expansion reveal any meaningful insight into the interactions with the deicer solutions.

4.1.3.1. Spratt Limestone

Figures 5A and 5B show the expansion of Spratt limestone mortar bars with fly ashes in the standard and the modified ASTM C 1567 tests, respectively. In addition, the expansion behavior of mortar bars with no fly ash (control) is also shown in both figures.

It is evident from figure 5A that the effectiveness of fly ashes in mitigating the expansion in the mortar bars depends on the composition of the specific fly ash used, particularly its lime content, and its dosage level. Low-lime and intermediate-lime fly ashes showed significant potential to mitigate expansions in the mortar bars at all the dosage levels evaluated. In contrast, high-lime fly ash was ineffective at all dosage levels in mitigating expansions in the mortar bars.

Another trend that is evident from the data shown in figure 5A is that the expansion in the mortar bars decreased with increasing levels of fly ash, regardless of the chemical composition of the fly ash. While certain combinations of fly ash type and dosage levels were successful in mitigating expansion at 14 days to below 0.1% (for instance 25% and 35% replacement levels of low-lime and intermediate-lime fly ashes), expansion at 28 days was over 0.1% for all combinations of fly ash types and dosages evaluated in this study. In particular, mortar bars containing low-lime and intermediate-lime fly ashes show an increase in the rate of expansion beyond 21 days. High-lime fly ash did not show any beneficial effect at each of the three dosage levels considered in this study, at either 14 days or 28 days of exposure to 1N sodium hydroxide solution.

Results from the modified ASTM C 1567 tests revealed similar trends as those observed in the standard ASTM C 1567 tests. Compared to control specimens, mortar bars containing low-lime and intermediate-lime fly ashes showed considerably less expansion in presence of potassium acetate deicer solution at all dosage levels, in particular at 25% and 35% replacement levels. In contrast, high-lime fly ash was not effective in mitigating expansion in mortar bars exposed to potassium acetate deicer, at all the three dosage levels evaluated in this study.

While the trends of mitigation offered by low-lime and intermediate lime fly ash in presence of potassium acetate deicer solution were similar to that observed in presence of 1N sodium hydroxide solution at early ages, the later age expansion in mortar bars (i.e. > 14 days) exposed to potassium acetate deicer solution were significantly lower than those observed in mortar bars exposed to presence of 1N sodium hydroxide solution. This suggests that low-lime and intermediate lime fly ash at 25% and 35% dosage level were more effective in suppressing mortar bar expansions in potassium acetate deicer solution compared to 1N sodium hydroxide soak solution.

Another trend that is evident from comparing the standard and the modified ASTM C 1567 tests is that at lower dosage levels of fly ashes, the expansion of mortar bars in potassium acetate deicer solution is considerably greater than that observed in mortar bars exposed to 1N sodium hydroxide solution. Similar differences in expansion behavior were observed in control mortar bars (i.e. without fly ash) exposed to potassium acetate deicer solution and 1N sodium hydroxide solution (see Figures 3 and 4).

4.1.3.2. NM Rhyolite

Figures 6A and 6B show the expansion of mortar bars containing NM Rhyolite aggregate and fly ashes in 1N sodium hydroxide and potassium acetate deicer soak solutions in the standard and the modified ASTM C 1567 tests, respectively. The expansion of mortar bars with no fly ash (control mortar) is also in these figures for reference.

Based on the data from the standard and the modified ASTM C 1567 tests shown in figure 6, only low-lime fly ash at 35% dosage appeared to have reduced expansion in mortar bars to less than 0.1% at both 14 days and 28 days of exposure to soak solution. Although, the mixture containing 25% fly ash was effective in suppressing the expansion in mortar bars up to 14 days in potassium acetate deicer solution, significant jump in expansion occurred at later ages. It is not clear at this time, the reason for the sudden increase in expansion of mortar bars containing low-lime fly ash beyond 14 days. Similar behavior was observed in case of intermediate-lime fly ash at 7 days.

With mortar bars containing high-lime fly ash very high levels of expansion were observed at very early ages in both 1N sodium hydroxide and potassium acetate soak solutions. In particular, the expansion of mortar bars containing 15% and 25% high lime fly ash and soaked in potassium acetate deicer solution (see figure 6B) exceeded that of the control mortar bars (i.e. without any fly ash).

Comparing the results from the standard and the modified ASTM C 1567 tests, it appears that low-lime fly ash at 35% dosage level is more effective in mitigating expansions in mortar bars exposed to potassium acetate deicer solution than those exposed to 1N sodium hydroxide solution. This trend with NM rhyolite mortar bars is similar to that observed with Spratt limestone mortar bars.

Another trend that is apparent from observing mortar bar expansions in figures 6A and 6B is that, at lower dosage levels of fly ash, the rate of expansion in mortar bars exposed to potassium acetate deicer solution in considerably greater than that observed in mortar bars exposed to 1N sodium hydroxide solution.

4.1.3.3. NC Argillite

Figures 7A and 7B show the expansion of mortar bars with North Carolina argillite with each of the three fly ashes at different dosage levels in the standard and modified ASTM C 1567 test methods, respectively. In addition, the expansion of control mortar bars is shown in these figures to provide a reference for the level of mitigation offered by the fly ashes.

It is evident from figures 7A and 7B that low-lime fly ash was effective in mitigating expansions in mortar bars exposed to 1N sodium hydroxide and potassium acetate deicer solutions at dosage levels of 25% and 35%. Low-lime fly ash was also effective in mitigating expansion in mortars exposed to potassium acetate deicer solution at 15% dosage level. Intermediate-lime fly ash was not effective in mitigating expansions in either 1N sodium hydroxide or potassium acetate deicer solutions at 15% dosage level. However, it was very effective at 25% and 35% dosage levels. High-lime fly ash was only effective in mitigating expansion in mortar bars exposed to potassium acetate deicer solution at 35% dosage level.

Results from the modified ASTM C 1567 tests also revealed that mortar bars with high-lime fly ash at 15% and 25% dosage levels expanded more than the control mortar bars. Similar behavior was observed with mortar bars containing NM rhyolite aggregate.

4.1.3.4. SD Quartzite

Figures 8A and 8B show the expansion of mortar bars containing SD quartzite in the standard and modified ASTM C 1567 tests, respectively.

The low-lime and the intermediate-lime fly ashes appear to be very effective in mitigating expansions in mortar bar exposed to potassium acetate deicer solutions and 1N sodium hydroxide solution at fly ash dosage levels of 25% and 35%. However, it is observed that the mitigation effect of these fly ashes at 25% and 35% dosage level is more pronounced and effective at later ages in mortar bars exposed to potassium acetate deicer solution than those exposed to 1N sodium hydroxide solution. In contrast, high-lime fly ash was ineffective in mitigation expansions of mortar bars exposed to 1N sodium hydroxide and potassium acetate deicer solution at all the replacement levels evaluated.

Mortar bars with high-lime fly ash exposed to potassium acetate deicer solution showed higher levels of expansion than those exposed to 1N sodium hydroxide solution, at all dosage levels. Also, it was observed that mortar bars with high-lime fly ash at 15% and 25% dosage levels showed more expansion than control mortar bars, when exposed to potassium acetate deicer solution. This behavior is similar to that observed with mortar bars containing NM rhyolite and NC argillite aggregate.

It should also be noted that in all the tests with potassium acetate deicer soak solution, the mortar bars containing SD quartzite reached an ultimate expansion in a finite time and thereafter showed no further expansion. In contrast, mortar bars soaked in 1N sodium hydroxide solution showed gradual increase in expansion even at later ages. Similar behavior was observed with mortar bars containing NM rhyolite and NC argillite aggregates.

4.1.3.5. IL Dolomite (Reference Non-Reactive Aggregate)

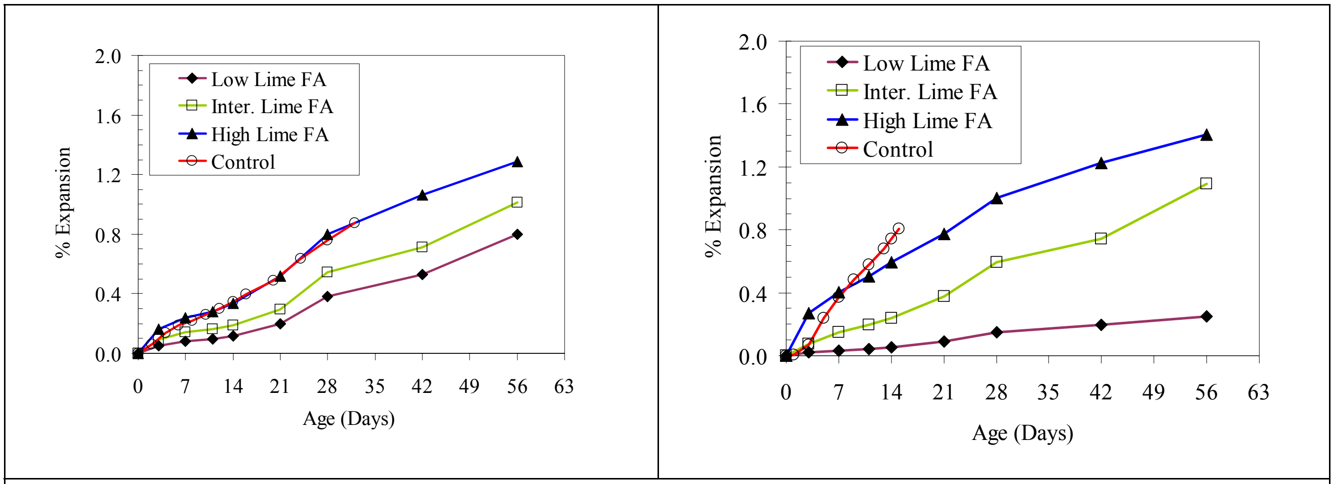
In this investigation mortar bars prepared with IL dolomite in combination with each of the three fly ashes at 25% dosage level were subjected to standard and modified ASTM C 1567 test. Other fly ash dosages (i.e. 15% and 35%) were not investigated for this aggregate.

Figures 9A and 9B show the expansion of mortar bars containing IL dolomite with each of the three fly ashes in 1N sodium hydroxide and potassium acetate deicer soak solutions, respectively.

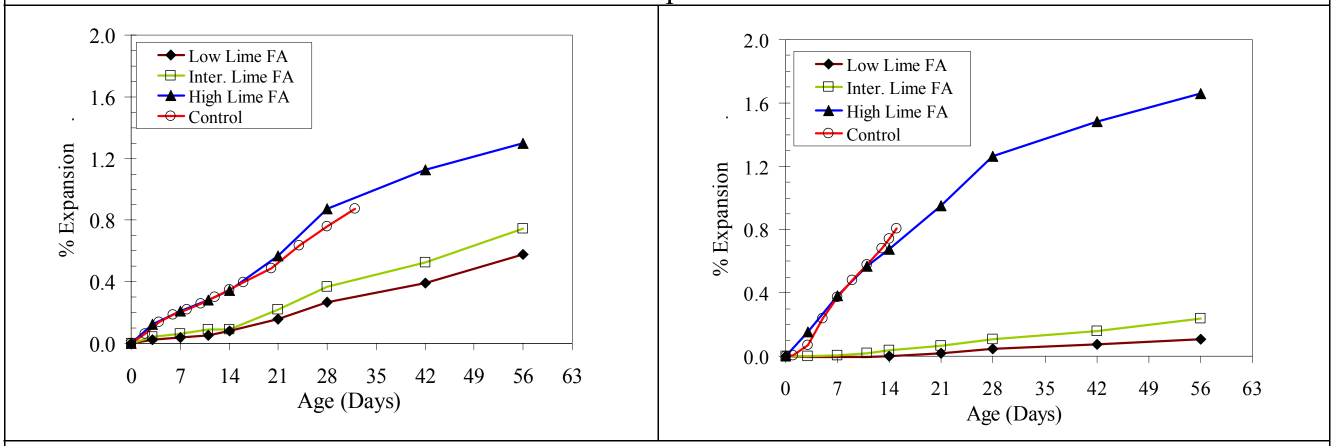
Modified ASTM C 1567 tests on IL dolomite mortar bars containing low-lime and intermediate-lime fly ashes at 25% dosage level showed no significant expansion in either 1N sodium hydroxide solution or potassium acetate deicer solution. While this result is to be expected with a non-reactive aggregate such as IL dolomite, these tests were conducted to determine if any deleterious interactions occurred between fly ashes and deicer solutions.

Based on the results from the modified ASTM C 1567 tests, mortar bars with low-lime and intermediate-lime fly ash showed no deleterious interactions when exposed to potassium acetate deicer solution. In contrast, mortar bars containing high-lime fly ash showed higher levels of expansion in potassium acetate deicer solution (> 0.11% at 14 days and 0.13% at 28 days) compared to control mortar bars.

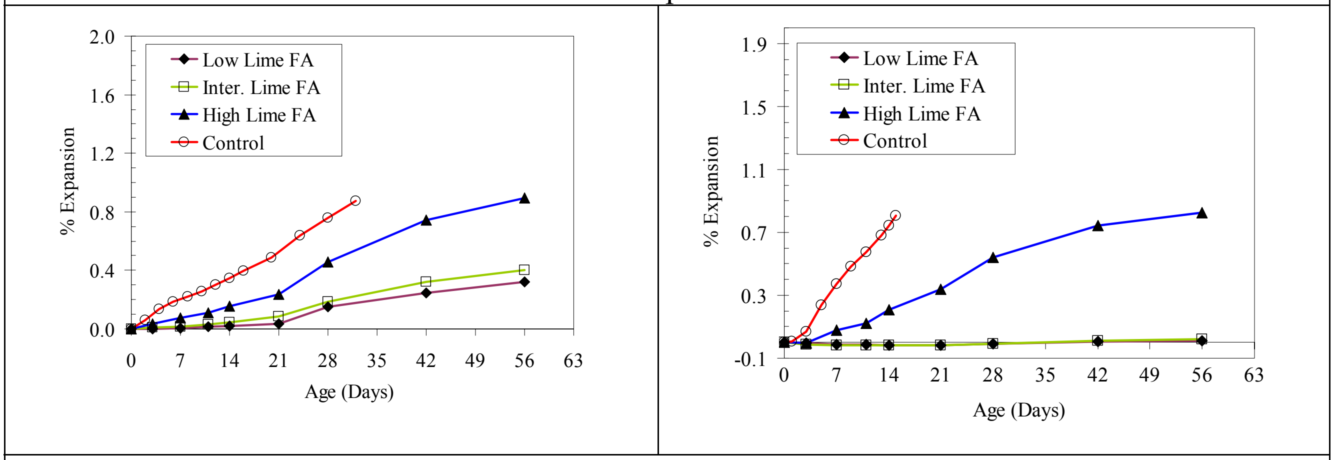
Based on the consistent evidence of higher expansion in mortar bars with high-lime fly ashes compared to control mortar bars in the modified ASTM C 1567 tests, it appears likely that the high-lime fly ash may be participating in some deleterious interactions with the potassium acetate deicer solution. Additional research is needed to isolate the mechanisms responsible for this negative interaction between high-lime fly ashes and potassium acetate deicer solution.



15% Cement Replacement Level



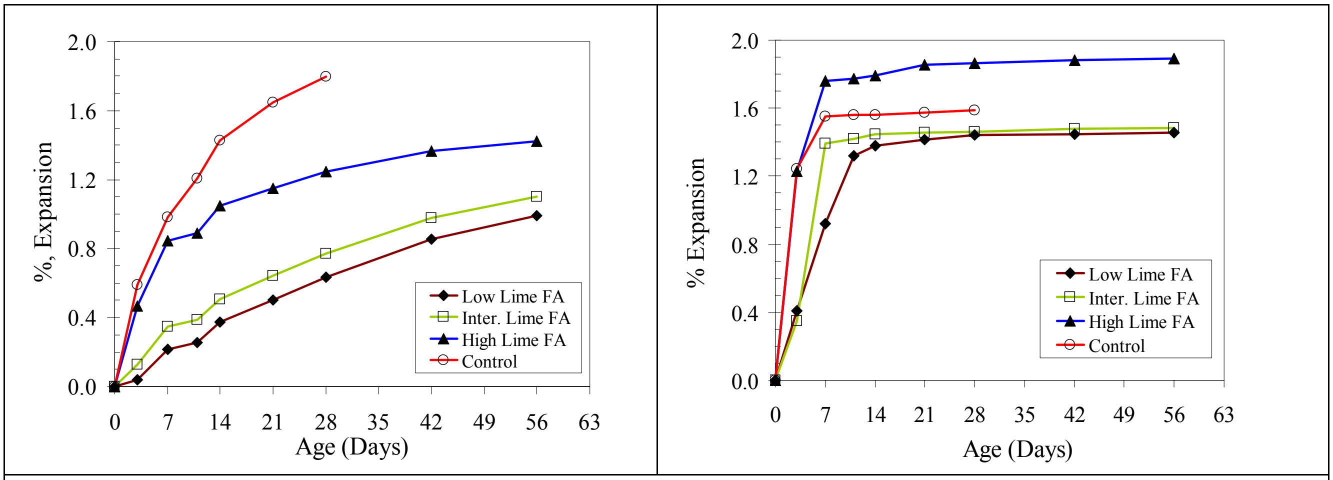
25% Cement Replacement Level



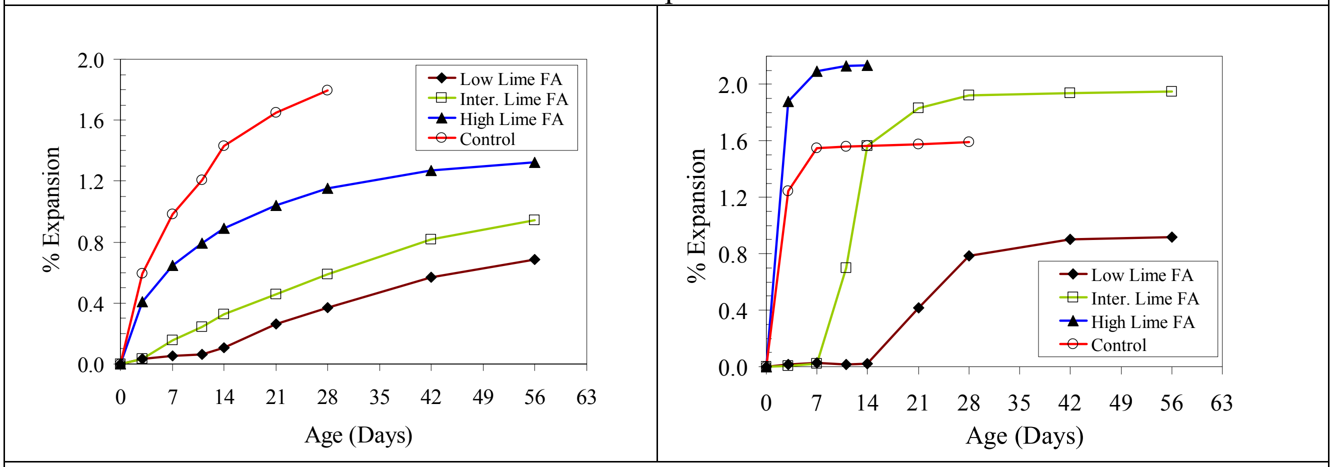
35% Cement Replacement Level

Fig. 5A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)	**Fig. 5B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)**

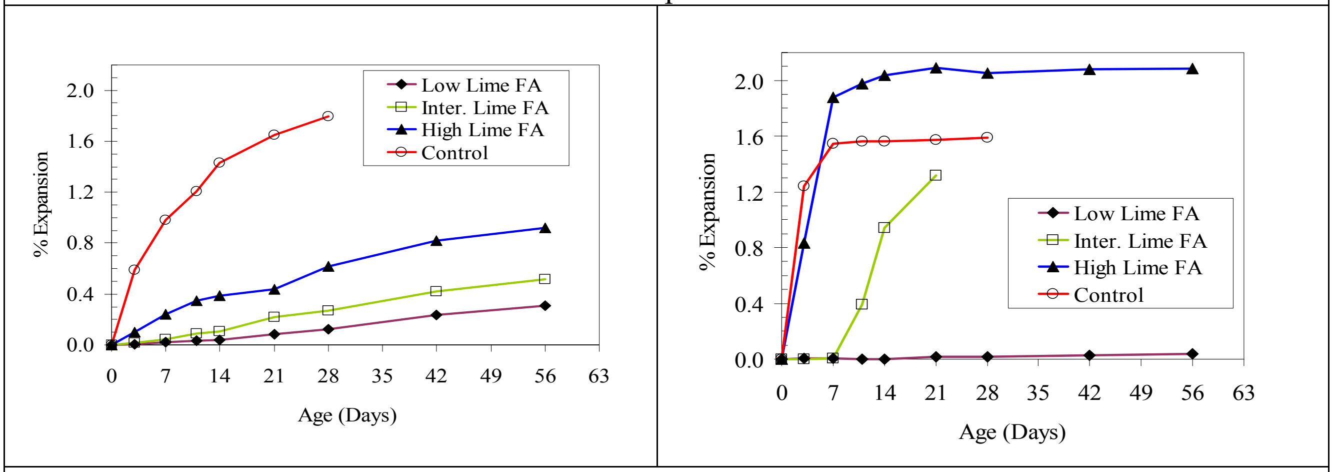
FIGURE 5. EXPANSION OF MORTAR BARS CONTAINING SPRATT LIMESTONE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH FLY ASHES AT 15%, 25% AND 35% CEMENT REPLACEMENT LEVEL



15% Cement Replacement Level



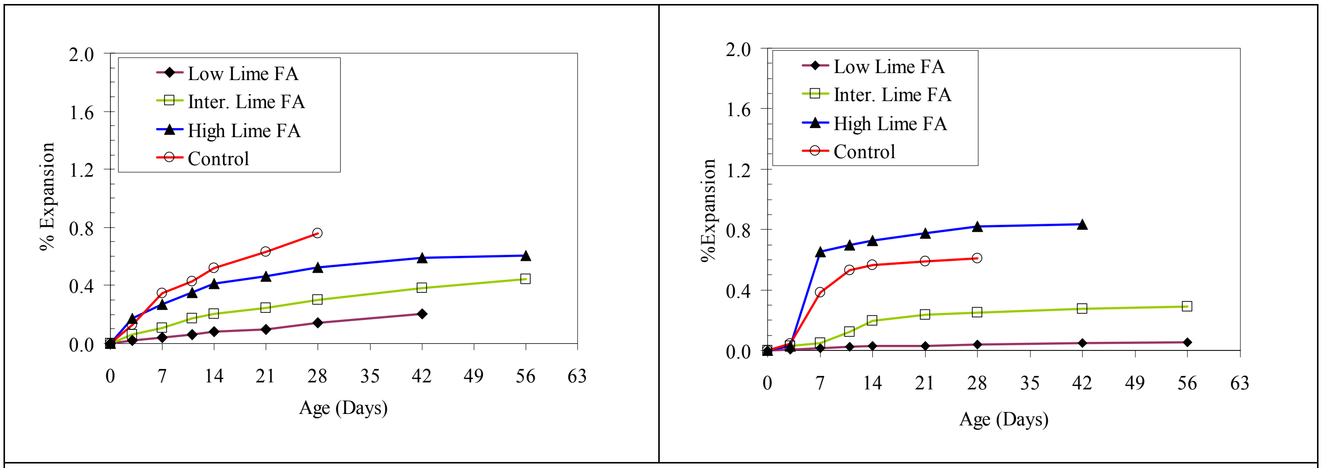
25% Cement Replacement Level



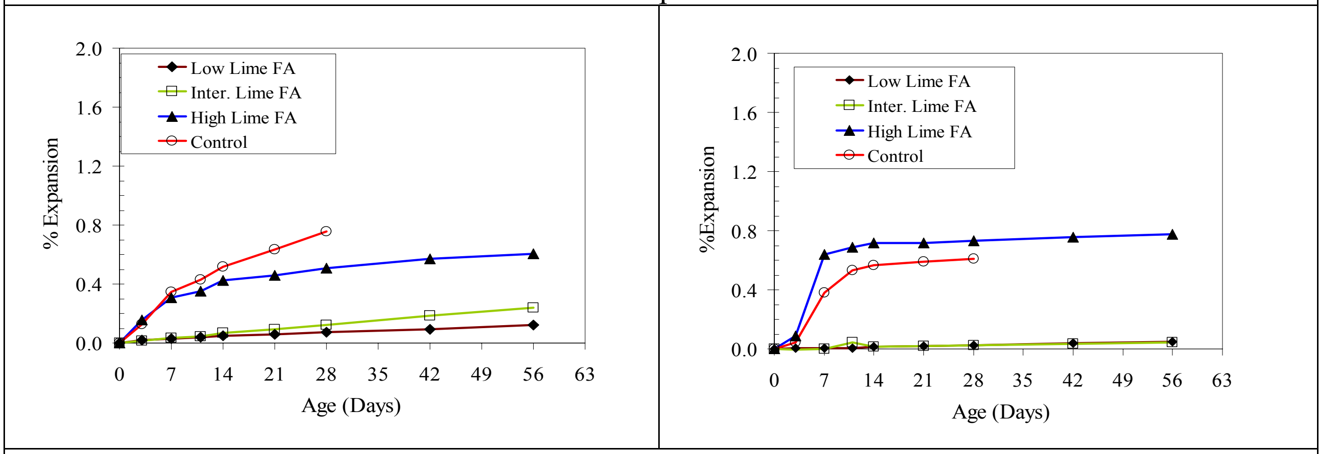
35% Cement Replacement Level

Fig. 6A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)	**Fig. 6B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)**

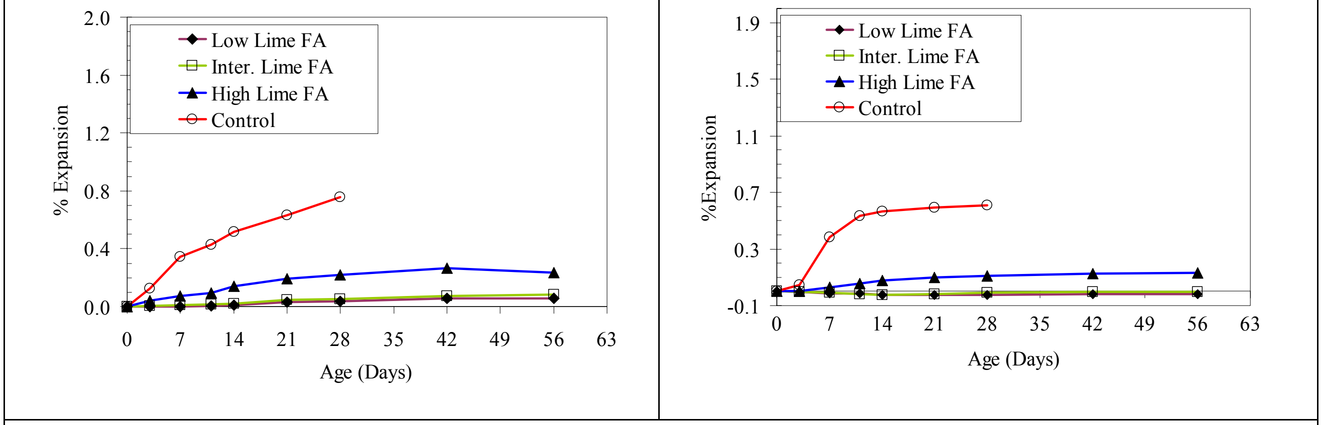
FIGURE 6. EXPANSION OF MORTAR BARS CONTAINING NM RHYOLITE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH FLY ASHES AT 15%, 25% AND 35% CEMENT REPLACEMENT LEVEL



15% Cement Replacement Level



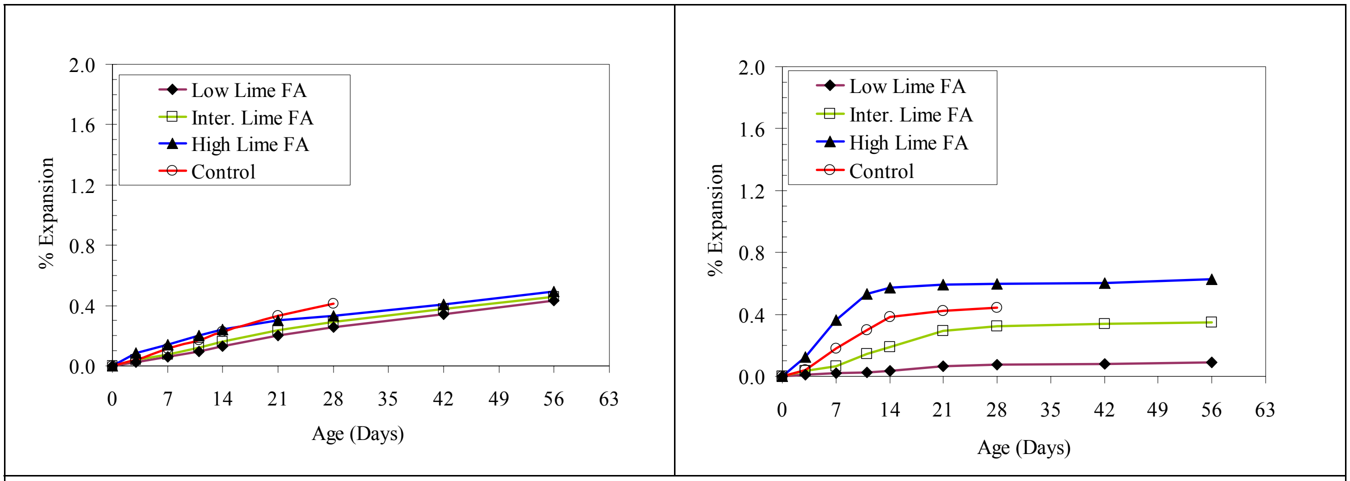
25% Cement Replacement Level



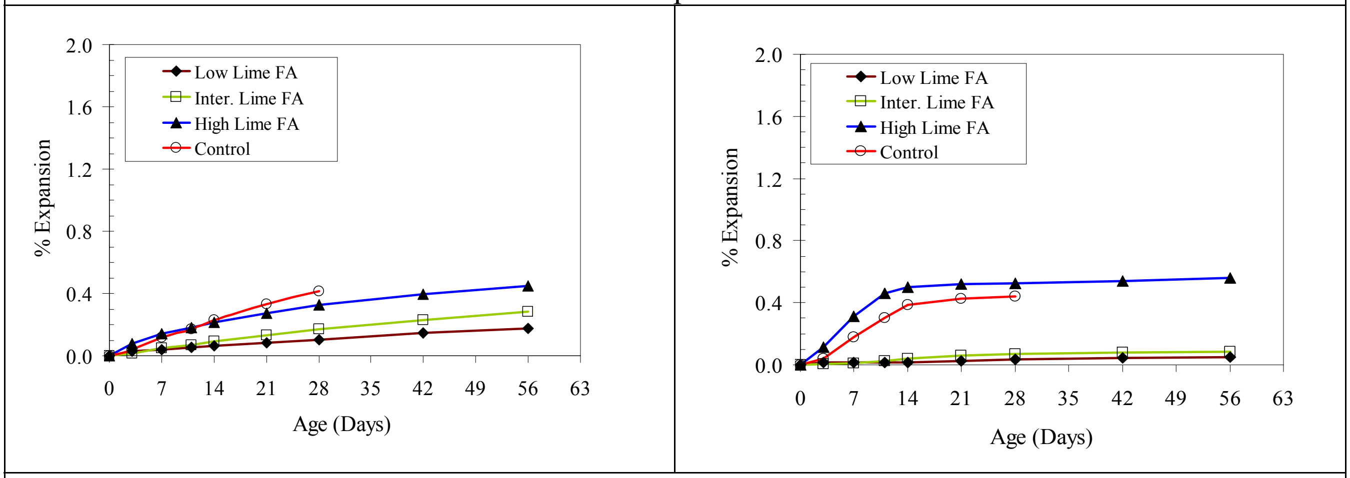
35% Cement Replacement Level

Fig. 7A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)	**Fig. 7B Modified ASTM C 1567 Tests (50% KAc Soak Solution)**

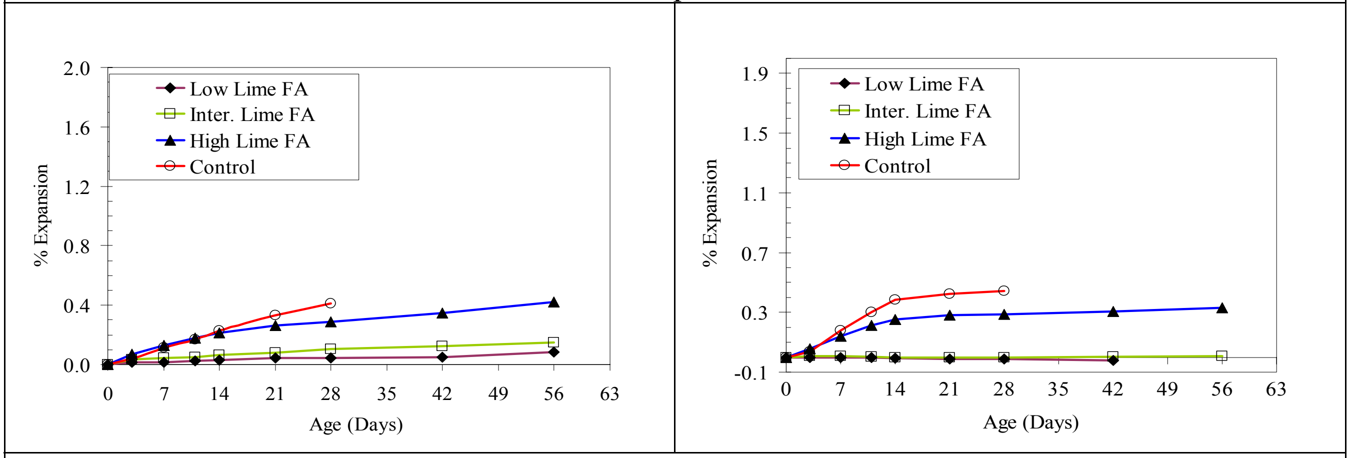
FIGURE 7. EXPANSION OF MORTAR BARS CONTAINING NC ARGILLITE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH FLY ASHES AT 15%, 25% AND 35% CEMENT REPLACEMENT LEVEL



15% Cement Replacement Level



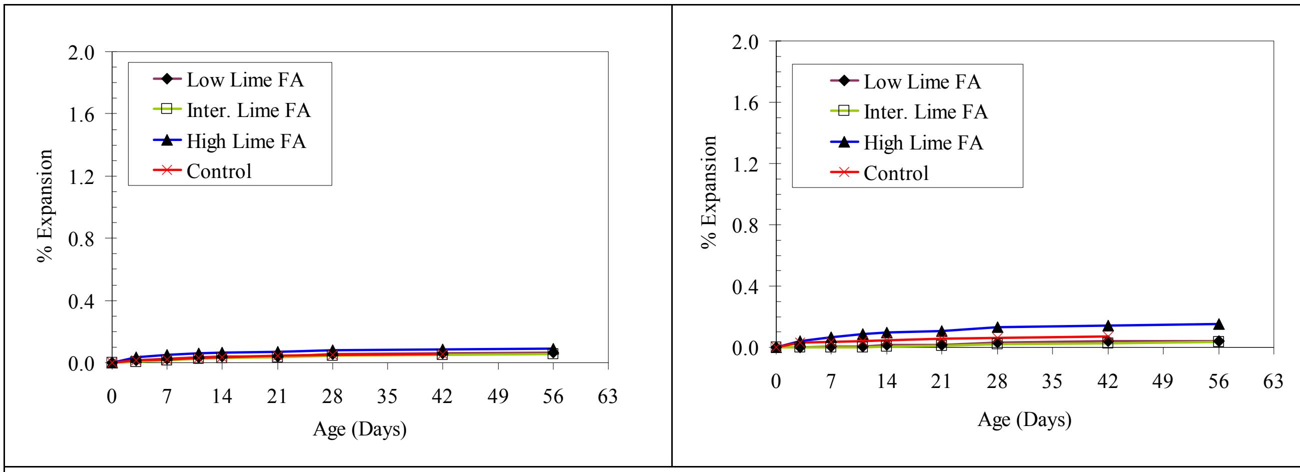
25% Cement Replacement Level



35% Cement Replacement Level

Fig. 8A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)	**Fig. 8B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)**

FIGURE 8. EXPANSION OF MORTAR BARS CONTAINING SD QUARTZITE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH FLY ASHES AT 15%, 25% AND 35% CEMENT REPLACEMENT LEVEL



25% Cement Replacement Level

Fig. 9A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)

Fig. 9B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)

FIGURE 9. EXPANSION OF MORTAR BARS CONTAINING IL DOLOMITE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH FLY ASHES AT 25% CEMENT REPLACEMENT LEVEL

4.1.4 Standard and Modified ASTM C 1567 Tests to Investigate Effectiveness of Slag

In this section results from the standard and the modified ASTM C 1567 tests are presented for mixtures that contain ground granulated blast furnace slag (slag) at 40% and 50% cement replacement levels. The results from these tests are presented for each of the four reactive aggregates. Comparative studies with the non-reactive IL dolomite aggregate were not conducted in this investigation. In these studies expansion measurements were taken up to 56 days.

In this study, a mortar bar expansion over 0.1% at 14 days was considered to represent an ineffective ASR mitigation alternative. While expansion at 14 day was considered as a benchmark, expansion behavior of mortar bars beyond 14 days was also monitored as a precautionary measure to reflect on the potential for deleterious interactions at later ages.

Although the role of slag in mitigating ASR is well established in literature, preliminary results from this investigation suggested that 40% slag dosage was not effective in mitigating expansions in mortar bars, particularly in presence of 50% potassium acetate deicer solution.

In an effort to assure repeatability of the results obtained in this investigation, the standard and the modified ASTM C 1567 tests were repeated on selected mixtures (Spratt limestone) containing 40% slag. Results from the recast samples are also presented in this section.

4.1.4.1. Spratt Limestone

Figures 10A and 10B show expansion behavior of Spratt limestone mortar bars containing slag at 40% and 50% cement replacement level in the standard and the modified ASTM C 1567 tests, respectively. In these figures the behavior of mortar bars containing slag is compared with that of the control specimens (with no slag).

The results from the standard ASTM C 1567 test in figure 10A show that slag in mortar bars at 40% and 50% cement replacement levels is able to significantly mitigate expansions at 14 days, compared to the control specimens. However, the increasing trend in the expansion of mortar bars beyond 14 days suggests that slag may not be effective in mitigating the effects of 1N NaOH soak solution at later ages. In fact, even at 56 days of exposure to 1N sodium hydroxide solution in the standard ASTM C 1567 test the mortar bars containing slag show a continued tendency to expand. Whether the expansion at ages beyond 14 days merits any consideration in reflecting the field performance is a question that is presently being debated.

Figure 10B reveals the behavior of Spratt limestone mortar bars containing slag in the modified ASTM C 1567 test. Compared to control specimen slag is able to significantly mitigate expansion at 40% and 50% cement replacement levels. However, at later ages Spratt limestone mortar bars continued to expand beyond 0.1%.

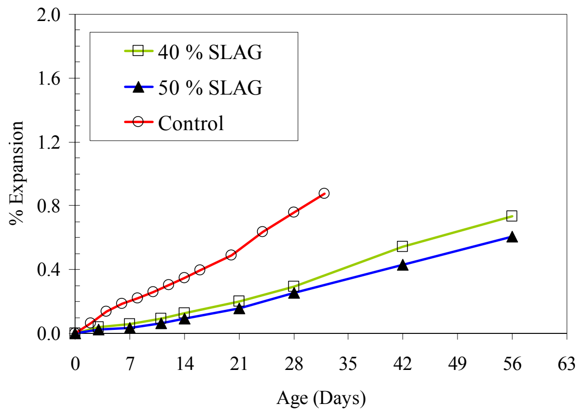


Fig. 10A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)

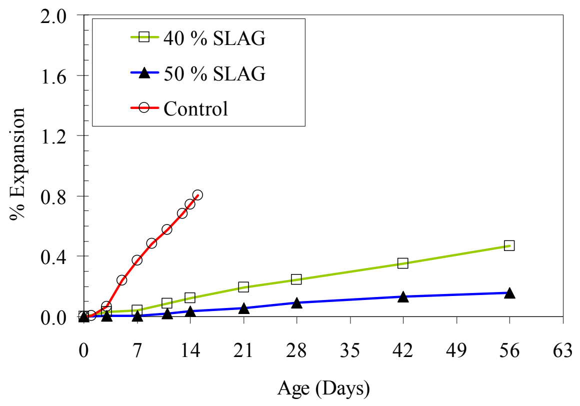


Fig. 10B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)

FIGURE 10. EXPANSION OF MORTAR BARS CONTAINING SPRATT LIMESTONE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH SLAG

4.1.4.2. NM Rhyolite

Figures 11A and 11B show expansion behavior of NM rhyolite mortar bars containing slag at 40% and 50% cement replacement level in the standard and modified ASTM C 1567 tests, respectively. In these Figures, the expansion behavior of NM rhyolite mortar bars without any slag is also presented for reference.

The results from the standard ASTM C 1567 test in figure 11A show that slag in mortar bars at 40% and 50% cement replacement levels is able to lower expansions at all ages compared to the control specimens. However, slag at either of the two replacement level is unable to completely mitigate expansions (i.e. below 0.1%) at 14 or 28 days in the standard ASTM C 1567 tests.

Figure 11B shows the behavior of NM rhyolite mortar bars containing slag in the modified ASTM C 1567 test. Compared to control specimen, slag is able to significantly mitigate expansion only at 50% cement replacement level. Slag at a 40% dosage level was not found to be effective.

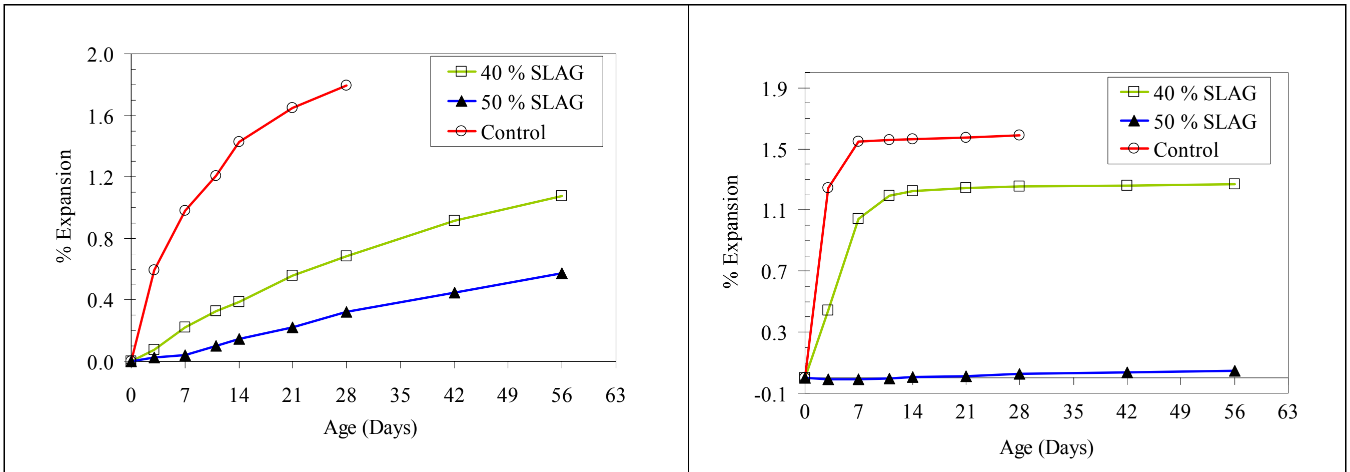


Fig. 11A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)

Fig. 11B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)

FIGURE 11. EXPANSION OF MORTAR BARS CONTAINING NM RHYOLITE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH SLAG

4.1.4.3. NC Argillite

Figures 12A and 12B show expansion behavior of NC argillite mortar bars containing slag at 40% and 50% cement replacement level in the standard and modified ASTM C 1567 tests, respectively. Results of standard and modified ASTM C 1260 tests with no slag (i.e. control) are also presented for reference.

The results from the standard ASTM C 1567 test in Figure 12A show that slag at 50% replacement level was able to mitigate expansions below 0.1% at 28 days compared to the control and 40% slag mortar bars. While mortar bars with 40% slag showed lower expansion than control mortar bars, the level of mitigation was not adequate at 28 days in presence of 1N NaOH soak solution.

Figure 12B shows the behavior of NC argillite mortar bars containing slag in the modified ASTM C 1567 test. Compared to control specimen, slag was able to mitigate expansion at both 40% and 50% cement replacement levels.

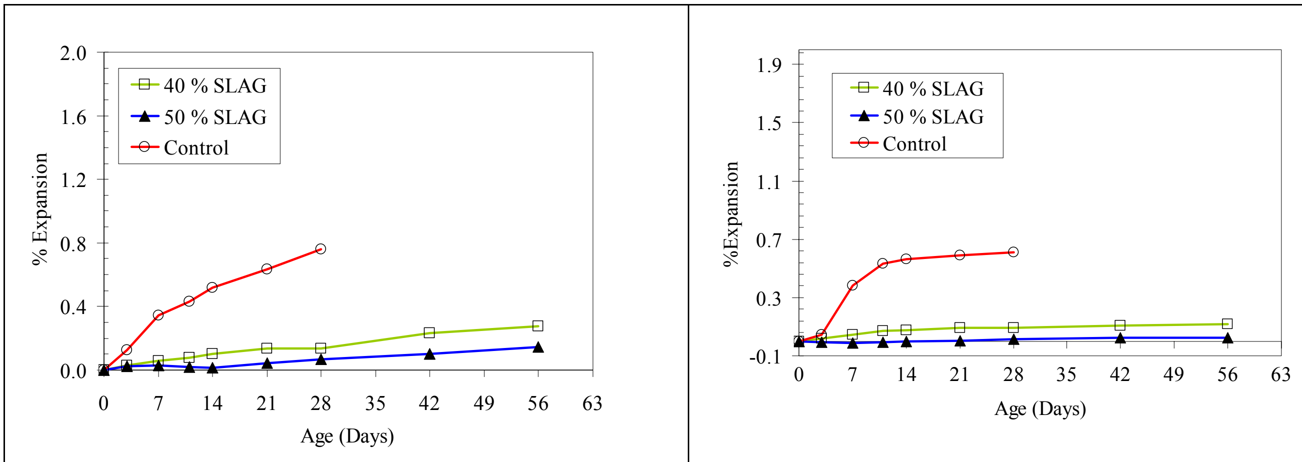


Fig. 12A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)

Fig. 12B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)

FIGURE 12. EXPANSION OF MORTAR BARS CONTAINING NC ARGILLITE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH SLAG

4.1.4.4. SD Quartzite

Figures 13A and 13B show expansion behavior of SD quartzite mortar bars containing slag at 40% and 50% cement replacement level in the standard and modified ASTM C 1567 tests, respectively. In addition, the results of standard and modified ASTM C 1260 tests with no slag (i.e. control) are also presented for reference.

The results from the standard ASTM C 1567 test in Figure 13A showed that increase in slag content of the mixture reduced the levels of expansion. However, even at 40% and 50% dosage levels slag was not able to mitigate expansions below 0.1% at 28 days.

Figure 13B shows the behavior of SD quartzite mortar bars containing slag in the modified ASTM C 1567 test. It is evident from the results that use of slag at 50% cement replacement level was able to adequately mitigate expansion in the modified ASTM C 1567 test. It should be noted that slag was not effective in mitigating expansions when used at 40% cement replacement level.

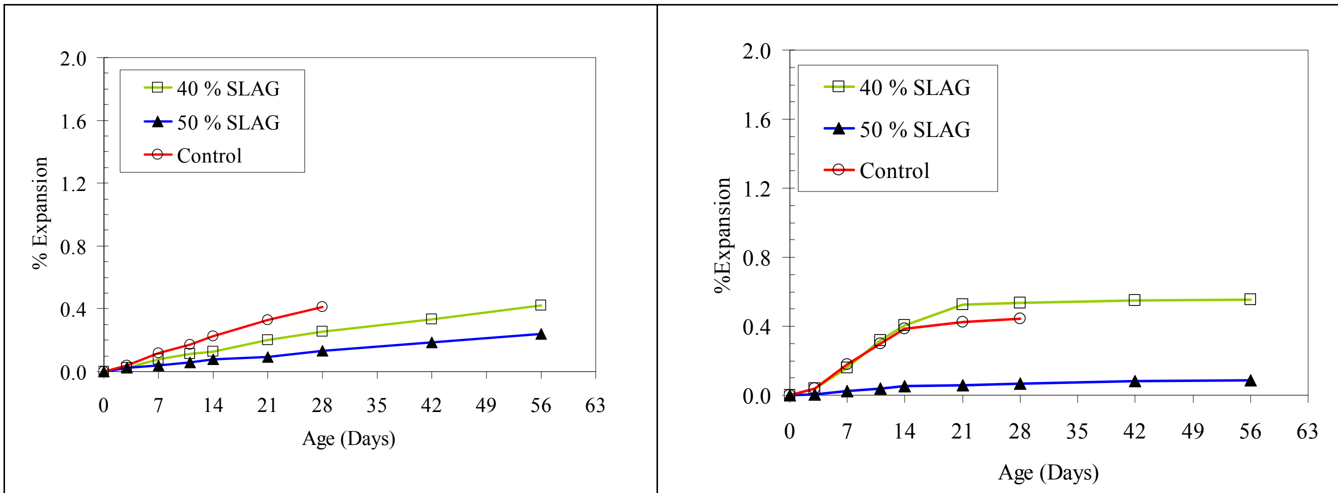


Fig. 13A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)

Fig. 13B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)

FIGURE 13. EXPANSION OF MORTAR BARS CONTAINING SD QUARTZITE AGGREGATE IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH SLAG

4.1.4.5. Recast Experiments

In an effort to study the repeatability of the results, modified ASTM C 1567 test was repeated on mortar bars prepared with Spratt limestone and 40% slag. The results from the original test and the recast test are shown in Figure 14.

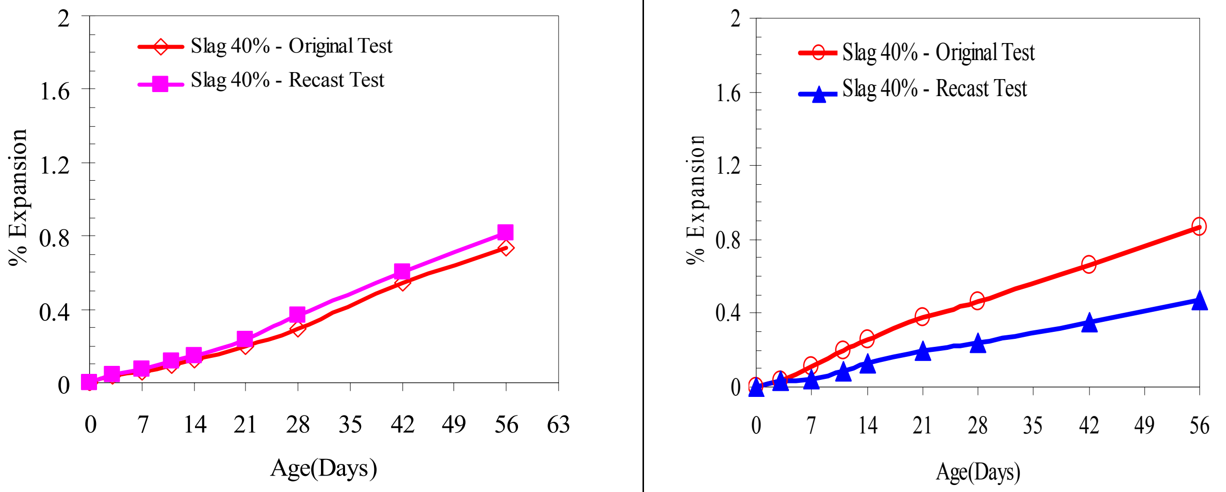


Fig. 14A Standard ASTM C 1567 Tests (1N NaOH Soak Solution)

Fig. 14B Modified ASTM C 1567 Tests (50% KAc Deicer Soak Solution)

FIGURE 14. REPEAT TESTING OF STANDARD AND MODIFIED ASTM C 1567 TESTS ON SPRATT LIMESTONE MORTAR BARS CONTAINING 40% SLAG

It can be observed from Figure 14A that the expansion of recast test specimens with 40% slag dosage is very similar to that of the original test specimens in case of the standard ASTM C 1567 test. However, in case of the modified ASTM C 1567 tests, expansions of the recast mortar bars were somewhat lower than those observed with the original test specimens. However, both the recast and the original test specimens exceeded 0.1% at 14 days and well over 0.2% at 28 days, thus failing to be effective in mitigating the deleterious expansions in presence of KAc deicer solution.

4.2 DYNAMIC MODULUS OF ELASTICITY (DME)

Figures 15 through 18 show the changes in dynamic modulus of elasticity of mortar bars for each of the four reactive aggregates containing the three fly ashes, in the standard (1N NaOH soak solution) and modified (50% KAc deicer soak solution) ASTM C 1567 test methods.

Based on the data presented in these Figures, it is evident that mortar bars containing high-lime fly ash showed a pronounced drop in DME values both in 1 N NaOH and potassium acetate deicer solutions for all the aggregates. This change in dynamic modulus was found to be consistent with the expansion behavior of mortar bars presented in previous Section.

Mortar bars prepared with Spratt limestone, NC argillite and SD quartzite and containing low-lime fly ash and intermediate-lime fly ash at 25% cement replacement level showed good performance in presence of KAc deicer solution compared to 1 N NaOH solution (for instance, DME values of NC argillite and SD Quartzite mortar bars at 28 days are 95% of the DME values at 0 days or greater). This behavior is consistent with the expansion data in the modified ASTM C 1260 test for the respective aggregate sources.

It should be noted that the 0 day and 3 day DME modulus data for Spratt limestone mortar bars was not available. However, it is evident that Spratt limestone mortar bars containing 25% low-lime fly ash performed satisfactorily.

NM rhyolite mortar bars containing low-lime and intermediate lime fly ashes showed a sudden drop in DME at 14 days and 7 days, respectively. The changes in DME of NM rhyolite mortars corresponded very well with the observed changes in expansion (see Figure 5).

Based on the DME data, it appears that low-lime and intermediate lime fly ashes are generally more effective in mitigating ASR induced by potassium acetate deicer compared to high-lime fly ashes.

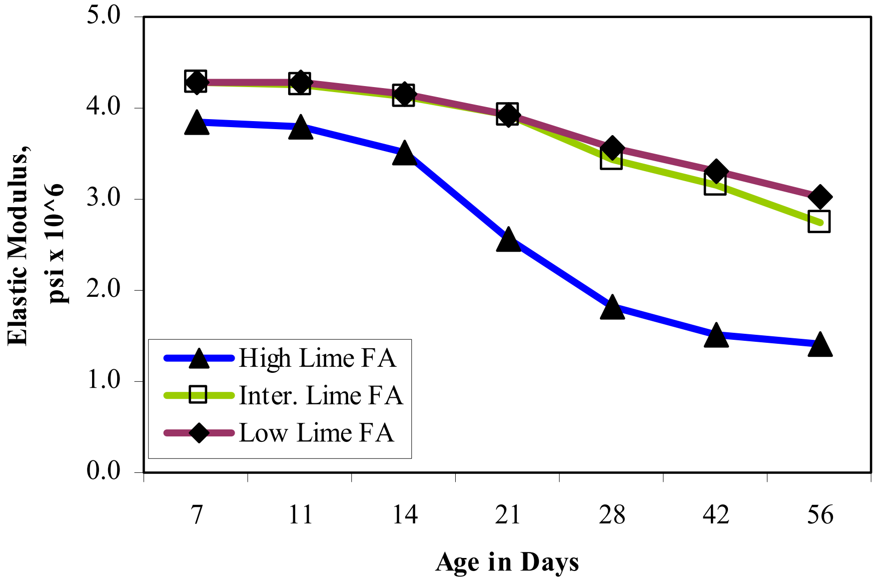


Fig. 15 A Standard ASTM C 1567 Test (1N NaOH Soak Solution)

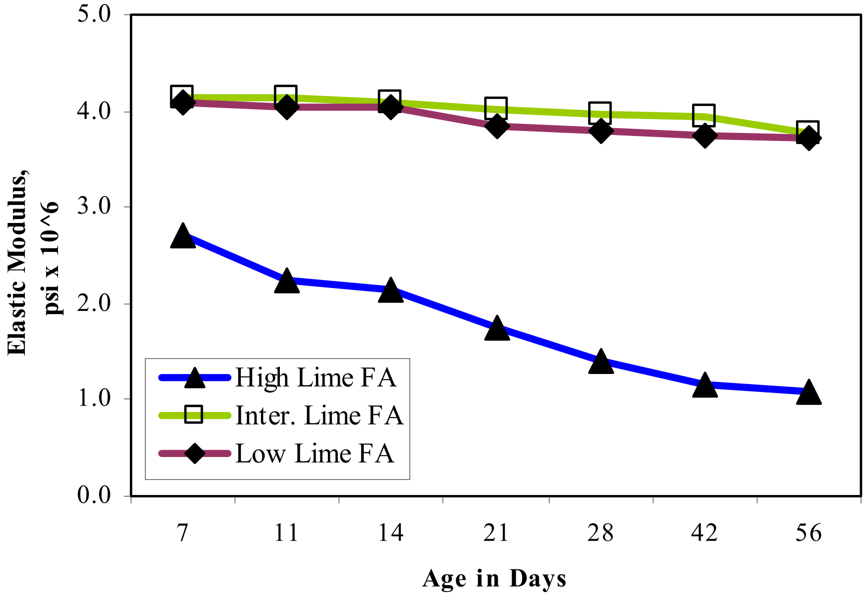


Fig. 15 B Modified ASTM C 1567 Test (50% KAc Deicer Solution)

FIGURE 15. CHANGES IN DYNAMIC MODULUS OF ELASTICITY OF SPRATT LIMESTONE MORTAR BARS IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH 25% FLY ASH DOSAGE

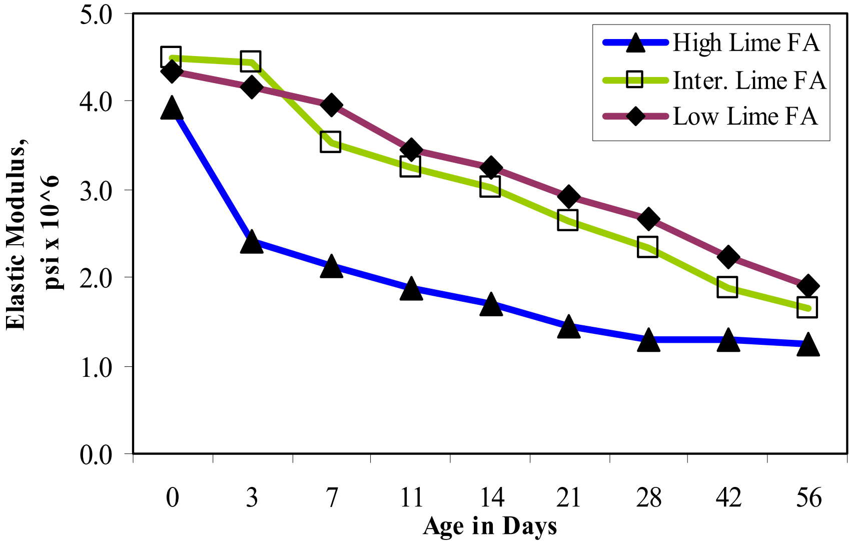


Fig. 16 A Standard ASTM C 1567 Test (1N NaOH Soak Solution)

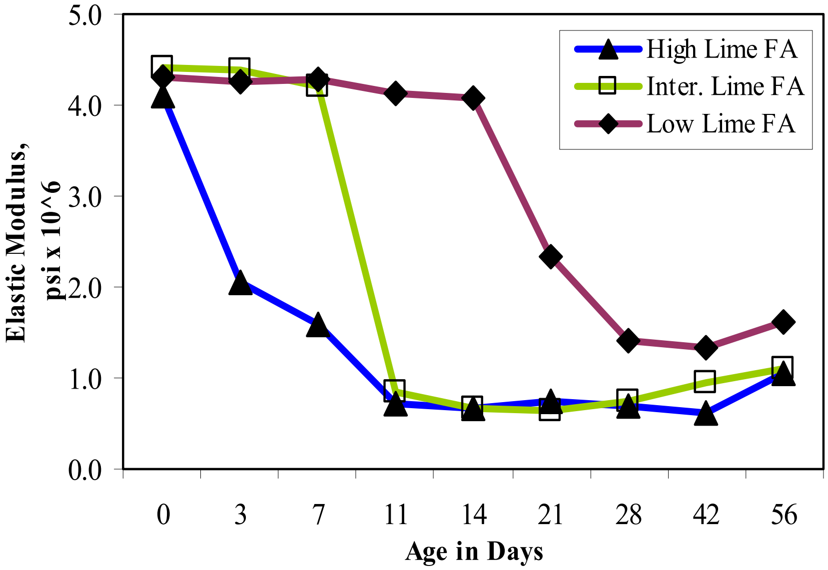


Fig. 16 B Modified ASTM C 1567 Test (50% KAc Deicer Solution)

FIGURE 16. CHANGES IN DYNAMIC MODULUS OF ELASTICITY OF NM RHYOLITE MORTAR BARS IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH 25% FLY ASH DOSAGE

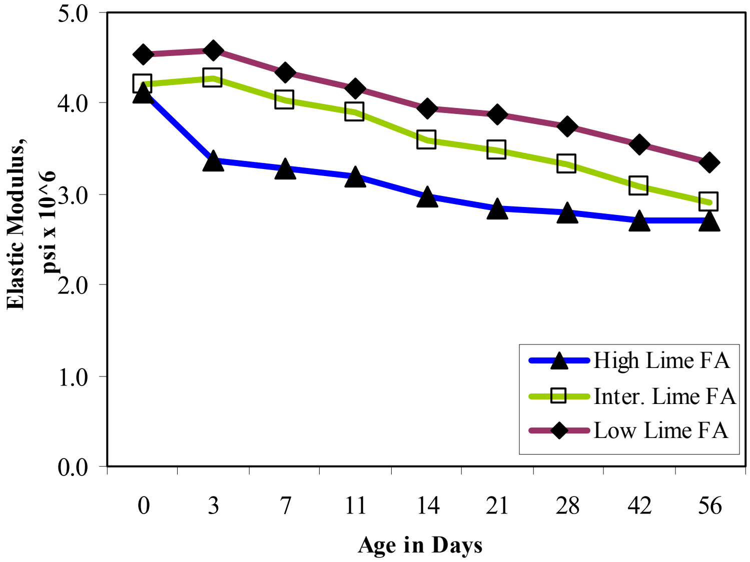


Fig. 17 A Standard ASTM C 1567 Test (1N NaOH Soak Solution)

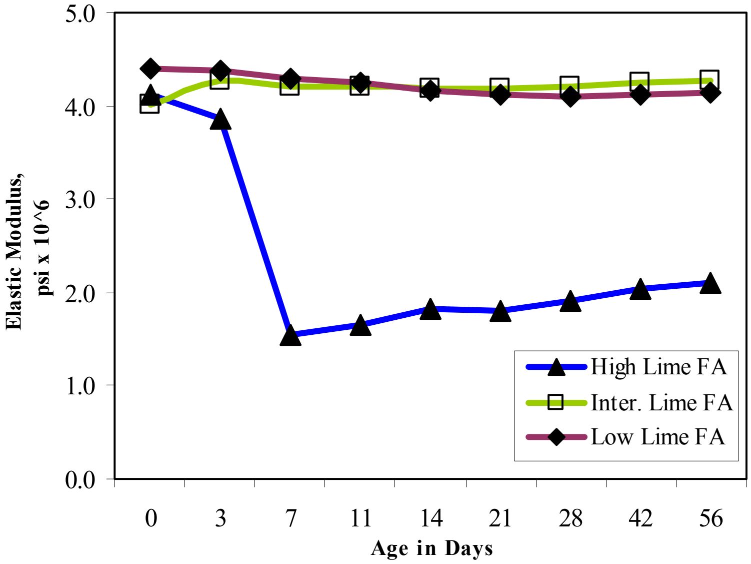


Fig. 17 B Modified ASTM C 1567 Test (50% KAc Deicer Solution)

FIGURE 17. CHANGES IN DYNAMIC MODULUS OF ELASTICITY OF NC ARGILLITE MORTAR BARS IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH 25% FLY ASH DOSAGE

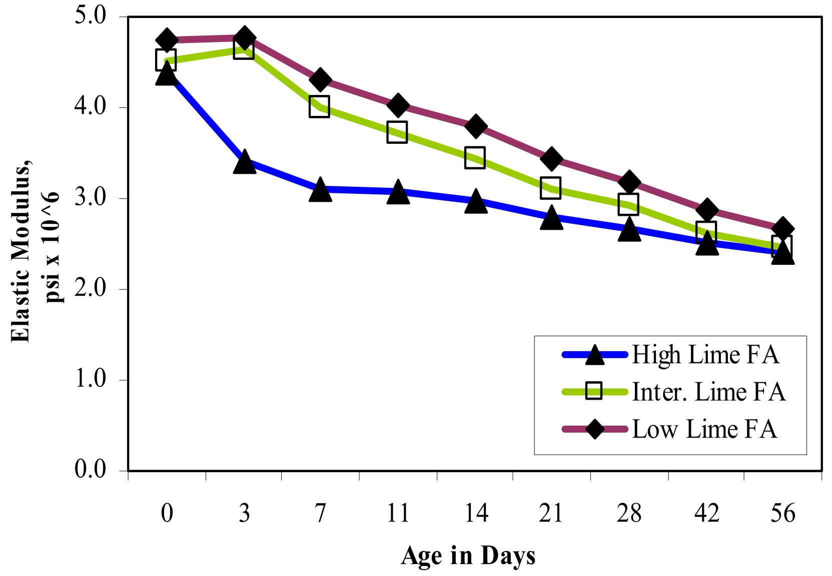


Fig. 18 A Standard ASTM C 1567 Test (1N NaOH Soak Solution)

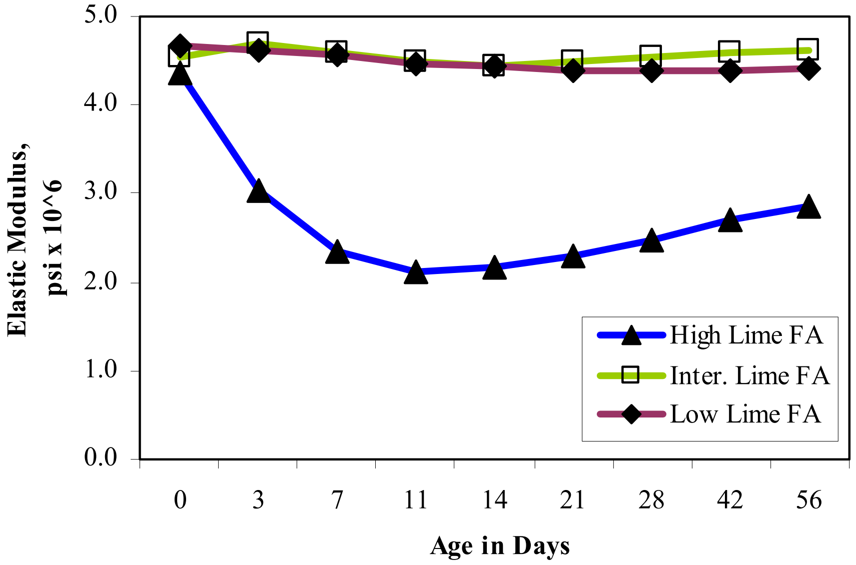


Fig. 18 B Modified ASTM C 1567 Test (50% KAc Deicer Solution)

FIGURE 18. CHANGES IN DYNAMIC MODULUS OF ELASTICITY OF SD QUARTZITE MORTAR BARS IN STANDARD AND MODIFIED ASTM C 1567 TESTS WITH 25% FLY ASH DOSAGE

4.3. MICROSTRUCTURE INVESTIGATION OF MORTAR BARS FROM STANDARD AND MODIFIED ASTM C 1567 TESTS

This section of the report summarizes the main features observed during the SEM examination of mortar specimens tested from:

- (i) Standard and modified ASTM C 1260 test methods as described in sections 3.3.1 and 3.3.2, and
- (ii) Standard and modified ASTM C1567 test methods as described in sections 3.3.3 and 3.3.4 of the report.

All the SEM specimens were prepared following the procedure described in Section 3.3.6 of this report.

In this investigation, microstructure of selected test specimens containing ASR mitigation measures was evaluated to document the distress in the mortar bars subjected to 1N NaOH soak solution and 50% KAc deicer solution and to corroborate the evidence from the length-change measurements (Section 4.1) and dynamic modulus of elasticity (Section 4.2) studies.

The microstructure study was limited to Spratt limestone mortar bars containing low-lime, intermediate-lime and high-lime fly ashes at a 25% dosage level. Test specimens from the standard ASTM C 1567 test (mortar bars soaked in 1N NaOH soak Solution) and the modified ASTM C 1567 test (mortar bars soaked in KAc deicer solution) were studied.

Most of the SEM images show a low-magnification image of an area of interest along with a high-magnification image where specific chemical information is obtained through EDX analysis. The EDX spot is clearly indicated in the high-magnification image. Remainder of the SEM images show a low-magnification image to capture the general extent of the damage observed in test specimens.

4.3.1. Spratt Limestone Mortar Bars (Control)

4.3.1.1. 1N NaOH Soak Solution

Figure 19 shows the microstructure of a Spratt limestone mortar bar (control) exposed to 1N NaOH solution. The microstructure reveals extensive damage that occurred in the mortar bar including cracks that completely extend through the aggregate particles. Typically, the cracks are in-filled with an ASR gel. In some cases, the ASR gel migrated from the aggregate particle into the surrounding paste as seen in the high-magnification area of Figure 19. The composition of the ASR gel within the paste adjacent to a reacted aggregate particle is identified in the EDX analysis shown in Figure 19.

4.3.1.2 KAc Deicer Soak Solution

Figure 20 shows the microstructure of a Spratt limestone mortar bar (control) exposed to KAc deicer solution. Extensive amount of cracking was observed in this specimen at the paste-aggregate interface along with moderate cracking in the aggregate particles and paste. The open (and mostly empty) cracks were predominately observed in the paste and at the interfaces with aggregate particles as illustrated in Figure 20. The presence of gaps around the aggregate implies the possibility of overall paste expansion, in addition to the expansion of the aggregate particles itself. The cracks present in the Spratt aggregate particles were typically in-filled with gel. The composition of the ASR gel in-filling the crack within the Spratt limestone particle is shown in the EDX spectra of Figure 20.

4.3.2. Spratt Limestone Mortar Bars with Low-Lime Fly Ash

4.3.2.1.1N NaOH Soak Solution

Figures 21 and 22 show the micrographs of a section from Spratt limestone mortar containing low-lime fly ash soaked in 1N NaOH solution for 28 days. While the presence of 25% low-lime fly ash in the matrix significantly mitigated expansion in mortar bars exposed to 1N NaOH solution (see Figure 4), some cracking was observed predominantly in the paste and at the paste-aggregate interface. Hairline cracks were also observed in the Spratt limestone aggregate particles, but the intensity of the cracking was significantly less compared to what was observed in the control specimens. The paste surrounding the aggregate particles appears to be particularly rich in sodium as observed in the EDX spectra shown in Figure 21.

4.3.2.2. KAc Deicer Soak Solution

Figure 23 shows a low-magnification image of Spratt limestone mortar bar with low-lime fly ash soaked in KAc deicer solution. Figures 24, 25 and 26 show specific information on microstructure of a region surrounding an aggregate particle (see Figure 26) and fly ash grain (see Figure 24 and 25).

From these figures it is evident that significant cracking of matrix has occurred with cracks predominantly in the paste and the paste-aggregate interface areas, and sometimes extending into the peripheral portions of aggregate particles (see Figure 23 and 24). The cracks observed in these specimens are characterized by a cluster of fine cracks, rather than a single large crack running across the aggregate or cement paste as observed in case of unmitigated test specimens (see Section 4.3.1.2 and Figure 20). The paste appears to be heavily infused with potassium ions from the deicer soak solution. Figures 25 and 26 show EDX spectra of a fly ash grain and of paste adjacent to the fly ash grain, respectively. It is evident from these Figures that the paste surrounding the fly ash grain is rich in potassium. The darker regions within these Figures were found to be rich in silica, and are assumed to be localized regions with in the paste that are decalcified.

4.3.3. Spratt Limestone Mortar Bars with Intermediate-Lime Fly Ash

4.3.3.1. 1N NaOH Soak Solution

Figures 27 and 28 show the microstructure of Spratt limestone mortar bar containing intermediate-lime fly ash that was soaked in 1N NaOH solution. Although the presence of 25% intermediate-lime fly ash in the matrix significantly mitigated expansion in mortar bars exposed to 1N NaOH solution (see Figure 4), significant cracking was observed predominantly in the paste and at the paste-aggregate interface. The paste surrounding the aggregate particles appears to be particularly rich in sodium as observed in the EDX spectra shown in Figures 27 and 28. It should be noted that very minimal cracking was observed within the aggregate particles.

4.3.3.2. KAc Deicer Soak Solution

Figures 29 and 30 reveal the microstructure of Spratt limestone mortar bar containing intermediate-lime fly ash and exposed to KAc deicer solution. These micrographs show a network of cracks predominantly in the paste and the aggregate-paste interface region. Sometimes, these cracks appear to continue into the aggregate particles (see Figure 29). It should be noted from the length-change data presented in Figure 4 that intermediate-lime fly ash resulted in significant mitigation in expansion compared to control specimen.

4.3.4. Spratt Limestone Mortar Bars with High-Lime Fly Ash

4.3.4.1. 1N NaOH Soak Solution

Figures 31 and 32 shows the microstructure of Spratt limestone mortar bar containing high-lime fly ash (25% dosage) soaked in 1N NaOH solution. The distress in this test specimen is characterized by the presence of a wide crack that traverses through both aggregate particles and paste, and is consistent with the expansion observed in the Standard ASTM C 1567 test method (see Figure 4). The EDX spectra of the deposits on the crack edges correctly identify the product to be ASR gel. The Mg and Al observed in the EDX spectra are likely from the dolomitic component of the Spratt limestone and fly ash particles, respectively. When the crack traverses through a aggregate particle, the walls of the crack are lined with distinct deposits of ASR gel (see Figure 31). The ASR gel appears to infuse into the surrounding paste when the crack traverses through the paste portion of the test specimen (see Figure 32). As a result, the surrounding paste exhibits desiccation cracks that are characteristic of the ASR gel in the SEM images.

4.3.4.2. KAc Deicer Soak Solution

Figures 33 and 34 show the microstructure of Spratt limestone mortar containing 25% high-lime fly ash soaked in KAc deicer solution. The expansion observed in these test specimens was very high, and similar to that of the test specimens that did not contain any fly ash.

The damage in these test specimens as observed in SEM images is characterized by the presence of an extensive network of cracks in the paste-aggregate interface. Minor amount of cracking was also noticed within some aggregate particles.

Figure 33 shows a distressed portion of paste-aggregate interface that is characterized by the presence of an extensive network of cracks. The EDX spectra obtained at three locations (A, B and C) shown in Figure 33 clearly indicates the transition from an aggregate particle unaffected by the presence of deicer soak solution into paste that is clearly influenced by the deicing soak solution. For instance the EDX spectra at location A that is away from the surface of the aggregate particle shows only calcite – the predominant mineral in Spratt limestone. The EDX spectra at location B within the aggregate particle but near the paste-aggregate interface shows minor amount of potassium that has likely migrated into the aggregate from the paste. The EDX spectra at location C in the paste but near the paste-aggregate interface shows the abundance of potassium that has likely migrated from the external soak solution into the paste. It is likely that the aggregate near the interface may have partially reacted to form ASR gel that has migrated into the surrounding paste.

Figure 34 shows evidence of a dense ASR gel like formation around the periphery of an affected aggregate particle. The EDX spectra shown in Figure 34 clearly show the decreasing levels of potassium from location A (in the paste) to C (in the aggregate). The hashed lines in Figure 34 show a tract of dense reaction product that is characteristic of ASR gel. However, this region is devoid of the intense cracking that is observed typically near the paste-aggregate interfaces, elsewhere in the sample.

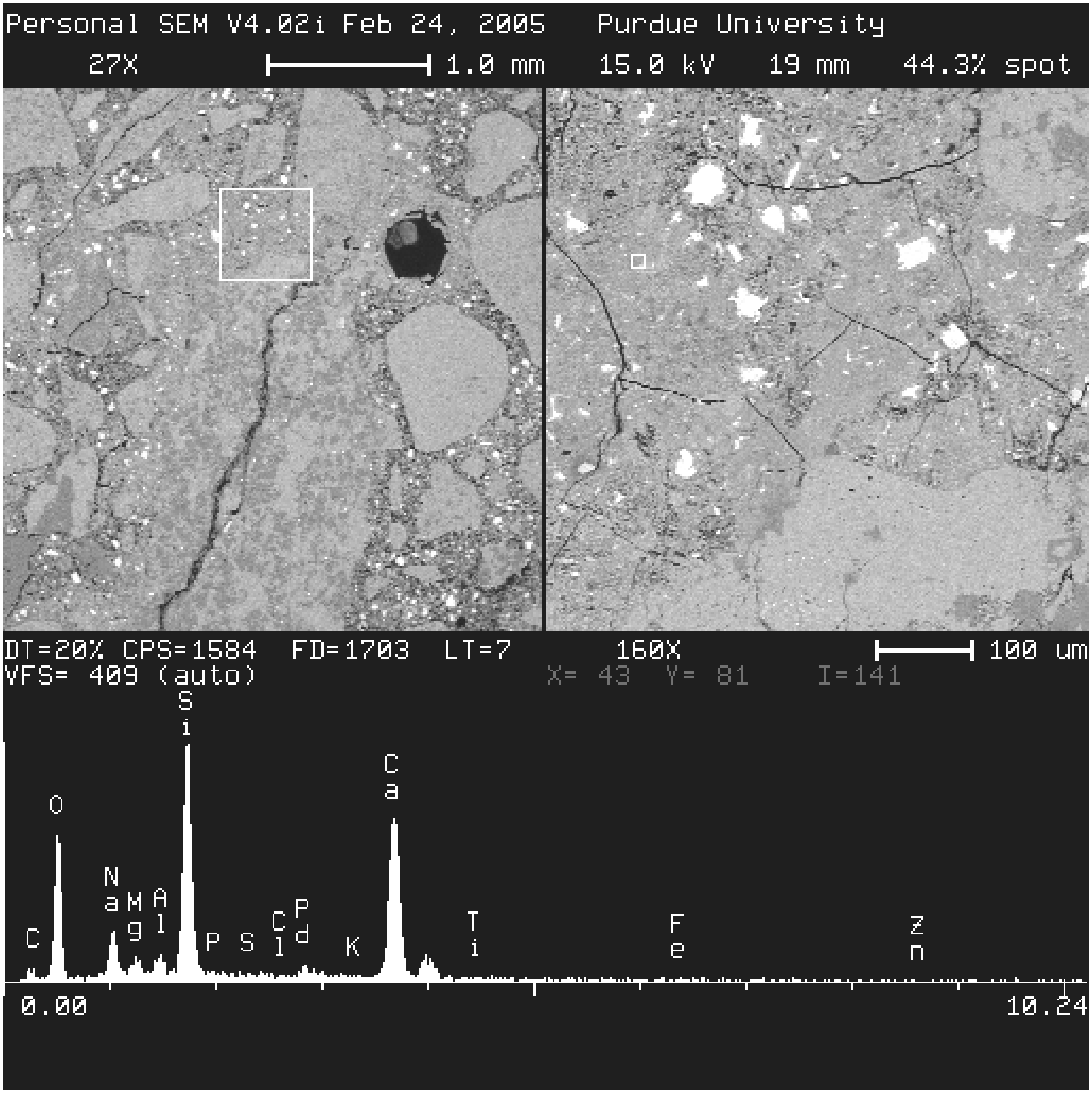


FIGURE 19. MICROSTRUCTURE OF SPRATT LIMESTONE MORTAR EXPOSED TO 1N NaOH SOAK SOLUTION

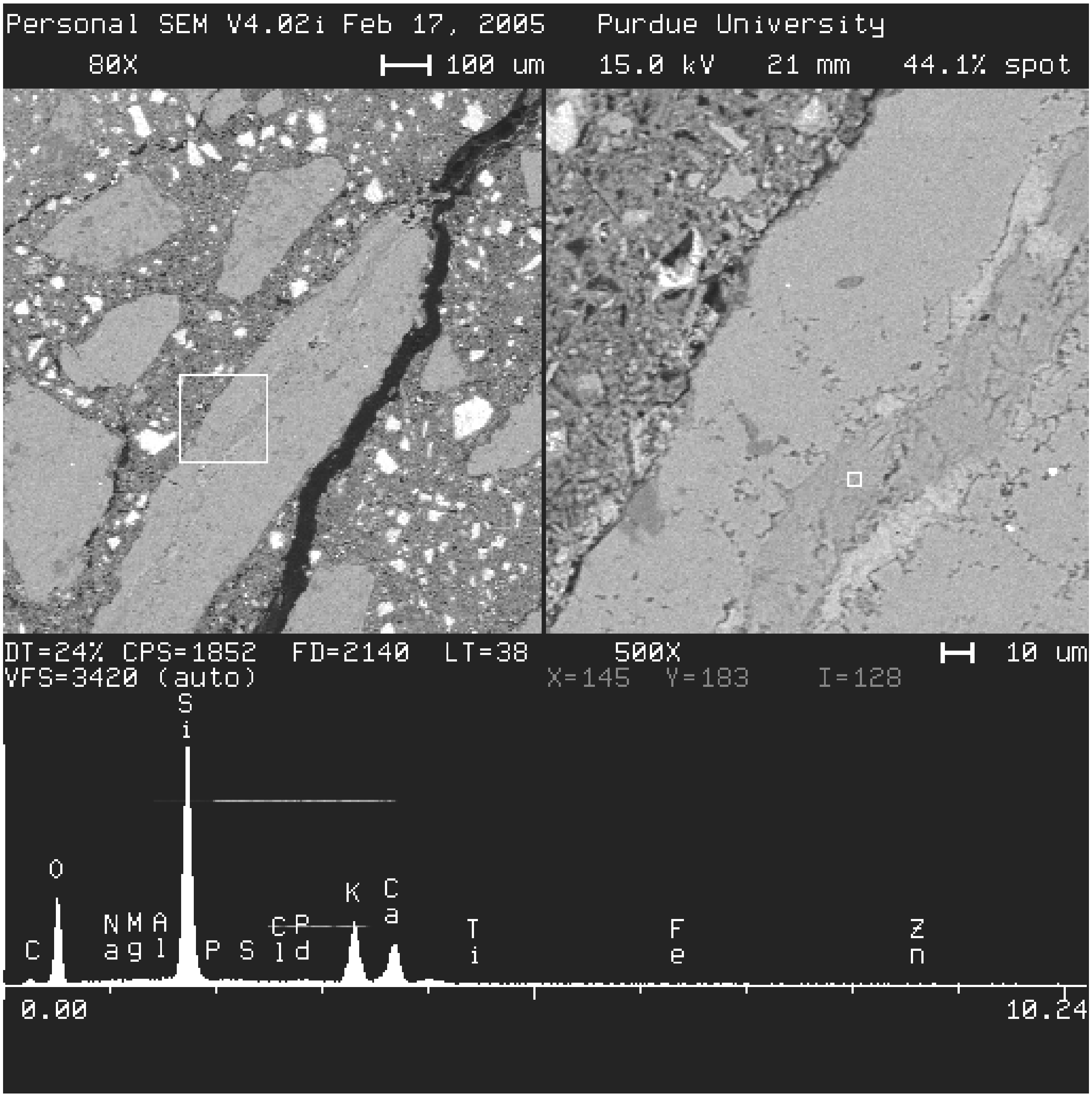


FIGURE 20. ASR GEL-FILLED CRACK IN THE PARTICLE OF A CONTROL SPRATT LIMESTONE MORTAR EXPOSED TO KAc SOLUTION

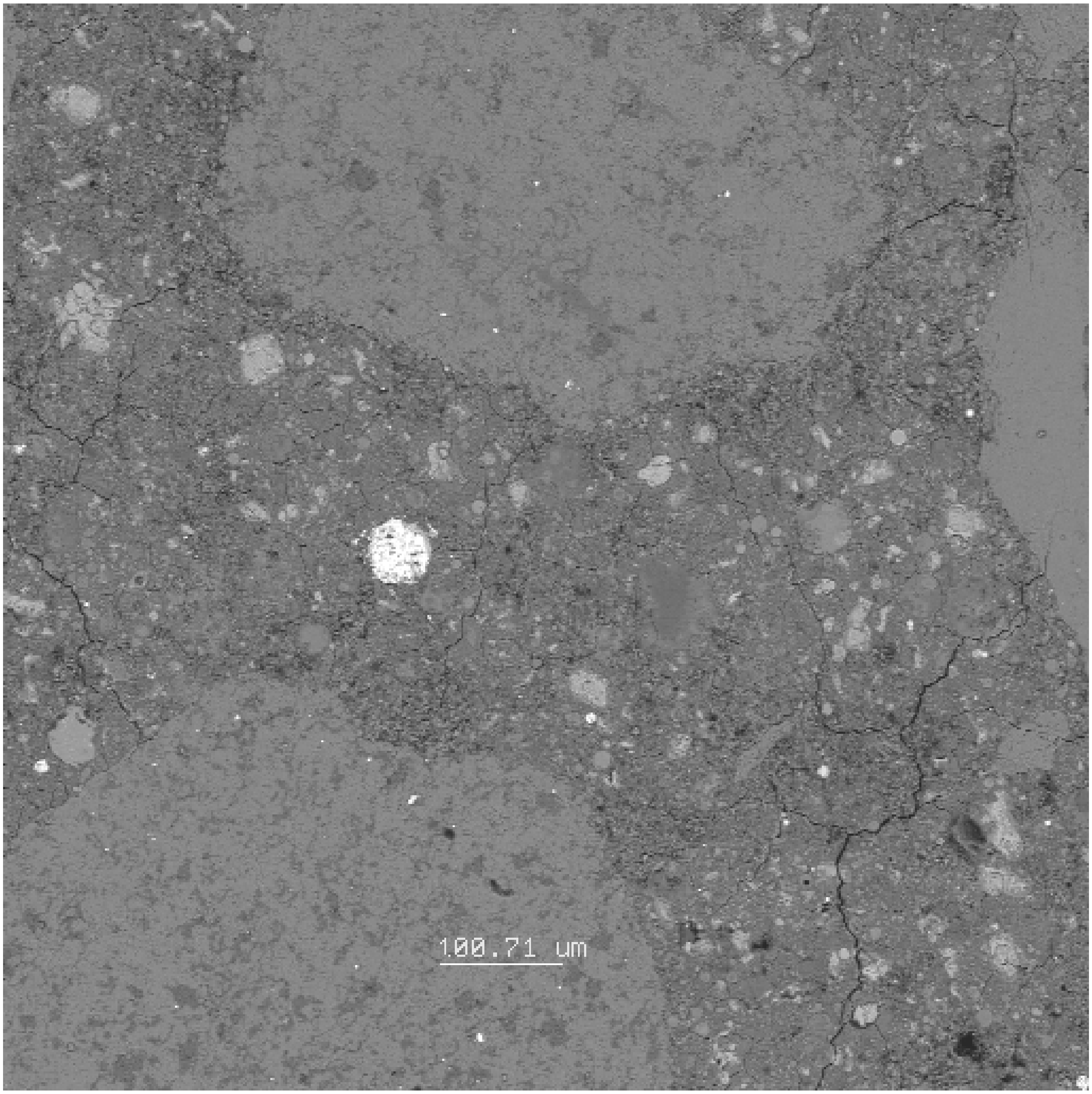


FIGURE 21. LOW MAGNIFICATION IMAGE OF SPRATT-LIMESTONE MORTAR BAR CONTAINING LOW-LIME FLY ASH EXPOSED TO 1N NaOH SOAK SOLUTION

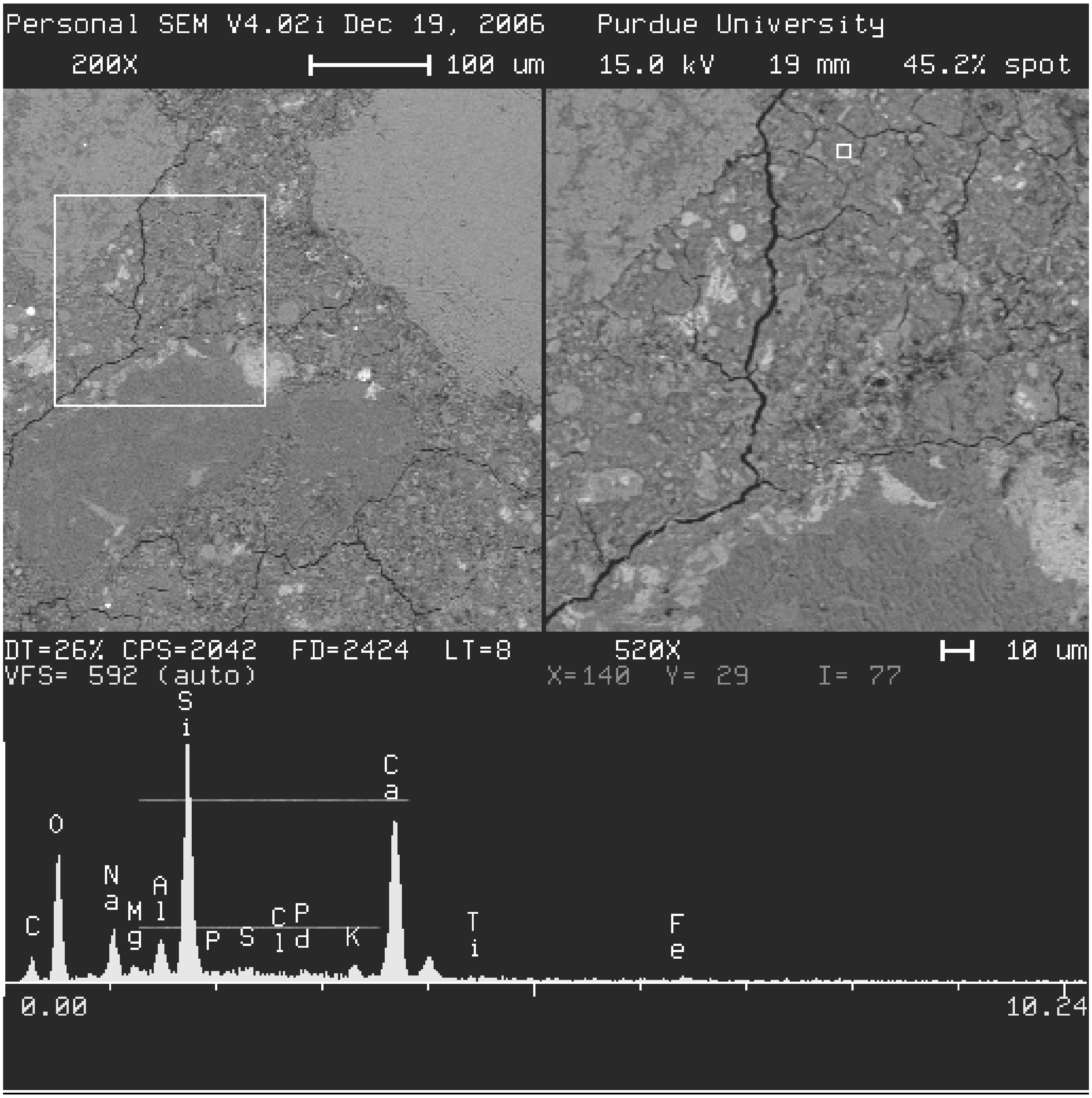


FIGURE 22. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING LOW-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN 1N NaOH SOAK SOLUTION

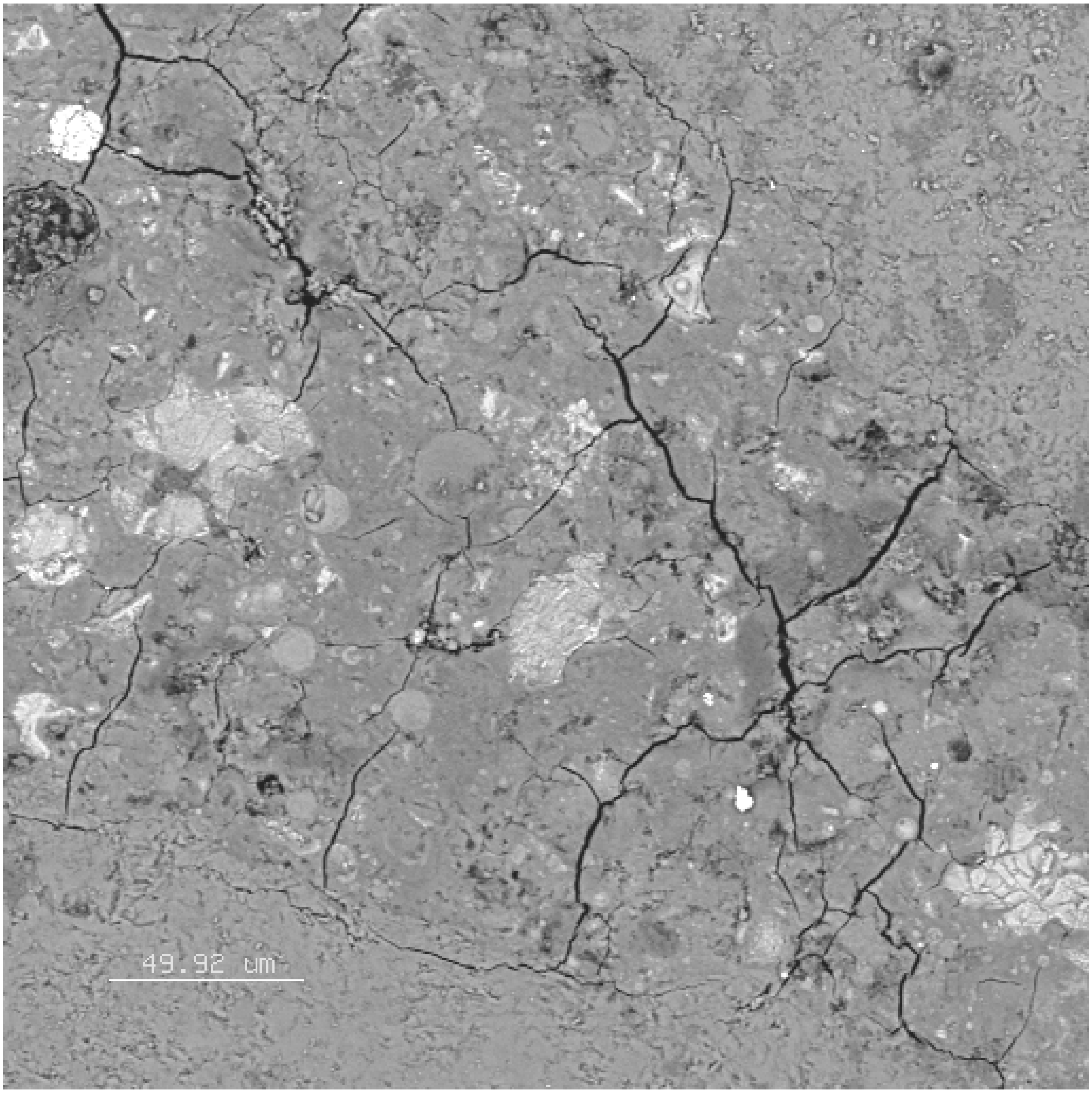


FIGURE 23. LOW MAGNIFICATION IMAGE OF SPRATT-LIMESTONE MORTAR BAR CONTAINING LOW-LIME FLY ASH EXPOSED TO KAc DEICER SOAK SOLUTION

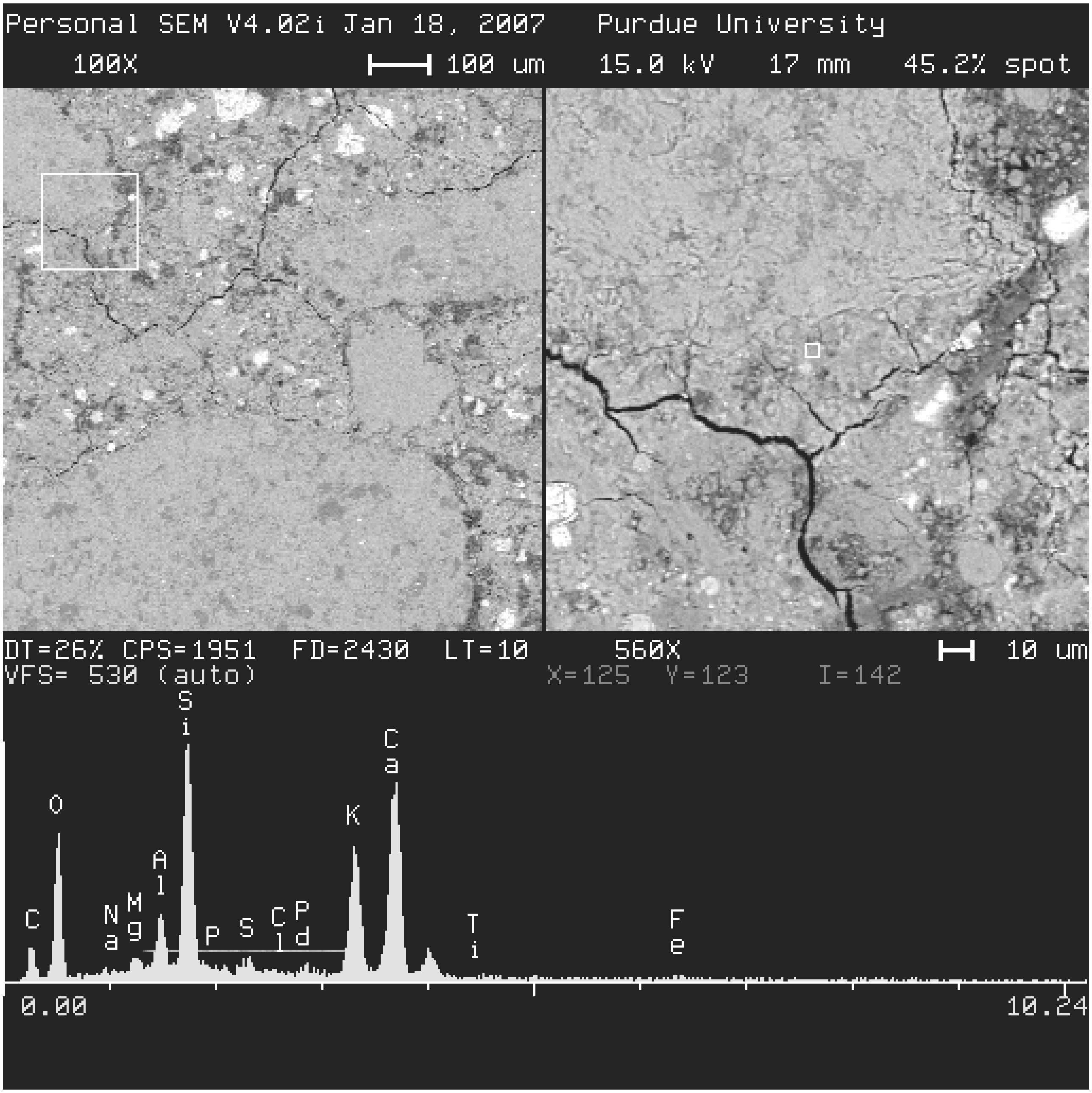


FIGURE 24. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING LOW-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN KAc DEICER SOAK SOLUTION.

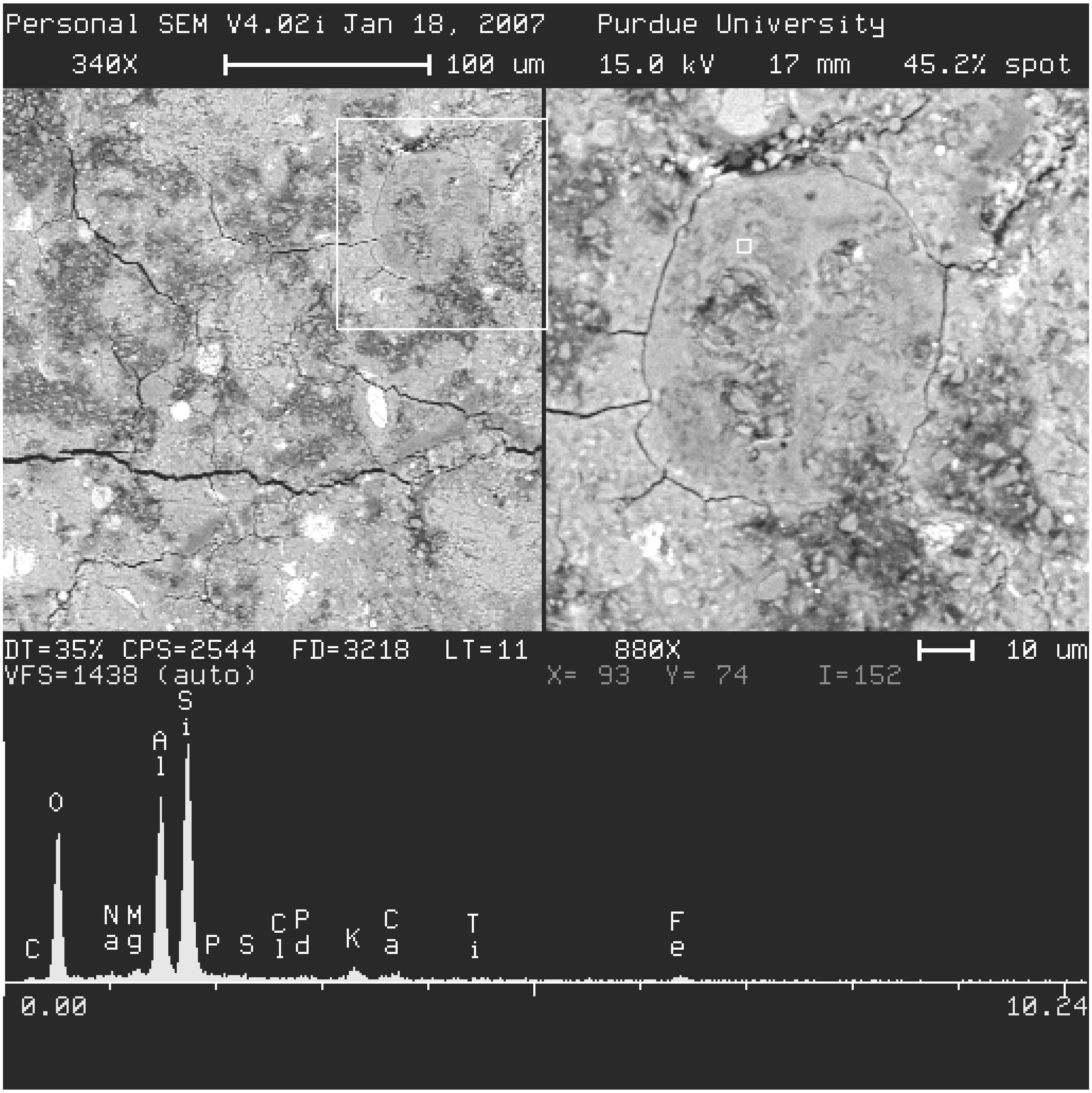


FIGURE 25. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING LOW-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN KAc DEICER SOAK SOLUTION (EDX SPOT ON FLY ASH GRAIN)

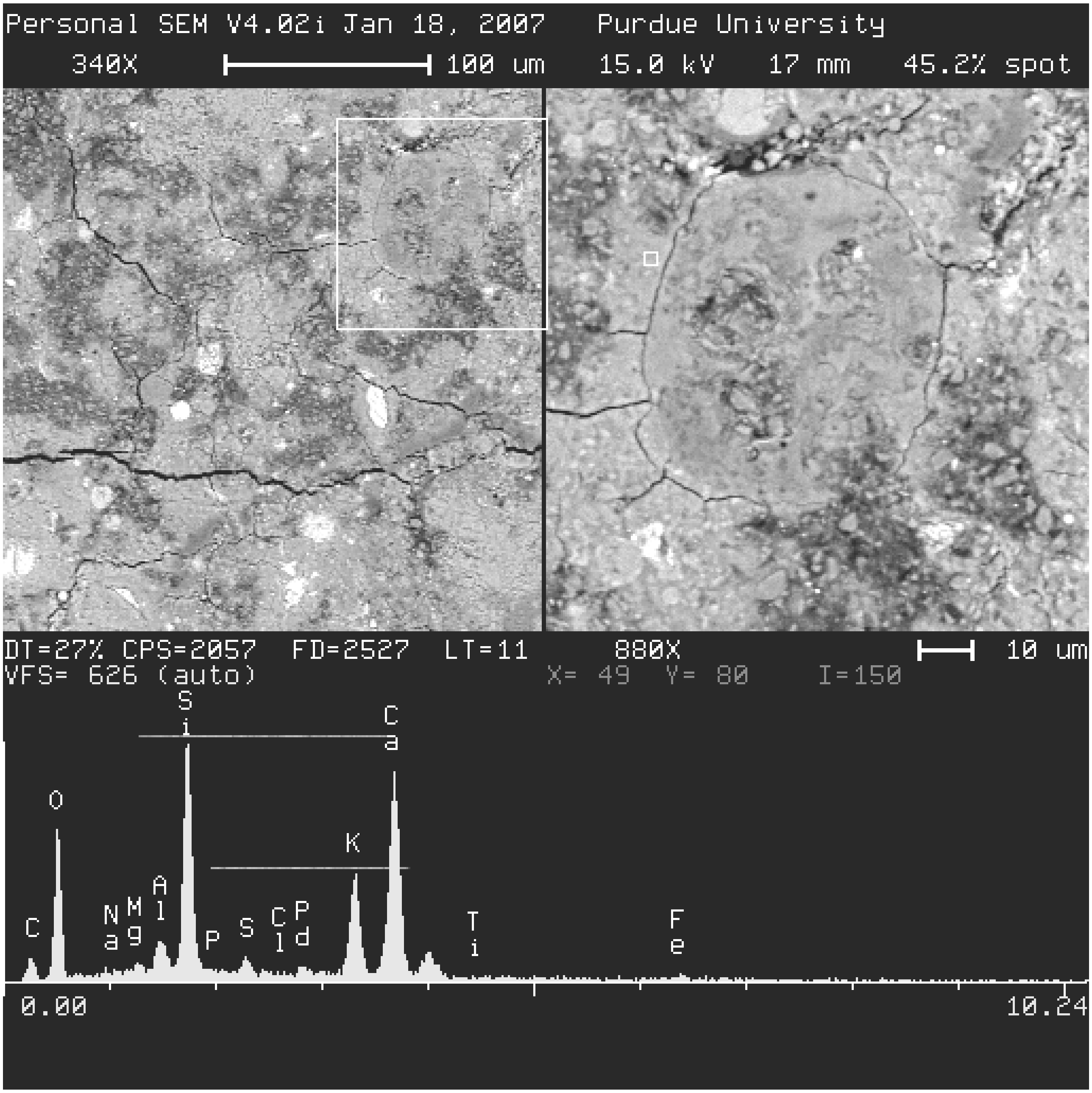


FIGURE 26. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING LOW-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN KAc DEICER SOAK SOLUTION (EDX SPOT ON PASTE ADJACENT TO FLY ASH GRAIN)

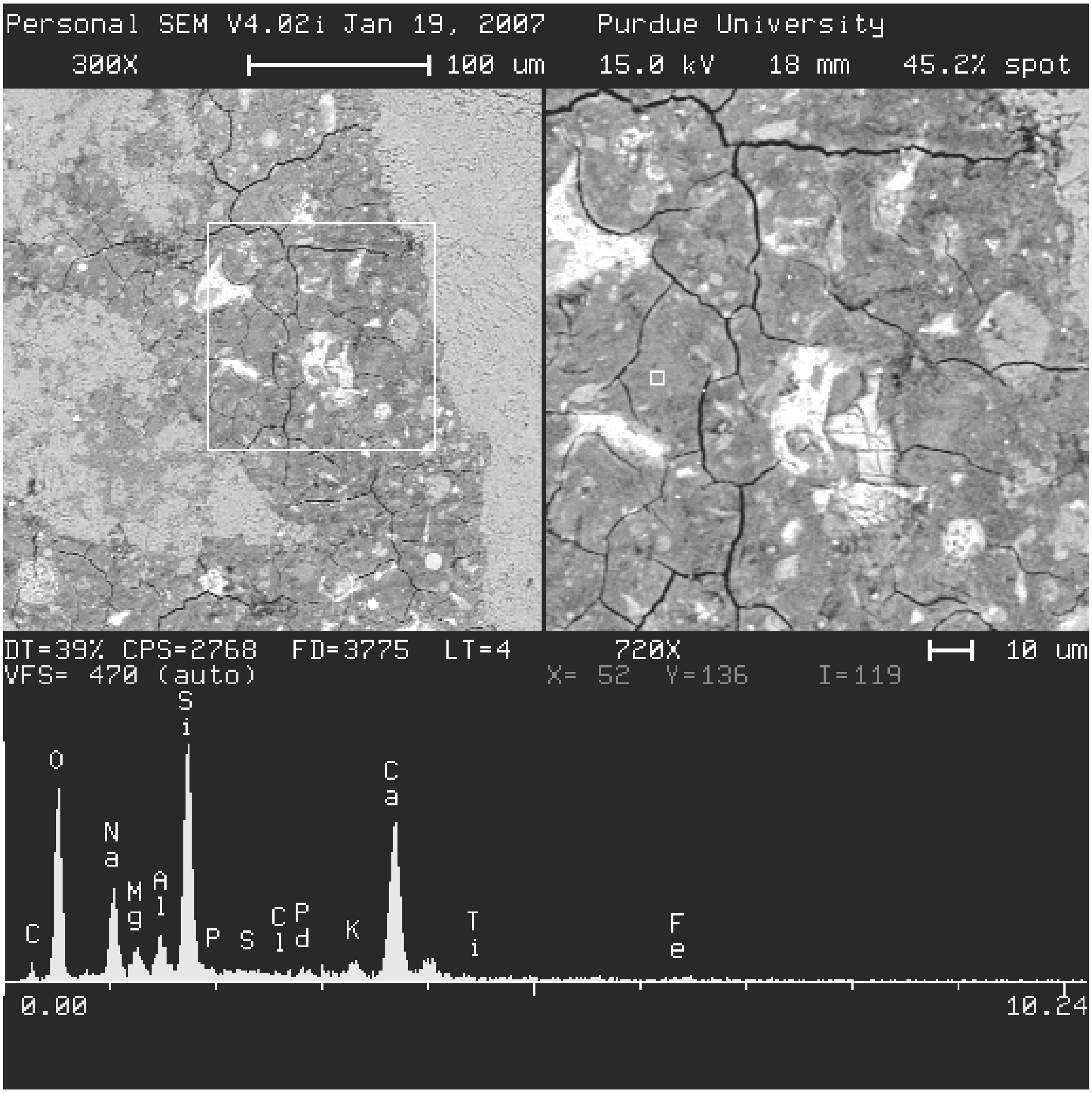


FIGURE 27. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING INTERMEDIATE-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN 1N NaOH SOAK SOLUTION.

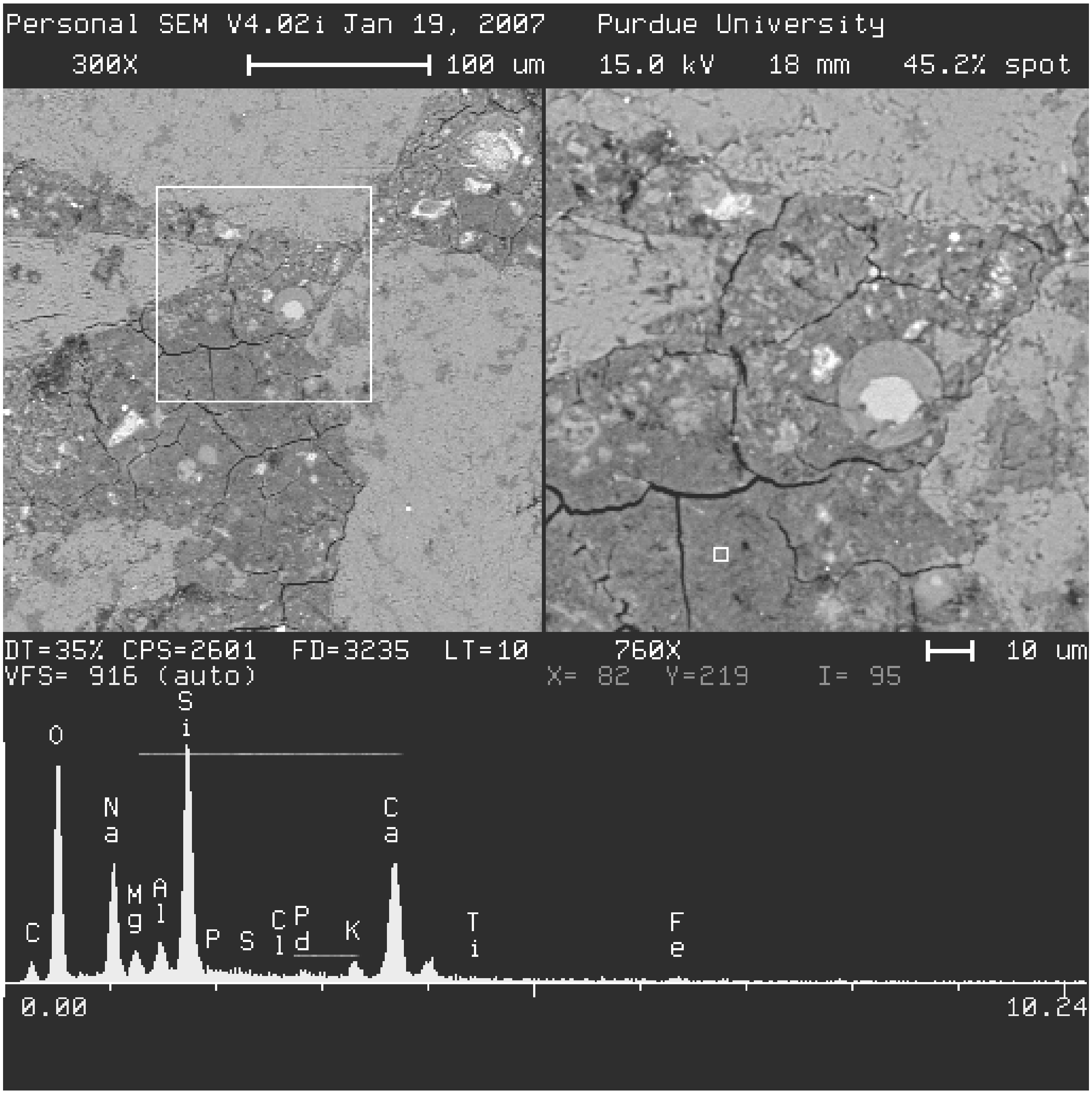


FIGURE 28. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING INTERMEDIATE-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN 1N NaOH SOAK SOLUTION.

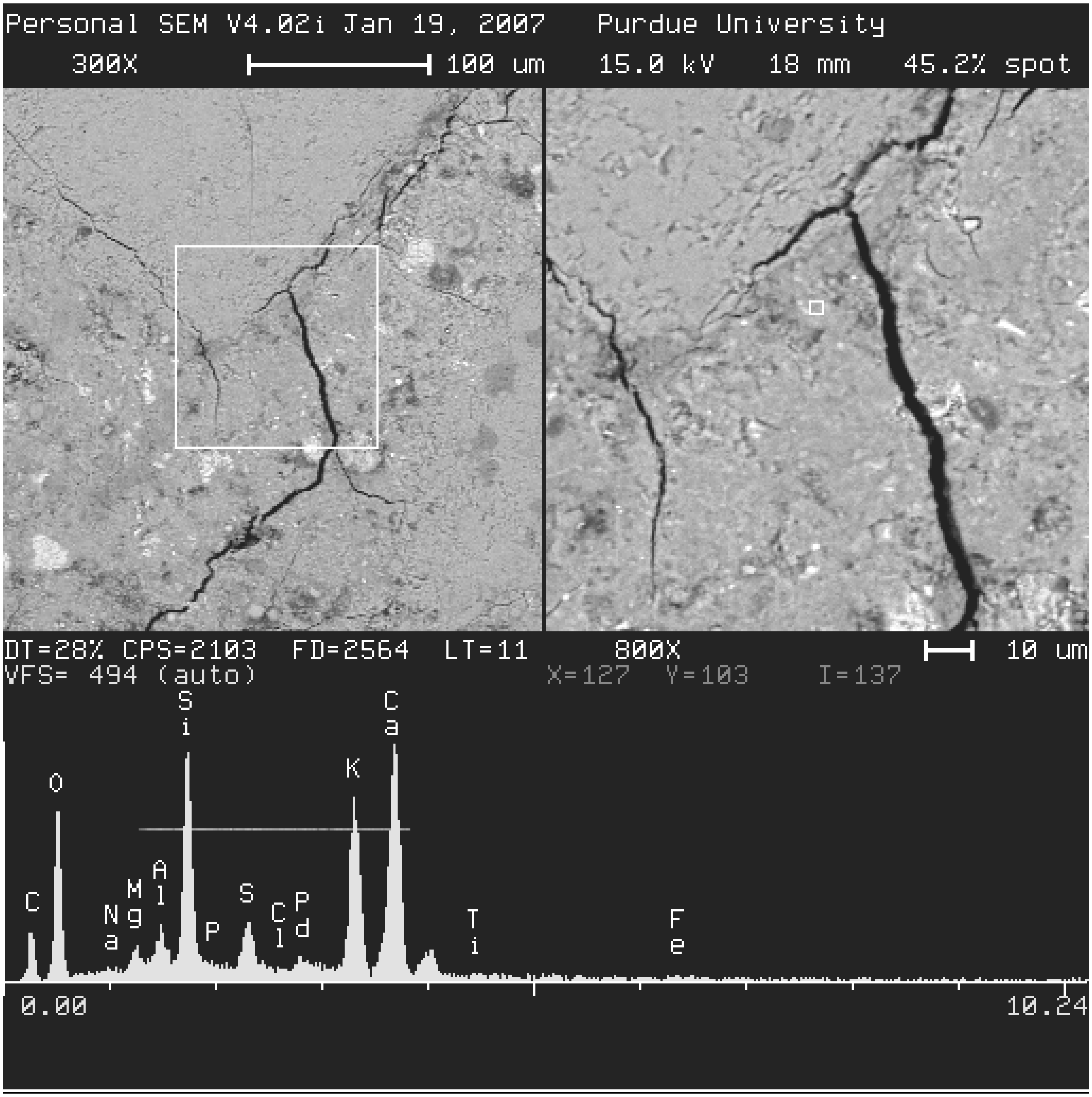


FIGURE 29. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING INTERMDIATE-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN KAc DEICER SOAK SOLUTION

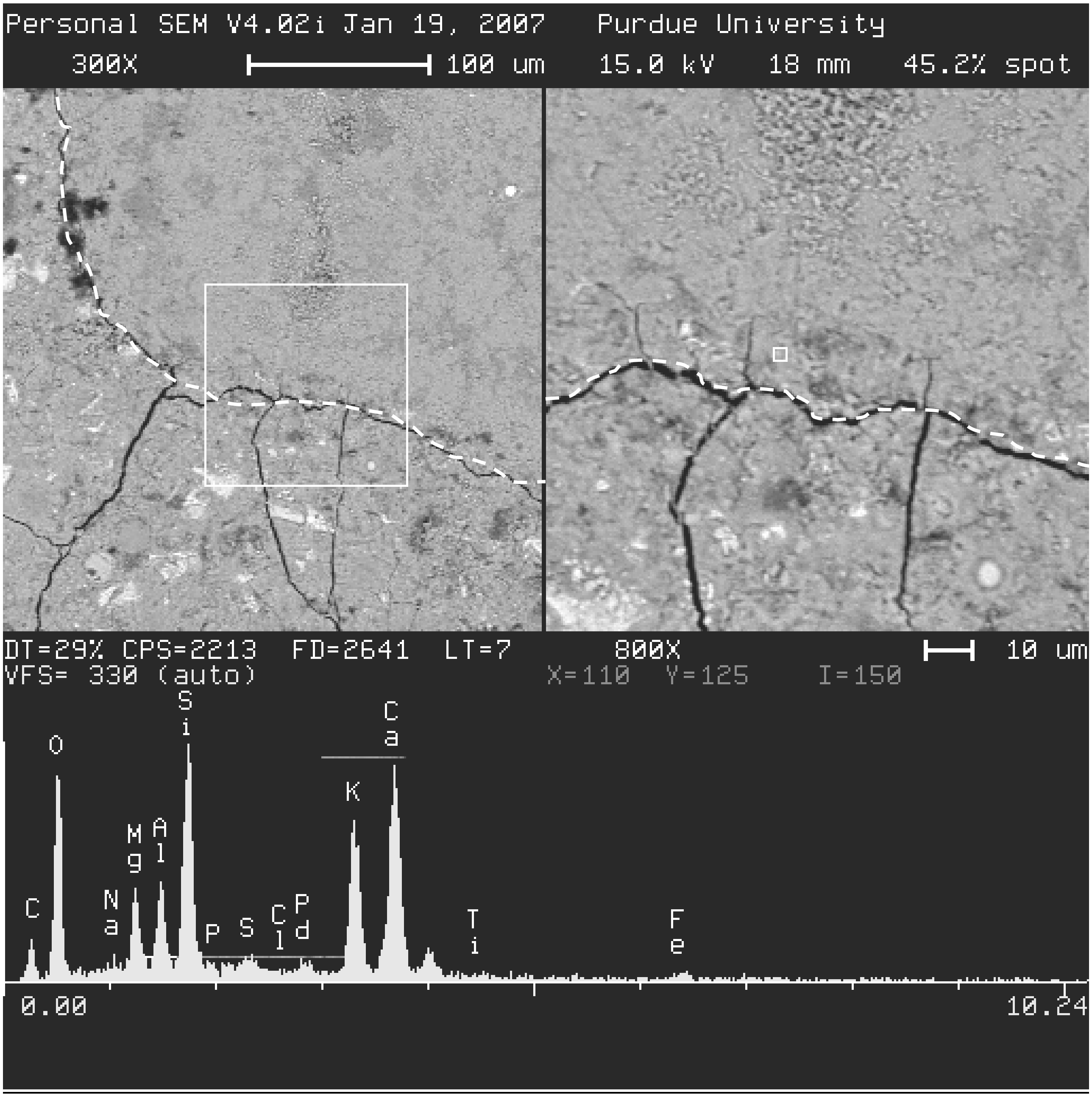


FIGURE 30. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING INTERMDIATE-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN KAc DEICER SOAK SOLUTION. THE PASTE-AGGREGATE INTERFACE IS IDENTIFIED WITH A HASHED LINE.

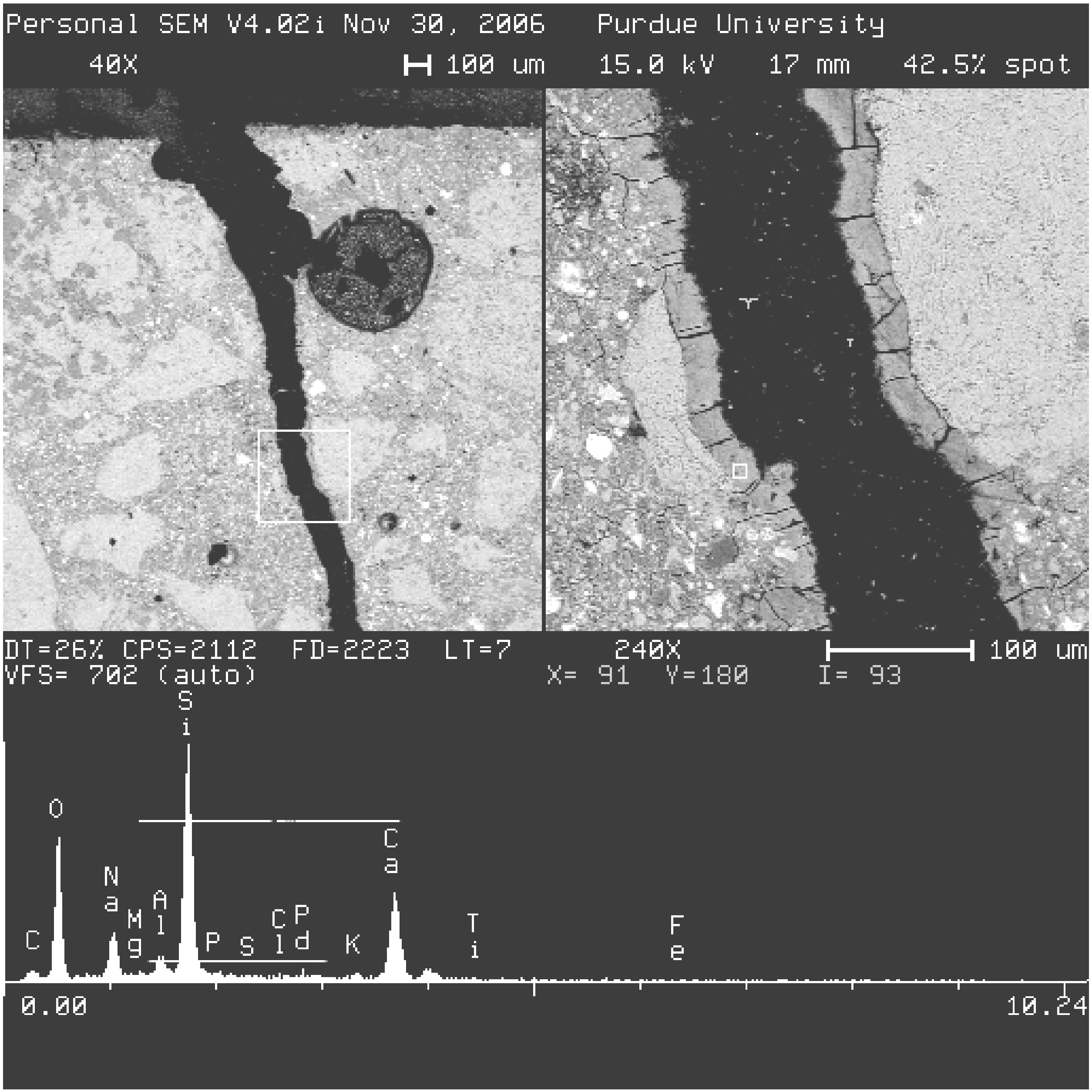


FIGURE 31 SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING HIGH-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN 1N NaOH SOAK SOLUTION.

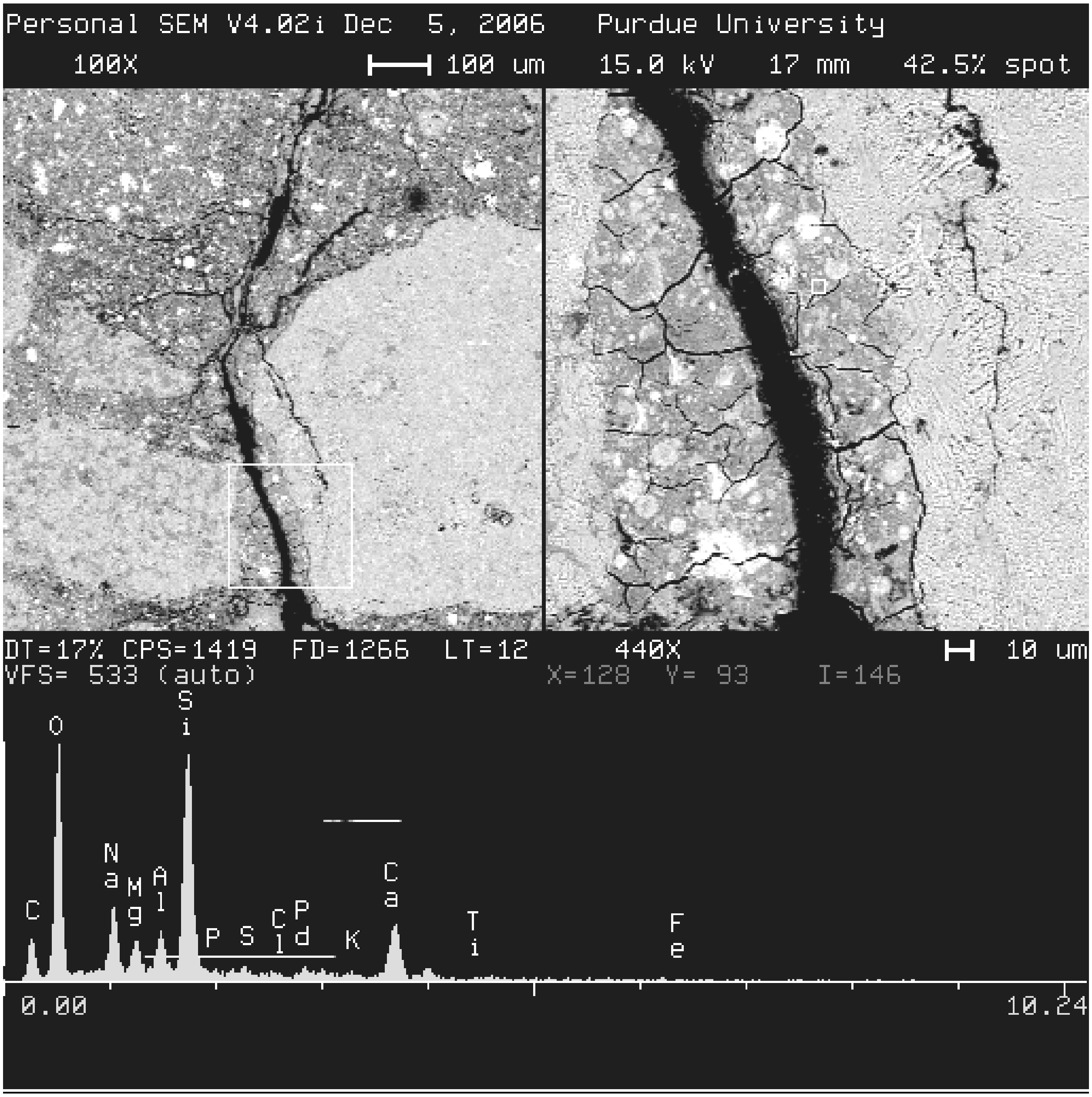


FIGURE 32 SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING HIGH-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN 1N NaOH SOAK SOLUTION.

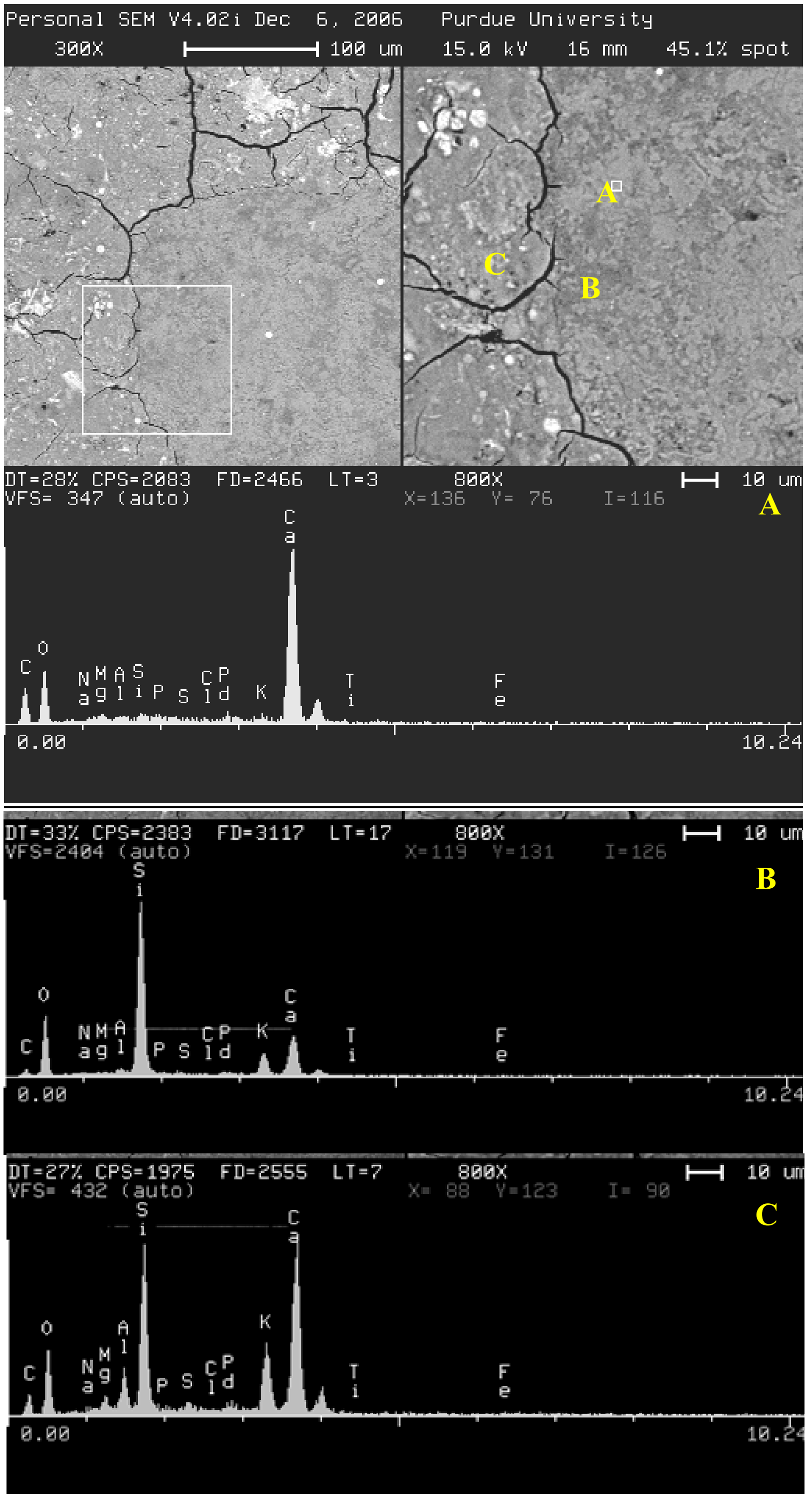


FIGURE 33. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING HIGH-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN KAc DEICER SOAK SOLUTION. (A – AGGREGATE; B – INTERFACE; C – PASTE)

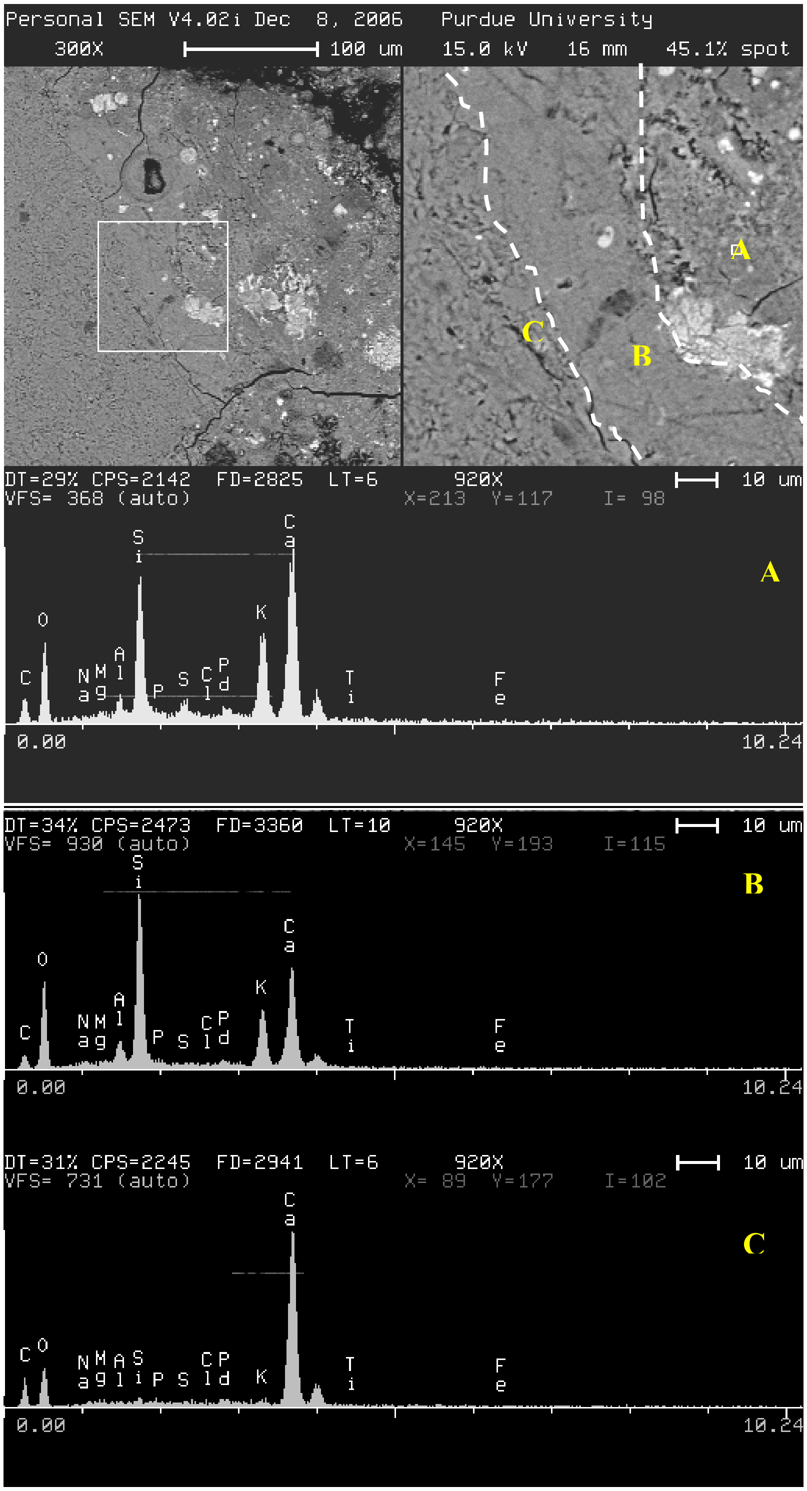


FIGURE 34. SEM MICROGRAPH OF SPRATT LIMESTONE MORTAR BAR CONTAINING HIGH-LIME FLY ASH AT 25% DOSAGE LEVEL SOAKED IN KAc DEICER SOAK SOLUTION. (A – PASTE ; B – INTERFACE; C – AGGREGATE)

5. RESULTS FROM STANDARD AND MODIFIED ASTM C 1567 TESTS TO EVALUATE ASR MITIGATION POTENTIAL OF LITHIUM ADMIXTURE

This section presents results from the standard and the modified ASTM C 1567 tests in which the effectiveness of lithium admixture was evaluated in presence of 1N NaOH and KAc deicer solutions. In these studies expansion of mortar bars over 0.1% at 28 days of exposure to soak solutions was considered to represent a deleterious level of expansion.

The matrix of tests involving lithium admixture was discussed in section 3.4.3. and presented in Tables 5, 6, 7 and 8. The material proportions used in preparing the mortar bars was discussed in Section 3.5.2 and presented in Tables 9 and 10. The composition of the soak solutions employed in these tests is presented in Section 3.5.3.

Standard ASTM C 1567 test method does not pertain to lithium admixtures. As a result, variations to the standard ASTM C 1567 test method were employed to assess lithium admixtures in this study. In tests with 1N NaOH soak solution, two different approaches to lithium addition were employed as discussed in section 3.4.3. The first series of tests included addition of lithium admixture to both soak solution and mortar bars at the same Li/Na molar ratio. The second series of tests involved addition of lithium admixture only to the soak solution alone at Li/Na molar ratios corresponding to those employed in first series. In addition, results from standard ASTM C 1260 tests (Control – 1N NaOH) are also shown in the figures for reference.

In tests with potassium acetate deicer soak solution, two different approaches to evaluating lithium admixtures were employed. In the first series, lithium admixture was added only to potassium acetate deicer soak solution. In the second series, lithium was added only to mortar bars. The reasons for employing such approaches and the details concerning the test matrix, material proportions and soak solution compositions are presented in Sections 3.4.3, 3.5.2, and 3.5.3.

5.1. STANDARD ASTM C 1567 TESTS (IN PRESENCE OF 1N NaOH SOAK SOLUTION)

5.1.1. Lithium Admixture in Mortar Bar and Soak Solution

Figures 35 through 38 show the expansion behavior of mortar bars containing lithium in bars and in 1N NaOH soak solution at four different Li/Na molar ratios. In addition, the results from standard ASTM C 1260 tests are shown in the figures for each aggregate to provide a reference for the level of mitigation offered by the lithium at different dosages.

From these figures it is evident that, compared to control mortar bar, addition of lithium nitrate admixture into mortar bars and soak solution significantly mitigates expansion, particularly when a certain minimum Li/Na ratio is maintained. At a dosage that yields a Li/Na ratio of 0.74 or greater, lithium nitrate admixture is extremely effective in mitigating expansions in mortar bars of all the aggregates evaluated in this study including NM rhyolite, one of the most reactive aggregate available.

The results from this investigation suggest that a Li/Na ratio of 0.23 is inadequate for all the reactive aggregates evaluated in this study under these test conditions. While this level of lithium dosage resulted in slightly lower levels of expansion with NM rhyolite and SD quartzite, it is important to observe that Spratt limestone showed no mitigation at this level. In fact, NC argillite showed higher levels of expansion with a Li/Na molar ratio of 0.23. These results suggest that certain reactive aggregates, such as NC argillite, may show a pessimum level of lithium dosage under these test conditions.

5.1.2. Lithium Admixture in Soak Solution Only

Figures 39 through 42 show the expansion behavior of mortar bars containing lithium in 1N NaOH soak solution only at four different Li/Na molar ratios. In addition, the results from the standard ASTM C 1260 tests are shown in the figures for each aggregate to provide a reference for the level of mitigation offered by the lithium at different dosages.

From these figures it is evident that addition of lithium nitrate admixture into soak solution alone provides a significant mitigation in expansion. The effectiveness of lithium addition into soak solution on its ability to mitigate expansion in mortar bars is particularly significant when Li/Na molar ratio of soak solution is 0.74 or greater for all the aggregates, perhaps with the exception of NM rhyolite.

Comparing the expansion behavior of respective mortar bars in tests with lithium in mortar bar and soak solution (Figures 35-38) and in tests with lithium in soak solution only (Figures 39-42), it is apparent that lithium is equally effective in mitigating expansion when used in soak solution alone or when used in mortar bar and soak solution, in case of Spratt limestone, NC argillite and SD quartzite aggregate. In case of NM rhyolite mortar bars, although significant mitigation in expansion was observed with a lithium dosage corresponding to a Li/Na molar ratio of 0.74, complete mitigation was achieved only at a lithium dosage of 1.00.

Lithium dosage, corresponding to Li/Na molar ratio of 0.23, was not adequate to mitigate expansion in case of all the aggregates evaluated. In these tests with lithium dosed in soak solution alone, NC argillite mortar bars did not exhibit a pessimum content.

The results presented in Sections 5.1.1 and Section 5.1.2 raise an important question that concerns the manner in which we evaluate the effectiveness of lithium admixtures in mitigating ASR expansions. Does adding lithium to soak solution at 100% dosage level (i.e. Li/Na molar ratio of 0.74) correlate with corresponding concrete mixtures placed in the field? (b) If not, to what extent should lithium be added to soak solution?

Findings reported in Sections 5.1.1 and 5.1.2 clearly suggest that for a given Li/Na ratio, the soak solution composition has a larger effect on the observed expansion. Additional research is needed to define the level of lithium admixture that should be added to the soak solution in order to correlate to the true potential of the admixture in mitigating ASR-induced expansions in mortar bars.

Although the focus of this research study was not on developing evaluating test methods to assess effectiveness of lithium admixtures, additional work is necessary to address this issue. It is suggested that parallel set of tests be conducted with lithium admixture in mortar bars alone, and develop an understanding of the amount of lithium that potential leaches into soak solution, and its possible influence on the mitigation.

As a result of the uncertainty that had developed over the appropriate methodology to evaluate lithium admixtures for ASR mitigation, the modified ASTM C 1567 tests to evaluate potassium acetate deicer solutions were based on applying lithium either to mortar bars alone or to soak solution alone. In this work, no tests were conducted in which lithium was added to both mortar bars and soak solution.

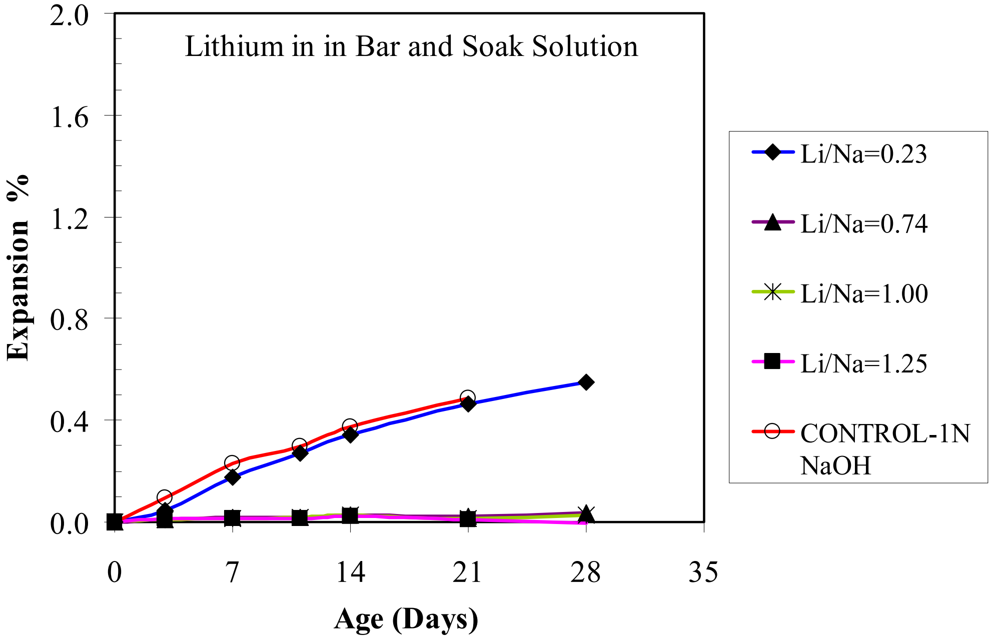


FIGURE 35. EXPANSION OF SPRATT LIMESTONE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN BARS AND 1N NaOH SOAK SOLUTION.

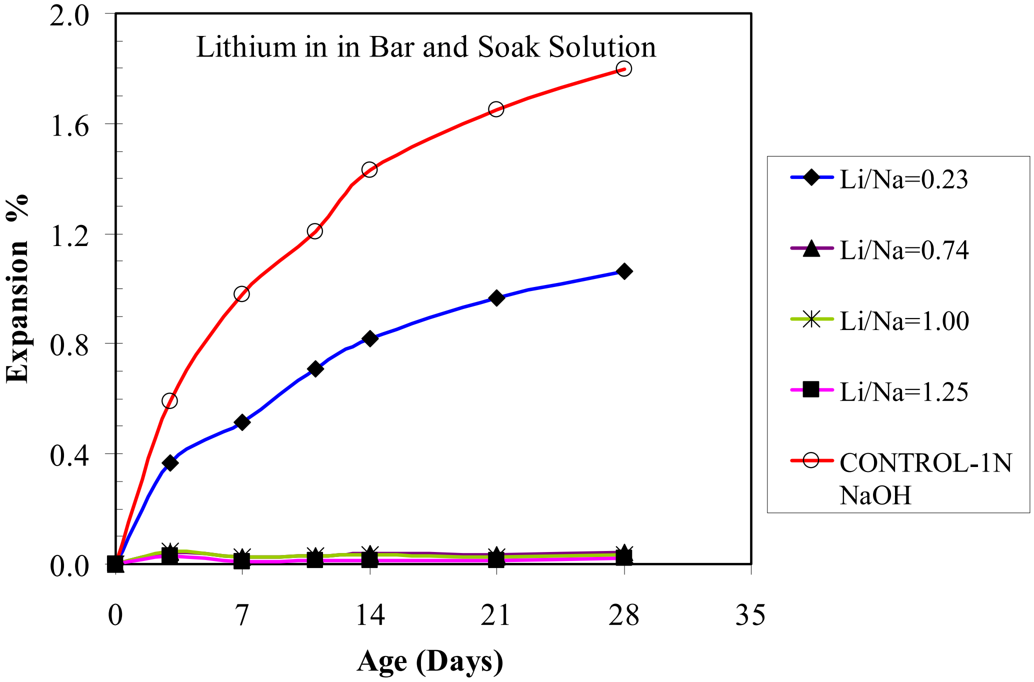


FIGURE 36. EXPANSION OF NM RHYOLITE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN BARS AND 1N NaOH SOAK SOLUTION.

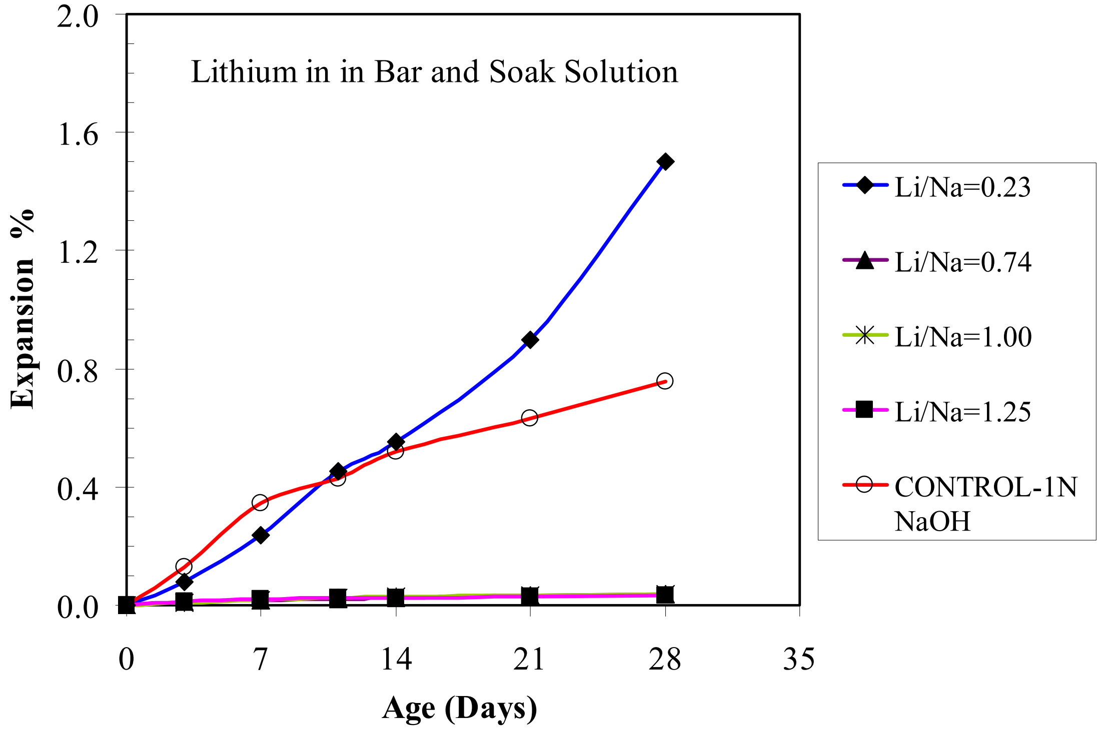


FIGURE 37. EXPANSION OF NC ARGILLITE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN BARS AND 1N NaOH SOAK SOLUTION.

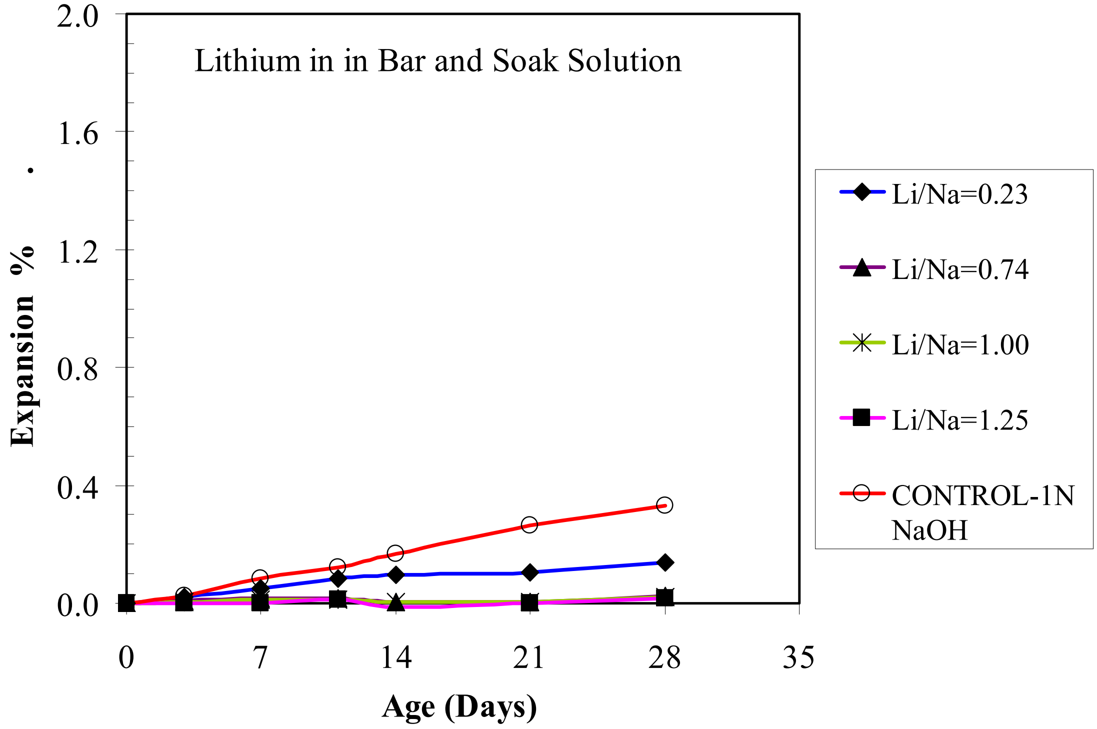


FIGURE 38. EXPANSION OF MN QUARTZITE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN BARS AND 1N NaOH SOAK SOLUTION.

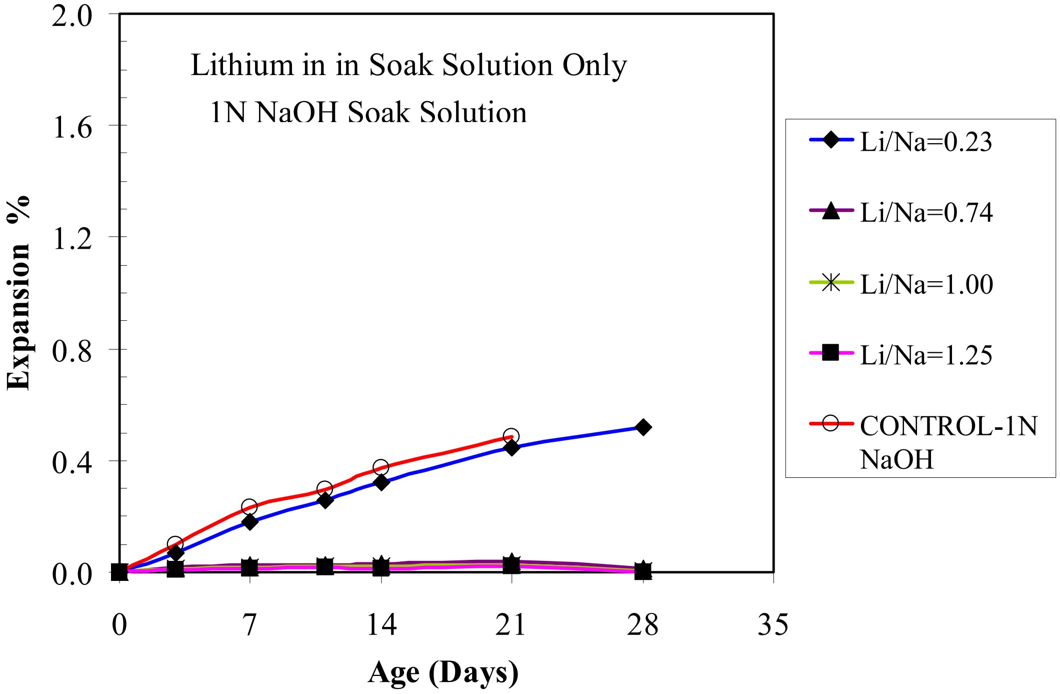


FIGURE 39. EXPANSION OF SPRATT LIMESTONE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN 1N NaOH SOAK SOLUTION ONLY.

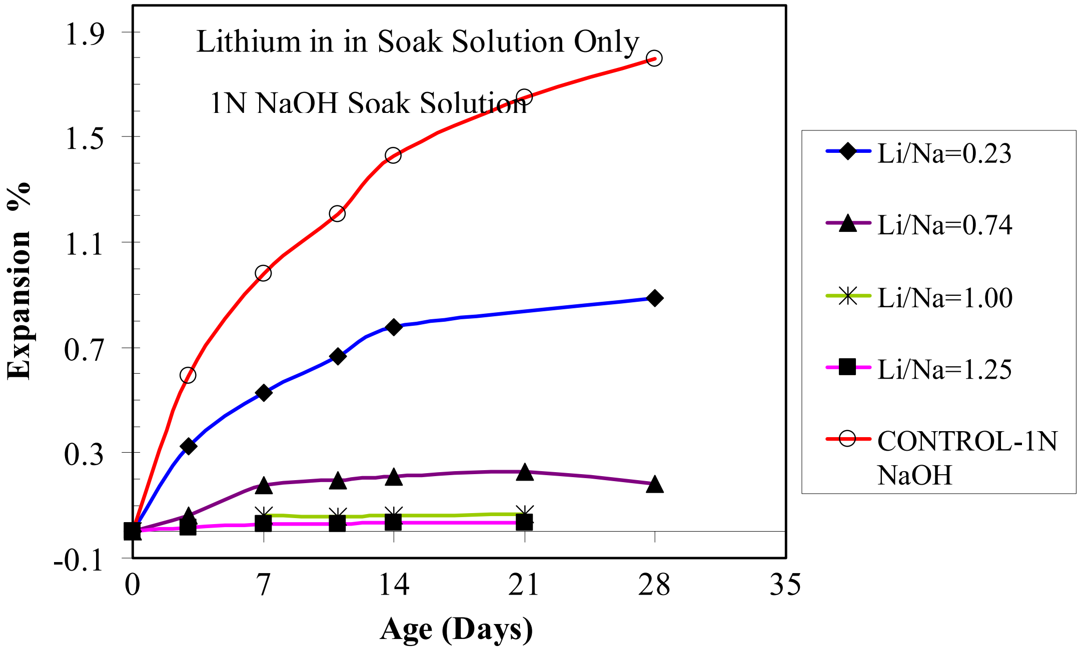


FIGURE 40. EXPANSION OF NM RHYOLITE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN 1N NaOH SOAK SOLUTION ONLY.

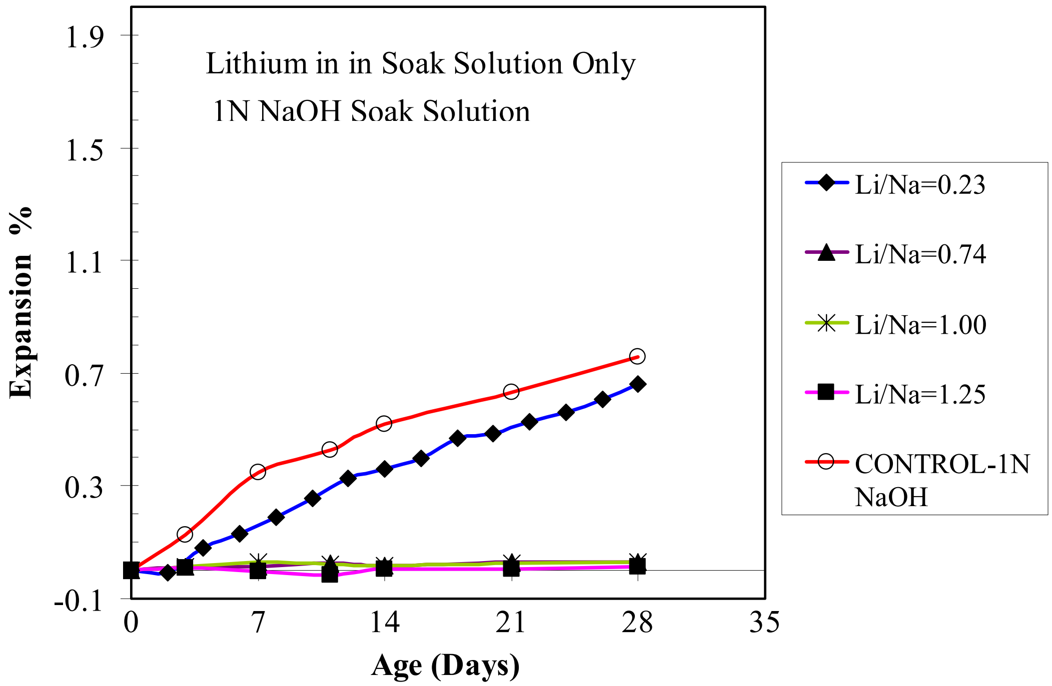


FIGURE 41. EXPANSION OF NC ARGILLITE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN 1N NaOH SOAK SOLUTION ONLY.

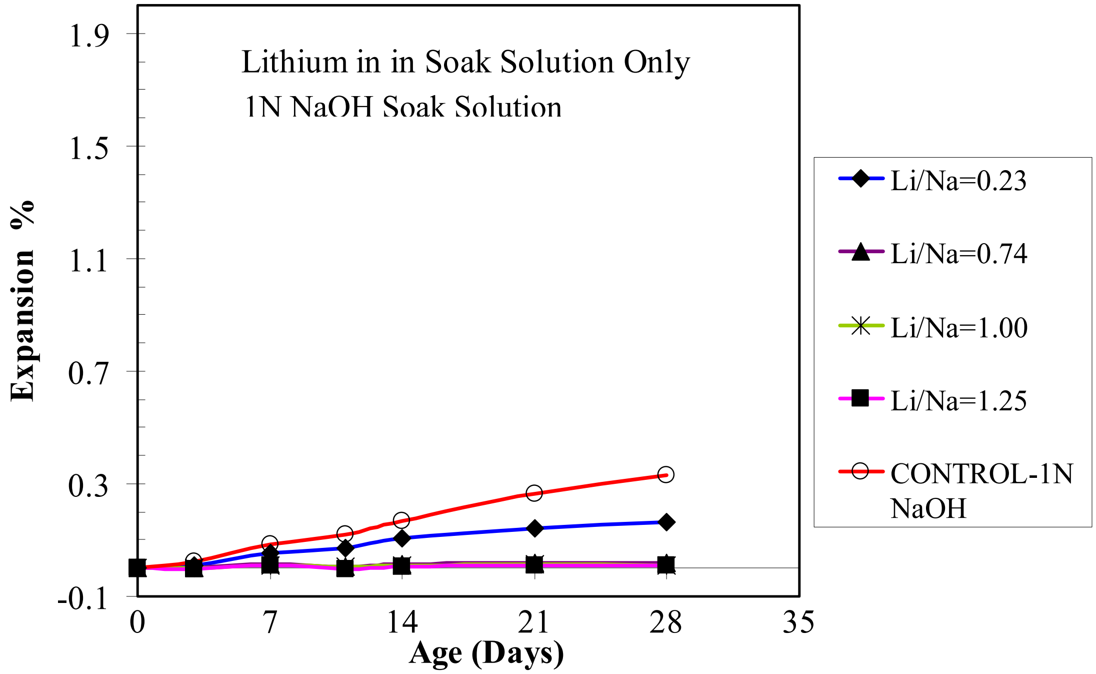


FIGURE 42. EXPANSION OF MN QUARTZITE MORTAR BARS IN STANDARD ASTM C 1567 TEST WITH LITHIUM IN 1N NaOH SOAK SOLUTION ONLY.

5.2. MODIFIED ASTM C 1567 TESTS (IN PRESENCE OF KAc DEICER SOAK SOLUTION)

5.2.1. Lithium Admixture in Mortar Bar Only

Figures 43 through 46 show expansion behavior of mortar bars containing lithium nitrate admixture in mortar bars alone and soaked in KAc deicer solution. No lithium was added to the KAc deicer soak solution. In these tests, lithium was dosed at four different Li/Na molar ratios of 0.25, 0.50, 0.75 and 1.0 based on the alkali content of the cement used. In addition, the expansion of the control mortar bars in KAc deicer (i.e. no lithium added to mortar bars) is also shown as a reference to assess the mitigation potential of lithium admixture.

Based on the results presented in Figure 43 through 46, it is evident that control mortar bars (i.e. without any lithium addition) showed the most expansion in presence of KAc deicer solution. With increasing dosage of lithium in mortar bars significant mitigation in expansion was observed with all the aggregates. However, with exception of NC argillite mortar bars, all the other aggregates expanded over 0.1% at 28 days even at the highest dosage of lithium evaluated in this study. In case of NC argillite, a lithium dosage corresponding to Li/Na molar ratio of 0.50 was effective in mitigating expansions to 0.1% at 28 days.

In case of NM rhyolite mortar bars, a lower dosage of lithium (Li/Na molar ratio of 0.25) appears to develop a pessimum condition that results in expansion higher than control test specimens. However, it should be noted that at the levels of expansion observed in NM rhyolite mortar bars (~ 2%) the pessimum dosage has no meaningful implication.

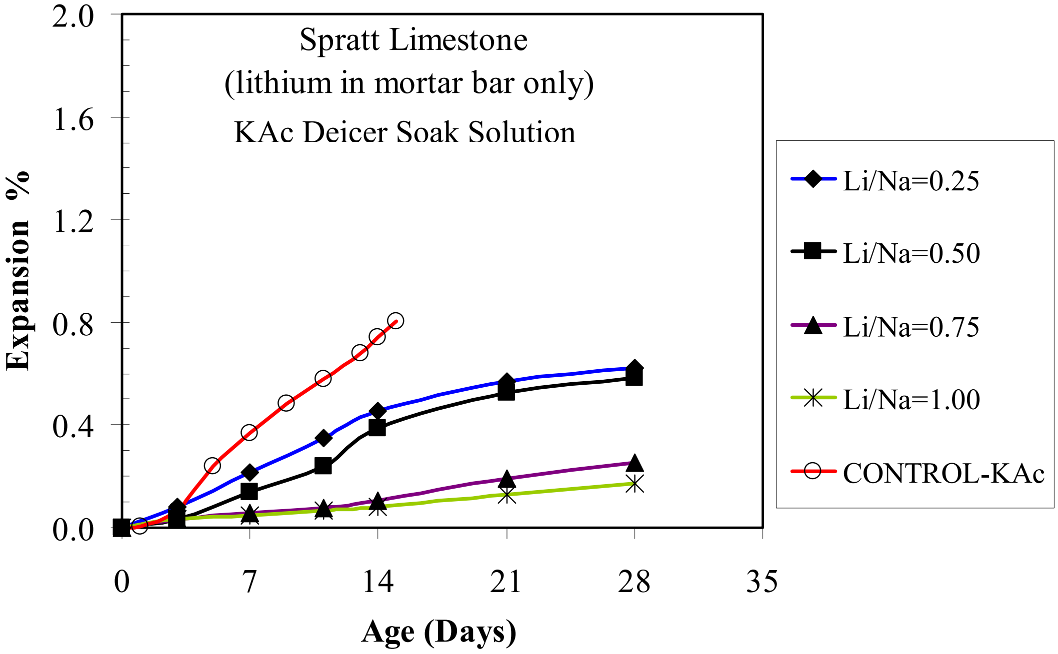


FIGURE 43. EXPANSION OF SPRATT LIMESTONE MORTAR BARS CONTAINING LITHIUM ADMIXTURE AND SOAKED IN KAc DEICER SOLUTION

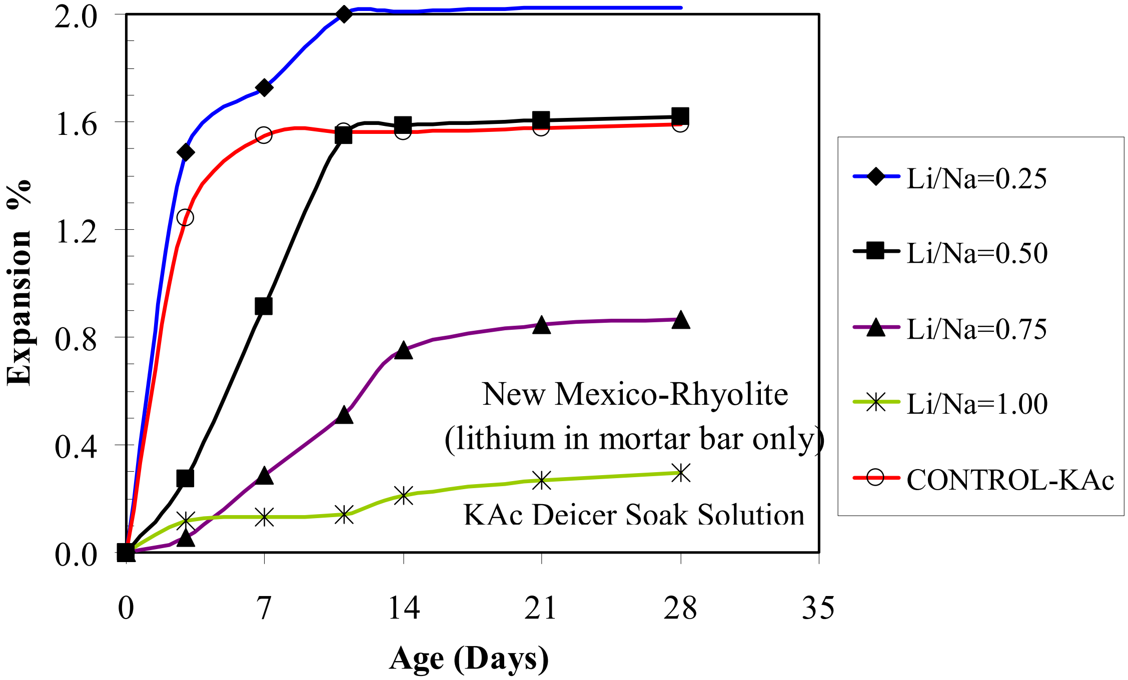


FIGURE 44. EXPANSION OF NM RHYOLITE MORTAR BARS CONTAINING LITHIUM ADMIXTURE AND SOAKED IN KAc DEICER SOLUTION

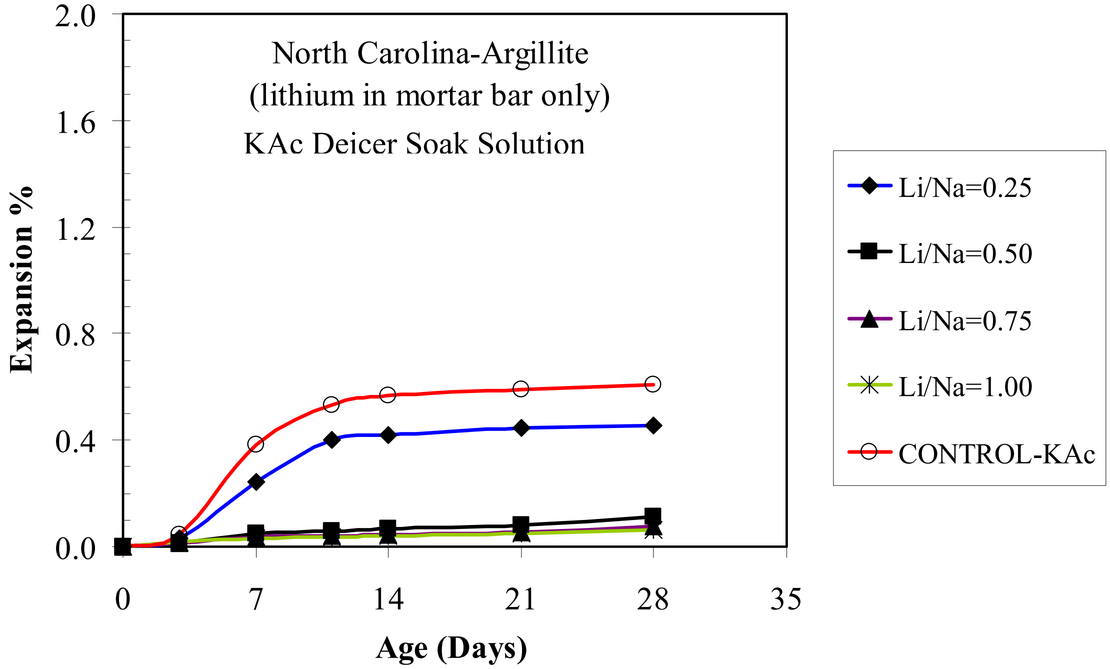


FIGURE 45. EXPANSION OF NC ARGILLITE MORTAR BARS CONTAINING LITHIUM ADMIXTURE AND SOAKED IN KAc DEICER SOLUTION

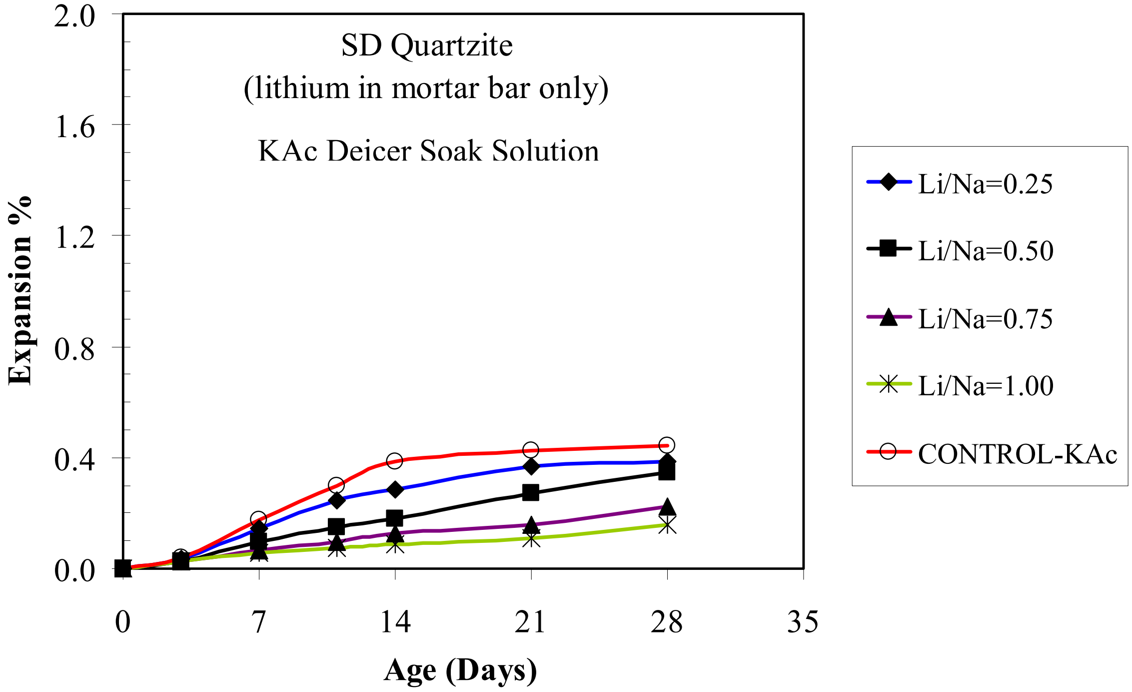


FIGURE 46. EXPANSION OF SD QUARTZITE MORTAR BARS CONTAINING LITHIUM ADMIXTURE AND SOAKED IN KAc DEICER SOLUTION

5.2.2. Lithium Admixture in Soak Solution Only

5.2.2.1. Li/K Molar Ratio of Soak Solution = 0.19

Figures 47 through 50 show the expansion behavior of mortar bars soaked in KAc deicer solution that is blended with lithium admixture to achieve a Li/K molar ratio of 0.19. In addition, expansion data of mortar bars soaked in each of the four different concentrations of KAc deicer solutions (1, 2, 3 and 6.15) are presented. Also, expansion results of control mortar bars soaked in 6.4 molar concentration KAc deicer solution and 1N NaOH solution are provided for reference.

Based on the results presented in Figures 47 through 50, it is evident that with exception of NM rhyolite, mortar bars prepared with all the other aggregates responded favorably to a lithium blended deicer soak solution at a Li/K molar ratio of 0.19. This finding was valid at each of the four concentrations of the KAc deicer solution evaluated in this study.

In case of NM rhyolite mortar bars, with exception of soak solution consisting of 1 molar KAc deicer solution with a Li/K molar ratio of 0.19, all the other blends of KAc deicer with lithium nitrate solution caused significant expansion.

5.2.2.2. Li/K Molar Ratio of Soak Solution = 0.74

Figures 51 through 54 show the expansion behavior of mortar bars soaked in KAc deicer solution that is blended with lithium admixture to achieve a Li/K molar ratio of 0.74. Expansion data of mortar bars soaked in each of the three different concentrations of KAc deicer solutions (1, 2 and 3 molar) are presented in these Figures. Also, expansion results of control mortar bars soaked in 6.4 molar concentration KAc deicer solution and 1N NaOH solution are provided for reference.

Results presented in Figures 51 through 54 reveal that mortar bars prepared with all aggregates, including NM rhyolite, showed no significant expansion in the presence of the lithium blended deicer soak solution at a Li/K molar ratio of 0.74. The concentration of KAc deicer soak solution did not prove to be a factor in influencing the expansions at Li/K molar ratio of 0.74.

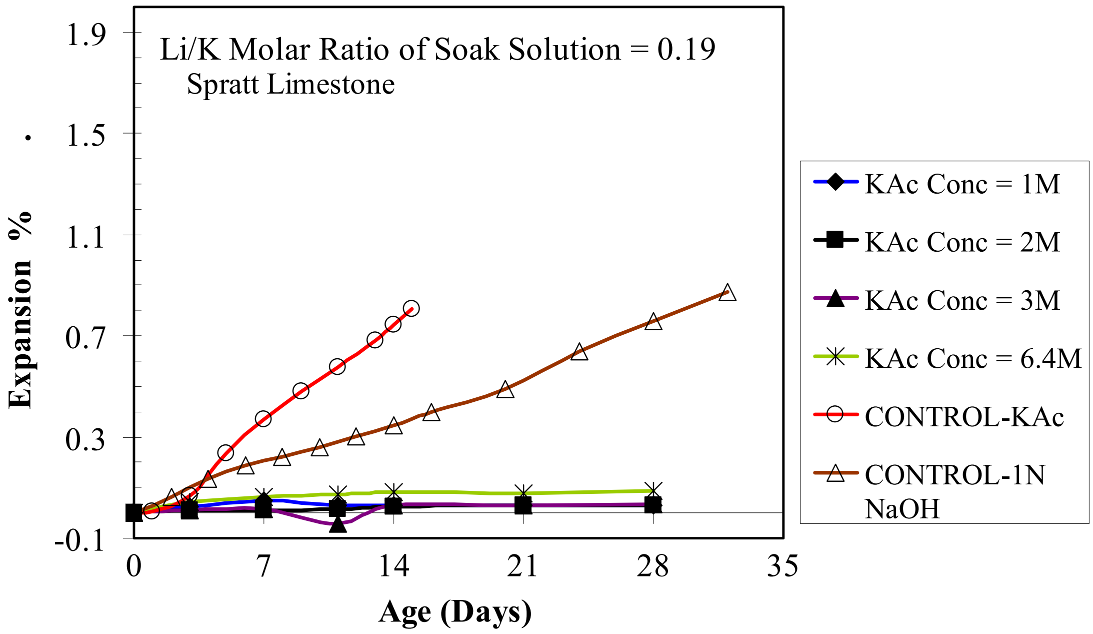


FIGURE 47. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF SPRATT LIMESTONE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.19)

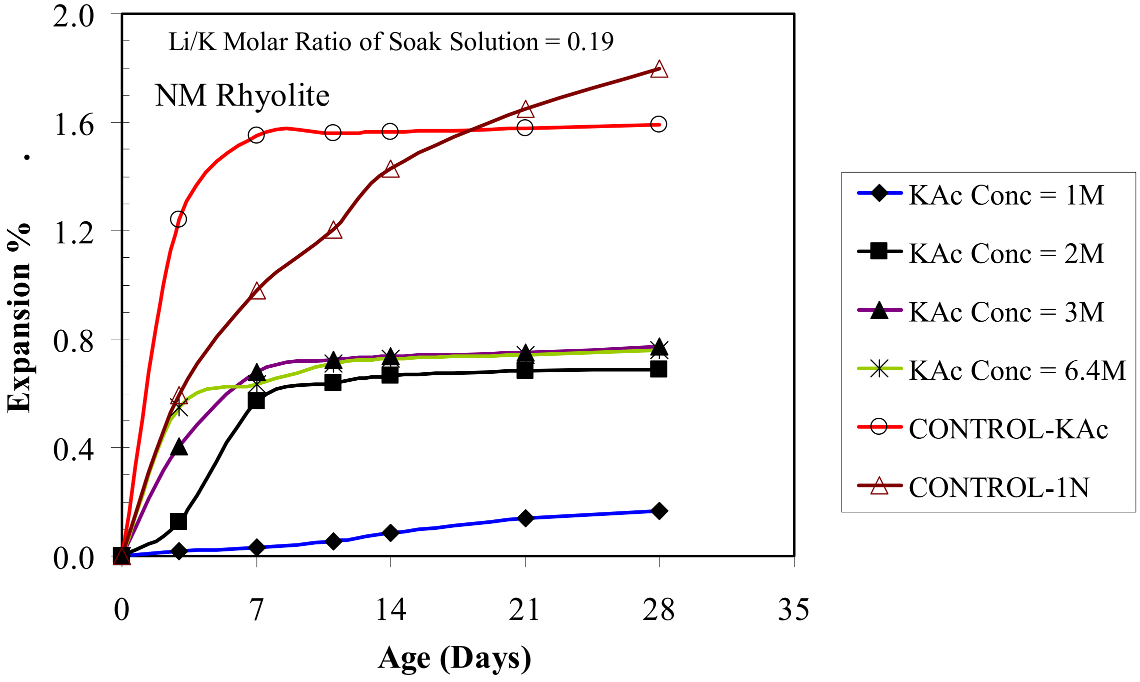


FIGURE 48. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF NM RHYOLITE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.19)

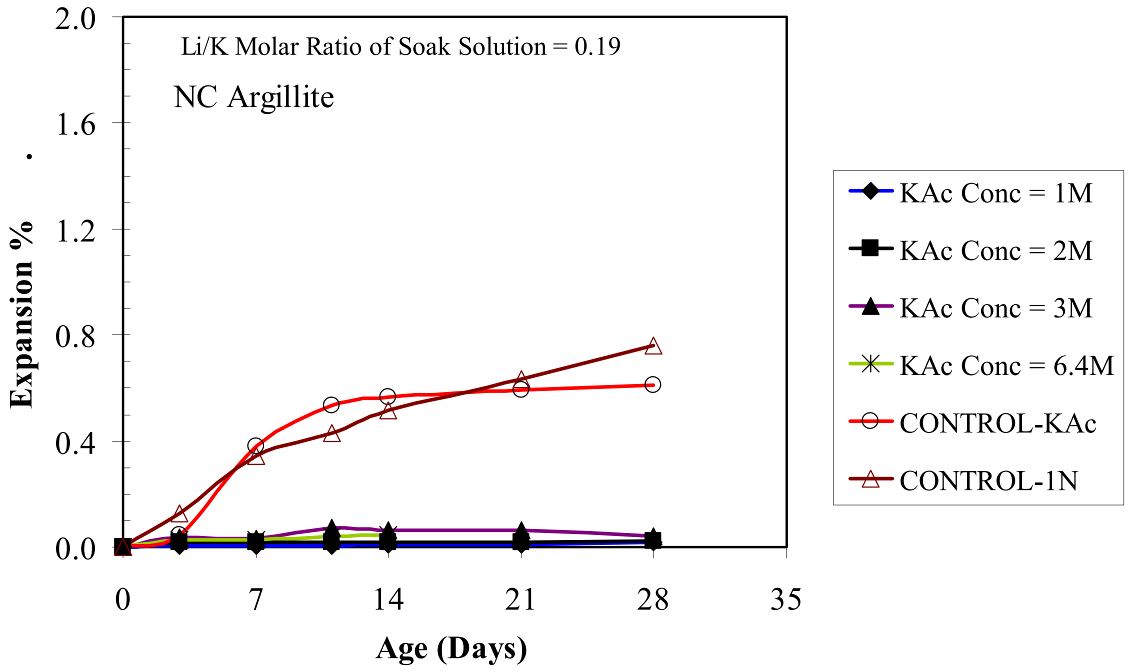


FIGURE 49. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF NC ARGILLITE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.19)

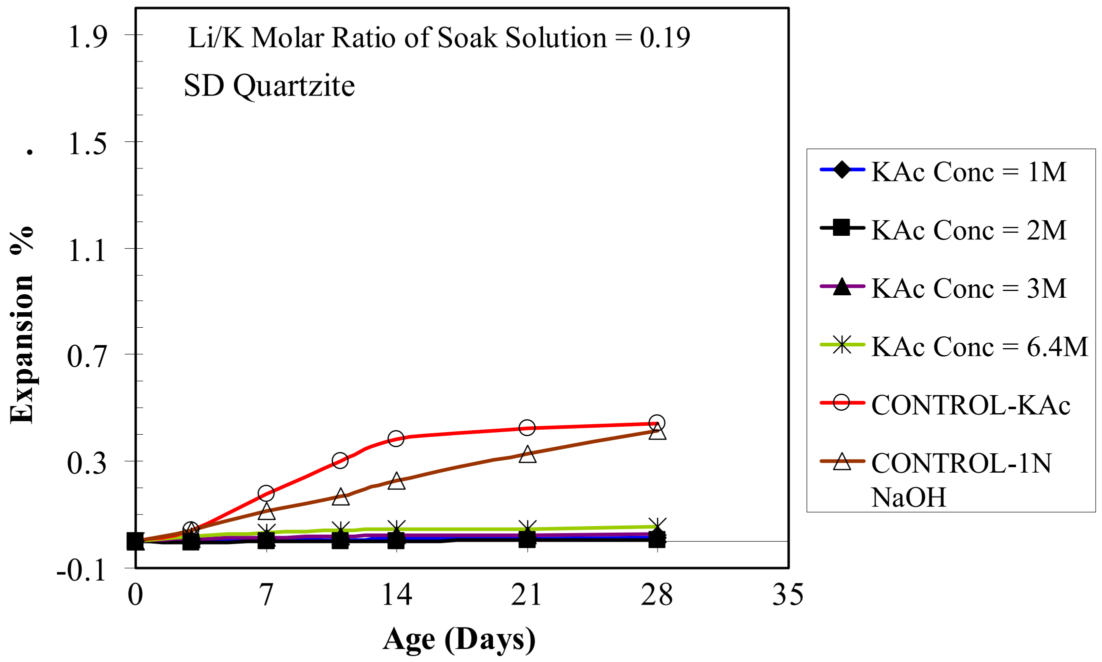


FIGURE 50. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF SD QUARTZITE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.19)

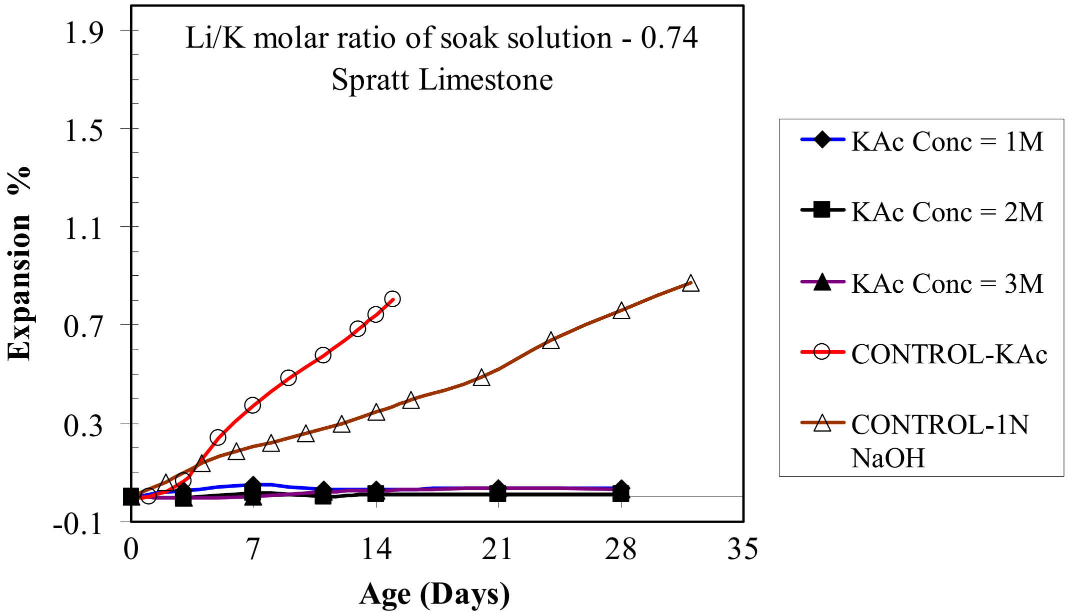


FIGURE 51. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF SPRATT LIMESTONE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.74)

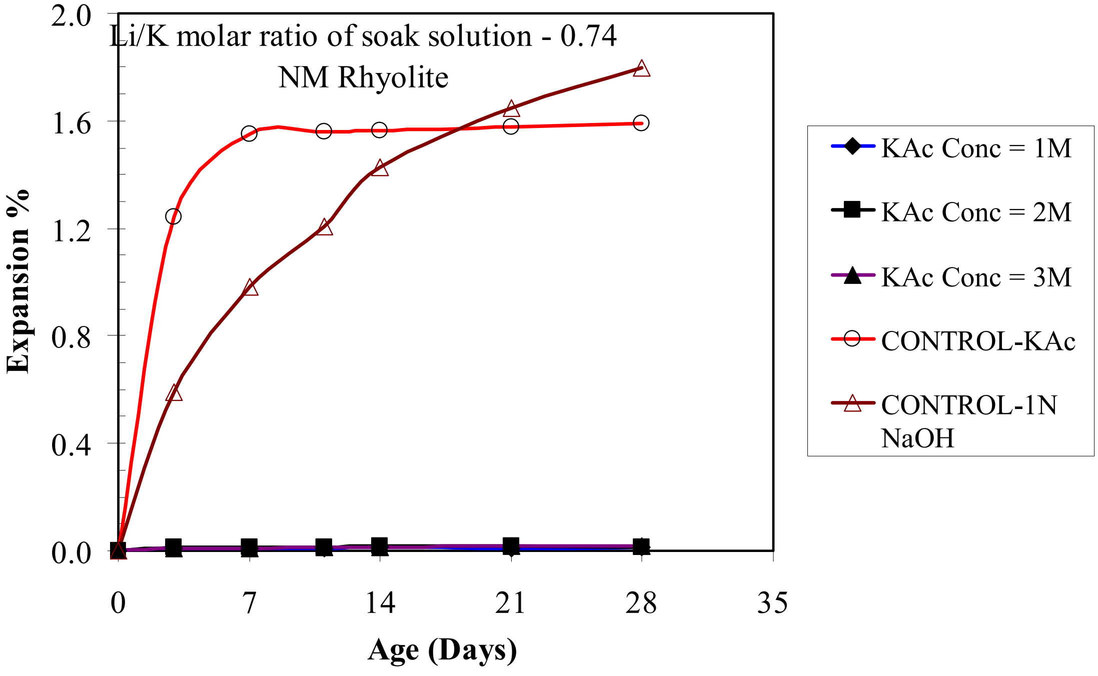


FIGURE 52. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF NM RHYOLITE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.74)

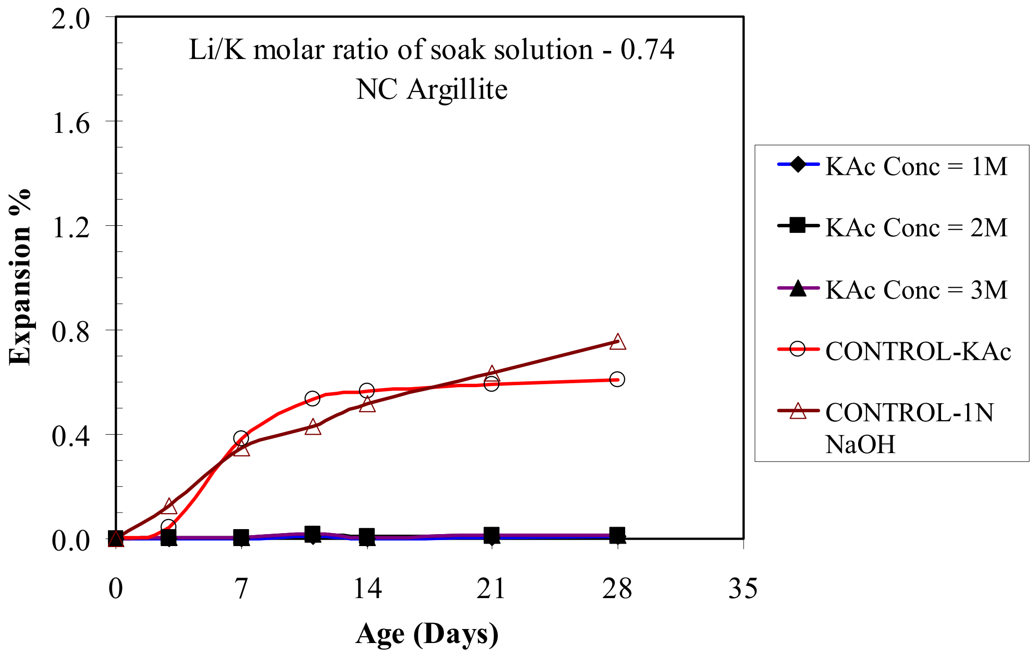


FIGURE 53. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF NM RHYOLITE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.74)

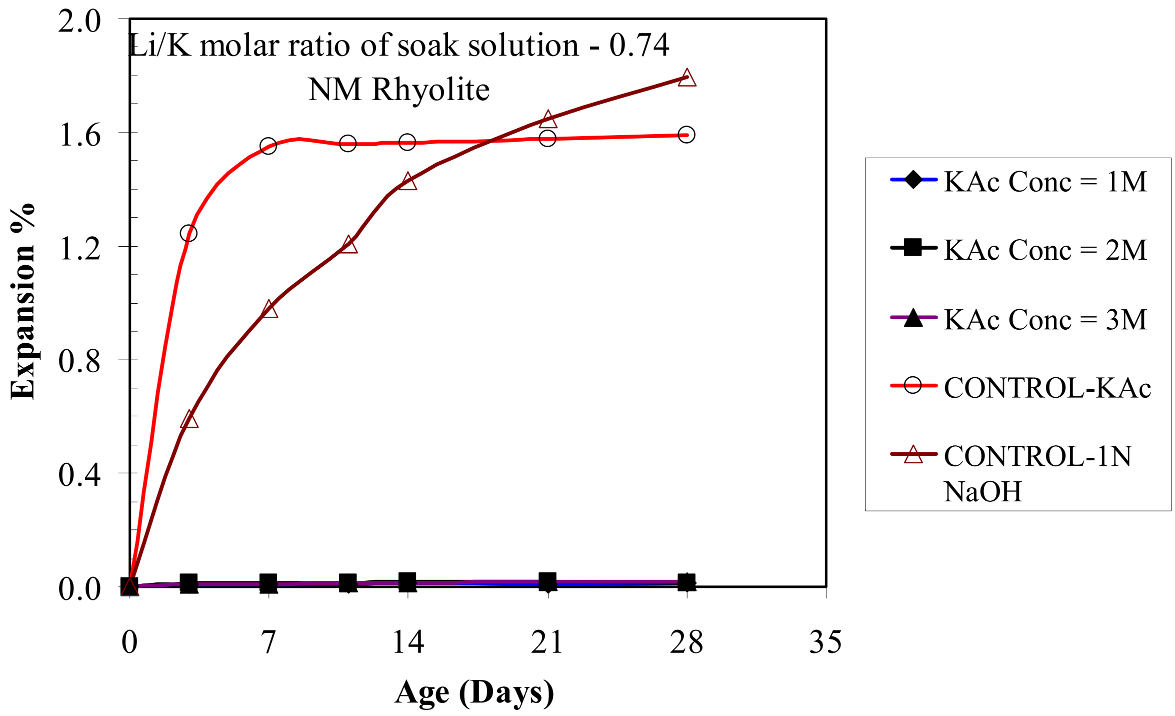


FIGURE 54. INFLUENCE OF LITHIUM NITRATE IN POTASSIUM ACETATE DEICER SOLUTION ON EXPANSION OF NM RHYOLITE MORTAR BARS (Li/K MOLAR RATIO OF SOAK SOLUTION = 0.74)

6. SUMMARY AND DISCUSSION OF PRINCIPAL FINDINGS

This section presents the summary of principal findings from the research conducted as part of IPRF 04-8 study. In this study a series of standard and modified ASTM C 1260 tests and ASTM C 1567 tests were conducted to establish (i) the alkali-silica reactive nature of selected aggregates (ii) the potential of four supplementary cementing materials (three fly ashes and one slag) and one lithium admixture (30% lithium nitrate solution) to mitigate ASR in presence of 1N NaOH and KAc deicer solutions.

In this study, a mortar-bar expansion limit of 0.1% at 14 days in the standard and the modified ASTM C 1260 and ASTM C 1567 tests was employed as the benchmark limit, against which performance of aggregates and mitigation potential of selected measures were evaluated. This expansion limit was based on the FAA Advisory Circular AC 150/5370-10B. The performance of aggregates and the ASR-mitigation measures were also evaluated against an expansion limit of 0.1% at 28 days, where relevant. This more stringent limit represents the specification imposed in the Northwest Mountain Region Airport Division.

The principal findings from this study are as follows:

6.1. EVALUATION OF AGGREGATE REACTIVITY IN THE STANDARD AND THE MODIFIED ASTM C 1260 TESTS

Mortar bars prepared with Spratt limestone, NM rhyolite, NC argillite and SD quartzite aggregates expanded well over 0.1% at 14 days in the standard ASTM C 1260 test, thus suggesting the reactive nature of these aggregates. Among these aggregates, mortar bars prepared with NM rhyolite showed the most expansion at 14 days (1.43%), followed by NC argillite (0.52%), Spratt limestone (0.35%) and SD quartzite (0.23%). With a 14-day expansion of 0.04%, IL dolomite aggregate proved to be a non-reactive aggregate in the standard ASTM C 1260 test. Application of the 28-day expansion limit criterion to all the aggregates (i.e. < 0.1% at 28 days), did not reclassify the reactivity of the aggregates.

1. In the modified ASTM C 1260 test with KAc deicer as the soak solution, mortar bars prepared with all the reactive aggregates (NM, Spratt, NC and SD) showed very high levels of expansion at 14 days (i.e. > 0.2%). Among these aggregates, mortar bars containing NM rhyolite showed the maximum expansion at 14 days (1.56%) followed by Spratt limestone (0.74%), NC argillite (0.57%) and SD quartzite (0.38%). In contrast to the behavior of reactive aggregates, mortar bars containing the non-reactive IL dolomite showed an expansion of only 0.05% at 14 days, and did not increase any further at later ages.

2. The magnitude of 14-day expansions of mortar bars containing reactive aggregates in the modified ASTM C 1260 test were significantly higher than those observed in the standard ASTM C 1260 tests. Also, the rate of expansion of mortar bars in the modified ASTM C 1260 tests was significantly greater than that observed in the standard ASTM C 1260 test for all the reactive aggregates. This indicates that the aggregate reactivity as characterized by the standard ASTM C 1260 test may not be very representative in situations where exposure to potassium acetate deicer solutions is to be expected.

6.2. EVALUATION OF FLY ASH IN THE STANDARD AND THE MODIFIED 1567 TESTS

6.2.1 Results from the Standard ASTMC 1567 Tests

Results from the standard ASTM C 1567 tests conducted to evaluate the ASR mitigation potential of three fly ashes suggested that low-lime fly ash was the most effective in controlling the expansions in presence of the 1N NaOH soak solution, followed by intermediate lime fly ash and high-lime fly ash. With low-lime and intermediate-lime fly ashes, a dosage of 35% by cement replacement yielded the highest level of mitigation, followed by dosages of 25% and 15%. In case of high-lime fly ash, although significant reduction in expansions of mortar bars was realized at 35% dosage level, the 14-day expansions of mortar bars were above 0.1% for all the reactive aggregates. At dosages of 25% and 15%, mortars containing high-lime fly ash yielded expansions similar to those observed in control mortar bars. Thus, high-lime fly ash was not effective as an ASR mitigation measure at the dosage levels considered in this study.

6.2.2. Results from the Modified ASTM C 1567 Tests

Results from the modified ASTM C 1567 tests indicate the use of low-lime fly ash at a dosage of 35% to be the most effective measure in controlling expansion in mortar bars exposed to KAc deicer solution at all ages. While low-lime fly ash at 25% dosage level was effective in mitigating expansions of mortar bars with Spratt, NC and SD aggregates to below 0.1% at both 14 and 28 days, mortar bars with NM aggregate showed expansions below 0.1% at only 14 days, but exceeded 0.1% at 28 days. At 15% dosage level low-lime fly ash was effective only with NC argillite and SD quartzite at 14 days.

Intermediate-lime fly ash proved to be effective in mitigating expansions at 35% and 25% dosage levels with all the aggregates at 14 and 28 days, with exception of NM rhyolite aggregate. High-lime fly ash was found to be ineffective in mitigating expansions of mortar bars with all aggregates, at all dosage levels. In specific cases (for instance with NM rhyolite, NC argillite and SD quartzite aggregates), mortar bars containing high-lime fly ash at 15% and 25% dosage level showed higher expansions than respective control mortar bars (i.e. with no high-lime fly ash).

Mortar bars containing non-reactive IL dolomite aggregate and high-lime fly ash showed higher expansions in presence of KAc deicer solution compared to control mortar bars. This behavior clearly suggests possible deleterious interactions between high-lime fly ashes and KAc deicer solution.

6.2.3. Microstructure of Specimens from Standard and Modified ASTM C 1567 Tests

Microstructural examination of mortar bars was conducted on specimens from the standard and the modified ASTM C 1260 and 1567 tests at 28 days. In these investigations, mortar bars prepared using Spratt limestone and the three fly ashes at 25% dosage level were considered. Microstructure examination of these specimens revealed the nature of the distress observed in the standard ASTM C 1260 and 1567 tests (using 1N NaOH soak solution) and the modified ASTM C 1260 and 1567 tests (using KAc deicer soak solution).

The following are the specific findings:

a) Spratt limestone mortar bars subjected to the standard ASTM C 1260 tests (i.e. in 1N NaOH soak solution with no mitigation measures) showed extensive cracking in the aggregate particles and the surrounding paste. The cracks typically traversed through aggregate particles and the paste. The cracks were in-filled or lined with ASR gel and often the paste surrounding the crack was infused with ASR gel. The predominant alkali ion present in the ASR gel was sodium (Na).

b) Spratt limestone mortar bars subjected to the modified ASTM C 1260 tests (i.e. in KAc deicer solution with no mitigation measure) showed extensive cracking in the aggregate-paste interface and paste, with relatively low intensity cracking in the aggregate particle. Often, the cracks in the aggregate particle were filled with dense ASR gel. However, the cracks in the paste and at the aggregate-paste interface were wide and empty. The paste located adjacent to the cracks was heavily infused with potassium from the KAc deicer solution.

c) Spratt limestone mortar bars with low-lime and intermediate-lime fly ashes at 25% dosage and subjected to the standard ASTM C 1567 tests (i.e. with 1N NaOH soak solution) showed presence of some moderate level of cracking, predominantly in the paste and at the aggregate-paste interface. This level of cracking was consistent with the expansion observed in the standard ASTM C 1567 test (~ 0.27% and 0.37% at 28 days). Although no isolated deposits of ASR gel were evident in these specimens, the paste surrounding the aggregate particles was heavily infused with sodium and exhibited composition similar to that of typical ASR gel.

d) In contrast, mortar bars with high-lime fly ash showed extensive cracking through aggregates and paste. Typically the cracks were found to be wider than those observed with low-lime or intermediate-lime fly ash mixtures. The walls of cracks in aggregate particles were typically lined with ASR gel. The paste adjacent to the cracks in the paste was infused with ASR gel. The cracks in the paste were characteristic of a desiccated ASR gel.

e) Spratt limestone mortar bars with low-lime and intermediate-lime fly ashes at 25% dosage and subjected to modified ASTM C 1567 tests showed moderate level of cracking in the aggregate-paste interface and paste regions, compared to that observed with high-lime fly ash. Some minor cracking was also observed in the peripheral region of the aggregate particles. The paste adjacent to the aggregate particle was heavily infused with potassium, presumably from the KAc deicer soak solution.

f) In the modified ASTM C 1567 tests with KAc deicer soak solution, mortar bars containing high-lime fly ash showed extensive cracking in the paste and aggregate-paste interface region. Often a dense ASR gel rim formation was observed at the aggregate-paste interface.

6.3. EVALUATION OF SLAG IN THE STANDARD AND THE MODIFIED 1567 TESTS

6.3.1. Results from the Standard ASTM C 1567 Tests

Results from the standard ASTM C 1567 tests indicated slag to be an effective ASR mitigation measure at 50% dosage level with all reactive aggregates evaluated, with exception of NM rhyolite. At 40% dosage, slag was found to be an effective ASR mitigation measure only with NC argillite aggregate at 14 days. With all other aggregates, 40% slag dosage was not adequate to mitigate expansion to below 0.1% at 14 days or 28 days.

6.3.2. Results from the Modified ASTM C 1567 Tests

Results from the modified ASTM C 1567 tests showed slag to be an effective ASR mitigation measure in presence of KAc deicer solution only at 50% dosage level with all reactive aggregates evaluated, including NM rhyolite aggregate. However, at 40% dosage level slag was effective only with NC argillite aggregate. Expansions in mortar bars prepared with all the other reactive aggregates and 40% slag, exceeded 0.1% at 14 days in presence of KAc deicer solution.

6.4. EVALUATION OF LITHIUM IN THE STANDARD AND THE MODIFIED 1567 TESTS

30% lithium nitrate admixture in mortar bars was found to be an effective ASR mitigation measure for all the reactive aggregates in the standard ASTM C 1567 test, when the Li/Na ion molar ratio in the mortar bar and the 1N NaOH soak solution was maintained at 0.74 or greater.

While the use of lithium admixture in mortar bars and soak solution at a dosage corresponding to Li/Na molar ratio of 0.23 was not effective in mitigating expansions of any reactive aggregates, this level of lithium dosage resulted in a pessimum condition in mortar bars containing NC argillite (i.e. caused more expansion in lithium containing mortar bars than control mortar bars).

Additional tests revealed that lithium was very effective in mitigating expansion in mortar bars, even when lithium was added to 1N NaOH soak solution alone. In these tests, it was observed that mortar bars containing Spratt limestone, NC argillite and SD quartzite were completely mitigated when soaked in solution with a Li/Na molar ratio of 0.74 or greater. However, mortar bars containing NM rhyolite required a Li/Na molar ratio of 1.0 for complete mitigation. This finding suggests that addition of lithium to mortar bar and soak solution at the same molar ratio in the standard ASTM C 1567 test may provide a very conservative response. Additional research is required to determine the appropriate dosage of lithium to be included in the soak solution to alleviate the influence of lithium leaching from mortar bars.

Results from modified ASTM C 1567 tests conducted to evaluate effectiveness of lithium admixtures in presence of KAc deicer solution revealed:

1. When lithium admixture was used in the mortar bars alone, at Li/Na molar ratio of 0.75 or greater, it was effective in mitigating expansions to below 0.1% at 14 days with Spratt limestone, NC argillite and SD quartzite aggregates. However, at 28 days even a higher

lithium dosage at Li/Na molar ratio of 1.0 was not effective in mitigating expansions to below 0.1% for any of the reactive aggregates evaluated in this study.

2. When lithium admixture was used only in the KAc deicer soak solution, it's effectiveness in mitigating expansions in mortar bars was dependant on the combination of KAc deicer solution concentration and the dosage of lithium admixture in the KAc deicer solution expressed as Li/K molar ratio.

   At KAc deicer concentrations of 1, 2, 3 and 6.15 molar solutions, a Li/K molar ratio of 0.19 was adequate to completely mitigate expansions in mortar bars containing Spratt limestone, NC argillite and SD quartzite aggregate. However, mortar bars with NM rhyolite could not be mitigated.

   At KAc deicer concentration of 1, 2 and 3 molar solutions, a Li/K molar ratio of 74 was adequate to completely mitigate expansions in mortar bars of all the reactive aggregates studied in this investigation.

It should be noted that due to significant differences in concentration of the external KAc deicer soak solution and the internal alkali levels within the mortar bars, no experiments were conducted in which lithium was added to both mortar bars and KAc deicer soak solution.

7. CONCLUSIONS

Based on the results presented in Sections 4 and 5 of this report, and the discussion in Section 6 the following general conclusions can be drawn on the effect of potassium acetate deicer on ASR, and effectiveness of fly ash, slag and lithium admixtures in mitigating ASR in presence of potassium acetate deicer solution:

1. Potassium acetate deicer solution caused deleterious levels of expansion in mortar bars containing reactive aggregates. However, no such deleterious expansion is evident in test specimens containing non-reactive aggregate.

2. The magnitude and rate of expansion in mortar bars exposed to potassium acetate deicer solution is significantly greater compared to 1N NaOH solution, particularly at early ages.

3. The effectiveness of fly ash in mitigating mortar bar expansion induced by KAc deicer solution was dependant on the chemical composition of fly ash used and its dosage level in the mixture. In this investigation, a low-lime (CaO = 5.19%) and intermediate-lime (15.74%) fly ash were found to be effective in mitigating expansion induced by KAc deicer solution at 25% cement replacement level, for three of the four reactive aggregates evaluated. Mortar bars with the highly reactive NM rhyolite aggregate could only be mitigated by using a low-lime fly ash at 35% cement replacement level. Similar trends in effectiveness of fly ash were observed in tests with the 1N NaOH soak solution.

4. High-lime fly ash was found to be ineffective in mitigating expansion of mortar bars induced by KAc deicer and 1N NaOH solutions, even at 35% cement replacement level.

5. Slag was effective in mitigating expansions in mortar bars induced by KAc deicer solution at a dosage level of 50% by mass replacement of cement for all the four reactive aggregates evaluated in this study. Studies conducted with a lower dosage level of 40% showed it to be not effective in controlling expansions with three of the four reactive aggregates evaluated.

6. The effectiveness of 30% lithium nitrate solution in mitigating expansion of mortar bars exposed to KAc deicer solution depended on the mode in which lithium was introduced into the mortar bars, the lithium dosage in mortar bar or soak solution, and the concentration of the KAc deicer soak solution.

    a. The addition of lithium into the KAc deicer soak solutions of 1, 2, 3 and 6.15 molar concentration alone at a Li/K molar ratio of 0.19 was very effective in mitigating expansion of mortar bars containing all the reactive aggregates, with exception of NM rhyolite aggregate. The addition of lithium into the KAc deicer soak solution of 1, 2 and 3 molar solutions at Li/K molar ratio of 0.74 was very effective in mitigating expansion of all the reactive aggregates, including NM rhyolite aggregate.
    b. The addition of lithium into mortar bars alone, without addition to KAc deicer soak solution resulted in significant reductions in expansion of mortar bars.

However, lithium admixture was unable to suppress the expansion of mortar bars to less than 0.1% at 14 days, at all the dosage levels investigated.

7. When lithium was added into the mortar bar and 1N NaOH soak solution, a lithium dosage corresponding to a Li/Na molar ratio of 0.74 was found to be adequate in completely mitigating expansion in mortar bars of all the reactive aggregates.

8. RECOMMENDATIONS

Based on the knowledge gained from this investigation, it is recommended that the following additional studies be conducted to develop better strategies to mitigate ASR effects of potassium acetate deicer solutions:

1. This study has employed a limited sample size of three fly ashes and one slag in evaluating the effectiveness of these supplementary cementing materials against the effects of deicing chemicals. Additional sources of fly ash and slag should be investigated before the findings from this study can be generalized.
2. The performance of the mitigation measures evaluated in this study is highly dependant on the nature of the reactive aggregate being considered. It should be noted that most aggregates used in the industry are likely to be far less reactive than those used in this study. Therefore, it is recommended that tests with local materials be conducted to arrive at a decision on the effectiveness of a particular ASR mitigation, rather than applying the findings from this study as a blanket statement.
3. In this study, evidence to suggest deleterious interactions between high-lime fly ash and KAc deicer solution was observed. Additional investigation should be conducted to evaluate these interactions.
4. Evidence from this study suggests that addition of lithium into mortar bars and soak solutions at equivalent molar ratios may not be appropriate in evaluating the performance of lithium admixtures for the combinations of bar dose and soak dose considered in this investigation. Additional research needs to be conducted to determine the lithium dosage that should be used in the mortar bar and the soak solution that will yield practical correlations with the field performance. No method is currently recommended for evaluating Li admixture for use in concrete exposed to deicer yet as a result of this study.

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