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EXAMINATION OF THE EFFECT OF LICI ON ASR GEL EXPANSION

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ABSTRACT

Lithium containing salts have been previously shown to affect expansion caused by ASR, but the mechanisms of action are not understood. The effect of LiCl on an alkali-silica reaction gel in 0.7M NaOH was examined by transmission soft x-ray microscopy. Interpretations of the x-ray images in consideration of quantitative elemental analysis and surface chemistry principles suggests two mechanisms by which lithium may act to reduce expansion of alkalisilica reaction gel. First, lithium may limit repolymerization, effectively reducing the potential for expansion. Second, lithium may to promote the aggregation of relatively larger (but still colloidal) silicate particles.

Keywords: alkali-silica reaction, chemical additives, expansion, lithium, prevention

INTRODUCTION

In new construction, damage to the concrete induced by alkali-silica reaction (ASR) can be avoided through careful materials selection and concrete mixture design and through good construction practices, following the holistic approach described by Mehta [1997, 1994]. However, for existing structures experiencing expansion caused by alkali-silica reaction. additional methods need to be developed to limit damage. Since McCoy and Caldwell [1951] first investigated the use of chemical salts and organic compounds to inhibit expansion caused by ASR, research to reduce expansion in existing structures has been focused on the use of chemical additives. Some lithium salts, including LiCl, have been shown to be particularly effective and have been the focus of much ongoing research [Prezzi et al. 1997, 1998; Ramachandran, 1998; Sakaguchi et al, 1989; Stark, 1992]. However, the mechanisms by which LiCl and other chemical additives decrease expansion in concrete are not understood. This lack of fundamental understanding of the effect of these additives is one obstacle to their use in existing structures to control expansion and damage by ASR. Without understanding the mechanism of control, it is difficult to predict the effectiveness of a chemical additive and to foresee the duration of its control. A more thorough understanding of these mechanisms would aid assessing which additives are most effective over long periods, which doses are optimal, and which delivery methods are most practical.

The objective of this research is to investigate the mechanisms by which one chemical additive, LiCl, may control expansion caused by ASR using two complementary techniques: transmission soft x-ray microscopy and inductively coupled plasma optical emission spectroscopy (ICP-OES). The reaction of alkali-silica reaction gel, obtained from a dam experiencing damage by the reaction, in 0.7M NaOH + 0.1M LiCl solution was observed using transmission soft x-ray microscopy. These images can be compared to images of the alkali-silica reaction gel in sodium hydroxide solution, without the additives, to qualitatively assess the effects of lithium chloride. To aid in the interpretation of the x-ray images, the concentration of silicon, calcium, lithium, and sodium ions in solution were measured by ICP-OES. These results can be used to assess the effect of the chemical additive LiCl on silica dissolution and the incorporation of those elements into a reaction product.

EXPERIMENT

Two techniques were used to investigate the mechanisms by which LiCl affects expansion during alkali attack of siliceous aggregate. Images of the reaction and the reaction product were obtained through transmission soft x-ray microscopy and quantitative elemental analysis was performed by inductively coupled plasma optical emission spectroscopy (ICP-OES). These techniques and the sample preparation performed for this investigation are described below.

Transmission Soft X-ray Microscopy

<u>Technique</u> - The transmission soft x-ray microscope XM-1 was used in this experimental investigation of the alkali-silica reaction (ASR). The Center for X-ray Optics (CXRO) built and operates XM-1 at the Advanced Light Source (ALS) at the E.O. Lawrence Berkeley National Laboratory (LBNL) in Berkeley, California. The Advanced Light Source (ALS) is a third generation synchrotron facility that generates intense light in the ultraviolet and soft x-ray regimes. Soft x-ray wavelengths range between 1 and 50 nm which is 50 to 100 times

longer than hard x-rays and 50 to 100 times shorter than visible light. XM-1 was originally developed to study biological specimens, but this technique has been shown to be advantageous for the study of wet chemistry reactions and hydrated reaction products in situ through application of this technique to the study of the alkali-silica reaction, the pozzolanic reaction, and cement hydration [Kurtis et al, 1997, 1999; Kurtis, 1998].

Transmission soft x-ray microscopy provides the required high spatial resolution to observe the interaction between ASR gel and alkali solutions in situ. In addition, the reactions can be observed over time, in a wet condition, and at normal pressures, features unavailable with most other high resolution techniques. Soft x-rays also reveal information on the internal structure of the sample. A discussion of x-ray optics can be found in Meyer-Ilse [in press], and a detailed description of XM-1 can be found in Meyer-Ilse [1995].

<u>Sample preparation</u> - X-ray microscopy samples were prepared as slurries containing a pessimum amount (SiO₂/Na₂O=3) of finely ground (~300 sieve) particles of alkali-silicate reaction (ASR) gel in alkali solution [Dent Glasser and Kataoka, 1981, 1982]. The ASR gel was obtained from the galleries of the FURNAS Dam, a hydroelectric facility located in Minas Gerais, Brazil. A chemical oxide analysis of the ASR gel, averaged from samples obtained from several on the dam, shows that the gel is composed primarily of SiO₂ with K_{20} , Na₂O, and CaO present in smaller quantities (Table 1).

Loss on Ignition	18.22%		
SO ₃	0.97%		
MgO	0.96%		
SiO ₂	62.10%		
Fe ₂ O ₃	0.31% 2.85% 1.27%		
Al ₂ O ₃			
CaO			
MnO	0.013%		
Na ₂ O	1.083%		
K20	15.73%		
Alkali Equiv.	11.21%		

TABLE 1: Average Composition by Mass of Alkali-Silica Reaction Gel Examined by Soft X-ray Microscopy

To examine the effect of LiCl on the ASR gel from FURNAS Dam, the ground gel was exposed to two solutions: (a) 0.7M NaOH (control/background) and (b) 0.7M NaOH + 0.1M. All solutions were made from deionized, low resistivity (17.8 M Ω ·cm) water and A.C.S. certified chemicals. While several Li-containing chemicals have been shown to decrease expansion, LiCl was selected for this investigation to complement the work of Prezzi et al [1997, 1998] and Rodrigues et al [1999]. The pH of 0.7M NaOH solution measured 12.6, and the pH of the 0.7M NaOH + 0.1M LiCl measured 12.4. Samples were stored in sealed polyethylene vials, and images were recorded within 1 week of sample preparation. Wet chambers [Kurtis et al, 1998; Kurtis, 1999; Kurtis et al 1999] were prepared and contained a 2 or 3 μ l drop of the sample. To prevent the windows from collapsing together and breaking due to the surface tension of the sodium hydroxide solution,

commercially available polystyrene beads of 6µm nominal diameter were inserted between the windows with the sample.

At XM-1, wet samples were observed at a wavelength of 2.4 nm (517 eV) and at an original magnification of 2400x. Exposure times measured 1 to 60 seconds. Images were recorded within 1 week of sample preparation.

Quantitative Elemental Analysis

<u>Technique</u> - Quantitative elemental analysis was performed using an ICP-OES (Thermo Jarrell Ash Iris Model). This instrument can measure ultratrace, trace, minor, and major concentrations of elements in solution under one set of experimental parameters [Fassel, 1978]. Detection limits for the instrument are in the parts per billion range for most elements, including calcium, lithium, sodium, and silicon [Barnard *et al.*, 1993].

Each element emits a characteristic optical ray pattern which can be described by a particular light wavelengths. To measure concentrations of elements in solution, wavelengths which are characteristic of the elements of interest must be identified in the experiment set-up. Table 2 shows the wavelengths selected for the quantitative elemental analysis described here. To produce the most accurate measurements, three wavelengths for a particular element of interest were selected when possible. The instrument was calibrated using Ca, Li, Na, and Si standards in a nitric acid matrix which were diluted to 100 ppm and 10 ppm. Calibration was checked periodically during the experiment and was repeated when necessary.

	Wavelength		
Element	(nm)		
Ca	184.006		
Ca	315.887		
Ca	317.933		
Li	610.362		
Li	670.784		
Na	588.995		
Na	589.592		
Si	212.412		
Si	251.612		
Si	288.158		

TABLE 2: Wavelengths Used to Detect Calcium, Lithium, Sodium, and Silicon in the Quantitative Elemental Analysis.

<u>Sample preparation</u> - To aid in interpretation of the x-ray images, a quantitative elemental analysis was performed using ICP-OES to measure the concentration of calcium, lithium, sodium, and silicon in solution. Because of the large quantity of silica required for the quantitative elemental analysis by ICP-OES, slurries were prepared using chemical grade silica gel. Four replicates, diluted 100x, were prepared for each sample: (a) filtrate from 0.7M NaOH + silica gel, and (b) filtrate from 0.7M NaOH + 0.1M LiCl + silica gel. The slurries were prepared with the pessimum amount (SiO₂/Na₂O=3) of chemical grade silica gel (70-230 mesh) [Dent Glasser and Kataoka, 1981, 1982]. Samples were stored in sealed

polyethylene vials in a water bath with constant temperature 22 C for 1 week. In addition, for each sample, background solutions of 0.7M NaOH and 0.7M NaOH + 0.1M LiCl, also diluted 100x and without silica gel, were reserved to act as controls. All solutions were made from deionized, low resistivity (17.8 M Ω ·cm) water, and all reagents were A.C.S certified and were obtained from Fisher. Solutions were prepared using Nalgene volumetric flasks (Class B) instead of glassware to avoid the introduction of silicon to the solutions.

After 1 week, the slurries were filtered to 0.45 μ m using polystyrene filtration units. Filtrates were diluted 100x and were stored in sealed Nalgene flasks until analysis by ICP-OES. Reaction products retained during filtration were placed in desiccators and reserved for analysis by x-ray diffraction.

Quantitative elemental analysis measurements were performed in duplicate for each sample. Thus, with four replicates, a total of eight measurements were made. For the background solutions, the analysis was performed in duplicate just prior to the analysis of the four replicate samples and just after the analysis of the four replicates.

RESULTS AND DISCUSSION

Alkali-silica reaction gel from the FURNAS Dam in Brazil was exposed lithium chloride in the presence of 0.7M NaOH to examine the effect of this chemical additive on the morphology of the gel with the aim of gaining a better understanding of the mechanisms by which LiCl affects expansion caused by ASR. Images of the gel in the presence of LiCl will be compared to images of the alkali-silica reaction gel dry and in 0.7M NaOH solution, a solution of alkali concentration similar to that of the concrete pore solution.

To examine the effect of LiCl on the dissolution of silica in 0.7M NaOH and to aid in the interpretation of the x-ray images, a quantitative elemental analysis was performed using ICP-OES. Concentrations of calcium, lithium, silicon, and sodium were measured in dilutions made from the filtrates of slurries containing silica gel in 0.7M NaOH with and without LiCl. Results from the quantitative elemental analysis are discussed first, and results from the transmission soft x-ray microscopy will be examined in relationship to the results from the quantitative elemental analysis.

Results from Quantitative Elemental Analysis

The alkali-silica reaction in concrete is initiated by the dissolution of silicates present in the aggregate. The product of the reaction is an alkali-silica reaction gel formed by repolymerization of the dissolved silica incorporating some of the alkalis from the concrete pore solution [Dent Glasser and Kataoka, 1981]. Smaller amounts of other ions present in the pore solution, generally in lower concentrations than the alkalis, may also become incorporated into the gel product. In this experiment, slurries containing the pessimum amount $(SiO_2/Na_2O=3)$ of chemical grade silica gel in 0.7M NaOH were allowed to react for 1 week in the presence and absence of lithium chloride. After this exposure period, the samples were filtered and concentrations of Ca, Li, Na, and Si in the filtrate were measured by ICP-OES. Measurements of Si concentration in solution may be interpreted simply as indicators of dissolution. However, when accompanied by a significant decrease in Na (and

possibly Ca and Li) concentration, repolymerization of an alkali-silica reaction gel product may be indicated.

Results from the quantitative elemental analysis are shown in Table 3. The results presented in Table 3 are averages calculated from the concentrations measured in duplicate at two (Li, Si) or three (Na, Ca) wavelengths for each sample. Standard deviations have also been calculated and are presented in parenthesis in units of ppm. All solutions and filtrates have been diluted 100x.

Samples diluted from 0.7M NaOH solution and the filtrate of the 0.7M NaOH + silica gel slurry serve as the controls in this experiment. For the 0.7M NaOH sample, the sodium concentration measures 150 ppm and trace amounts of silicon are measured. Because this sample contains no silica gel, the trace amount may have been dissolved in the water used to make the solutions or may simply reflect the error tolerances of the instrument. Silicon concentrations are much higher in the filtrate of the 0.7M NaOH + silica gel slurry and measure nearly 400 ppm. The sodium concentration is approximately equal to the concentration in the 0.7M NaOH solution. Trace amounts of calcium are measured in the sample containing silica gel, suggesting that the chemical grade silica gel itself may contain some calcium, although this is not indicated on the label. These results indicate that dissolution of the silica gel has occurred in the 0.7M NaOH solution, but that little sodium, if any (depending on instrument error), from the solution has been adsorbed onto the silica gel or incorporated into an alkali-silica reaction gel.

Element	Average Concentration in ppm (with Standard Deviation in ppm)				
	0.7 M NaOH ¹	0.7M NaOH & silica gel ²	0.7 M NaOH + 0.1M LiCl ¹	0.7 M NaOH + 0.1M LiCl & silica gel	
Ca 0	0	0.48	0	0.39	
		(0.061)		(0.054)	
Li 0	0	0	7.46	6.48	
			(2.28)	(1.44)	
Na 150 (6.0	150.13	147.61	152.3	137.98	
	(6.67)	(10.12)	(9.94)	(5.66)	
Si (0	0.17	397.00	0.05	339.48	
	(0.084)	(31.33)	(0.010)	(19.58)	

TABLE 3: Results from Quantitative Elemental Analysis; Concentrations of Calcium, Lithium, Sodium, and Silicon and Standard Deviations Are Given in ppm for Filtrates Diluted 100x.

Control solution.

² Control slurry.

The diluted 0.7M NaOH + 0.1M LiCl solutions are the third control. The ICP-OES measurements show the sodium concentration in the samples to be 152 ppm which is approximately the same as in the 0.7M NaOH solution, demonstrating the accuracy of the experiment methodology. As in the control sample, only trace amounts of silicon are measured. In the diluted 0.7M NaOH + 0.1M LiCl solution, lithium concentration measures 7.5 ppm.

In the dilution from the slurry of 0.7M NaOH + 0.1M LiCl + silica gel, lithium concentration measures 6.5 ppm. Measurements of lithium by ICP-OES are known to drift, as is evidenced by the relatively high standard deviations for these measurements. As a result, no conclusions can be drawn from these measurements. However, silicon and sodium concentration in the diluted filtrate are 15% and 6.8% less than in the 0.7 M NaOH + silica gel control sample. These results indicate that less silica is present in solution in the presence of lithium chloride, and that the product of the reaction of silica gel in the 0.7M NaOH + 0.1M LiCl solution contains some sodium or that sodium has been adsorbed at the silica gel surface.

Results from Transmission Soft X-ray Microscopy

Previous x-ray microscopy studies have shown that alkali-silica reaction gel from FURNAS Dam partially dissolves and repolymerizes as an expansive gel in the presence of 0.7M NaOH solution in slurries prepared in the pessimum proportion [Kurtis et al, 1998; Kurtis, 1999; Kurtis et al, 1999]. While x-ray images show that the interior or central regions of the original gel particles remain dense, edges of the of the particle are more transparent to x-rays. In addition, regions near the center of the original particle appear to have no discernible internal structure, while the cloud-like forms around the perimeter of the particle appear to have regions of varying density. These areas of decreased and varying density have suggested that the silica network, originally dense and less pervious, has become less ordered, allowing the ions in the surrounding solution to permeate more freely into the silicate network. Solubility of silicates is relatively high in strong alkali solutions, but the dissolution process may become limited because of low diffusivity in the sample (as in concrete). Thus, the area near the site of dissolution may reach a saturation concentration for the silicate, in this case an alkali-silica reaction gel, at a given pH value. As a result, the silica species in solution repolymerize, incorporating alkali cations into the relatively less dense alkali-silicate reaction gel.

Figures 2 and 3 are x-ray images of alkali-silica reaction gel in 0.7M NaOH + 0.1M LiCl solution. The images show products of varying morphology. Small particles ranging from approximately 0.25μ m to 3μ m in size, like those in Figure 2, are typical of the sampl. In Figure 2, some repolymerization has occurred near the perimeter of the small particle, but the volume of gel appears to be less than in was apparent in previous studies with 0.7M NaOH and no lithium ions present [Kurtis et al, 1998; Kurtis, 1999; Kurtis et al, 1999]. Figure 3 shows two distinct morphologies produced by the reaction of alkali-silica reaction gel in 0.7M NaOH + 0.1M LiCl solution. First, small particles similar to those in Figure 2 have aggregated. Some repolymerized gel appears to have formed within the cluster of small particles. In addition, structures similar to the spherulitic or near-spherulitic "sheaf of wheat" structures produced by the reaction gel with calcium hydroxide solution [Kurtis et al, 1998] and sodium hydroxide and calcium chloride solution [Kurtis et al, 1999]. The sheaf of wheat morphology formed in such systems is believed to be calcium silicate hydrate (C-S-H) [Zampini et al, 1998; Kurtis et al, 1998]. The presence of the lath-

like spherulites implies that the ASR gel has dissolved in the 0.7M NaOH + 0.1M LiCl solution, and that calcium present in the gel (Table 1) has reacted with the dissolved silicate species to form the sheaf of wheat structures. Furthermore, the presence of repolymerized gel and the general smaller size of the gel particles as compared to dry gel particles [Kurtis et al, 1998] indicate that dissolution of the ASR gel has occurred. Quantitative elemental analysis confirms that silica dissolution occurs in the 0.7M NaOH + 0.1M LiCl solution.





Fig. 2: X-ray image of alkali-silica reaction gel after 1 week in 0.7M NaOH + 0.1M LiCl. The image was taken with a 11.190 s exposure time with a beam current of 267.5 mA at an original magnification of 2400x. scalebar = 1 μ m. 71015011



With the evidence of dissolution, the formation of a relatively small amount of repolymerized gel is remarkable. Of the alkali metal cations, lithium is unique because it stabilizes colloids and prevents gelling [ller, 1956]. In a system containing both Na⁺ and Li⁺, such as the one under investigation, the alkalis compete for adsorption at negatively charged sites on the silicate surface. Since adsorption affinity increases with cation radius, it is expected that sodium adsorption with be preferential to lithium adsorption. However, measurements of the concentration of alkali ions in expressed pore solution show that over time the Li⁺ concentration decreased while the Na⁺ and K⁺ concentration remained constant [Sakaguchi, 1989]. The Eisenman model of cation exchange includes a limiting case, the strong field case, where cations are in direct contact with the surface [McBride, 1994]. Because of strong field exchange behavior, the negatively charged surface of iron oxide, for example, exhibits a preference for smaller cations, with a stronger attraction for Li⁺ as compared to K^{*}. Thus, the apparent preference of the alkali silica reaction gel for Li as compared to Na⁺ and K⁺, as described by [Sakaguchi, 1989] may be the result of strong field exchange behavior. If lithium adsorption is more favorable than Na^+ and K^+ adsorption, a physical mechanism for prevention of gel repolymerization may exist. Iler [1956] postulates that the highly hydrated lithium ions are not adsorbed as near to the silicate surface as a cation with smaller hydrated radius, such as sodium or potassium. Thus, the net repulsion between the silicate particles remains high and repolymerization does not occur. The effect of the lithium would be dependent upon its relative concentration in the solution. In this system, the concentration of Na⁺ is five times greater than the concentration of Li⁺, indicating that some repolymerization will occur, but the amount of gel repolymerization may be decreased by the presence of the lithium-containing additive.

While the formation of the expansive gel by repolymerization may be limited by the presence of lithium, aggregation of $0.25-1\mu$ m particles occurs (Figure 3). Rodrigues et al. [1999] found that in a system with both sodium and lithium ions present, lithium reduced the surface charge density of alkali-silica reaction gel. These results indicate that lithium may act to reduce repulsion forces between colloidal silicate particles. The small particles clustered in Figure 3 may be considered to be colloidal according to Tan [1982] who describes a colloid as a state of matter consisting of very fine particles that approach, but never reach, molecular sizes (the upper size limit of colloids is 0.2 mm and the lower size limit is approximately 50 Å or 5 nm). A decrease in the repulsive forces between colloidal particles of the gel.

Interpretations of the x-ray images in consideration of quantitative elemental analysis and surface chemistry principles suggests two mechanisms by which lithium may act to reduce expansion of alkali-silica reaction gel. First, lithium may limit repolymerization, effectively reducing the potential for expansion. Second, lithium seems to promote the aggregation of relatively larger particles.

CONCLUSION

Transmission soft x-ray microscopy was used to examine the reaction of ground ASR gel, obtained from a Brazilian dam, in 0.7M NaOH solution and 0.7M NaOH +0.1M LiCl to gain further insight mechanisms by which ASR-induced expansion is reduced in the presence of LiCl. Concentrations of Ca, Li, Na, and Si in solution were measured by inductively coupled plasma optical emission spectroscopy, and these results were used to aid in the interpretation of the x-ray images. Images of the reaction of the ASR gel in 0.7M NaOH + 0.1M LiCl solution showed that dissolution of the original gel particles had occurred, which was supported by the quantitative elemental analysis. However, in the presence of lithium chloride, the repolymerization into an expansive gel was decreased as compared to the reaction of the ASR gel in 0.7M NaOH solution.

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