Coventry University and The University of Wisconsin Milwaukee Centre for By-products Utilization, Second International Conference on Sustainable Construction Materials and Technologies June 28 - June 30, 2010, Università Politecnica delle Marche, Ancona, Italy. Main Proceedings ed. J Zachar, P Claisse, T R Naik, E Ganjian. ISBN 978-1-4507-1490-7 http://www.claisse.info/Proceedings.htm

# Lithium Salt for Reactive Aggregates in Concrete

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# ABSTRACT

The objective of this study was to investigate the extent to which lithium salt can inhibit the excessive expansion caused by alkali-silica reactivity. The test specimens of ASTM C 1260 were prepared with different dosages of lithium nitrate salt having lithium-to-alkali molar ratios of 0.00, 0.59, 0.74, 0.89, 1.04, 1.19 and 1.33, and three reactive aggregates. They were tested for the linear expansions at the immersion ages of 14, 28 and 56 days, for which the minimum amount of lithium dosage required to suppress ASR expansion of each trial aggregate was evaluated. Test results revealed that the optimum lithium dosage to suppress ASR expansion varied mainly depending upon the extent of aggregate reactivity and the mineralogy of the aggregates. The statistical analyses to determine the minimum amount of lithium-to-alkali molar ratio in controlling ASR expansion showed a good correlation with the amount of experimental lithium salt used.

# **INTRODUCTION**

Alkali-silica reactivity (ASR) is one of the major durability problems, which result in premature deterioration of concrete structures, in the United States and throughout the world. ASR is the process in which certain minerals of aggregate react, in the presence of sufficient moisture, with the hydroxyl ions of the alkalis present in concrete. The combined product (gel) expands creating tensile forces in the concrete resulting in concrete cracks, spalling and other deterioration mechanisms.

The main factor in controlling or eliminating alkali-silica reaction is through the choice of nonreactive aggregates for concrete construction. The reactive aggregates can also be used without affecting the ASR-induced damaged in the concrete by using mitigation techniques. One of the main techniques is the use of lithium compounds to suppress excessive expansion due to ASR. The idea was first reported in 1951 [McCoy and Caldwell]. For the next 40 years, a few studies were conducted on the effectiveness of lithium-bearing compounds to control ASR. However, in the past two decades, awareness of using lithium as an admixture in new concrete for ASR suppressive purposes has been renewed among the researchers.

The amount of  $LiNO_3$  needed to mitigate ASR in fresh concrete is a function of sodium oxide equivalent ( $Na_2O_{eq}$ ) of Portland cement and the amount of cement used per cubic yard of concrete. The standard lithium dose (sometimes referred 100% Li) is the amount of Li admixture that supplies enough Li ion to achieve a molar ratio of 0.74 of Li ion to the sum of Na and K ions supplied by the Portland cement. The dose refers as approximately 4.6 liters of Li admixture for every kg of sodium equivalent supplied by the cement. In general, the Li:(Na+K) of 0.74 is adequate to suppress excessive ASR expansion for the majority of the aggregates [McCoy and Caldwell 1951; Thomas 2000; Ekolu et al. 2007; Millard and Kurtis 2008]. However, some highly reactive aggregates require substantially more dosage [Folliard et al. 2006] and some less reactive aggregates may need less [Lane 1994; Touma et al. 2001; Collins et al. 2004]. The efficacy of lithium in suppressing expansion due to ASR strongly depends on the nature of aggregate, form of lithium, the amount of alkalis present in the concrete and the use of supplementary cementitious materials.

Feng et al. (2005) studied the influence of various lithium salts, such as LiF, LiCl, LiBr, LiOH, LiOH.2H<sub>2</sub>O, LiNO<sub>3</sub>, LiNO<sub>2</sub>, Li<sub>2</sub>CO<sub>3</sub>, Li<sub>2</sub>SO<sub>4</sub>, Li<sub>2</sub>HPO<sub>4</sub>, and Li<sub>2</sub>SiO3, in suppressing ASR expansion in new concrete. The study pointed out that the trial lithium salts were effective in suppressing ASR at the appropriate dosages. However, LiNO<sub>3</sub> appeared to be the most effective. The minimum lithium-to-alkali dosage to inhibit ASR expansion was generally in the range of 0.72 - 0.93 for LiNO<sub>3</sub> and 0.67 - 1.20 for other lithium salts.

Tremblay et al. (2007) studied the use of  $LiNO_3$  to control ASR expansion of twelve reactive aggregates. The study showed that the standard dosage of  $LiNO_3$  (Li/Na+K = 0.74) was effective in suppressing ASR expansion for only six aggregates, three aggregates required a higher  $LiNO_3$  dosage of between 0.75 and 1.04, while the expansion of the remaining three aggregates were not suppressed even by a lithium to alkali molar ratio of 1.11.

Based on the past research investigations, a wide range of lithium-to-alkali molar ratios that was shown effective for suppressing ASR were suggested. They were: 0.60 - 0.90 [Collins et al., 2004]; 0.72 - 0.92 [Feng et al. 2005]; 0.74-0.93 [McKeen et al. 1998 and Fournier et al. 2004], and 0.74 - 1.04 [Tremblay et al. 2007].

Past investigations on the influence of lithium nitrate in suppressing the alkali-silica reactivity were mostly confined to the test duration of 14 days, and limited to types of aggregate that did not produce a wide range of ASR-induced expansions. The purpose of this study was to enhance the previous research investigations by extending the test duration from 14 to 98 days, using three highly reactive aggregates with a large variation in ASR expansion, and introducing statistical correlations in support of the experimental results.

#### **RESEARCH SIGNIFICANCE**

This study highlights the impact of under, optimum and over lithium nitrate dosages on the ASR expansion of the mortar bars containing three potentially reactive aggregates at the immersion

ages of 14, 28 and 56 days. Additionally, the proposed analytical model demonstrates the minimum required lithium nitrate dosage to reduce the excessive expansion of the trial reactive aggregates below the suggested expansion limits for the above mentioned three immersion ages.

#### **EXPERIMENTAL PROGRAM**

The raw materials utilized in this investigation were reactive aggregate, Portland cement, lithium nitrate salt and sodium hydroxide pellet. The aggregates (NV-1, NV-2 and NV-3) were acquired from three distinct quarries within the State of Nevada. Table 1 illustrates the compositions of the trial aggregates (rock type). The aggregates were prepared as per requirements of ASTM C 1260. The moisture content and absorption of the graded aggregates were also accounted in mixture design. The compositions of Portland cement used in this investigation are shown in Table 2. Lithium nitrate (LiNO<sub>3</sub>), a specially formulated admixture (commercially known as Lifetime) contained 30 percent solution of LiNO<sub>3</sub> in water, was used in this study. The amount of water contained in the lithium solution added to the concrete mixture was removed at a rate of H<sub>2</sub>O:LiNO<sub>3</sub> equal to 70:30 from the mixing water to maintain the targeted water-cement ratio.

Agg. ID	$SiO_2$	$Al_2O_3$	$Fe_2O_3$	CaO	MgO	Na <sub>2</sub> O	$K_2O$	LOI	Roc	k Type
NV-1	13.50	0.40	0.29	32.55	13.09	0.03	0.14	39.4	Dolo Lim	omite- estone
NV-2	60.82	15.89	5.37	4.34	2.49	3.57	3.73	2.14	And	esite
NV-3	52.50	18.45	8.35	8.36	4.59	3.74	1.22	0.98	Basaltic- Andesite	
Table 2. Chemical compositions of Portland cement										
SCMs	Si	$O_2 Al_2$	O <sub>3</sub> F	$e_2O_3$	CaO	MgO	Na <sub>2</sub> O	D <sup>a</sup>	SO <sub>3</sub>	LOI

63.1

4.7

0.42

2.6

1.3

Table 1. Physio-chemical compositions of trial aggregates

 $^{a}Na_{2}O_{eq} = Na_{2}O + 0.658K_{2}O$ 

Cement

21

3.6

3.4

Previous research studies illustrated that about half of the lithium added in the mortar bars to suppress ASR-induced expansion is absorbed by the hydrating cement and the uptake of lithium by C–S–H, and the remaining half is available for the suppressive purpose [McKeen et al. 1998; Bérubé el al. 2004; Feng et al. 2005; Li 2005]. To prevent excessive leaching of lithium from the bars into the surrounding solution, the ASTM C 1260 test was modified by adding lithium into the soak solution to maintain the same lithium-to-alkali molar ratio in the mortar bars and the soak solution [Berra et al. 2003; Folliard et al. 2003; Collins et al. 2004 and Li 2005]. The amount of lithium nitrate used in the mortar bars, corresponding to lithium-to-alkali molar ratios of 0, 0.59, 0.74, 0.89, 1.04, 1.19 and 1.33, and in the 1N NaOH solution is shown in Table 3.

The four 1 in. x 1 in. x 12 in. mortar bars containing each trial aggregate and lithium dosage were prepared from the 7-bar batch size mixture based on the proportions of ASTM C 1260. The mixing procedures were accomplished based on the requirements of ASTM C 305. The mortar bars were molded within a total mixing time of not more than 2.25 minutes. After 24 hours of

moist curing, the specimens were demolded, and initial readings were taken before immersing in tap water at 80°C for 24 hours for which the zero readings were recorded. Afterward, the mortars were submerged in the 1N NaOH solution having the half lithium concentration of the companion mortar bars, in an air-tight plastic container held in an oven at the temperature of 80°C. Subsequent readings were taken at the immersion ages of 3, 6, 10, 14 days and, thereafter, one reading per week until the 98-day immersion age was reached.

soak soluti	ion constitu	lents						
	Mortar Ba	r		1.0L of 1N soak solution				
Lithium Mixture	Li	Li dose	LiNO <sub>3</sub> <sup>a,b</sup>	Li	Water <sup>c</sup>	LiNO <sub>3</sub>	NaOH	
winxture	Na + K	(%)	(gm)	Na + K	(gm)	(gm)	(gm)	
#1	0.59	80	37.78	0.295	952.50	67.93	_	
#2	0.74	100	47.23	0.370	940.56	84.92	-	
#3	0.89	120	56.67	0.445	928.70	101.90	- 40	
#4	1.04	140	66.12	0.520	916.80	118.90	- 40	
#5	1.18	160	75 56	0.590	904 90	135.86	_	

0.665

893.00

152.90

Table 3. Lithium-to-alkali molar ratios (%Li dose) in the mortar bars and corresponding soak solution constituents

<sup>a</sup>The amount of lithium was computed based on 0.84%  $Na_2O_{eq}$  cement alkali

85.01

<sup>b</sup>Based on 7-bar size mixture; <sup>c</sup>Distilled water was added to prepare NaOH solution

## **RESULTS AND DISCUSSIONS**

180

1.33

#6

#### ASR expansion as related to the immersion age and lithium dosage

The results of the study clearly revealed that the selected lithium nitrate dosages reduced the adverse effect of alkali-silica reactivity for the three reactive aggregates. A typical ASR expansion as a function of the immersion age and lithium-to-alkali molar ratio is shown in Figure 1. As can be seen, the expansion of control mortar bars expanded rapidly at the early age of immersion, and the expansion rate decreased with an increase in the immersion age. In the case of lithium-bearing mortar bars, the expansion rate was fairly small throughout the test duration. Additionally, the mortar bars containing lithium salt showed little tendency to expand after the immersion age of two months.

Figure 1 also demonstrates that, at the immersion age of 14 days, the control expansion (0.890%) was reduced to 0.277, 0.166, 0.076, 0.046, 0.036 and 0.032% for the lithium-to-alkali molar ratios of 0.59, 0.74, 0.89, 1.04, 1.19 and 1.33, respectively. For the same aggregate, the expansion progression at the immersion ages of 28, 56 and 98 days as compared to the 14-day expansion was 1.33, 1.49 and 1.60 times for 0%Li, 2.0, 3.05 and 3.61 times for 80%Li, 2.42, 4.34 and 5.22 times for 100%Li, 3.26, 8.09 and 10.51 for 120%Li, 3.22, 10.09 and 15.20 for 140%Li, 2.28, 10.78 and 15.20 for 160%Li, 2.06, 10.53, 16.44 for 180%Li, respectively. A higher rate of expansion in the early phase of immersion was because the lithium was readily available and incorporated in cement during the more active phase of hydration. While aggregate mineralogy plays an important role in suppressing ASR, the characteristics of the remaining two aggregates (NV-1 and NV-2) followed a similar pattern to that of the NV-3 aggregate.

#### Reduction in expansion as related to the immersion ages and lithium dosages

The influence of the selected lithium dosages in suppressing the ASR-related expansion of the trial reactive aggregates was also expressed in terms of the Reduction in Expansion (RIE) of the untreated mortars (having no lithium content) at various immersion ages. A typical reduction in expansion as related to the immersion age and lithium dosage is illustrated in Figure 2. As can be seen, the RIE increases with an increase in the amount of lithium content in the mortar bars for any immersion age. For each of the trial lithium dosages, the RIE at the early immersion age of 3 days was highest and then gradually decreased with increases in the test duration. As seen in Figure 2 and with increases in lithium-to-alkali molar ratio from 0.59 to 1.33 with an increment of 0.19, the 14-day control expansion of NV-3 aggregate was reduced by 51.4, 69.7, 83.8, 93.7, 95.4 and 96.7% with the use of lithium-to-alkali molar ratios of 0.59, 0.74, 0.89, 1.04, 1.19 and 1.33, respectively.



Fig. 1. The expansion of the mortars made with NV-3 aggregate and various lithium-toalkali molar ratios respondent



Reduction in expansion of the mortar bars of NV-3 aggregate

Fig. 2.

For the same period of test duration (14 days), the RIE of the mortar bars made with lithium-toalkali molar ratios of 0.59, 0.74, 0.89 and 1.04 was recorded as 31.6, 76.8, 87.3 and 90.7% for NV-1, and 68.9, 81.3, 91.5 and 94.8% for NN-2 aggregates, respectively (Fig. 3). It is revealed that the variation in the RIE among the trial aggregates was more for the mortar bars containing lowest lithium content of 0.59 than those made with the higher lithium dosages. The 14-day RIE of the mortar bars containing the trial aggregates was fairly equal for the lithium-to-alkali molar ratios of 0.89 or more.



Fig. 3. Reduction in expansion at the immersion age of 14 days

The performance of various amount of lithium salt in arresting the ASR expansion of the trial aggregates at the extended immersion age of 98 days is illustrated in Figure 4. The 98-day reduction in expansion versus the lithium dosages revealed a similar pattern to that for the immersion age of 14 days. More precisely, the 98-day RIE over the lithium dosage increased rapidly for NV-1 aggregate, and gradually for the NV-2 and NV-3 aggregates. However, the rate of RIE was more for the NV-3 aggregate than that of NV-2 aggregate. The influence of each trial lithium nitrate dosage in suppressing ASR varied depending on the aggregate mineralogy.

The cracks on the surface of the test mortar bars due to alkali-silica reactivity were also examined. The ASR-related cracks of the mortar bars reduced with an increase of the amount of lithium salt present in the test specimens.

#### Optimum lithium dosage to control alkali-silica reactivity

Above discussions summarized that the influence of lithium dosage in arresting alkali-silica reactivity primarily depended on the aggregate mineralogy, lithium dosage, and the immersion ages. Table 4 documents the levels of alkali-silica reactivity of the mortar bars having five to seven lithium dosages based on the three suggested expansion criteria. The criteria of ASTM C 1260 to evaluate the alkali-silica reactivity were: 14-day expansion of lower than 0.10% was considered innocuous and greater than 0.10% was reactive. Past research investigations [Hooton 1991 and Rogers and Hooton 1993) proposed the expansion limits of 0.33% at 28 days and 0.48% at 56 days. It can be recommended that the minimum amount of lithium dosage that is

sufficient to keep the ASR expansion below the expansion limits of 0.10, 0.33 and 0.48% at the immersion ages of 14, 28 and 56 days, respectively. As can be seen, the 14- and 28-day expansion criteria resulted in the lithium-to-alkali molar ratios of 0.74, 0.89 and 1.04 for NV-1, NV-2 and NV-3 aggregates, respectively. To meet the failure criteria 0.48% at 56 days, however, the required lithium dosage for each selected aggregate was more than that needed for the 14- and 28-day expansion criteria. The optimum lithium-to-alkali molar ratios to encounter alkalisilica reactivity of NV-1, NV-2 and NV-3 aggregates were 0.89, 1.04 and 1.19, respectively. The results concluded that the amount of lithium required to suppress ASR was a function of the expansion characteristics over the test duration of 14, 28 and 56 days, which remained dependent on the mineralogy of the aggregate.



Fig. 4. Reduction in expansion at the immersion age of 98 days

Agg. ID	$\frac{Li}{Na+K}$	Alkali-Silic	Alkali-Silica Reactivity				Optimum lithium-to-alkali molar ratio		
	Deces	14 Days <sup>a</sup>	28 Days <sup>b</sup>	56 Days <sup>b</sup>	14	28	56		
	Dosage	(0.10%)	(0.33%)	(0.48%)	Days	Days	Days		
NV-1	0 Reactive		Reactive Reactive						
	0.59	Reactive	Reactive	Reactive	0.74	0.74	0.89		
	0.74	Innocuous	Innocuous	Reactive	-0.74	0.74			
	0.89-1.04	Innocuous	Innocuous	Innocuous	_				
	0	Reactive	Reactive Reactive						
NIV 2	0.59-0.74	Reactive	Reactive Reactiv		0.90 0	0.80	1.04		
IN V-2	0.89	Innocuous	Innocuous	cuous Reactive		0.89	1.04		
	1.04-1.33	Innocuous	Innocuous Innocuous		_				
	0	Reactive	Reactive	Reactive Reactive					
NV-3	0.59-0.89	59-0.89 Reactive		active Reactive		1.04	1 10		
	1.04	Innocuous	Innocuous	Reactive	-1.04	1.04	1.19		
	1.19-1.33	Innocuous	Innocuous	Innocuous	_				

Table 4. Optimum dosage of lithium nitrate to control ASR

<sup>a</sup>Based on ASTM C 1260; <sup>b</sup>Hooton (1991), and Rogers and Hooton (1993)

# Analytical model to determine the optimum lithium dosage to control ASR of the trial aggregates

The expansion of the test mortar bars made with each trial reactive aggregate and lithium dosage was evaluated at the immersion ages of 14, 28 and 56 days. The most suitable relationship between the dependent and independent variables is presented in Equation 1. The regression parameter *a*, *b* and *c*, the Prob(t) of each parameter, Prob(F) and the coefficient of multiple determination ( $\mathbb{R}^2$ ) for Equation 1 for all trial aggregates are shown in Table 5. The  $\mathbb{R}^2$  value of the regression line for each trial aggregate and three distinct immersion ages showed a good fit.

$$Y = a + bX^3 + \frac{c}{X^{1.5}}$$
(1)

Where: Y is the lithium-to-alkali molar ratio; X is the expansion of the mortar bars made with lithium-to-alkali molar ratio of Y; a, b and c are the regression parameters

The expansion of the control mortar bars containing each trial aggregate at three distinct immersion ages of 14, 28 and 56 days showed a good correlation with the above mentioned regression parameters a, b and c as presented in Equations 2, 3 and 4, respectively.

In order to evaluate the minimum dosage of lithium salt to arrest the alkali-silica reactivity of a reactive aggregate at the immersion ages of 14, 28 and 56 days, the parameters a, b and c for Equation 1 can be evaluated from Equations 2, 3 and 4, respectively, with the known expansion of the control mortar bars. The optimum analytical lithium-to-alkali molar ratio to suppress ASR expansion of the trial aggregates below 0.10% at 14 days, 0.33% at 28 days, and 0.48% at 56 days was determined by substituting the above mentioned three parameters for the companion aggregate source at their respective immersion age in Equation 1. Table 6 illustrates the procedures for determining the optimum analytical lithium-to-alkali molar ratio for the trial aggregates. As can be seen, there is a good agreement existed between the optimum experimental data and analytical results for all three reactive aggregates.

Agg. ID	Immersion Age (Days)	Parameters of Eq. 1			Prob(t) of	f Parameter	Drah (E)	$\mathbf{p}^2$	
		а	b	С	а	b	С	-P100(F)	K
NV-1	14	0.730	-19.548	0.0016	0.0102	0.0224	0.1259	0.0155	0.97
	28	0.890	-3.745	0.0021	0.0201	0.0556	0.5588	0.0647	0.94
	56	0.945	-1.158	0.0028	0.0215	0.0583	0.7207	0.0706	0.93
NV-2	14	0.654	-0.936	0.0038	0.0000	0.0004	0.0003	0.0001	0.99
	28	0.757	-0.467	0.0102	0.0001	0.0016	0.0036	0.0006	0.98
	56	0.634	-0.316	0.1394	0.0000	0.0001	0.0001	0.0000	0.99
NV-3	14	0.766	-0.940	0.0033	0.0001	0.0009	0.0021	0.0003	0.98
	28	0.894	-0.291	0.0061	0.0002	0.0038	0.0348	0.0033	0.94
	56	0.870	-0.150	0.0370	0.0007	0.0076	0.0600	0.0059	0.92

Table 5. The statistical data for Equation 1 for the trial aggregates

$$a = 0.78 + 0.0537X \tag{2}$$

$$b = -0.77 + \frac{4.98}{X^{1.5}} - \frac{4.94}{X^2}$$
(3)

$$c = -\frac{0.00356X}{(-2.004 + X)} \tag{4}$$

Where: X is the expansion of the untreated mortar bars made with the reactive aggregate at various immersion ages

Tuble 6. That yield and experimental number dosage to artest ABR expansion									
Agg.	Immersion	Control Exp. (%)	<b>Regression Parameters</b>			Suppre-	Analytica	Experi-	
ID	Age (Days)		0	h	c	ssed Exp. (%)	1	mental	
			a (Eq. 2)	$(\mathbf{E}_{\mathbf{z}}, 2)$			Li Dose	Li Dose	
			(Eq. 2)	(Eq. 3)	Eq. 4)		(Eq. 1)		
	14	0.33	0.80	-19.55	0.0007	0.08	0.822	0.74	
NV-1	28	0.89	0.83	-1.07	0.0029	0.29	0.820	0.74	
	56	0.94	0.83	-0.89	0.0032	0.33	0.815	0.89	
	14	0.60	0.81	-3.79	0.0015	0.08	0.883	0.89	
NV-2	28	1.19	0.84	-0.42	0.0052	0.25	0.879	0.89	
	56	1.47	0.86	-0.26	0.0099	0.46	0.865	1.04	
	14	0.90	0.83	-1.03	0.0029	0.06	1.032	1.04	
NV-3	28	1.32	0.85	-0.32	0.0070	0.25	0.900	1.04	
	56	1.83	0.88	-0.23	0.0378	0.41	1.006	1.19	

Table 6. Analytical and experimental lithium dosage to arrest ASR expansion

## CONCLUSIONS

Based on the results of this study, the following conclusions can be made.

1. The reduction in expansion (RIE) of the mortar bars made with various lithium dosages peaked at the early immersion age of 3 days and then gradually decreased with an increase in the test duration. Additionally, the study also revealed that the RIE decreased rapidly, with an increase in immersion age, for the mortar bars containing lower lithium dosage than that prepared with a higher lithium content.

2. A good agreement existed between the required experimental and analytical lithium dosages to control the excessive expansion of the trial reactive aggregates at the ages of 14, 28 and 56 days. The proposed analytical method may be used in selecting lithium dosage that is capable of limiting the ASR expansion of reactive aggregates below the suggested failure criteria. However, since aggregates can vary in their mineralogies, it is recommended that each reactive aggregate be tested with various lithium dosages before it is used in Portland cement concrete.

3. The required lithium dosage to suppress excessive expansion of the trial reactive aggregates due to alkali-silica reactivity below 0.1% at 14 days was less than that required to reduce the expansion below 0.33% at 28 days and 0.48% at 56 days. Since the alkali-silica reactivity of an aggregate depends on a number of factors, therefore, it is recommended to use the minimum amount of lithium nitrate to arrest the ASR-related damages based on the 56-day expansion criteria.

#### ACKNOWLEDGEMENTS

The authors would like to acknowledge Nevada Department of Transportation (NDOT) for supplying materials and financial support throughout the research investigation. Thanks are extended to the Southern Nevada Aggregate Association (SNAA) that acted as a liaison between the aggregate producers and researchers. Material contributions made by lithium and cement producers are also acknowledged.

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