

# *Evaluation of Lithium Nitrate as an alternative to reduce the expansive effect of the Alkali-Silica Reaction in concrete made with reactive aggregates in Arequipa – Perú*

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**Abstract**— The Alkali-Silica Reaction (ASR) is an expansive reaction of concrete, caused by the combination of three simultaneous conditions: high Alkali content in Portland cement, high relative humidity, and aggregates with reactive Silica. The objective of this research is to evaluate the effect of Lithium Nitrate on the Alkali-Silica Reaction of concrete made with reactive aggregates from southern Peru (Arequipa). An evaluation of the expansion reduction was made applying the methodology of accelerated testing of mortar bars according to the American standards ASTM C1260 and ASTM C1567 with the purpose of analyzing the reduction of expansion by ASR due to the addition of Lithium Nitrate. Also, to present a Certified and Documented record of the Alkali-Silica reaction in Peru to evidence a possible problem in the future.

**Keywords**— Alkali-Silica Reaction, Lithium Nitrate, percent expansion, reactive aggregate.

## I. INTRODUCTION

The alkali silica reaction (ASR) phenomenon generates a fairly important concrete erosion problem in various countries. The reaction affects the strength, durability and consequently the time and useful life of concrete structures [1].

To add, the network of cracks is a characteristic qualitative indicator of expansion by alkali-silica reaction, also, whitish exudations and eruptions [2]. Furthermore, the presence of cracks generates concrete that is more exposed to erosive agents from the environment, for example, exposure to common agents in marine environments such as chlorides and sulfates [3-4].

However, the alkali silica reaction occurs in two stages. In the first stage, the hydrated paste together with the reactive aggregate silica forms an alkaline silicate gel, the gel formed reacts with the calcium ions in the cement, which generates a product called alkaline calcium silicate hydrate gel [ 5]. In the second stage, the gel begins to absorb moisture from the environment, which produces an increase in volume and consequently the expansion generates internal tensile stresses in the structure [6-7]. In this regard, for the alkali-silica reaction to occur, 3 conditions must be met simultaneously; high alkali

equivalent content in portland cement, exposure to high humidity and aggregates with reactive silica [8-9-10].

Likewise, in the concrete mix, the largest source of alkali is the cement, represented as equivalent oxide content. To calculate the equivalent alkali content, the equation [%Na<sub>2</sub>O eq = Na<sub>2</sub>O + 0.658 \* K<sub>2</sub>O], is used, but this equation can vary between values of 0.20 to 1.30% with respect to the mass of cement [8]. The equivalent alkali or [%Na<sub>2</sub>O eq] corresponds to a small part of all the oxides that make up the cement, but they are quite soluble and determines the alkalinity in the pore solution [8]. Although a large part of the alkali is provided by the cement, there are other sources that can contribute to the development of the alkali-silica reaction, for example; the environment, supplemental cementitious materials and aggregates. But, although these sources can aggravate or contribute to the formation of the ASR gel; Currently there is no method to account for this contribution [11].

Likewise, there are various recommendations on how to avoid the expansive effect of the alkali-silica reaction, for example, limiting the alkali content in the mixture, use of non-reactive aggregates and control of exposure to humidity. All these suggestions are focused on preventing one of the conditions necessary for the production of the reaction from being met [12].

In this regard, the use of supplementary additions is recommended as a partial replacement of cement, which would reduce the amount of alkali available in the mixture to react with the reactive silica of the aggregates [13]. Furthermore, it is advisable to use concrete with a low water-cementitious material ratio and thus obtain a less porous, denser and more uniform mixture [14]. Therefore, the U.S. Department of Transportation Federal Aviation Administration in its advisory circular, confirms that, due to the porosity of concrete, these are filled with the alkaline solution of Na<sup>+</sup> and K<sup>+</sup> and hydroxyl ions (OH<sup>-</sup>), the which gives way to the formation of the expansive gel. Therefore, it is recommended to limit the content of [%Na<sub>2</sub>O eq] to less than 0.60%.

Likewise, the use of non-reactive aggregates is recommended as a preventive method, based on the results of accelerated tests and petrographic tests to confirm the results [11-15]. But, changing aggregates is not always feasible, since the use of non-reactive aggregates further away would negatively affect the project objectives.

But lithium nitrate does not act directly on any of the conditions that generate ASR. In this regard, the benefits of lithium were studied for a long time. But, the effect of  $\text{Li}^+$  ions on ASR reduction are still not completely defined [16-6]. Furthermore,  $\text{LiNO}_3$  compared to other lithium compounds, has the advantage compared to other additions, it is superior due to its neutrality and high solubility [17].

To evaluate the effect of lithium nitrate on expansion, reactive aggregates from the "La Poderosa" quarry in Arequipa, Peru were used. ASTM C1260 [18] and ASTM C1567 [19] were used to estimate the validity of lithium nitrate. For this,  $\text{LiNO}_3$  was used in quantities of 17.6 g, 19.9 g, and 22 g with type I cement and 44 g, 47.1 g, and 50.6 g with HE cement, carrying out a water correction according to specifications from the supplier SIKA, to determine if it is equally effective with conventional cement and with added cement.

Currently in Peru there is no record of history that can evidence the existence of concrete structures that have presented damage due to alkali-silica reaction, as stated by Sims & Poole [20]. However, the company SUPERMIX S.A. has demonstrated the presence of quarries with reactive aggregate in various areas of southern Peru such as; Arequipa, Tacna, Moquegua, Ilo, Nazca, Juliaca, Cusco, Puerto Maldonado and Abancay, which could generate future problems if adequate precautions are not taken.

The non-existence of registered information does not imply that the problem cannot arise in our area, Peru, even more so with the existence of reactive aggregates. Therefore, the implementation of lithium nitrate as an addition to concrete is sought in order to reduce the expansive effect of the alkali-silica reaction and show a documented record of its existence.

## II. MATERIALS Y MÉTODOS

### A. Materials

Reactive aggregates from the "La Poderosa" quarry located in Arequipa; Peru were used. This belongs to the SUPERMIX concrete company. The fine aggregate used has a specific weight of  $2625 \text{ kg/m}^3$ , absorption percentage of 2.46% and a passing percentage of mesh # 200 of 8.41%. The aggregates were determined as reactive due to the accelerated tests carried out by the SUPERMIX concrete company, following the standards of the regulations [18-19].

In addition, type I and HE cement from the YURA company was used. Type I cement presented an amount of  $[\% \text{Na}_2\text{O eq}]$  equal to 0.80% and HE cement 1.9%. The equivalent oxide content was used to calculate the lithium nitrate dose, which was 4.63 liters of lithium nitrate per kilogram of sodium oxide equivalent of the cement. To add, in order not to alter the water-cement ratio, an adjustment was made in the amount of water following the recommendation of the SIKA supplier, the

correction consists of reducing 0.84 liters of water for each liter of lithium nitrate ( $\text{LiNO}_3$ ).

The percentage of lithium nitrate addition that was used for the accelerated expansive tests regulated by [18-19] was 4%, 4.5% and 5% with respect to the mass of cementitious material for type I cement and for type HE cement. 10%, 10.7% and 11.5% addition of lithium nitrate was used. The author Deng [21] states that with an addition of 1%  $\text{LiNO}_3$  the expansive effect of ASR can be reduced by 85.12%. But, to carry out the proportions of this research they were carried out in accordance with the recommendations of the supplier SIKA.

To add, American standards [18-19] establish the quantities of aggregate retained in meshes No. 8, 16, 30, 50 and 100 necessities for the production of mortar bars. Table I shows the quantities in grams. for each type of mesh.

TABLE I. AGGREGATE QUANTITY REQUIREMENTS

Retained in each sieve	Mass (%)	Mass (gr)
2,36 mm N° 8	10.00%	99
1,18 mm N° 16	25.00%	247.5
600 m N° 30	25.00%	247.5
300 m N° 50	25.00%	247.5
150 m N° 100	15.00%	148.5
Total	100.00%	990

Likewise, American standards [18-19] establish the necessary quantities of cement, water and sand to make 3 bars of mortar according to the relative density of the aggregates. Therefore, as the aggregates used had a relative density greater than  $2.45 \text{ g/cm}^3$ , the proportions described in Table II were used for the mortar designs.

TABLE II. QUANTITY OF MATERIALS FOR MORTAR MIX DESIGNS

Cement:	440 g	Aggregate:	990 g
Mortar designs	Water (g)	$\text{LiNO}_3$ (g)	Sika Fume (g)
Cement HE	206.8	-	-
HE - $\text{LiNO}_3$ al 10%	176.1	44	-
HE - $\text{LiNO}_3$ al 10.7%	174.0	47.1	-
HE - $\text{LiNO}_3$ al 11.5%	171.5	50.6	-
Cement Tipo I	206.8	-	-
Type I - $\text{LiNO}_3$ 4%	194.5	17.6	-
Type I - $\text{LiNO}_3$ 4.5%	193.0	19.9	-
Type I - $\text{LiNO}_3$ 5%	191.5	22	-
Type I - Sika fume 5%	206.8	-	22
Type I - Sika fume 10%	206.8	-	44
Type I - Sika fume 15%	206.8	-	66

### B. Methods and experimental process

For the implementation of lithium nitrate, use was made of the standard test standards for aggregate alkaline reactivity potential ASTM C1260 [18] and ASTM C1567 [19]. The

aggregates were sieved until obtaining the masses indicated in Table I for each mesh; the amounts retained on each sieve were washed to eliminate fine particles. Once washed, they were dried in an oven at a constant temperature of  $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  for 24 h.

For the expansion analysis of the mortar with lithium nitrate, 3 size bars (25 mm x 25 mm x 285 mm) were made according to the quantities for each design in Table II. In each mold, a guide steel pin was placed at the ends for subsequent measurements. The samples unmolded after 24 h were immersed in water at  $80\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  for 24 h. The samples were removed after 24 hours to obtain a zero reading. The bars were then stored in sodium hydroxide solution at  $80\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  for 28 days with 5 intermediate readings at 1, 3, 7, 10, 14 and 28 days.

Finally, the expansion of the mortar bars over time was calculated using equation 1, and subsequently the average of the readings of the 3 bars was taken to compare with the 0.10% and 0.20% limits. In this regard, the standards [18-19] classify the expansion as harmless if, after 16 days of evaluation, it is less than 0.10%, but if an expansion greater than 0.20% is obtained after 16 days, it will be classified as harmful. To add, if the expansion is within the limits of 0.10% and 0.20%, it is recommended to perform the analysis at 28 days, maintaining the previous parameters.

$$\text{Mortar bar expansion (\%)} = \frac{(L_n - L_0)}{L_c} * 100 \quad (1)$$

Donde:

$L_n$  = Length measured over time

$L_0$  = Zero length

$L_c$  = Calibrated bar length (250 mm)

### III. RESULTS AND DISCUSSION

The accelerated tests of mortar bars were carried out for the mixture designs shown in table II, where the expansion results were obtained at a frequency of readings taken at 1, 3, 7, 10, 14 and 28 days respectively, immediately after zero reading. The ASTM C1567 [18] and ASTM C1260 [19] regulations indicate that the final result of the test ends 14 days after taking the zero reading and in case the results of the expansions are greater than 0.10% stipulated by the aforementioned standards, this would indicate that the aggregate is potentially reactive. However, there are results with false positives and/or false negatives as a result of the lack of reliability of the accelerated test. Therefore, expansive tests were carried out at 28 days according to the recommendation of Menéndez [11] due to the possibility of false negatives.

On the other hand, it should be noted that the results are closely related to how the alkali-silica reaction develops. Of the factors that promote the reaction, it is known that conventional type I cement from the company YURA S.A. It has an equivalent alkali content of 0.80% with respect to the mass of the cement and the equivalent alkali content of HE cement is approximately 1.9%, which represent high values compared to what the U.S. regulations indicate. Department of Transportation Federal Aviation Administration on reduction of alkali silica reactivity. This airport standard recommends that the alkali equivalent content of cement should be below 0.60%.

TABLE III. EXPANSION PERCENTAGES DUE TO ALKALI SILICA (ASR) REACTION

Mortar Bar Design		Average ASR Expansion Percentages (%)						
Cement	Addition	1 day	3 day	7 day	10 day	14 day	21 day	28 day
HE	-	0.0075	0.0215	0.0493	0.0861	0.1219	0.1740	0.2123
HE	NL al 10%	0.0049	0.0095	0.0191	0.0227	0.0335	0.0480	0.0663
HE	NL al 10.7%	0.0087	0.0136	0.0221	0.0276	0.0369	0.0475	0.0683
HE	NL al 11.5%	0.0060	0.0163	0.0189	0.0243	0.0297	0.0392	0.0577
Type I	-	0.0376	0.2049	0.3916	0.4491	0.5013	0.5660	0.6211
Type I	NL al 4%	0.0151	0.0532	0.1879	0.2559	0.3175	0.3959	0.4475
Type I	NL al 4.5%	0.0116	0.0467	0.1608	0.2243	0.2889	0.3603	0.4107
Type I	NL al 5%	0.0119	0.0375	0.1347	0.1996	0.2595	0.3349	0.3871
Type I	SF al 10%	0.0172	0.0741	0.2524	0.3405	0.4649	0.5776	0.6376
Type I	SF al 15%	0.0500	0.0616	0.0831	0.1202	0.1820	0.2718	0.3606

As can be seen in table III, the expansion results at 28 days with respect to type HE cement without addition is 0.21% and in the case of Type I cement, the expansion at 28 days was 0.62%, exceeding the limit of 0.20. % of American regulations [18-19] and this would indicate that the aggregate is highly reactive.

Figure 1 in correlation with table III shows that the expansion of the mortar bar with HE type cement with the addition of lithium nitrate at 10%, 10.7% and 11.5%, the expansion at 28 days was 0.07%, 0.07% and 0.06%. These results indicate that silica alkali reactivity is mitigated when using these dosages. Furthermore, it is shown that the addition

of 10% lithium nitrate was sufficient to mitigate the expansive effect according to the parameters of the standard [19].

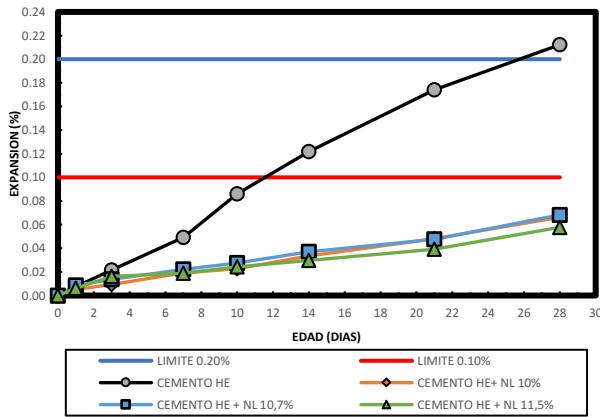


Fig. 1. ASR expansion with HE cement and addition of Lithium Nitrate at 10%, 10.7% and 11.5% with respect to cementitious mass.

As can be seen in Table IV, the greatest reduction at 28 days was obtained with an addition of LiNO<sub>3</sub> of 11.5%, achieving a mitigation of the ASR at 28 days. The high addition content of LiNO<sub>3</sub> is because the content of [% Na<sub>2</sub>O eq] is very high. Furthermore, as seen in Figure 1, the HE cement without addition exceeds the limit of 0.10% indicated by the ASTM C1567 standard [19], which would indicate the effectiveness of the lithic nitrate evaluated in those additions.

TABLE IV. EXPANSION REDUCTION PERCENTAGES IN MORTARS WITH HE TYPE CEMENT AND LITHIUM NITRATE

Mortar Design	% Expansion 28 days	% Reduction
Type HE	0.21	
Type HE y NL 10%	0.07	68.77%
Type HE y NL 10.7%	0.07	67.83%
Type HE y NL 11.5%	0.06	72.82%

To add, the alkali-silica reaction is being mitigated with type HE cement and not with type I cement, due to the pozzolans contained in the HE cement. In this regard, Menéndez [13] states that pozzolans contribute to increasing the mechanical resistance of concrete, they form a calcium silicate with a low [Ca/Si] ratio, which would suppress the amount of Silica dissolution, a preliminary requirement for the formation of ASR.

Furthermore, according to Oey [22], the most important step for ASR is the dissolution of reactive silica, because dissolved silica forms hydrate gels that cause internal stresses in the structure. Therefore, any addition or supplemental material that inhibits or reduces the rate of silica dissolution serves as a viable mechanism for ASR.

In this regard, pozzolans release Ca and Al, contributing to the aforementioned reduction [13-6]. Also, Oey [22] as well as Shi [5] affirm that sufficient calcium, dissolved aluminum and with interaction with the reactive sites could promote the C-S-H and thus partially cover and passivate the extension of the aggregate in solution.

Therefore, due to the pozzolan content in the HE cement and in combination with lithium nitrate, which contributed to the reduction of silica dissolution, the reaction was mitigated after 28 days.

According to Figure 2, it is shown that in only 3 days the limit of 0.10% was exceeded with type I cement without addition. In this regard, Figure 2 shows that with greater amounts of LiNO<sub>3</sub> addition, a greater reduction in expansion is obtained compared to the mortar mixture with type I cement without the addition of LiNO<sub>3</sub>. However, although a large reduction in expansion was achieved, the maximum limits of the standard were not met. But there is a possibility that; at a higher dose of lithium nitrate, mitigation of expansion is achieved below the limit of 0.10%.

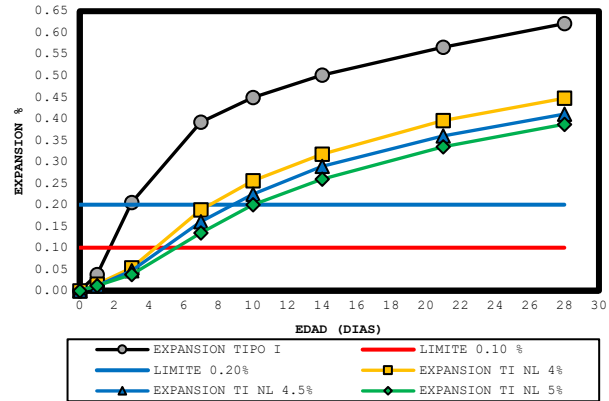


Fig. 2. ASR expansion with Type I cement and addition of Lithium Nitrate at 4%, 4.5% and 5% with respect to cementitious mass.

Figure 2 in correlation with table III shows that the expansion of the mortar bar with type I cement at 28 days was 0.62%, with the addition of 4% lithium nitrate the expansion at 28 days was 0.45%, with 4.5% the expansion was 0.41% and finally with 5% an expansion of 0.39% was obtained. These results clearly indicate the reduction of expansion, however, they exceed the limit of 0.10% in terms of alkali silica reaction mitigation.

TABLE V. EXPANSION REDUCTION PERCENTAGES IN MORTARS WITH TYPE I CEMENT AND LITHIUM NITRATE.

Mortar Design	% Expansion 28 days	% Reduction
Type I	0.62	
Type I y NL 4%	0.45	27.95%
Type I y NL 4.5%	0.41	33.88%
Type I y NL 5%	0.39	37.68%

As seen in table V, with an addition of LiNO<sub>3</sub> at 5%, a great reduction in expansion was achieved by 37.68% compared to the expansion value at 28 days of a mortar design with conventional type I cement without addition.

Consequently, the effectiveness of lithium nitrate would be related to the behavior that this addition has in dissolving the ratio (Na/Si) because lithium cations have a greater union

compared to sodium cations due to having a hydrated radius. more extensive. However, by reducing the ratio (Na/Si), the water absorption of the expanding gel is reduced, which means that the potential for cracking or swelling is limited [21].

Furthermore, the most viable lithium compound to reduce the expansive effect of the alkali-silica reaction is lithium nitrate because it has different types of advantages such as ease of handling of the additive, the pH of the pore solution does not increase and it does not affect the mechanical properties of concrete in the fresh and hardened state [17]. And, some mechanisms of lithium nitrate operation focus on: reducing the dissolution of silica, formation of a dense silica alkali reactivity gel to protect the reactive minerals from a future reaction and the formation of a crystal coating of (Li – Si) on the surface part of the aggregate to inhibit the dissolution of silica [23].

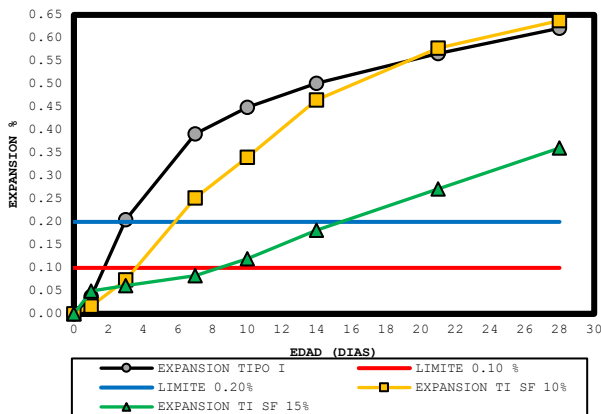


Fig. 3. ASR expansion with Type I cement and addition of Micro silica at 10% and 15% with respect to cementitious mass

According to Figure 3, a maximum expansion reduction was achieved between the two additions of SIKAFUME by 41.94% with a microsilica addition of 15% compared to the expansion obtained with type I cement without addition. But it is outside the limit of 0.10%. Furthermore, as seen in Figure 3 at 28 days, the reduction in expansion with an addition of 10% is not significant, that is, there is no substantial contribution to the reduction of expansion.

In addition, Mahyar [14] states that the micro silica pozzolana reacts with the calcium hydroxide constituted by the hydration of the calcium silicate, consequently, it forms C-S-H with a low ratio of (Ca/Si), obtaining mixtures more homogeneous and less porous concrete. Furthermore, there is the possibility that this reduction in expansion was due to the increase in the properties of the mixture, reducing the water-cementitious material ratio and only occurs at early age.

To add, it was necessary to analyze the effectiveness of the use of lithium nitrate compared to other environmental options, that is, obtaining a reduction in expansion, but with the lowest use of resources and its viability for projects. For this reason, the use of micro silica was analyzed as an option. In this regard, in Peru obtaining  $\text{LiNO}_3$  is quite expensive, because there is no lithium production in the environment and its import is necessary. Additionally, micro silica is less expensive and more applicable for projects in Peru.

#### IV. CONCLUSIONS

A tendency was obtained to not exceed the limit of 0.10% stipulated by the ASTM C1260 [18] and ASTM C1567 [19] standards for HE cement with nitrate additions at 10%, 10.7% and 11.5% at 28 days of evaluation. On the other hand, type I cement exceeded the 0.10% limit at 28 days even with the addition of lithium nitrate; indicative of danger to failures due to ASR expansion. But, although the expansion of the alkali-silica reaction has been mitigated, the use of lithium nitrate in proportions greater than 10% is technically not profitable for use in projects due to the high cost that its import into Peru would entail.

It was shown that; To reduce the expansion due to alkali-silica reaction with the use of supplementary materials, it is not necessary to limit the available alkali content, that is, replace the cement. In this regard, with the use of micro silica as an addition, the expansion at 28 days was reduced by 41.94%. Although it is true that the limit stipulated by the standards of 0.10% was exceeded, it was confirmed that, with the densification of the mixture, a low cementitious material-water ratio and low porosity, the expansion by ASR can be reduced.

The effectiveness of nitrate addition was shown with a tendency to obtain better results at higher addition amounts. In this regard, the maximum reduction in expansion with respect to the expansion obtained with type I cement was 37.68% for an addition of 5% at 28 days. Although, in the doses proposed for type I cement, this phenomenon was not mitigated, the results suggest that, with a dose significantly higher than those proposed in this article, it would be possible to comply with the stipulations of the standards [18 - 19].

The south of Peru runs the risk of problems due to alkali-silica reaction if the 3 simultaneous conditions for its production occur. Because the existence of reactive aggregates and the high expansion they cause when used with type I cement have been demonstrated. Therefore, although there are no documented records of projects affected by ASR, precautions can be taken if it is considered that the conditions of ASR.

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