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## Empirical Modelling of Lithium Mitigation for Alkali-Silica Reactivity



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### ABSTRACT

The Alkali-Silica Reaction (ASR) is a deleterious concrete chemical reaction that has been studied a lot since its discovery in 1940. The reaction produces a highly hygroscopic silica gel that swells into the pores of concrete, resulting in the critical decrease of the mechanical performances of the affected structures. Some methods have been investigated to limit expansion under a certain limit, among them lithium mitigation. However, the mechanisms are not really understood yet and determining the right amount of lithium is still arduous. An attempt for a method in coherence with experience is presented in this study, using the chemo-mechanical model of Larive and curve-fitting. The results provide accuracy to experimental measurements from several protocols, and call for further research.

**Key words:** Concrete, Alkali-Silica Reaction, Lithium mitigation, Modified chemo-mechanical model.

## 1. INTRODUCTION

Numerous studies on lithium mitigation have been performed since its discovery by McCoy & Caldwell [1], using different lithium compounds, under equally diverse experimental conditions [2]. These studies have demonstrated the effectiveness of lithium in inhibiting ASR. Indeed lithium, that is also an alkali like sodium or potassium, seems to react with silica in a less expansive way. Since then, many scientists investigated the mechanisms under this mitigating property and three main theories are discussed:

- Lithium is somehow more reactive than other alkali ions and the silica gel formed by the reaction is non-expansive [2] [3].
- The silica gel formed with lithium constitute a protective layer on the surface of the aggregate, making the reactive silica unavailable for other alkali ions [2] [3].
- In presence of lithium, the dissolution rate of reactive silica is slower and consequently less silica gel is formed [4] [3].

However, this effectiveness varies in terms of the necessary quantities to be added according to the experimental parameters and the materials used. Some applications with lithium have been tested such as fresh concrete formulation admixtures with lithium salts to prevent ASR from happening. Lithium solution injection in the surface of hardened concrete under operation is also a fledging technique. Consequently only empirical data are available without a general model describing the effect of lithium mitigation on expansion behaviour of concrete. Calculating the right amount of lithium needed to have a significant reduce of expansion for a given concrete formulation is then approximate, which makes field application difficult to design.

The main purpose of this study is the establishment of a statistical method based on chemo-mechanics theory to determine the optimum quantity of lithium to reduce the expansion for a fresh concrete admixture. It will be based on reviews, experiments that have been carried out over the years and field experience from industry about alkali-reactivity tests and standards, adapted to lithium mitigation. They are mainly composed of mortar/concrete tests measuring the global free expansion of calibrated samples. Each of these standards fixes its own maximum expansion limit varying according to the different test conditions. Data from these tests are collected in this article and adapted to Larive's chemo-mechanical model to predict necessary lithium dosage and resulting mitigation of deleterious ASR-expansion. Since the results are only based on test specimen where Li-compounds were mixed into the samples, they do not reflect any relevance for concrete under operation which is treated from the surface with Li-containing solutions.

## 2. METHODS

The amount of lithium needed is generally calculated in two different ways: based on mass of cement percentage or with a ratio over alkali content (lithium ratio  $[Li]/[Na+K]$ , where ions are counted one for one). Since the alkali content is the main parameter to reduce ASR expansion, the latter is the most convenient way to display data over quantity of lithium needed.

Thomas et al. [3] reviewed the different tests that are used to assess the potential of mitigation using lithium and the optimal lithium ratio for suppressing ASR, considering a particular mortar or concrete formulation with reactive aggregates.

According to this, data were collected from studies using ASTM C 1293, 1260 & 227 which are the most used and reliable standards, even if only ASTM C 1293 is eff recommended for lithium mitigation. Indeed some relative issues can be encountered for example with ASTM C 1260, that can misestimate the critical  $[Li]/[Na + K]$  ratio (*ie* the necessary amount of lithium to reduce expansion under a defined limit), because of the approximate composition of the soak solution, see [3]. The objective of this paper is to link expansion as function of lithium dosage and to design and perform statistical methods: the perfect reliability of the standards are in the first place left aside to obtain enough data.

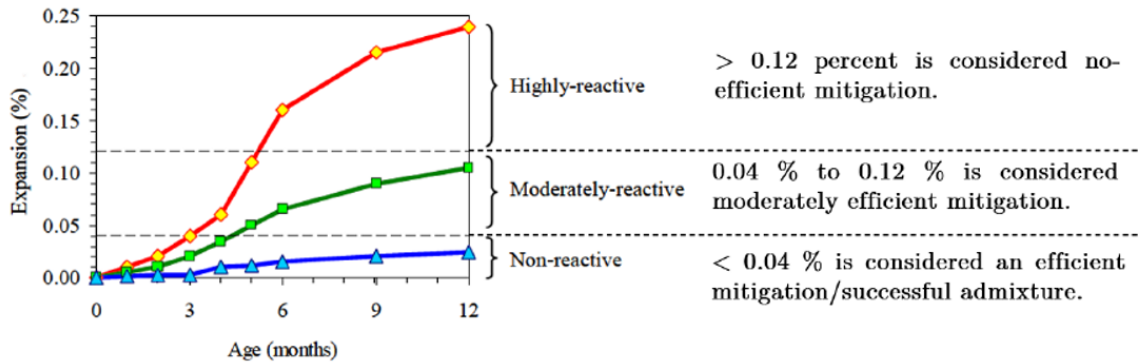


Figure 1 – Example of test classification: ASTM C 1293 expansion limits [6].

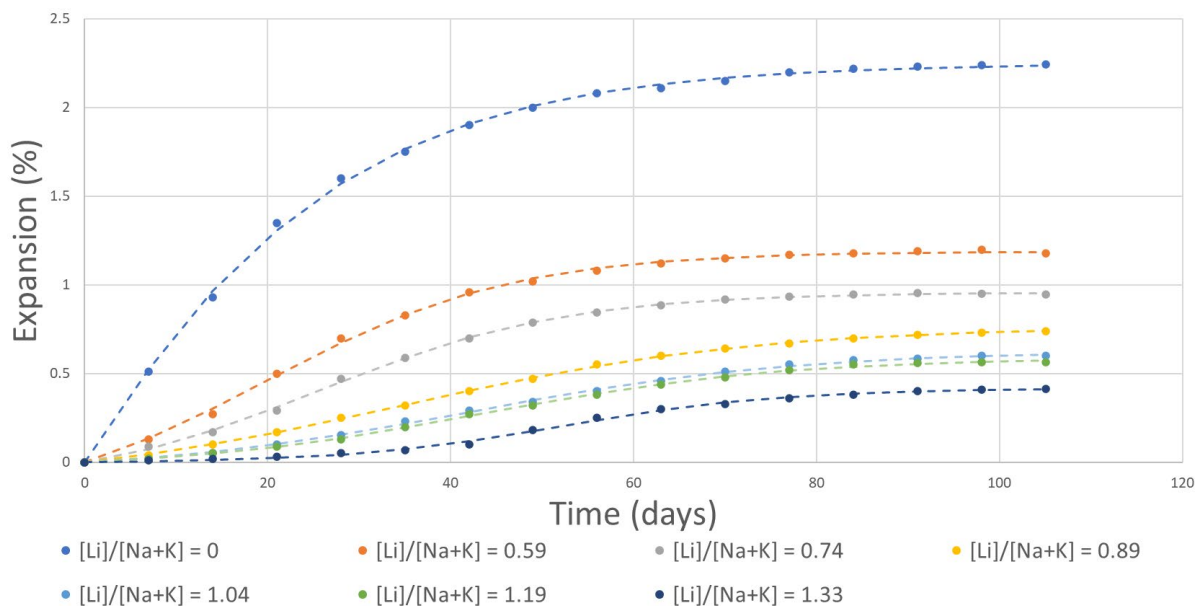


Figure 2 – Expansion of the mortar bars prepared with various  $[Li]/[Na+K]$  ratios, from Islam & Ghafoori [7] - Example of data used to design the method.

The idea is to find a law that models these expansion curves (see example in Figure 2) linked to different parameters, in particular lithium dosage, and try to determine the optimum verifying the expansion criteria (defined by the selected standards, see example in Figure 1). To achieve this, the chemo-mechanical model of Larive will be used [8].

## 2.1 Larive chemo-mechanical model

Larive [8] established from a chemo-mechanical analysis a first-order kinetic ordinary differential equation (ODE) that can be expressed as:

$$\varepsilon_{\infty} - \varepsilon_{ASR} = \lambda(\varepsilon_{ASR})\varepsilon_{ASR} \quad (1)$$

where:

- $\varepsilon_{\infty}$  is the ultimate expansion due to ASR.
- $\varepsilon_{ASR}$  is the measured expansion due to ASR through time, it is the unknown variable of the ODE.
- $\lambda(\varepsilon_{ASR})$  represents the diffusion of the reaction products.  $\lambda$  must be a decreasing function of  $\varepsilon_{ASR}$  to reflect the fact that the more  $\varepsilon_{ASR}$  increases, the easier the diffusion of the reaction products is. It is usually considered that  $\lambda(\varepsilon_{ASR}) = \frac{a}{b + \varepsilon_{ASR}}$  with  $a$  &  $b$  material constants.

Solving this equation gives the time-dependant law presented below, without further details:

$$\varepsilon_{ASR} = \varepsilon_{\infty} \frac{1 - \exp\left(-\frac{t}{\tau_c}\right)}{1 + \exp\left(-\frac{t - \tau_l}{\tau_c}\right)} \quad (2)$$

which describes the expansion due to ASR through time with three parameters. Along with  $\varepsilon_{\infty}$  are introduced a time of latency  $\tau_l$  linked to the initiation of the chemical reaction (giving the time of the inflection point, see Figure 3) and  $\tau_c$ , the characteristic time of the reaction itself.

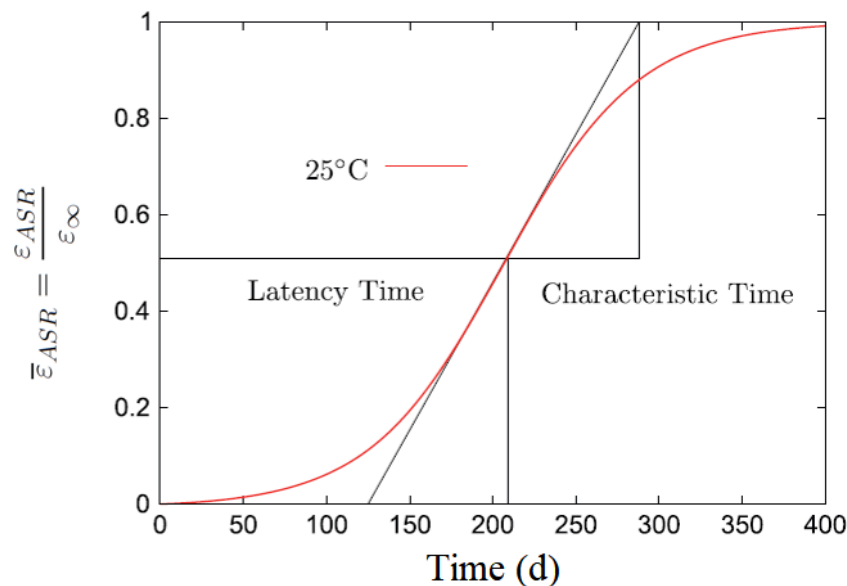



Figure 3 – Model of chemical expansion by Larive [7].

These time parameters have been experimentally linked by Larive [8] to temperature and saturation, and models have been established later [10] [9]. Even if these physical quantities are important when considering ASR, they have constant values in each standard, only lithium ratio is varying. Consequently, curve fitting were done only with those three parameters  $\varepsilon_{\infty}$ ,  $\tau_c$  &  $\tau_l$ .

It should be  in mind that models that will be presented in the following study are only tendencies because of the differences between methods and Li-compounds used in the analysed studies.

## 2.2 Data collection

To properly identify parameters for this model, some criteria were chosen. Only the studies working with one of the three following standards were selected: ASTM C1293, 1260 & 227. For the remaining studies, the following selection criteria were applied:

- Expansion tests have to be carried out for a 1-year duration at least - or samples have to reach 95% of their theoretical final expansion.
- At least two expansion measurements have to be done during the first month and at least one measurement by month after.
- At least four different combinations have to be tested with  $[Li]/[Na+K]$  ratio and/or  $Na_2O_{eq}$  varying.

In 2005, Feng et al. wrote a very precise review on lithium mitigation [2]. It constituted a starting point for this research. The different sets of expansion samples that were selected in this study are presented in Table A in Appendix. Some other studies were analysed to verify global tendency but were not further analysed due to a lack of statistical relevance (too few measure points, not verifying the “four different combinations” criteria). Even though ASTM C 227 was withdrawn in 2018, studies using it were kept because of the great amount of data it provided for statistical purposes.

## 2.3 Curve-fitting routine

The regression to identify  $\varepsilon_{\infty}$ ,  $\tau_c$  &  $\tau_l$  in Eq. 2 is processed with the Curve Fitting Tool of MATLAB.

Curve Fitting Toolbox is an additional app in MATLAB that provides functions for fitting curves and surfaces to data. It conducts regression analysis with both linear and nonlinear models. The regression technique is based on a least-squares equation to minimize. The Levenberg-Marquardt algorithm is used to solve the equations because this algorithm has been proven to work most of the time for a wide range of nonlinear models and starting values.

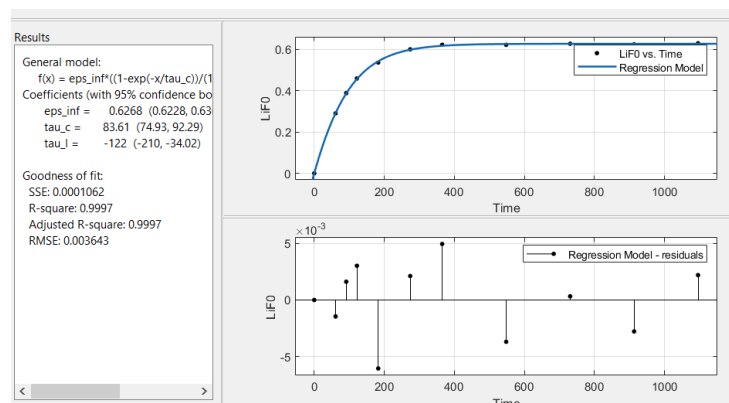


Figure 4 – Screenshot of MATLAB Curve Fitting Tool.

### 3. RESULTS & DISCUSSION

#### 3.1 Expansion curves

Sets of expansion curves like the one presented in Figure 2 were analysed with the curve fitting routine presented above and results were obtained for each of the parameters  $\epsilon_{\infty}$ ,  $\tau_c$  &  $\tau_l$ . The results are plotted in the Figures 5, 6 & 7.

#### 3.2 Larive’s model parameters

$\epsilon_{\infty}$  - Final expansion & pessimum effect

A clear and expected global tendency is observed on Figure 5 with pessimum effect [23] (positive value of  $[Li]/[Na+K]$  where  $\epsilon_{\infty}$  reaches a maximum value and then decreases toward a limit value, see Eq. 3, Figure 5 and Figure 9). A limit value is observed for every study, at various  $[Li]/[Na+K]$ . However, only four of the studies presented here (K\_LiOH, K\_Li2CO3, S\_LiF, S\_Li2CO3, see Table A in Appendix for correspondence) show a maximum value of expansion.

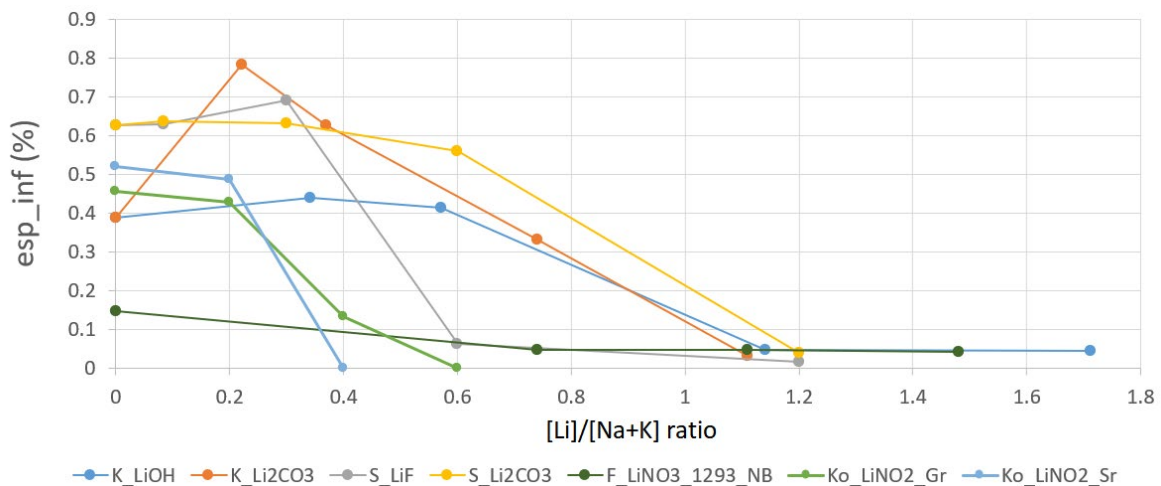


Figure 5 –  $\epsilon_{\infty}$  vs.  $[Li]/[Na+K]$ .

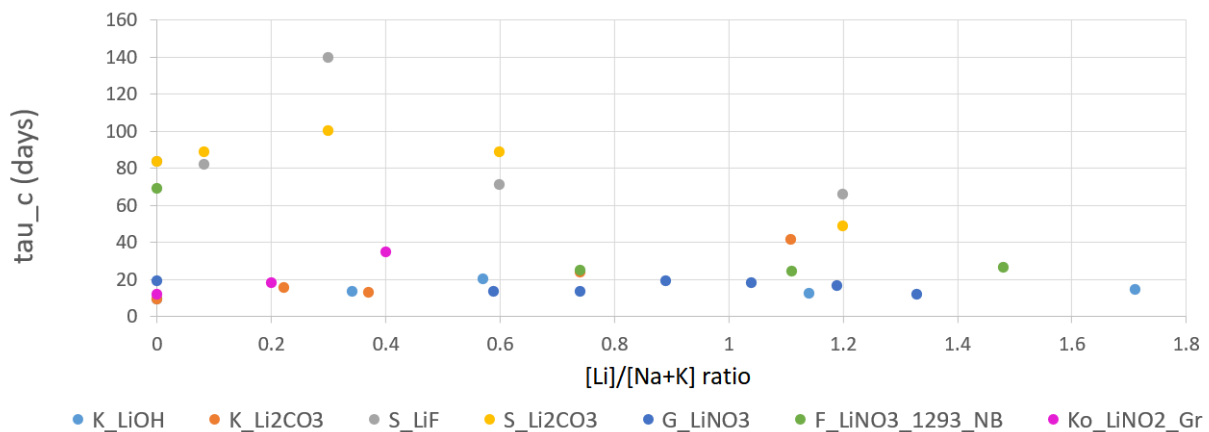


Figure 6 –  $\tau_c$  vs.  $[Li]/[Na+K]$ .

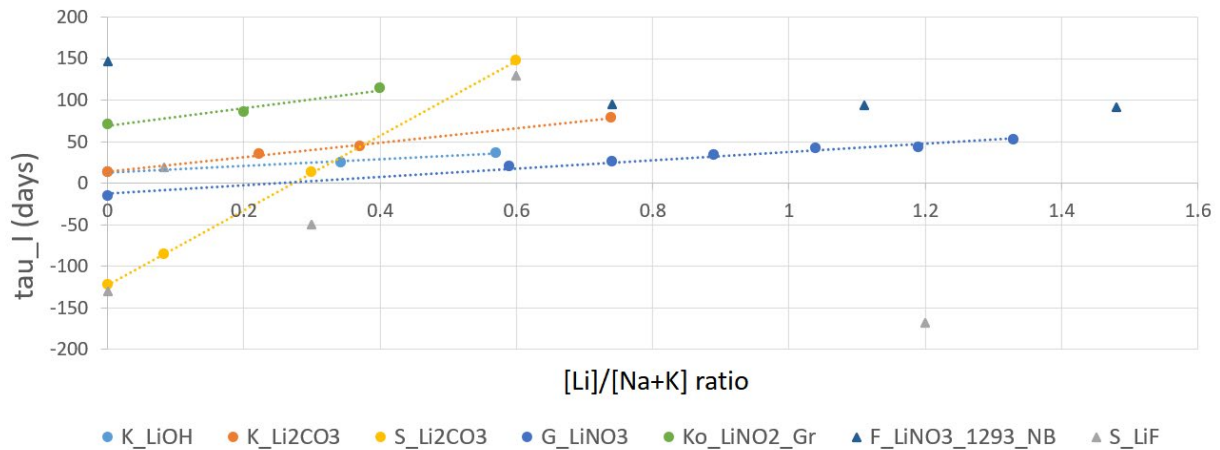


Figure 7 –  $\tau_l$  vs.  $[Li]/[Na+K]$ .

The three other studies (F\_LiNO3\_1293\_NB, Ko\_LiNO3\_Gr, Ko\_LiNO3\_Sr), and some other not displayed here (F\_LiNO3\_1260\_NS, G\_LiNO3) do not present this pessimum. A model of  $\epsilon_\infty$  can be deduced from the data that both describes the pessimum value and threshold effect seen in Figure 5. The following equation was found to describe this with as few parameters as possible:

$$\epsilon_\infty = \epsilon_{max} \times \exp\left(\frac{-([Li]/[Na+K]-p)^2}{c}\right) \quad (3)$$

Eq. 3 is a pseudo-gaussian model where:

- $\epsilon_{max}$  is the maximum final expansion, obtained for a particular  $[Li]/[Na+K]$  ratio.
- $p$  is the pessimum  $[Li]/[Na+K]$ , abscissa where  $\epsilon_\infty = \epsilon_{max} \cdot p$  can be negative to properly model the cases where no pessimum is observed.
- $c$  is a mathematical dilation factor controlling the rate of decrease.

The Figure 8 & 9 show the adequacy between model and experience for two different studies. One series (Figure 8) shows monotonic reduction of final expansion with increasing lithium dosage up to the critical  $[Li]/[Na + K]$ . The other (Figure 9) shows a pessimum value of the final expansion as lithium dosage increases before the critical  $[Li]/[Na + K]$  is reached. Clearly, eq.(3) is able to model both types of behaviour

### $\tau_c$ – Characteristic time

Figure 6 shows a clear difference in the behaviour with one showing monotonic reduction of final expansion and another shows a pessimum value of the final expansion as lithium dosage increases.

### $\tau_l$ – Latency time

Figure 7 reveals a possible linear correlation with  $\tau_l$  and  $[Li]/[Na+K]$ , unless some studies don't follow this tendency. For these studies, an average value of  $\tau_l$  will be taken. Table 2 presents the values used for  $\tau_c$  and  $\tau_l$  in the final model.

Table 1 – Regression values for  $\tau_c$  and  $\tau_l$  - Final model

Article Id	$\tau_l = a \times x + b$		$\tau_c = \overline{\tau_c}$
	a	b	-
K_LiOH	33	13.327	13.9752
K_Li2CO3	87.6	14.064	20.4652
S_LiF	0	-38.504 ( $\overline{\tau_l}$ )	88.422
S_Li2CO3	449	-122.31	81.982
G_LiNO3	49.978	-12.274	15.9729
F_LiNO3_1293_NB	0	106.635 ( $\overline{\tau_l}$ )	36.155
Ko_LiNO2_Gr	107.68	68.998	21.6767
Ko_LiNO2_Sr	157.55	22.11	20.745

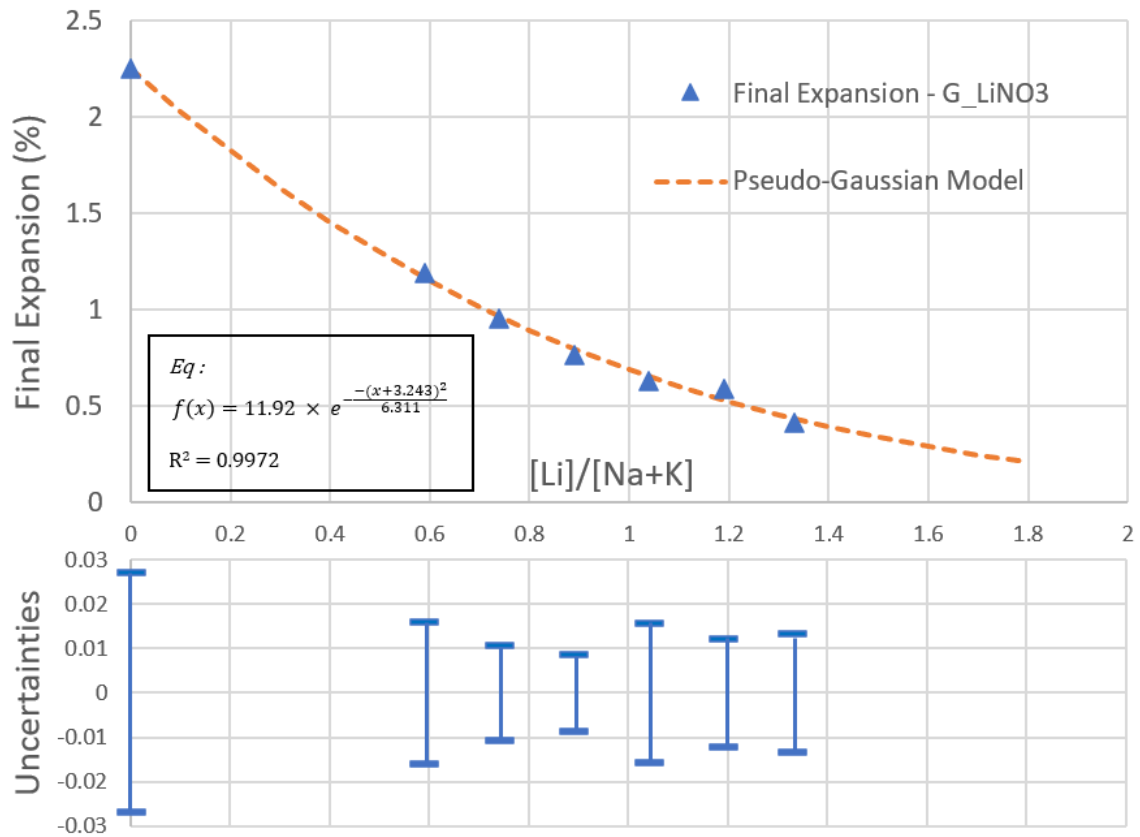


Figure 8 – G\_LiNO3 [7]: pseudo-gaussian model for  $\epsilon_\infty$  function of  $[Li]/[Na+K]$ .



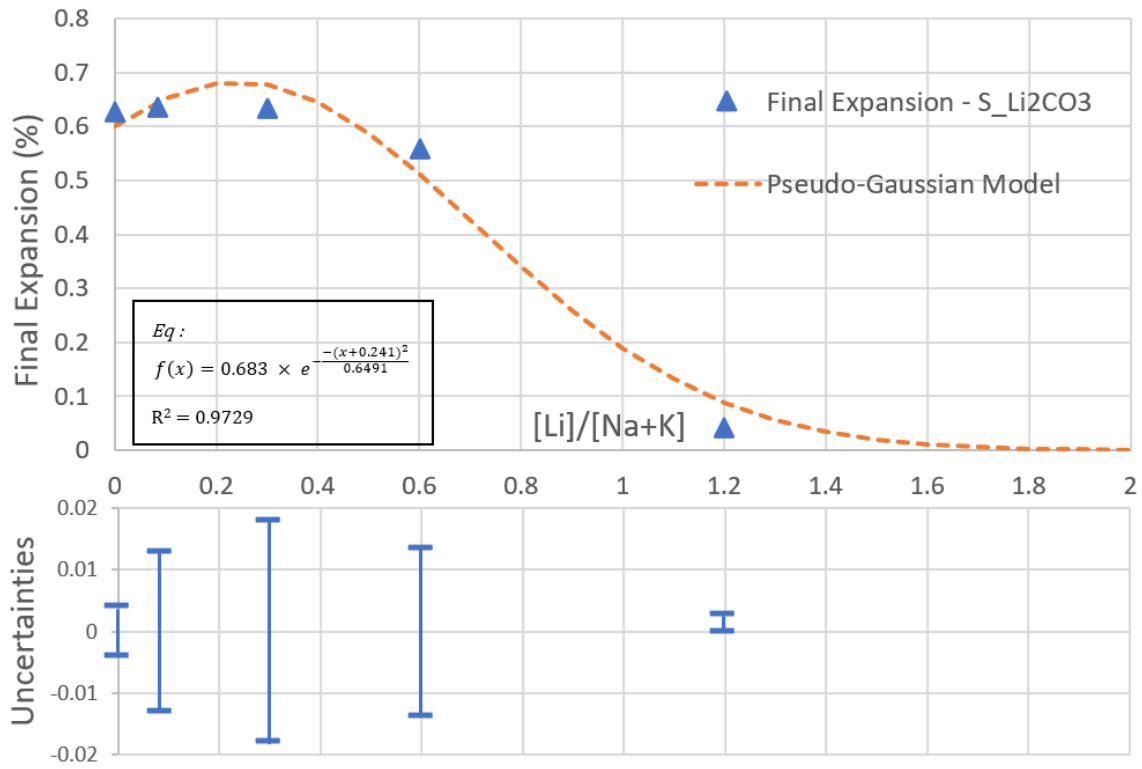


Figure 9 –  $S_{Li_2CO_3}$  [11]: pseudo-gaussian model for  $\varepsilon_{\infty}$  function of  $[Li]/[Na+K]$ .

### 3.3 Critical $[Li]/[Na+K]$ model construction and final results

With all the results presented above, we can finally rewrite the model of Larive for lithium mitigation. For each set of expansion curves of a given study, the global expansion due to ASR is:

$$\varepsilon_{ASR}(t, x) = \varepsilon_{\infty}(x) \frac{1 - \exp\left(-\frac{t}{\tau_c}\right)}{1 + \exp\left(-\frac{t - \tau_l(x)}{\tau_c}\right)} \quad (4)$$

with:

- $x = [Li]/[Na+K]$ .
- $\tau_c = \overline{\tau_c}$  for the given set of expansion curves.
- $\tau_l(x) = a \times x + b$
- $\varepsilon_{\infty} = \varepsilon_{max} \times \exp\left(\frac{-(x-p)^2}{c}\right)$

The equation to solve to determine the critical  $[Li]/[Na+K]$  is then  $f(\varepsilon_{st}, t_{st}, x) = 0$  with  $\varepsilon_{st}$  and  $t_{st}$  describes the expansion criterion defined by the selected standard and where  $f$  is:

$$f(\varepsilon_{st}, t_{st}, x) = \varepsilon_{ASR}(t_{st}, x) - \varepsilon_{st} \quad (5)$$

$g(x) = \varepsilon_{ASR}(t_{st}, x)$  is plotted on Figures 10 & 11 for two given studies to illustrate the equation resolution.

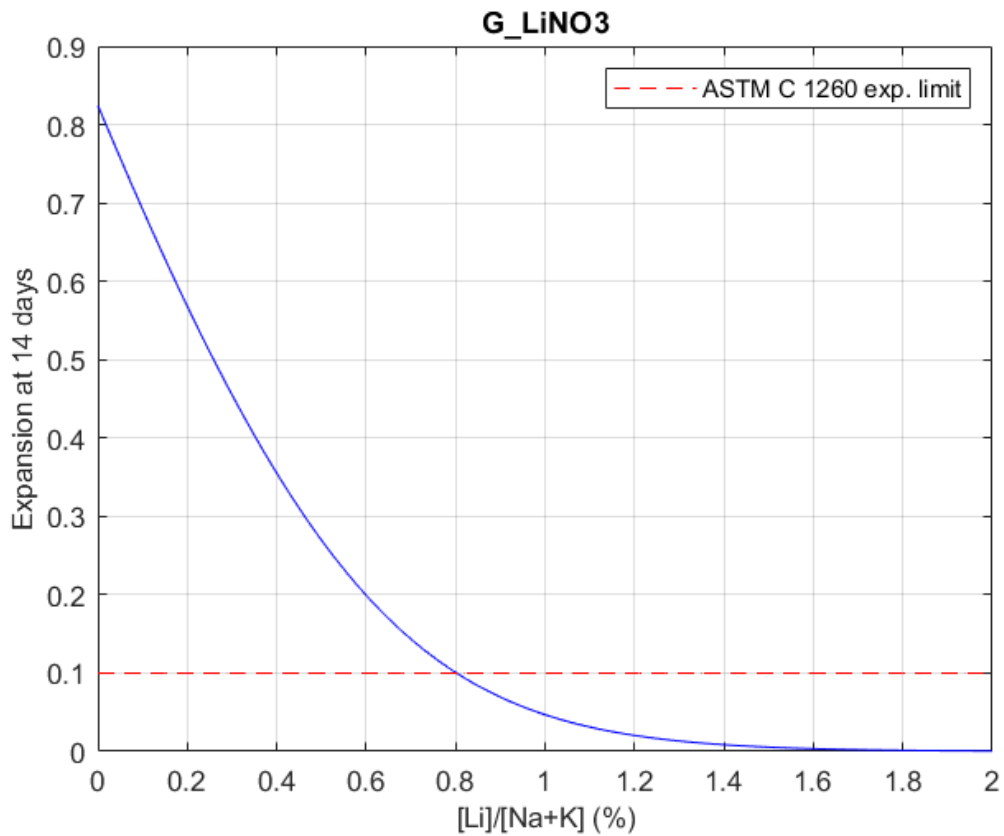


Figure 10 – Result for G\_LiNO3 - ASTM C 1260 exp. criteria: 0.1 % at 14 days.

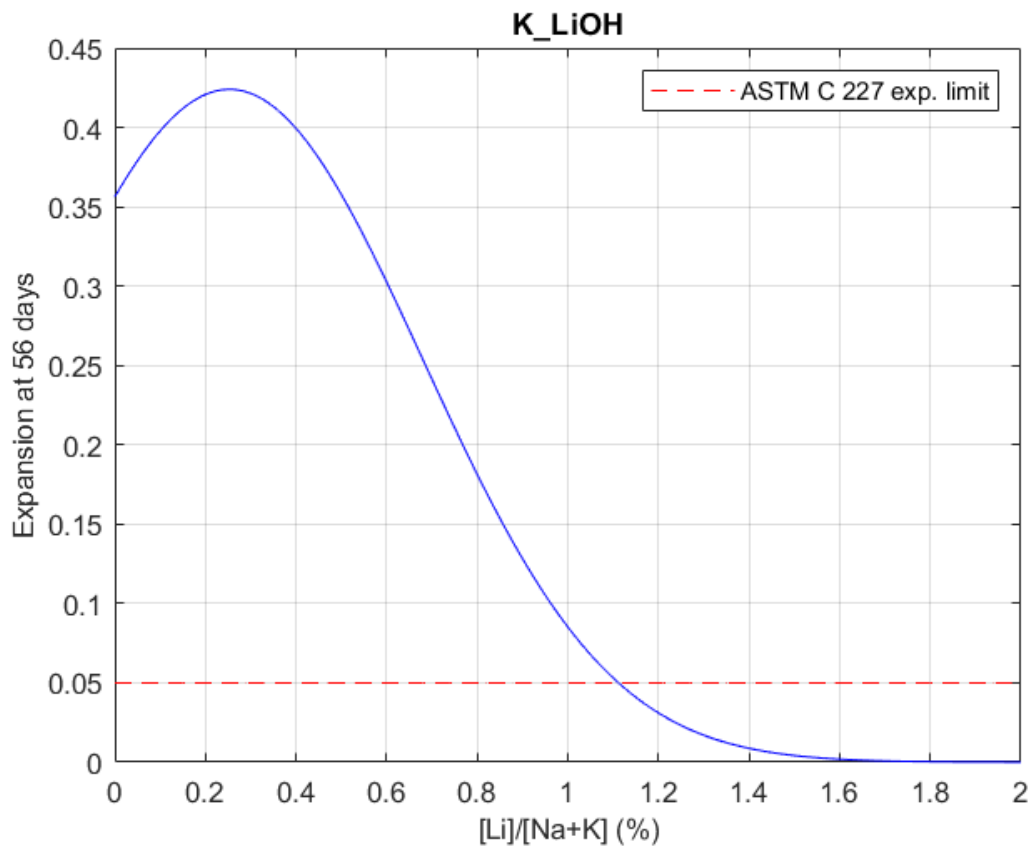


Figure 11 – Result for K\_LiOH - ASTM C 227 exp. criteria: 0.05 % at 56 days.

The results displayed in Table 2 all give reliable prediction compared to the original data. Model prediction always gives results that are into the confidence interval of the experience. Of course, the statistical part of the model allow to find back the experimental results, but the practical part here is to link it with a chemo-mechanical model that allows a quantitative determination of mitigating lithium dosage for real cases. Some researchers such as Islam & Ghafoori [7] [24] also did regression to determine critical  $[Li]/[Na+K]$  but used only mathematical models with no link with mechanics such as the model of Larive has.

Table 2 – Final Results - Critical  $[Li]/[Na+K]$

<b>Id</b>	<b>Experimental Observation</b>	<b>Model Prediction</b>
S-LiF	0.5 $[\pm 0.05]$	0.52
S-Li <sub>2</sub> CO <sub>3</sub>	0.6 $[\pm 0.05]$	0.64
K-LiOH	1.14 $[\pm 0.05]$	1.11
K-Li <sub>2</sub> CO <sub>3</sub>	0.8 $[\pm 0.05]$	0.78
Ko-LiNO <sub>2</sub> -Gr	0.3 $[\pm 0.1]$	0.36
Ko-LiNO <sub>2</sub> -Sr	0.5 $[\pm 0.1]$	0.42
G-LiNO <sub>3</sub>	0.89 $[\pm 0.05]$	0.92

#### 4. CONCLUSION AND PERPECTIVES

A mechanistic based model for the decrease of final expansion of potentially ASR affected concrete mortars with Li-compounds admixture was developed from Larive's model. It was designed applying a wide range of experimental data for ASR expansion measured in accelerated expansion tests. It also allows to study the influence of multiple influences with a relatively small number of three main independent parameters. The main benefit of this study is that it brings a behavioural model to describe and compare data from very common tests performed to evaluate the potential of an ASR expansion for a given concrete formulation, model that apparently did not existed in the literature at this time.

The principal limitation is that it remains difficult to predict the critical ratio of Lithium without an experimental test, for example one of the standard tests presented in this study. It is known that even with a precise understanding of the reaction, the nature of aggregate plays a huge role in the final expansion. More study is needed to investigate the parameters of influence linked to the nature of aggregates. Also the effect of leaching of alkalis (and possibly lithium) during accelerated testing of small specimens was ignored.

Further work should involve both full-scale pressurisation tests on sections of ASR-affected concrete bars as well as measurements on cores of the concrete structures. All of this in order to analyse to what extent the results for experimental mortar bars can be generalized for real hardened concrete exposed to real climate conditions.

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**APPENDIX**

*Table A – Extract: Lithium mitigation tests selected for this study - Selected data ; Analysed data for global comparison but not selected ; Rejected data*

Article	Id	Testing Method	Reactive Aggregate	Li. Salt	$\frac{[Li]}{[Na+K]}_{crit}$	Specifications
Stark (1992) [11]	S-LiF	ASTM C 227	Andesite	LiF	0.5	Ok
	S-Li <sub>2</sub> CO <sub>3</sub>	ASTM C 227	Andesite	Li <sub>2</sub> CO <sub>3</sub>	0.6	
Kawamura (2003) [12]	K-LiOH	ASTM C 227	Calcined Flint	LiOH	1.14	Ok.
	K-Li <sub>2</sub> CO <sub>3</sub>	ASTM C 227	Calcined Flint	Li <sub>2</sub> CO <sub>3</sub>	0.8	
Ghaafori (2016) [7]	G-LiNO <sub>3</sub>	ASTM C 1260	Andesite	LiNO <sub>3</sub>	0.89	Ok.
Kobayashi (2019) [13]	Ko-LiNO <sub>2</sub>	JIS A 1146	Var. Aggregates	LiNO <sub>2</sub>	0.4 & 0.6	Ok. Only non-ASTM method selected.
Lane (2002) [14]	La-LiOH	ASTM C 1293	Quartz	LiOH	0.925	Ok, but only two ratios tested.
	La-LiNO <sub>3</sub>	ASTM C 1293	Quartz	LiNO <sub>3</sub>	?	
Ramyar (2002) [15]	Ra-LiF	ASTM C 1260	?	LiF	>0.9	Ok, but only one ratio tested.
	Ra-Li <sub>2</sub> CO <sub>3</sub>	ASTM C 1260	?	Li <sub>2</sub> CO <sub>3</sub>	1.54	
Feng et al. (2010) [16]	F-LiNO <sub>3</sub> -1293	ASTM C 1293	Rhyolithe	LiNO <sub>3</sub>	>0.74	Ok, but only two ratios tested.
	F-LiNO <sub>3</sub> -1260	ASTM C 1260	Rhyolithe	LiNO <sub>3</sub>	0.74	
Tremblay (2010) [17]	T-LiNO <sub>3</sub> -1293	ASTM C 1293	Var. Aggregates	LiNO <sub>3</sub>	0.74	Ok, but only two ratios tested.
	T-LiNO <sub>3</sub> -1260	ASTM C 1260	Var. Aggregates	LiNO <sub>3</sub>	?	
Ekolu et al. (2003) [18]	E-LiNO <sub>3</sub>	ASTM C 1293	Volcanic Glass	LiNO <sub>3</sub>	?	Ok, but only one ratio tested.
Mo et al. (2003) [19]	Mo-LiOH	Autoclave	Microcryst. Quartz	LiOH	0.3 & 0.6	Less relevant data but interesting tendencies
Collins et al. (2004) [4]	C-LiOH	ASTM C 227	Borosilicate glass	LiOH	0.6	Less relevant data.
	C-LiCl	ASTM C 227	Borosilicate glass	LiCl	0.9	
	C-LiNO <sub>3</sub>	ASTM C 227	Borosilicate glass	LiNO <sub>3</sub>	0.8	
Hargis (2013) [20]	H-LiOH	ASTM C1260	Various Aggregates	LiOH	?	Less relevant data
Liu (2019) [21]	Liu-LiNO <sub>3</sub>	ASTM C589	Sandstone	LiNO <sub>3</sub>	1.66	Less relevant, Liu tests rocks and not concrete
Rousselet (2016) [22]	R-LiOH	Own Method	Various Aggregates	LiOH	0.2 -> 0.4	Less relevant results but very interesting tendencies