



# The influence of the anolyte solution type and concentration on lithium migration in mortar specimens

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## HIGHLIGHTS

- A study on the influence of the anolyte solution on lithium migration is presented.
- Results indicate that anolyte concentration, rather than type, affected migration.
- Anolytes with highest concentrations led to highest levels of lithium in specimens.

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## ABSTRACT

Electrochemical lithium migration has been suggested as repair technique for alkali-silica reaction affected concrete structure. In this method, an electric field is used to transport lithium into the material. Current studies have used anolyte solutions with various lithium salts at different concentrations. However, little has been said on the effect of the anolyte on lithium migration. In this paper, an experimental study on the influence of the type of lithium compound and its concentration in the anolyte is presented. Results point out that the concentration of the solution, rather than the type of lithium salt, affected migration. The anolytes with the highest concentrations provided the highest final levels of lithium in the specimens.

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## 1. Introduction

Even though alkali-silica reaction (ASR) affects many concrete structures worldwide, currently, there are limited repair options available [1,2]. In this framework, electrochemical lithium migration has been suggested as an intervention method.

The use of lithium-based admixtures to suppress ASR expansion has been known for decades (e.g. [3–5]). It has been proposed that lithium ions alter the reaction mechanism either by hindering the reaction or by altering the product into a less-expansive one [1,6–8]. In existing concrete structures, however, lithium ions can no longer be incorporated into the fresh mixture. In this case, the ions need to be transported into the material and electrochemical lithium migration has shown to be the most effective technique to do so [9,10].

Driving lithium ions into concrete by means of an electrical field was first suggested by Page [11]. He theorized that, if a lithium

solution was used as anolyte during an electrochemical chloride extraction treatment of a structure, lithium ions would migrate towards the reinforcing steel and mitigate the effects of ASR. Since then, a number of studies have been published (e.g. [9,10,12–19]), with divergent conclusions.

In current literature, several different lithium salts have been used in the anolyte solution, at different concentrations, under voltages up to 60 V. Nevertheless, little has been discussed on the reason behind the choice of those solutions or on whether the choice would influence the final results. In fact, Ueda [20] investigated the effect of different lithium compounds. However, the concentration of the anolyte solutions was not discussed. In this article, the influence of different lithium solutions on migration will be addressed.  $\text{Li}_2\text{CO}_3$ ,  $\text{LiOH}$  and  $\text{LiNO}_3$  were the lithium salts chosen to be tested at concentrations varying from 0.2 to 7.8 M.  $\text{LiOH}$  and  $\text{LiNO}_3$  were considered because of their high solubility in water. Although  $\text{Li}_2\text{CO}_3$  has very low solubility in water, Ueda [14] noted in his work that  $\text{Li}_2\text{CO}_3$  solution leads to higher effective diffusion coefficient than  $\text{LiOH}$  (when tested in a two-chamber set-up).

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## 2. Experimental program

### 2.1. Materials and specimen preparation

Mortar specimens were prepared with water to cement ratio (w/c) of 0.5 and sand to cement proportion of 3:1. The mixing procedure followed the standard NEN-EN 196-1 [21]. The air void content was measured as 1.0% (NEN-EN 12350-7 [22]) and the flow value, obtained by the flow table test (NEN-EN 12350-5 [23]), was 270 mm.

Ordinary Portland cement type CEM I 42.5 N, commercially available in the Netherlands (ENCI), was used. Its chemical composition is shown in Table 1. In addition, CEN standard sand with  $D_{max}$  of 2 mm (according to EN 196 1:2005) and deionized water were used. Cylindrical specimens, with diameter of 98 mm and height of 50 mm, were cast and cured in a fog room ( $20.0 \pm 2.0^\circ\text{C}$  and R.H. of  $96 \pm 2\%$ ) for 36 days before the beginning of the experiment.

### 2.2. Methods

Lithium migration testing was performed in the set-up described by ASTM 1202 [24]. As shown in the scheme of Fig. 1(a), a specimen was placed between two acrylic chambers filled with solution, each with a stainless steel mesh as electrode. Each chamber contained 270 ml of electrolyte solution. Once the electric potential was applied between the electrodes, cations were attracted by the cathode (negative electrode), whereas anions moved in the opposite direction, towards the anode (positive electrode). Fig. 1(b) shows one of the experimental cells. The experiments were carried out in a climate controlled laboratory, at  $20.0 \pm 2.0^\circ\text{C}$  and R.H. of  $50 \pm 5\%$ .

Saturated  $\text{Ca}(\text{OH})_2$  solution (0.02 M) was used as catholyte in all tests. The anolytes, on the other hand, were solutions of different lithium compounds, at different concentrations, as shown in Table 2. The highest concentration of each lithium compound solution is its saturation (or near saturation) concentration. Lithium compounds with higher solubility were also tested in lower concentrations, as the table shows. It is worth noting that  $\text{Li}_2\text{CO}_3$  0.2 M solutions had lithium concentration of 0.4 M while the other 0.2 M solutions had 0.2 M of lithium. The range of anolyte concentrations was chosen so that the solutions would be tested at their saturation (or near saturation) concentration and at a wide range. Each solution was tested with two replicates, except in the case of the  $\text{LiOH}$  0.2 M solution. In this case, due to experimental problems, the results of one specimen will be presented. The specimens were tested during one week under 40 V (electric field of 0.8 V/mm). This voltage was chosen as it is maximum voltage usually used in the field in treatments such as electrochemical chloride removal [25].

Passing current and catholyte temperature were continuously monitored and recorded by a data logger, while electrical resistance of the specimens and electrolyte pH were measured four times during the experiments. The electrical resistance was measured with a LCR-meter, in resistance mode at 120 Hz, while the specimens were still in the cells. During the experiment, the resistance was

**Table 2**  
Lithium solutions used as anolytes.

Salt	Concentration (M)	Salt	Concentration (M)
$\text{Li}_2\text{CO}_3$	0.2 (saturated)	$\text{LiNO}_3$	0.2
$\text{LiOH}$	0.2	$\text{LiNO}_3$	4.9
$\text{LiOH}$	4.9 (near saturation)	$\text{LiNO}_3$	7.8 (near saturation)

measured immediately after switching off the current. From the resistance, the specimen resistivity can be calculated with Eq. (1) [26], assuming that the resistance outside the specimens is zero:

$$\rho = \frac{RA}{L} \quad (1)$$

where  $R$  is the electrical resistance ( $\Omega$ ),  $A$  is the specimen surface area ( $\text{m}^2$ ) and  $L$  is the thickness of the specimen (m). Care should be taken when interpreting resistivity values, as they are strongly affected by temperature variations. Increase in temperature leads to drop in resistivity and vice versa. In fact, temperature may influence up to 5% with every K degree of variation [26]. The measurement on the eighth day was performed after 24 h without power, at room temperature.

The pH values of the electrolytes were obtained with a pH-meter, when it was possible. In the case of high pH (above 11) or high lithium concentration, pH test strips were used, in order to avoid pH-meter reading errors such as alkaline error [27]. Electrolyte samples were collected three times during the test and were analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES), in order to obtain the concentration of sodium, potassium, lithium and calcium (the last only in anolyte).

Ionic concentration profiles in mortar were obtained after the end of the test. To obtain these, the specimens were ground in a profile grinder in steps of 5.0 mm. The obtained powder (10–20 g) was then dissolved in boiling 3.0 M  $\text{HNO}_3$  (100 ml) and filtered to obtain a clear solution. The filtrate was washed with four parts of 10 ml of 1.0 M  $\text{HNO}_3$ . The obtained clear solution was then analyzed by ICP-OES for lithium, sodium and potassium.

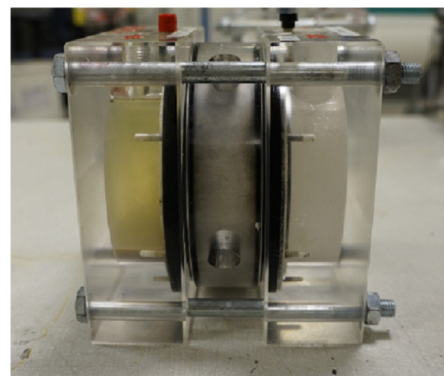
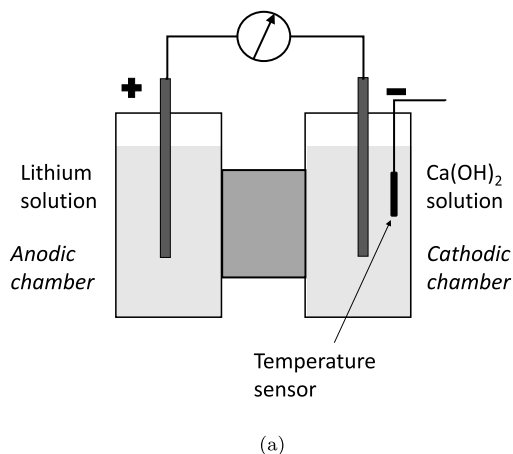
## 3. Results and discussion

Fig. 2(a) shows the current density that passed through all specimens during the migration experiment. The test with  $\text{LiNO}_3$  4.9 M (2) presented connection problems, as can be seen in the current density plot. The general behavior can be divided into three parts: in the first couple of hours, there was a rapid current increase, followed by a slower drop until around the third day. Finally, the

**Table 1**  
Cement composition, wt.% of cement.

CaO	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$\text{SO}_3$	MgO	$\text{P}_2\text{O}_5$	$\text{K}_2\text{O}$	$\text{TiO}_2$	$\text{Na}_2\text{O}$	Other	L.O.I. <sup>a</sup>
65.00	18.33	4.42	3.38	3.01	2.02	0.57	0.46	0.37	0.28	0.53	1.60

<sup>a</sup> L.O.I.: loss on ignition.



**Fig. 1.** Schematic diagram of the experimental set-up (a) and an experimental cell (b). Each electrolyte chamber contained 270 ml of solution.

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