

STUDY OF SILANE COMPOSITIONS TO MITIGATE ALKALI-SILICA REACTION

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Abstract

There are well-established preventive measures to reduce the potential damage to a concrete structure when reactive aggregates are used. However, there are only few efficient measures to apply to a structure once the deleterious reaction has already been established. So as to extend the ASR repair strategies, the present study aimed to verify, in laboratory, the mitigating potential of silane compositions in residual expansion of affected mortars cast with reactive aggregates. The results indicated that silane-based treatments, mainly the one containing octyltriethoxysilane, are very promising. Water absorption measurements and microstructural characterization bywith scanning electron microscopy of the mortar bars treated with silanes were also included in the experimental program.

Keywords: expansion; mitigation; alkali-silica reaction; silane; treatments.

1 INTRODUCTION

The alkali-silica reaction (ASR) produces a siliceous-alkaline gel which adsorbs water and expands, causing damages to concrete. Considering this reaction installed and at advanced deterioration stages, it may compromise the stability of the structure even generating problems at operational level. Concrete structures, such as those of hydropower plants, dams and bridges, are particularly more vulnerable to this reaction due to the continuous presence of water. The measures adopted to prevent the expansions deriving from this reaction have long been known and routinely applied to hydropower plants structures when reactive aggregates are present. However, there is still a gap to fill: the lack of options for treatments that stop the ASR mechanisms in already affected structures, or that at least mitigate residual expansions and, consequently, the effects of the reaction. The costs for repairing these structures and for minimizing the problems deriving from ASR are extremely high, and the measures adopted are generally palliative.

The methods for mitigating ASR effects in a structure can be divided into two categories: attenuation of the symptoms and treatment of the causes. The methods for attenuating the symptoms include caulking cracks; making cuts to allow expansion, thus relieving the stresses within the concrete or adding structural elements that restrain expansion. As to the treatment of the causes, the two only practical means for retarding or for preventing future reaction in existing structures is by drying the concrete to eliminate the necessary moist for the reaction to occur or by changing the nature of the reaction by introducing products that stabilize it [1]. In this context, there are the lithium-based compositions, applied to concrete already affected by ASR, whose proven efficacy is known in the technical environment due to the number of works published [1, 2, 3, 4, 5, 6, 7, 8, 9, 10], be it approaching the application in laboratory tests or, in some cases, field applications.

The present work aims to present the results of a research [11, 12] that investigates alternatives to ASR mitigation, seeing that there is still a lack of knowledge as regards fighting the phenomenon when it is already installed in the concrete. Hence, silanes were chosen due to the easiness in forming Si-O-Si chemical bonds, with the silanol groups present in mortars and in concretes. These bonds are very stable and may present good resistance to bad weather and to some chemical products. Therefore, the effect of silanes was assessed viewing their application to concretes already affected by

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ASR in an attempt to minimize residual expansions. An efficient and safe treating method to fight ASR may increase the service life of the structures affected, which are several in Brazil and innumerable abroad.

2 MATERIALS AND METHODS

2.1 General

Firstly, a bibliographic review was conducted, focusing on the objectives of the research, significantly contributing both to the experimental program elaboration stage and for analyzing and discussing the results. In some of the publications surveyed, the application of the lithium-based compositions in concretes already affected by the ASR was observed to have been conducted in laboratory tests or, in some cases, in field applications. Some authors applied the treatment with topic (pulverization), vacuum impregnation, electrochemical migration and pressure injection [1, 3, 5, 6, 8, 13]. Among the works investigated, some applied the treatment by immersion, which served as a base for elaborating the procedures adopted herein [2, 3, 7, 9]. It can be generally verified that there is not a fixed protocol of this form of application in treatments in the samples affected by ASR, be it for mortar or for concrete, the moment of treatment application, the drying and saturation times varying in each study, besides the number of cycles adopted. Furthermore, [14,15,16,17], regarding the use of silanes for civil construction applications, some of the studies discuss their application as painting, forming a surface protection system (water-resistant silicones); therefore, we could not find a single protocol for applying these products that met the expectation of this research, that is, laboratory application by immersing mortar bars. Hence, the methodology developed was based on the applications of lithium nitrate-based (LiNO_3) compositions. Also noticed was the lack of research directed to using silane as an ASR mitigating agent, not only owing to its hydrophobic effect, but also to the formation of chemical bonds during the condensation reaction, with silanol groups present in mortars and concretes [18, 19].

2.2 Materials and mix designs

The criteria for choosing the materials were:

a) Aggregate: proven reactive potentiality originating in a region with a history of cases of structures affected by ASR and the availability of a sufficient amount for conducting all the tests provided in the experimental program. Thus, a reactive minelite, originating in Recife (PE), Brazil was selected. The aggregate was characterized by petrographic analysis, following a Brazilian methodology established by NBR 15577-3/2008, from ABNT (Brazilian Association of Technical Standards), and AMBT (Accelerated mortar bar test method), employing ABNT NBR 15577-4/2008, similar to ASTM C 1260. The petrography indicated that the aggregate sample has mineral and deleterious phases that can lead to ASR, according to ABNT NBR 15577-3/2008, such as highly deformed quartz (with undulating extinction exceeding 30°) and over 15% of microgranular quartz associated to perthitic and myrmekitic intergrowth textures. The main mineralogical composition obtained from the microscopic examination estimated the presence of 50% of feldspar, 30% of quartz, 15% of micas, 3% opaque and traces of zircon. Figure 1 shows the photomicrography of the aggregate studied. As regards the AMBT test, the results indicated high expansions, reaching 0.31% at 16 days and 0.65% at 30 days of age. Based on the limits established by NBR 15577-1/08, the aggregate is considered Potentially Reactive, corroborating the petrographies.

b) Portland Cement: free from any kind of admixture, according to ABNT NBR 5732 and/or ABNT NBR 5733, presenting high total alkaline equivalent (1.53% expressed in $\text{Na}_2\text{O}_{\text{eq}}$) and autoclave expansion below 0.20%, determined by the method provided in ASTM C 151. High Initial Strength Portland Cement, of the CP-V type (similar to III-ASTM Type), was therefore used. The major physical, chemical and mechanical characteristics of the cement employed in the study were determined and the results are presented in Tables 1 and 2. According to these results, the cement abides by the limits of Brazilian standard ABNT NBR 5733/91.

c) Treatment products: silane-based compositions (of the methyltrimethoxysilane and octyltriethoxysilane types). The two types of silanes were chosen mainly because of having excellent bond with silica-based substrates, due to the length of their chains, thus facilitating their penetration in the mortar sample to be treated, along with their hydrophobic characteristics. Besides the silanes, employed in 3% and 10% dilutions, three different solvents were tested, namely: deionized water, ethanol and dimethyl sulfoxide (DMSO), to verify the response of each of them to the expansions generated. As regards lithium, a commercial product was chosen, composed of 30% lithium nitrate (LiNO_3) diluted in water, whose action to fight ASR is already known. This treatment was used as a reference and for

comparing the efficiency of the silane treatments. The lithium content was based on an optimization performed by different authors, whereas for silane, only two concentrations were used, there not being, therefore, an optimization of content.

In the flowchart shown in Figure 2, the materials used in the treatments are detailed, including the combinations of the solutions prepared, considering the following variables studied:

- Different treatment solutions (with lithium and with silanes), being that for the solutions with silanes, the following variations occurred:
 - Silane types: methyltrimethoxysilane (MTMS) and octyltriethoxysilane (OCTEO);
 - Silane dilutions in 3% and 10% solutions;
 - Solvent types: For MTMS, two types of solvents were tested: deionized water and dimethyl sulfoxide (DMSO). For OCTEO, two solutions, the first composed of ethanol and deionized water, and the second composed of ethanol, deionized water and DMSO; and
- Different treatment cycles: single treatment x multiple treatment.

2.3 Methods for assessment and analysis

The method for applying the treatment products was defined having as a reference some of the protocols available in the literature [2, 3, 7, 9] and based on the forms of applying the lithium-based product. Besides, laboratory essays were conducted to investigate the times necessary for drying and saturating the mortar bars (for applying the treatments), comparing them with the data obtained from the literature. From these studies, the procedures were elaborated, considering the particularities of each type of silane, obtained from the bibliographic review [11]. The methodology focused on the application of the treatment products in mortar bars cast according to AMBT (NBR 15577-4/2008, similar to ASTM C 1260); however, the bars were exposed in the condition employed in the accelerated concrete prism test (CPT), that is, they were stored at high moisture condition (> 95%) at a 60°C temperature [20] and the tests for monitoring the expansions took up to 60 days. The methodology used was as follows:

After 3 days of casting, the 60 bars were cured for 24 hours in the wet chamber and were then demolded. After the first reference reading, the bars were placed vertically in the storing containers, hermetically sealed, so as to keep high internal moist and placed in a controlled temperature ambient of $60^{\circ}\text{C} \pm 2^{\circ}\text{C}$, where they remained until the date for applying the treatments (60 days), only being removed to perform their length readings. The choice of the 60 days of age for removing the bars from the box and treating them derived from the observation of their expansive behavior up to that date; the bars still presented growing expansions and at 2 months they had already reached a 0.13% value. Considering that each type of treatment was applied to a sample composed of 3 bars, the trio was chosen by means of statistical sampling (randomized blocks design) so as to ensure the representativity of the data obtained on the group. After separation by blocking (from statistics), the bars were placed for drying in laboratory environment for 10 days. The reference bars were immersed in deionized water and the others were immersed in the treatment solutions for 7 days; there were 6 bars for each container (3 for single treatment and 3 for multiple treatment). They were then removed for drying in laboratory environment for another 10 days. At the end of the 10-day drying, the expansions were measured and 3 bars from each treatment were reserved and taken to the reactivity box containing a sodium hydroxide solution NaOH ($1.00 \pm 0.01 \text{ N}$) and at $60 \pm 2^{\circ}\text{C}$ temperature. The expansions were monitored for a 60-day period. The reference bars immersed in deionized water also followed the same criterion. The 3 bars remaining in each container were returned to the treatment solution for continuing the multiple application cycles. In this case, two more saturation (7 days) and drying (10 days) cycles were conducted, intercalated between themselves. At the end of the final drying cycle, the expansions were measured and the bars were also taken to the reactivity box for continuing with the monitoring of the expansions over time. Both for the single and for the multiple treatment, which occurred before returning to the reactivity box (post-treatment), the bars also underwent a drying process for 10 days so as to allow more time for the increase in the number of chemical bonds to occur; these were made available by the presence of silanes introduced at the treatment stage. After the bars were returned to the attack, the residual expansions were monitored for 60 days more. The flowchart in Figure 3 presents the experimental program carried out for this research.

Finally, some post-treatment mortar bars were reserved for performing the complementary analyses, namely water absorption tests and analyses by scanning electron microscopy (SEM), aiming to verify the effect of the treatments on the mortar pores, and to gain a better understanding of its actuation, be it by means of physical or by microstructural observations.

3 RESULTS

3.1 Single application treatment

Figure 4 shows the results of the residual expansions in the mortar bars treated with the solutions in a single application. By analyzing the average residual expansive behavior it is observed a significant gain of expansion on the first day, after placing mortar bars in the attack box; after that period, the expansions have continued to increase gradually, but with a less intense development. For each treatment, the influence of products on the expansions can be verified to have reduced the latter in all the cases. At 60 days, the silanes can be observed to be well grouped between the reference and the lithium solutions. The lithium-based solution presented the best result regarding the reduction of expansions (0.047%), followed by the OCTEO-4 solution (0.065%); the worst result was obtained for the OCTEO-1 solution (0.084%). The reference one presented average residual expansion of 0.089%.

3.2 Multiple application treatment

Figure 5 presents the results for the multiple application. By analyzing the average residual expansions there is a similar behavior to the single treatment; a significant gain of expansions on the first day and after that, along testing, at a less intense rate. At 60 days, the results of each treatment can be observed to be well detached from one another. The OCTEO-4 treatment (0.050%) kept presenting the best result among the silane-based products, practically matching that of lithium (0.045%), the more efficient, therefore in multiple application. As regards the silanes of the methyltrimethoxysilane type, at the same age, they presented the worst results verified for the MTMS-1 (0.080%) type, as occurred for the single application. As shown in Figure 5, the bars treated with OCTEO-2 and up to a 27-day age it was verified an average residual expansion slightly smaller than the one of the bars treated with lithium. Nevertheless, after this age, there was an increment in the expansions, reaching higher levels than the expansions of the bars treated with OCTEO-4. Lithium reached 4% in the reduction of the expansions of multiple treatments in relation to the single treatment, whereas for the silane treatments and with OCTEO-4 this difference was 23%. For the best treatment with the silane-based product, MTMS-4 generated a 13% difference. For the case of MTMS-1 and MTMS-2, the differences were minimal.

3.3 Complementary analyses - Water absorption by immersion

Figure 7 shows the values found for the water absorption tests, conducted after 60 days of residual expansion test, by comparing single and multiple applications. The bars treated with the silane-based products of the MTMS type were observed to present higher absorption values than those presented by the bars treated with the silane of the OCTEO type. Concerning the dilutions studied (3% and 10%), for all the treatments, that of 10% was observed to yield the lowest average absorption values. The opposite occurred when the DMSO solvent was introduced in the treatment solutions, which caused them to yield higher average absorption values. Both for MTMS and for OCTEO, greater absorption value was obtained for lower silane dilution (3%) and with the presence of the DMSO solvent, employing the MTMS-3 and OCTEO-3 treatments for each case. Considering the bars treated by multiple applications, the ones that obtained the lowest water absorption, the graph presented also shows the average absorption values correlated with the average results of the residual expansions for each treatment. Even though it is not possible to obtain a direct correlation between the expansion percentage and absorption, for the silane treatments, the OCTEO type was verified to be the one which presented the least water absorption with the trend and the smallest residual expansions.

3.4 Complementary analyses – Scanning Electron Microscopy

The analyses by means of SEM/EDS were performed in fragments extracted from the following bars: reference, MTMS-4 and OCTEO-4 and the results showed the formation of ASR products, particularly for the reference samples, in which several pores (located in the closest portion to the surface exposed to the NaOH solution) were verified to exist, containing cracked massive gel with alkaline silica-calcium composition, as can be observed in Figure 8. Neither rosette-like crystallized products or with lacy aspect, nor the aggregate interface containing cracked gel could be observed. In all the samples analyzed, be it for the reference or treated ones, cement hydration products were identified mainly inside the pores, such as prism crystals of calcium hydroxide $\text{Ca}(\text{OH})_2$ and acicular ones of ettringite (aluminat trisulfate hydrate). The micrographs presented in Figures 9 and 10, regarding the samples treated with MTMS-4 and with OCTEO-4, respectively, illustrate the products identified as being solely from hydration, which are mostly present.

4 DISCUSSION

The results from the treatments indicated that the silane-based products tested are promising for mitigating residual expansions deriving from the alkali-aggregate reaction (ASR). The significant gain of expansions on the first testing day observed in Figures 4 and 5 can be attributed to both gel swelling by water adsorption and also to mass gain saturation by mortar water absorption (due to the previous drying cycles) along with thermal contribution [11]. It is really difficult to separate those phenomena accurately. The approach presented in [7] indicates similar behavior; samples containing the pre-existing ASR gel expand not only due to its swelling, but also by some water absorption. According to [7], considering two similar concretes cast with the same materials, but one is attacked by ASR and the other not, the one with ASR can absorb the same water, but generating different levels of expansion due to the gel sorption effect in addition to their thermal expansion. *Thus, in the present research, expansion associated only to saturation was not deducted at any stage from the global expansions.*

As regards the silane types, after the lithium-based product, the silane-based treatment of the octyltriethoxysilane type, OCTEO-4, was the one which presented the best result concerning the reduction of expansions. Nonetheless, the mitigating potential of methyltrimethoxysilane was also evidenced, once the MTMS-4 treatment was the third best among all the treatments. This behavior was observed both for single application and for multiple applications. By analyzing the influence of the types of application of the products, which were single application and multiple application, it was verified that, although for the latter the majority of the results had been more efficient for reducing expansions for the lithium as well as for the silanes, this behavior was not verified for the MTMS-1 and MTMS-2 treatments. For MTMS-1, the values are very close; their average reduction percentage differs by only 2% between the two types of applications. For the MTMS-2, a single application caused the same effect as for the multiple application. For the treatments in which the multiple application was better than the single application, when these two applications were compared, lithium obtained a 4% difference in the reduction of expansions, whereas for the best result among the silane treatments, OCTEO-4, this difference was far greater, 19%. For the best treatment with the MTMS silane-based product, MTMS-4, the difference was with 13% also large. These results can be verified in Figure 6.

Concerning the dilutions used (3 and 10%), comparing the pairs (MTMS-1 with MTMS-2; MTMS-3 with MTMS-4 and so forth) and considering the two types of application (single and multiple), it is generally verified that the 10% ones presented the best results.

As to the introduction of the DMSO-type solvent in the MTMS-3, MTMS-4, OCTEO-3 and OCTEO-4, despite the most efficient results for reducing the residual expansions having been obtained by the OCTEO-4 solutions, the introduction of the DMSO-type solvent showed to be more active in the mortar bars treated in multiple applications with the solution with methyltrimethoxysilane at 10%, whose reduction of expansions went from 25% (MTMS-2 - without DMSO) to 38% (MTMS-4 - with DMSO). The second best result of the DMSO action was obtained from the mortar bars treated in multiple applications with the solution containing methyltrimethoxysilane at 3%, whose reduction of expansions went from 14% (MTMS-1 - without DMSO) to 24% (MTMS-3 - with DMSO). In the case of OCTEO-4, the presence of DMSO had just a 4% improvement in the reduction of expansions, going from 42% (OCTEO-2 - without DMSO) to 46% (OCTEO-4 - with DMSO). The presence of DMSO did not alter the results for the solutions containing octyltriethoxysilane at 3% (OCTEO-1 and OCTEO-3). Although the introduction of the DMSO-type solvent showed to be more active in the mortar bars treated in multiple applications with the solution containing MTMS methyltrimethoxysilane at 10% than in those containing OCTEO, with the reduction of expansions going from 25% to 38%, generally the most efficient results for mitigation were those obtained with OCTEO-4 (46% reduction).

Hence, after lithium, which already has proven its efficacy for mitigating the alkali-aggregate reaction [1, 2, 4, 6], the OCTEO silane-based treatment, at a 10% dilution, with the DMSO (OCTEO-4)-type solvent, in multiple application, showed to be more efficient; The mitigating potential of methyltrimethoxysilane (MTMS-4) silane-based treatment could also be observed. The approach presented in [11] indicates that these products can even be more economically viable than lithium-based products.

Finally, by selecting the two treatments which presented the best performance in this study, that is lithium and silane OCTEO-4, and considering the multiple application at 60 days of age, the reductions in expansions were verified to be expressive, being of the order of 52% for the first and of 46% for the second, in relation to the reference, as can be observed in Figure 6. Hence, the first aspect that may have contributed to this positive result is connected to the fact that the post-treatment bars were taken back to the drying ambient for 10 days before they were once again placed under

attack. Thus, there was probably an enhancement in the stability of the chemical bonds occurring between the products and the penetration areas, consequently improving the hydrophobic structure of the surface. This occurs because during the treatment stage with the silanes, there is a chemical bond due to the condensation reaction, forming other stable products (Si-O-Si bond), which besides sealing the concrete pores, make it hydrophobic. As reported in some publications [16,19], the bonds formed are very stable, and may well resist meteorological conditions and some chemical products. However, if the bars are placed in an aggressive attack ambient soon after the treatments, the bonds formed may be broken, therefore reducing the treatment efficiency. Figure 6 allows observing the percentage of reduction in expansions, as regards the reference and for each type of treatment and application, at the end of the test (60 days).

It is worth stressing that, once the mortar bars were previously taken to expansion treatments, forming the ASR gel, the silanes action occurs both by bonding with the reactive silica still available (although part of the aggregate is less reactive owing to the gel formation), and by the interaction of the silane with the gel, modifying its structure [21]. Added to that is the fact that, even if there is gel formation, the silane-based treatment is expected to contribute to prevent expansion, seeing that the gel needs water to expand and the silane will form a water repellent layer [19]. Another consideration regarding the condition of the bars' exposition is the post-treatment period. For the single application, the bars return to the attack 10 days after the treatment and, for the multiple application, the bars only return to the reactivity box 44 days after the treatment.

Concerning the water absorption test by immersion, when the periods of pre-returning to the attack were extended due to the multiple application cycles, the treatments were verified to present better performances, that is, besides being more efficient in reducing expansions, they also decreased water absorption. For the bars treated with silanes, this behavior can be attributed mainly to the strengthening of the surface treatment layer caused by the drying and saturation cycles in the treatment solutions [16, 18, 19]. Lastly, the SEM analysis allows verifying a differentiated microstructure between the reference sample and the treated ones; the ASR products were not easily observed after the treatments (MTMS-4 and OCTEO-4), as occurred for the reference sample.

5 CONCLUSIONS

Based on the studies conducted, in the presence of the reactive aggregate selected, parameters were obtained which may help choosing a treatment for fighting the additional residual expansions of ASR so as to mitigate the harmful effects and the residual expansions. The results from the treatments tested are promising both in preventing and in mitigating the residual expansions of ASR (study presented herein) and, although lithium seems to produce greater efficiency in reducing the expansions up to the age assessed, the silane-based treatments, and especially the octyltriethoxysilane type, showed good potential and performance for such and may also be more economically viable. Let us highlight, however, that the choice for one of these treatments is connected not only to the factors studied, but also to other important aspects related to the durability of concrete structures, their mechanical properties and others; further studies are therefore necessary. Considering that the presented studies were conducted with mortar bars cast in laboratory, additional studies on concrete, as well as their validation in real structures, in localized areas, and concretes already affected by ASR, are required.

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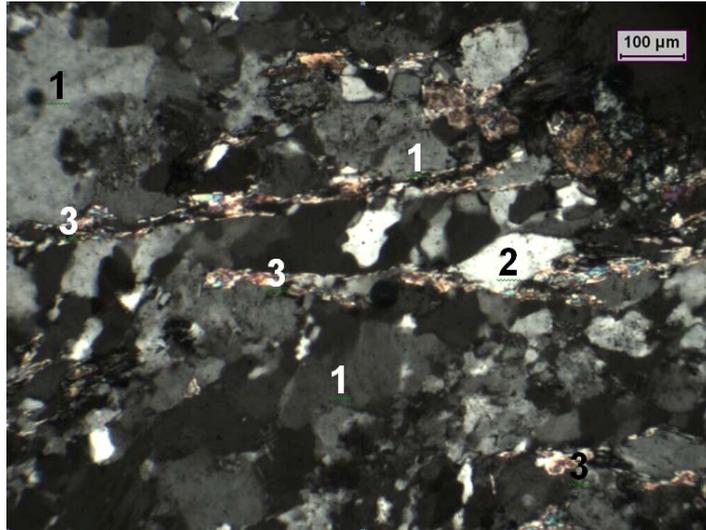


FIGURE 1: Optical micrograph (polarized light microscope) of rock: feldspar (1), strained quartz (2), mica (3).

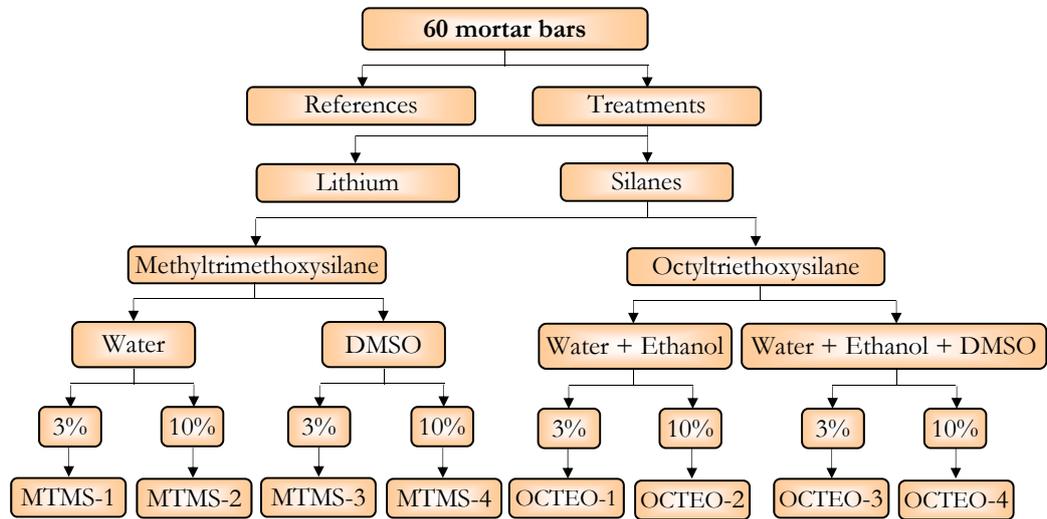


FIGURE 2: Experimental program – Treatment products.

TABLE 1: Chemical characteristics of cement.

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Total alkalis			Soluble alkalis			Loss on ignition	SO_3	Free lime	Insoluble residue
					Na_2O	K_2O	Na_2O_{eq}	Na_2O	K_2O	Na_2O_{eq}				
19.03	3.8	2.5	61.73	2.93	0.46	1.62	1.53	0.17	0.75	0.66	2.43	3.03	1.9	0.56

Note (1): $Na_2O_{eq} = 0.658 K_2O + Na_2O$

TABLE 2: Physical and mechanical properties of cement.

Cement	Specific Gravity (g/cm^3)	Blaine Fineness (cm^2/g)	Autoclave expansion (%)	Setting time (h:min)		Compressive strength (MPa)		
				initial	final	3 days	7 days	28 days
CP-V	3.13	4460	0.1	03:10	04:00	33.2	38.0	43.1

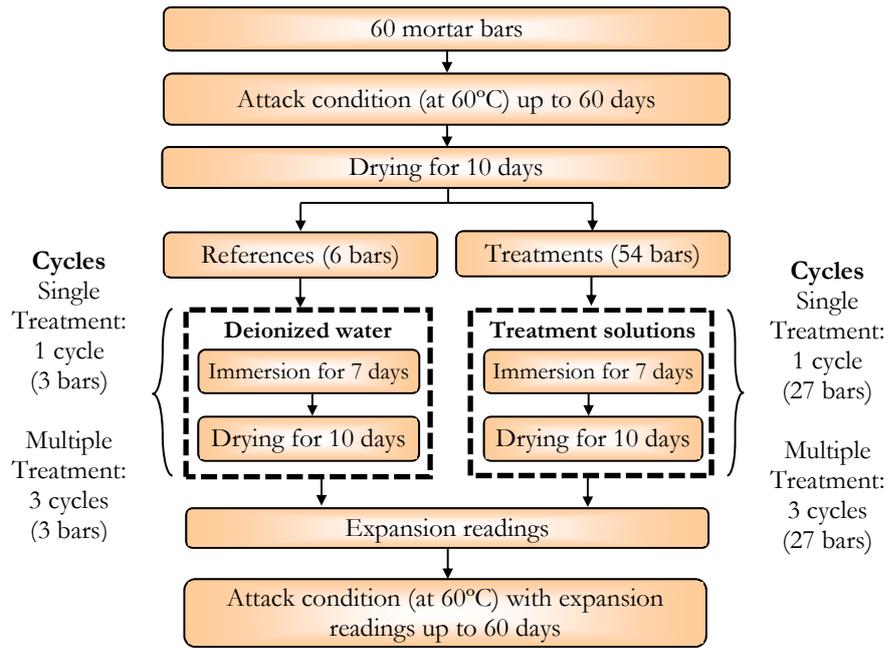


FIGURE 3: Flowchart of experimental program.

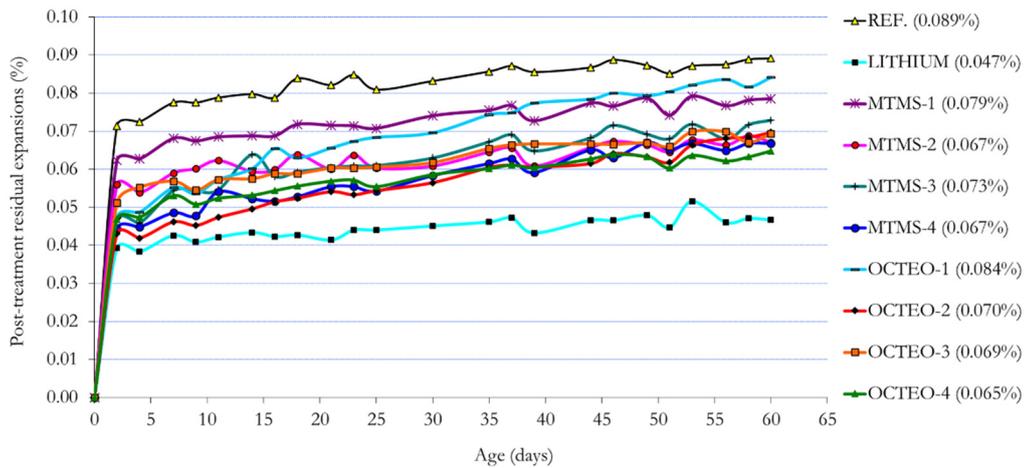


FIGURE 4: Post-treatment residual expansions – Single treatment.

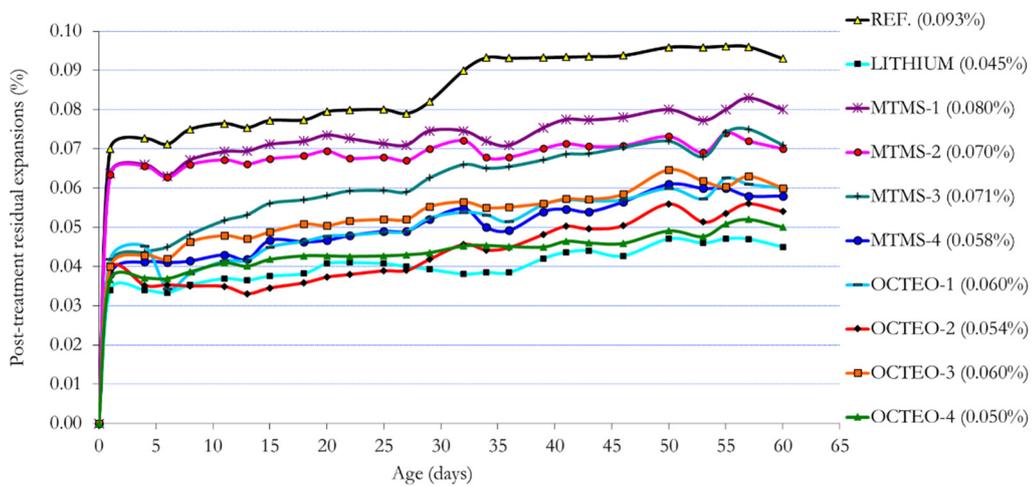


FIGURE 5: Post-treatment residual expansions – Multiple treatment.

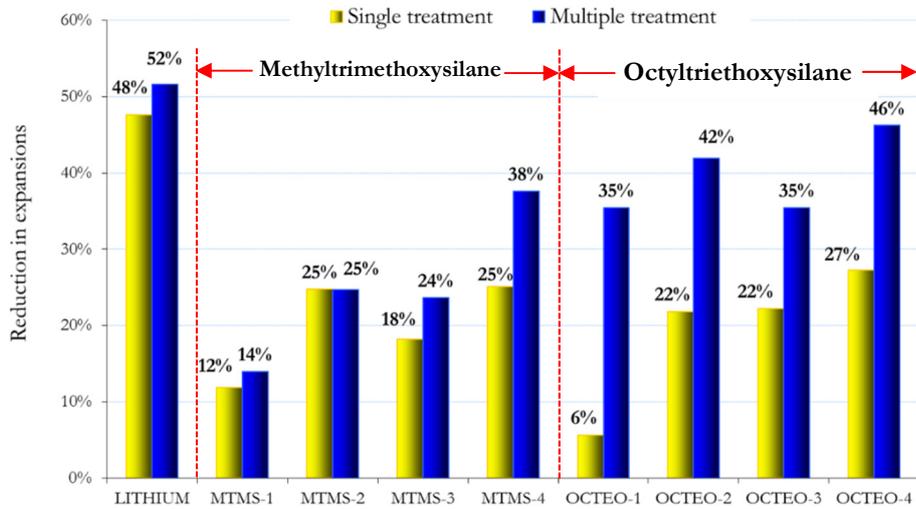


FIGURE 6: Reduction in expansions at 60 days – comparative results between single and multiple treatments.

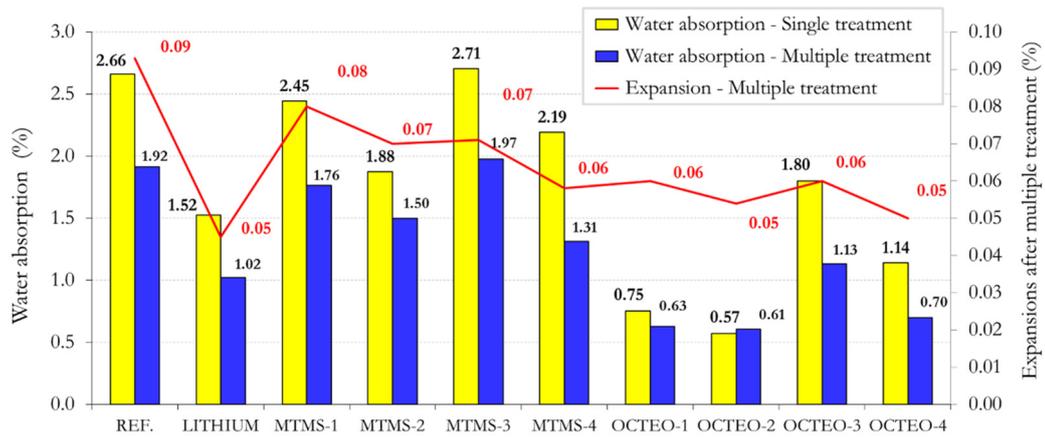


FIGURE 7: Mortar bars water absorption.

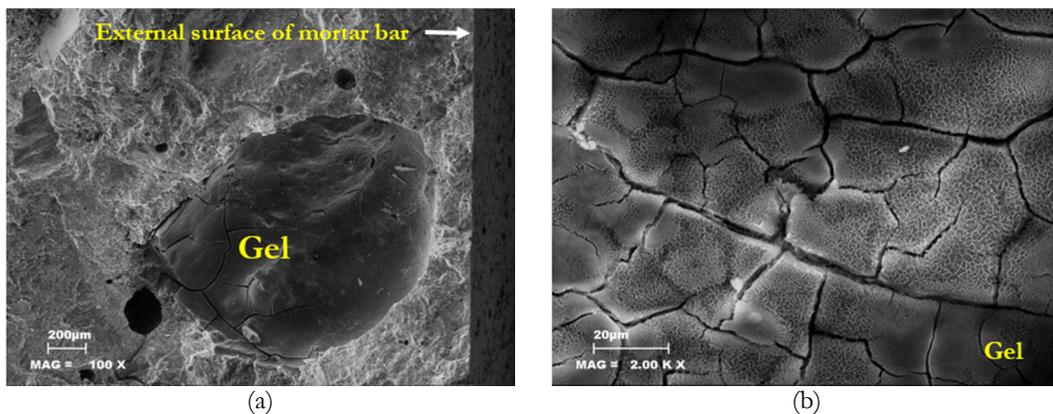


FIGURE 8: AAR products found in the reference mortar by SEM. (a) Massive cracked gel in the pore; (b) Detail of a cracked gel inside a pore.

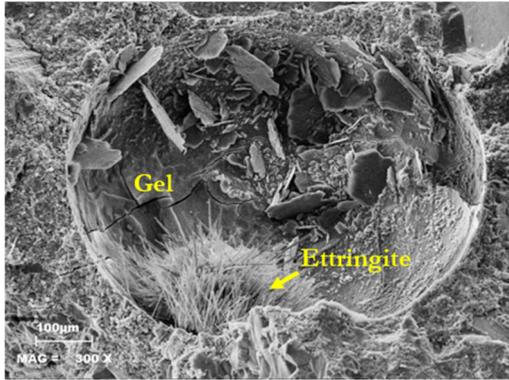


FIGURE 9: Micrograph by SEM of MTMS-4 mortar bar: Hydrated products in the pore (ettringite and portlandite) and some gel on the left.

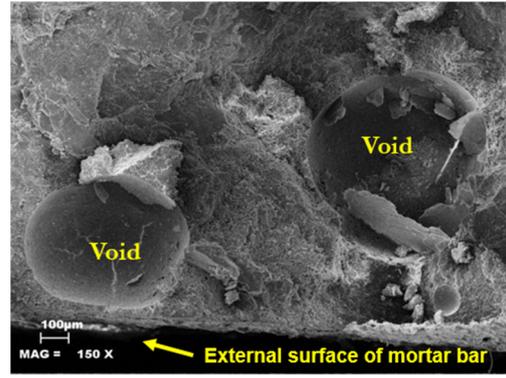


Figure 10: Scanning electron micrograph of OCTEO-4: Empty voids, with no gel.