

ASSESSMENT OF ALKALIS RELEASED BY AGGREGATES. CONTRIBUTION TO THE ALKALINITY INCREASE AND AAR DEVELOPMENT IN CONCRETE.

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Abstract. *Concrete swelling processes are one of the main degradation mechanisms of hydro concrete structures, such as dams. Included in this degradation phenomenon, the reactions that involve the aggregates when exposed to the alkali rich environment of the cement paste are probably the ones that deserve more concern worldwide. The alkali concentration of the concrete pore solution could also be modified with time due to the alkalis release from some aggregates or mineral phases present in the aggregate particles, a fact which perhaps could explain the delaying occurrence of AAR in some dams.*

This paper presents the first results of the evaluation of the alkalis released by different types of aggregates. The results obtained evidence that aggregates containing alkali minerals in their composition are susceptible to alkalis releasing in alkaline solution. This release is thought to be dependent on the mineralogy of the aggregate, the particle size and the type of solution employed. The reactivity of an aggregate, should take into account the alkali content which can release and that may contribute to the AAR.

1 INTRODUCTION

Concrete swelling processes are one of the main degradation mechanisms of hydro concrete structures, such as dams. Included in this degradation phenomenon, the attack of the alkali rich environment of the cement paste to aggregates is probably the one that deserves more concern worldwide. These alkali-aggregate reactions (AAR) are very dependent on water or humidity, which is permanently available in hydraulic structures, and originate deleterious expansive reaction products that may compromise the safety of these structures.

Despite the efforts made in recent decades to mitigate or inhibit this form of concrete degradation, there are several dams worldwide that show deterioration due to AAR. Most intriguing has been the fact that many of these dams start to show signs of degradation

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several decades after construction, or worse, when diagnosed, the expansive process progresses over several decades without showing signs of slowing down.

To develop AAR in a concrete structure it is necessary the simultaneous presence of three conditions: alkali reactive aggregates, a highly alkaline pore solution in the concrete and sufficient moisture. Prevention of AAR is therefore intended to eliminate at least one of the three aforementioned conditions. However, in the case of concrete dams some of these conditions may be difficult or impracticable to implement (e.g. reduce the access of moisture or the availability of local non-reactive aggregates). Taking into consideration these limitations and the long service life expected for these structures, the recommendations to avoid AAR in such structures prescribe normally the use of mineral additions or “supplementary cementitious materials” (SCMs), such as low-lime fly ash and other pozzolans demonstrated to be effective¹. However, some of these SMCs can supply significant amounts of alkalis to the concrete pore solution that promote an alteration on the alkali control level of AAR.

The alkali concentration of the concrete pore solution might also be modified with time due to the alkalis release from some aggregates or mineral phases present in the aggregate particles. According to Bérubé et al.² some aggregates can supply over $3 \text{ kg/m}^3 \text{ Na}_2\text{O}_e$, which is the alkali level normally admitted to AAR control, a fact which perhaps could explain the delaying occurrence of AAR in some dams. This issue constitutes an important development in the AAR prevention but unfortunately there is presently no consensus on how to test for releasable alkalis in aggregates. To overcome this limitation, it is under development by the RILEM Committee TC AAA a set of tests to establish a general procedure for the assessment of the alkali content released for a given aggregate. This paper presents the first results of the evaluation of the alkalis released by different types of aggregates, including the influence of particle size, and type of extraction solution.

2 MATERIALS AND METHODS

2.1 Selected aggregates and samples preparation

Five Portuguese granitic aggregates with different mineralogy were selected (Figure 1), including two which were used in a bridge and in a dam suffering AAR; a third was recently used in the construction of a new dam; the remaining two aggregates are widely used in several structures in northern part of Portugal. To keep the confidentiality of the quarries locations, aggregates have been codified in accordance to the nomenclature established by the IMPROVE project as GR1, GR2, GR3, GR17 and GR26.

The aggregates were previously characterized by petrographic analysis and chemical analysis performed to determine the total and soluble in water alkali content. Subsequently, the releasable alkali from granites was evaluated over time.

For petrographic analysis, hand samples of different granitic aggregates were selected for production of thin-section with a thickness of $30 \mu\text{m}$. The chemical analysis was performed on a representative sample of the crushed aggregate with a particle size $<106 \mu\text{m}$. In the test for evaluating the alkali release by aggregate, samples were crushed into different grain size fractions, namely, 0-2 mm, 4.75-9.5mm, 12.5-20mm and a fraction $<4.75\text{mm}$, resulting from the mixture of six grain size fractions in different percentages: $<0.15\text{mm} - 10\%$; $0.15-0.30\text{mm} - 15\%$; $0.30-0.60\text{mm} - 25\%$; $0.60-1.18\text{mm} - 20\%$; $1.18-2.36\text{mm} - 20\%$; $2.36-4.75\text{mm} - 10\%$. These four grain size fractions were respectively designated by F1, F3, F5 and M.

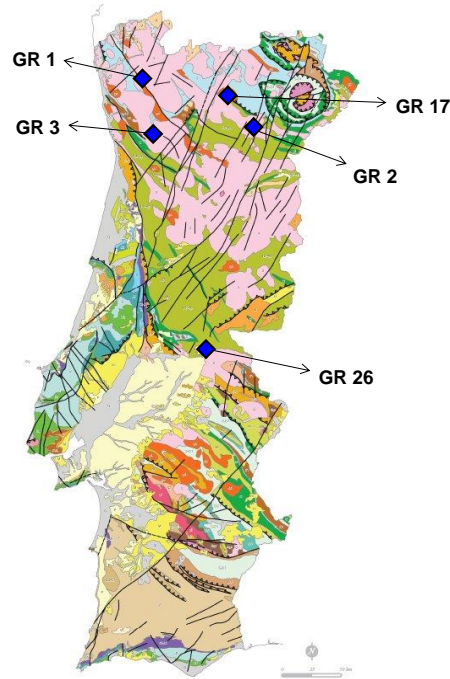


Figure 1: Localization and nomenclature of the granitic aggregates used in this study.

2.2 Test methods

2.2.1 Petrographic characterization of the aggregates

Petrographic characterization was performed by analysis of thin-sections under the a Nikon Eclipse E400 POL polarizing microscope, coupled with automatic counterpoint PELCON (version 01) and a camera Zeiss Axio Cam, available on the University of Porto Geology Center (CGUP). The assessment of reactivity to alkalis was based on local experience with granitic aggregates, namely on the dimensions of the quartz crystals (presence of quartz crystals $<100\ \mu\text{m}$ = microcrystalline quartz³ and according to the classes established by RILEM AAR-1.1³ and by the Portuguese specification LNEC E 461⁴. Myrmekitic quartz was included in the microcrystalline quartz group and considered as a potentially reactive form of silica⁵.

2.2.2 Total and soluble alkalis of aggregates

The determination of total alkali content was performed at Activation Laboratories (ACTLABS), in Canada, by $\text{LiBO}_2/\text{Li}_2\text{B}_4\text{O}_7$ fusion – inductively coupled plasma (ICP).

The analysis of the water-soluble alkali content of aggregates was based on Portuguese standard NP 1382⁶ which consists in an attack with hot water of then sample ground to pass the $106\ \mu\text{m}$ sieve. Soluble alkali was subsequently analyzed by atomic absorption spectroscopy (AAS) for determination of sodium and potassium contents. Analyzes were performed using a spectrophotometer “Shimadzu AA-6300”.

2.2.3 Alkalis released by aggregates

The method used to evaluate alkali content released by aggregates aims at the extraction of Na^+ e K^+ ions in alkaline solutions simulating the interstitial pore solution of concrete⁷. Therefore, an amount of representative aggregate sample was immersed in a

specific volume of alkaline solution (Table 1) at a ratio of 1:4 aggregate/solution. All sample materials were placed into hermetically sealed polyethylene bottles, and conditioned in a chamber at 38° C and RH > 95% (test conditions of RILEM AAR-3⁸ test-method). Periodically, the test containers were gently rolled back for about 10 seconds, and in predefined time intervals (e.g.: 7, 14, 28, 56 days 3, 6, 9 and 12 months) a sample was taken from the test solution (25 mL), and this volume replaced with a solution of same concentration (blank solution) maintained under the same test conditions, in order to maintain the 1:4 ratio between the aggregate and the solution.

Different alkaline solutions were tested: saturated Ca(OH)₂, NaOH and KOH in two different concentrations (1M and 0.7M) and with, or without, addition of saturated Ca(OH)₂. A total of 7 alkaline solutions were applied, as shown in Table 1, which is also part of the test conditions used, in particular the particle size of aggregates and alkaline solutions selected. After filtration and acidification, the determination of alkali released content (Na⁺ and K⁺) of the extraction solutions was performed by atomic absorption spectrometry (AAS).

Particle size \ Solution	Fraction "F1" (0–2 mm)	Fraction "F3" (4.75–9.5 mm)	Fraction "F5" (12.5–20 mm)	Fraction "M" (0–4.75 mm)
Ca(OH) ₂ saturated	GR1	GR1	GR1	-
	GR2	GR2	GR2	-
	GR3	GR3	GR3	-
	-	GR17	-	-
	-	GR26	-	-
NaOH 1M + Ca(OH) ₂ sat.	GR1	GR1	GR1	-
	GR2	GR2	GR2	-
	GR3	GR3	GR3	-
	-	GR17	-	GR17
	-	GR26	-	GR26
KOH 1M + Ca(OH) ₂ sat.	GR1	GR1	GR1	-
	GR2	GR2	GR2	-
	GR3	GR3	GR3	-
	-	GR17	-	GR17
	-	GR26	-	GR26
NaOH 0.7M + Ca(OH) ₂ sat.	-	-	-	GR26
KOH 0.7M + Ca(OH) ₂ sat.	-	-	-	GR26
NaOH 0.7M	-	-	-	GR26
KOH 0.7M	-	-	-	GR26

Table 1: Test conditions used (particle size of aggregates and type of alkaline solution) in each aggregate studied⁹.

3 RESULTS AND DISCUSSION

3.1 Petrographic characterization of aggregates

The petrographic characterization of aggregates allows the identification of potentially reactive forms of silica and of minerals that can contribute with alkalis. A brief petrographic description of the aggregates is shown in Table 2. Microphotographs are also presented (Figure 2) that illustrates some textural and mineralogical aspects of the studied aggregates.

Aggregate	Brief petrographic description	Undulatory extinction angle (°) of quartz crystals*	Potentially reactive silica forms (< 100 µm) (% vol)*
GR1	Medium-grained granite with hypidiomorphic porphyritic texture. Quartz, K-feldspar and plagioclase are the essential minerals. Biotite, muscovite, zircon, apatite, opaque minerals, chlorite, rutile, leucoxene, monazite, fluorite, fibrolite and epidote are the accessory minerals. Presents frequent microfractures, especially in quartz crystals and plagioclase, with microcrystalline muscovite and oxides.	16.0° Moderate	Globular and micrographic quartz; myrmekites 2.4% - Class II
GR2	Medium to coarse-grained granite with hypidiomorphic porphyritic texture and evidence of deformation. Quartz, K-feldspar and plagioclase are the essential minerals. Biotite, muscovite, tourmaline, zircon, apatite, opaque minerals, leucoxene, chlorite and rutile are the accessory minerals.	20.0° Moderate	Tectonized, microcrystalline and micrographic quartz; myrmekites 21.7% - Class II
GR3	Medium to coarse-grained granite with hypidiomorphic texture. Quartz, K-feldspar and plagioclase are the essential minerals. Muscovite, biotite, zircon, apatite, andalusite, opaque minerals, chlorite, rutile and leucoxene are the accessory minerals. Abundant intergranular microcracks filled with oxides and microcrystalline muscovite.	18.0° Moderate	Microcrystalline and micrographic quartz; myrmekites 1.0% - Class I
GR17	Fine to medium-grained granite with hypidiomorphic texture. Quartz, K-feldspar and plagioclase are the essential minerals. Biotite, muscovite, apatite, zircon, rutile, opaque minerals, chlorite and leucoxene are the accessory minerals.	15.0° Moderate	Microcrystalline and micrographic quartz; myrmekites 1.5% - Class I
GR26	Medium to coarse-grained granite with hypidiomorphic texture. Quartz, K-feldspar and plagioclase are the essential minerals. Biotite, muscovite, apatite, zircon, tourmaline, andalusite and chlorite are the accessory minerals.	11.0° Weak	Microcrystalline and micrographic quartz 2.0% - Class II

*Note: Undulatory extinction: Weak – [0° - 14°]; Moderate – [15° - 24°]; Strong – [25° - 35°];
Reactivity class: Class I - Potentially reactive silica < 2%; Class II - Potentially reactive silica > 2% (RILEM AAR1.1, 2013³; LNEC E461, 2007⁴)

Table 2: Results of petrographic characterization of the studied granitic aggregates⁵.

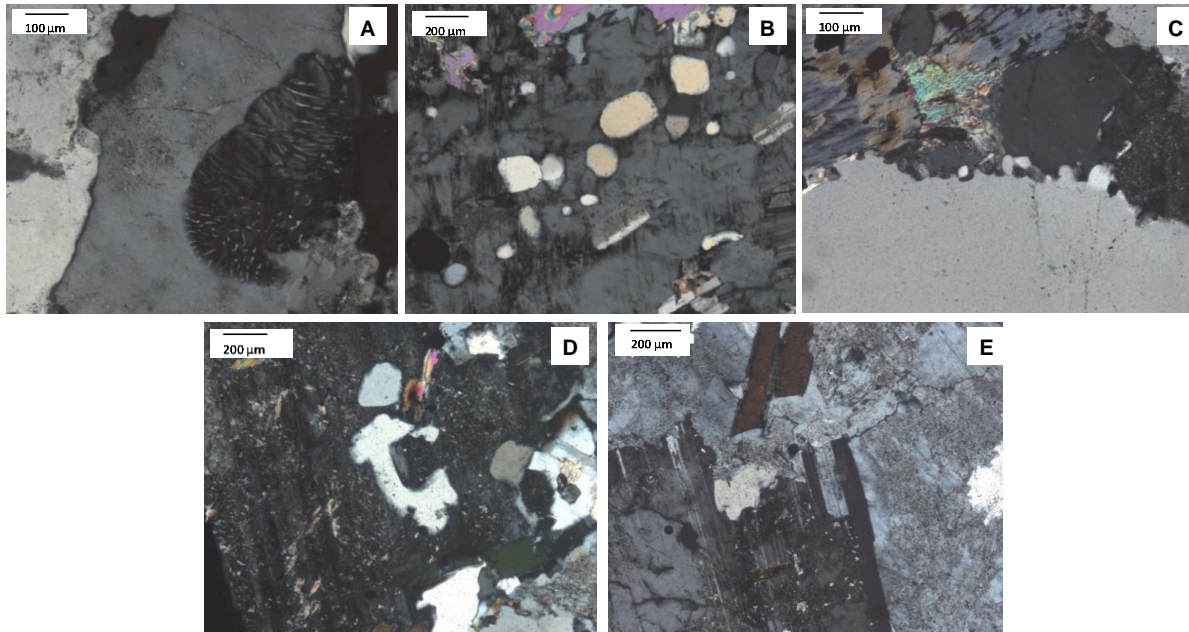


Figure 2: Microphotographs of the investigated aggregates in cross-polarized light (XPL): a) GR1: Myrmekites in plagioclase crystal neighbouring microcline; b) GR2: Microcrystalline quartz in microcline megacryst; c) GR3: Reaction texture between quartz and K-feldspar; d) GR17: Micrographic quartz in an altered plagioclase; e) GR26: K-feldspars altered, with more intense alteration in core of plagioclase crystals; fractures in the rock are visible⁵.

3.2 Total and soluble alkalis of aggregates

Table 3 shows the results obtained for the major elements where it can be seen that all granites have a SiO₂ content greater than 65%, and Al₂O₃ content similar and of around 14%. With respect to alkalis, it is found that the Na₂O content (between 2.57% to GR17 and 3.47% to GR26) is in all granites lowest than K₂O content (between 4.43% to GR2 and 5.56% for GR 17).

To complete the characterization of the aggregates the content of water-soluble alkali was further determined (Table 4), expressed as a percentage of Na₂O_e, which is obtained through the contents of Na⁺ and K⁺ after conversion to their respective oxides by the following relationship:

$$\text{Na}_2\text{O}_e (\%) = \text{Na}_2\text{O} (\%) + 0,658 \times \text{K}_2\text{O} (\%)$$

	GR1	GR2	GR3	GR17	GR26
SiO ₂ (%)	67.05	72.76	71.17	70.92	71.70
Al ₂ O ₃ (%)	14.87	14.13	14.37	14.10	14.72
Fe ₂ O ₃ (%)	3.74	1.58	1.81	2.92	2.40
MnO (%)	0.05	0.03	0.04	0.04	0.04
MgO (%)	1.18	0.48	0.56	0.51	0.56
CaO (%)	1.90	0.57	0.90	1.04	0.76
Na ₂ O (%)	2.71	2.81	2.88	2.57	3.47
K ₂ O (%)	5.09	4.43	4.80	5.56	4.84
TiO ₂ (%)	0.74	0.21	0.25	0.77	0.29
P ₂ O ₅ (%)	0.36	0.33	0.29	0.64	0.32
LOI (%)	0.88	1.63	1.57	1.20	0.80

Table 3: Chemical analysis (major elements) of the aggregates (weight %) ⁵.

Through the analysis of the results it can be seen that the Na_2O_e values range from 0.031% for GR17 to 0.041% for GR1. It also appears that for most granites, except GR26, the K_2O content (between 0.019% for GR26 to 0.034% for GR 1) is greater than the content of Na_2O (between 0.018% for GR1, GR3 and GR17 to 0.023% for GR26).

Comparing the results obtained for the water-soluble alkalis with the total alkali values, it appears that only $\approx 0.6\%$ (% total on average) of the alkalis of granitic aggregates is soluble in water.

Aggregate	Total alkalis ^a			Water-soluble alkalis ^b				
	Na_2O (%)	K_2O (%)	Na_2O_e (%)	Na_2O (%)	K_2O (%)	Na_2O_e (%)	(% total) ^c	(kg/m^3) ^d
GR1	2.71	5.09	6.06	0.018	0.034	0.041	0.67	0.75
GR2	2.81	4.43	5.72	0.020	0.024	0.036	0.64	0.67
GR3	2.88	4.80	6.04	0.018	0.029	0.038	0.62	0.70
GR17	2.57	5.56	6.23	0.018	0.021	0.031	0.50	0.58
GR26	3.47	4.84	6.65	0.023	0.019	0.036	0.53	0.66

^a Determined by chemical analysis by the FUS-ICP method; ^b Water-soluble alkalis according to NP 1382, 1976; ^c Percentage of total water-soluble alkalis (Na_2O_e) in the aggregate; ^d Based on a total concrete aggregate content of 1850 kg/m^3 .

Table 4: Total and water-soluble alkalis of the investigated granitic aggregates⁹.

3.3 Alkalis released by aggregates

The results of alkalis released by granitic aggregates obtained are presented in summarized form in Table 5⁹.

3.3.1 Influence of particle size

Concerning the influence of particle size of aggregates, it is found that for granites GR1, GR2 and GR3 the smaller fraction (0-2 mm) shows greater alkalis release in the alkaline solution. This is due to the increase in specific surface that is produced in the crushing process, which means that the dissolution reactions occur faster by increasing the area available for reaction^{10,11,12}. However, despite the higher extraction occurs in the fraction 0-2 mm, it may be more realistic to test aggregates with a particle size distribution closer to that used in the concrete mixes of. The same is observed for GR26, which provides greater alkalis release for the sample with 0-4.75 mm particle size, in comparison to the fraction 4.75-9.5 mm. Granite GR17 is the only one that does not show the same behavior.

Another important observation is that all the aggregates have a higher extraction of Na than K in the alkaline tested solutions, although this difference is less significant in saturated $\text{Ca}(\text{OH})_2$.

Taking into account the petrographic analysis (Table 2) it can be seen that all granites have plagioclase and K-feldspar as main minerals.

In summary, the obtained results up to 180 days of test show that all aggregates have a significant alkali release, some of which show a trend of stabilization, or even reduction, with age, which can be related to the formation of ASR precipitate products in the test solutions. This can happen especially in long duration tests^{13,14}.

		Alkalis released by aggregates									
Aggregate	Size (mm)	Saturated Ca(OH) ₂					NaOH or KOH 1M + saturated Ca(OH) ₂				
		Na ₂ O (%)	K ₂ O (%)	Na ₂ O _e (%)	(% total) ^a	(kg/m ³) ^b	Na ₂ O (%)	K ₂ O (%)	Na ₂ O _e (%)	(% total) ^a	(kg/m ³) ^b
GR1	0-2	0.007	0.004	0.010	0.16	0.18	0.077	0.011	0.084	1.39	1.56
	4.75-9.5	0.003	0.004	0.006	0.11	0.12	0.080	0.006	0.083	1.38	1.54
	12.5-20	0.003	0.001	0.003	0.05	0.06	0.054	0.004	0.056	0.93	1.04
GR2	0-2	0.012	0.008	0.017	0.29	0.31	0.085	0.017	0.096	1.67	1.77
	4.75-9.5	0.003	0.003	0.005	0.09	0.10	0.055	0.009	0.061	1.07	1.13
	12.5-20	0.003	0.001	0.004	0.07	0.07	0.049	0.004	0.052	0.90	0.96
GR3	0-2	0.010	0.006	0.014	0.23	0.26	0.097	0.014	0.106	1.76	1.96
	4.75-9.5	0.003	0.002	0.004	0.07	0.08	0.061	0.006	0.065	1.08	1.20
	12.5-20	0.004	0.001	0.004	0.07	0.07	0.062	0.005	0.065	1.08	1.21
GR17	0-2	-	-	-	-	-	-	-	-	-	-
	4.75-9.5	0.005	0.002	0.007	0.10	0.12	0.073	0.008	0.079	1.26	1.45
	12.5-20	-	-	-	-	-	-	-	-	-	-
	0-4.75	-	-	-	-	-	0.055	0.008	0.061	0.97	1.12
GR26	0-2	-	-	-	-	-	-	-	-	-	-
	4.75-9.5	0.003	0.002	0.005	0.07	0.08	0.036	0.007	0.040	0.61	0.75
	12.5-20	-	-	-	-	-	-	-	-	-	-
	0-4.75	-	-	-	-	-	0.037	0.015	0.047	0.70	0.86
		NaOH or KOH 0.7M + saturated Ca(OH) ₂									
		Na ₂ O (%)	K ₂ O (%)	Na ₂ O _e (%)	(% total) ^a	(kg/m ³) ^b					
GR26	0-4.75	-	-	-	-	-	0.126	0.012	0.134	2.01	2.48
		NaOH or KOH 0.7M									
		Na ₂ O (%)	K ₂ O (%)	Na ₂ O _e (%)	(% total) ^a	(kg/m ³) ^b					
GR26	0-4.75	-	-	-	-	-	0.121	0.011	0.128	1.92	2.36

^a Percentage of total alkalis (Na₂O_e) in the aggregate; ^b Based on a total concrete aggregate content of 1850 kg/m³.

Table 5: Alkalis released by aggregates at 180 days of testing⁹.

3.3.2 Influence of mineralogy

The influence of mineralogy was evaluated by testing the F3 fraction in different solutions (Table 5). Granite GR17 is the aggregate showing higher alkalis release in Ca(OH)₂ solution, while GR1 has the higher release in KOH or in NaOH 1M + Ca(OH)₂ saturated. However, the differences in the amount of alkali released are very low, being the differences more evident in the Na content. Taking into account the petrographic analysis (Table 2) it was observed that all granites have plagioclase as sodium mineral supplier and K-feldspar, biotite and muscovite as potassium mineral suppliers. GR17 and GR26 granites were further tested in M fraction, being apparent that GR17 presents greater amount of Na released, while the GR26 has a higher amount of K released.

3.3.3 Influence of type of extraction solution

The influence of concentration of the extraction solution was evaluated by immersing the GR26 granite in KOH and NaOH solutions at two different concentrations, with or without $\text{Ca}(\text{OH})_2$ addition. By analyzing the Na release in KOH solutions it can be seen that the lower concentration solution (0.7 M) is more extractive, however it is not possible to evaluate the benefits effect of $\text{Ca}(\text{OH})_2$ in the extraction, since the results obtained are similar (2.48 kg/m^3 and 2.36 kg/m^3). When the concentration of KOH solution is too high or when there is no $\text{Ca}(\text{OH})_2$ in excess, the alkalis release happens by K-Na ion exchange, producing a deficit of K ions in the solution and an increase in the Na ions content¹⁴.

In the case of NaOH solutions, the most concentrated solution (1M NaOH + $\text{Ca}(\text{OH})_2$) was the more efficient. In this case, there was also a Na-K ion exchange causing the replacement of K by Na in the aggregates. Also here, the role of $\text{Ca}(\text{OH})_2$ presence is not clarifying, however it seems to be able to come out beneficial for a higher extraction.

Anyway, the two solutions mentioned above have an higher alkali extraction than the saturated $\text{Ca}(\text{OH})_2$ solution (e.g., 0.75 kg/m^3 vs 0.08 kg/m^3 in the F3 fraction of GR26).

4 CONCLUSIONS

The work carried out evidences that aggregates containing alkali minerals in their composition are susceptible to release alkalis in alkaline environment, being this release dependent on the mineralogy and particle size of aggregate, and also on the type of solution employed. It has been found that reducing the particle size of the aggregates leads to an increase of alkalis release to the solution, which is higher in the alkaline solutions of KOH and NaOH.

For the five granites tested it has been found a higher Na extraction, which is attributed to the higher instability of the sodium mineral constituents (e.g. plagioclase), in comparison to K-feldspars or micas, in highly alkaline conditions. Moreover, it is noteworthy that feldspars promote the release of minority ions, which is associated to the instability of crystalline network. The migration of minority alkaline ions is more evidence with sodium ion, due to that its ionic radius (0.95 \AA) permits its substitution by Ca (with ionic radius 0.99 \AA), while the ionic radius of potassium is much higher (1.33 \AA)¹³. This fact, associated with other possible risk factors for AAR, could indicate that aggregates containing Na minerals will be those at higher susceptibility in terms of AAR. Regarding the alkaline concentration effect, the KOH 0.7M solution was the most extractive, while for NaOH solution the 1M concentration was more efficient.

In summary, the reactivity of an aggregate shall not take into account only the reactive silica constituents, but also the alkali content which can be released. This fact may be very important for slowly reactive aggregates that did not show the presence of potentially reactive silica minerals in its constitution, and can justify the long-term reactivity presented by some aggregates in concrete structures.

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