# STUDY ON THE FACTORS AFFECTING ALKALIS RELEASE FROM AGGREGATES INTO ASR

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#### Abstract

Alkaline minerals, like feldspars and micas, present in aggregates can release alkalis with time for concrete pore solution, being a contributor factor for the delaying ASR occurrence in some concrete structures (e.g. dams). Since there is presently no consensus on how to test for releasable alkalis in aggregates, a research program has been conducted to evaluate the factors that can affect the alkalis release in laboratory test conditions.

This paper presents the results after 455 days evaluation of alkalis released by igneous aggregates with different grain sizes, in different alkaline solutions (Ca(OH)<sub>2</sub>, NaOH and KOH), in two different concentrations (1M and 0.7M) -with and without addition of saturated Ca(OH)<sub>2</sub>.

The results obtained show 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 with saturated Ca(OH)<sub>2</sub>. The sodium extraction is higher in 0.7M KOH, while potassium is higher in 1M NaOH.

Keywords: Concrete, ASR, Alkalis release, Prevention, Mitigation

# 1 INTRODUCTION

Previous investigations [1,2,3,4,5] have shown that the alkali content of the concrete pore solution increases with time due to the alkalis release by aggregates, and suggests that this may result in increased risk of alkali-silica reaction (ASR). Furthermore, it has been found that several concrete structures start to show signs of degradation several decades after their construction, and also that the expansive process can progress over time without showing signs of slowing down [6].

Alkali minerals, like feldspars and micas, are potential sources of alkalis and are very common minerals in many rocks, like granites, gneisses, greywackes, etc. According to Bérubé et al. [7] some igneous aggregates can supply over 3 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>e</sub>, which is the alkali level normally admitted to avoid AAR, a fact that could explain the delay in the occurrence of ASR in some Canadian dams.

This issue constitutes an important development in the ASR 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 results from an ongoing project about the evaluation of the alkalis released by different granitic aggregates, including the study of the influence of aggregate particle size, the type of extraction solution and the aggregate mineralogy.

### 2 MATERIALS AND METHODS

# 2.1 Selected aggregates and samples preparation

In this paper six Portuguese granitic aggregates were selected which differ slightly in terms of their mineralogy and more in texture. To keep the confidentiality of the granitic quarries origins, these aggregates have been codified as GR1, GR2, GR3, GR17, GR18 and GR26.

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The aggregates were previously characterized by chemical and petrographic analysis. The chemical analysis was performed on a representative sample of aggregate, crushed and grinded to pass a 106  $\mu$ m sieve. For petrographic analysis, hand samples of different granitic aggregates were selected for production of thin-sections with a thickness of 30  $\mu$ m.

For evaluating the alkalis released by aggregates, the granitic samples were crushed and screened into different grain sizes, namely 0-2 mm (designated as "F1"), 4.75-9.5 mm (designated as "F3"), 12.5-20 mm (designated as "F5") and < 4.75 mm (designated as "M"). This M size is obtained from the mixture of six grain size fractions in different percentages: <0.15 mm – 10%; 0.15-0.30 mm – 15%; 0.30-0.60 mm – 25%; 0.60-1.18 mm – 20%; 1.18-2.36 mm – 20%; 2.36-4.75 mm – 10%.

# 2.2 Methods for assessment and analysis

Chemical analysis of aggregates (ICP)

The chemical analyses of main oxides were performed by LiBO<sub>2</sub>/Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> fusion at inductively coupled plasma (ICP).

#### Petrographic analysis of aggregates

Petrographic characterization was performed by analysis of thin-sections under polarizing microscope, coupled with automatic point-counter. This method enables to quantify the relative volume of potentially reactive components to alkalis, as well as the mineral content which may provide the alkalis for the system (e.g. plagioclase, K-feldspars, muscovite and biotite).

The assessment of reactivity to alkalis was based on local experience with granitic aggregates, namely on the dimensions of the quartz crystals (<100  $\mu$ m = microcrystalline quartz) and according to the classes established by RILEM AAR-1.1 [8] and by the Portuguese specification LNEC E 461 [9]. Myrmekitic quartz was included in the microcrystalline quartz group and considered as a potentially reactive form of silica [10].

#### Alkalis released by aggregates

The method used to evaluate alkali content released by aggregates aims at the extraction of Na<sup>+</sup> e K<sup>+</sup> ions in alkaline solutions simulating the interstitial pore solution of concrete [11]. 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  $38 \pm 2^{\circ}$  C (temperature test condition of RILEM AAR-3 test-method [12]). 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 the same concentration (blank solution) maintained under the same test conditions, in order to maintain the 1:4 ratio between the aggregate and the solution. Once at one year a plateau was not reached, subsequent measurements were carried out with periodicity of 3 months.

Different alkaline solutions were tested: lime-saturated solution with an excess of solid  $Ca(OH)_2$  (for the measurement of K and Na supplies), NaOH (for the measurement of K supply) and KOH (for the measurement of Na supply) 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, where the test conditions used are also presented, in particular the particle size of aggregates and the alkaline solutions selected [13]. After filtration and acidification, the determination of alkali released content (Na<sup>+</sup> and K<sup>+</sup>) of the extraction solutions was performed by atomic absorption spectroscopy (AAS).

#### 3 RESULTS

#### 3.1 Chemical analysis of aggregates

Table 2 shows the results obtained for the main oxides, where it can be seen that all granites have a SiO<sub>2</sub> content greater than 65%, and Al<sub>2</sub>O<sub>3</sub> content similar and of about 14%. With respect to alkalis, it is found that the Na<sub>2</sub>O content (between 2.57% and 3.47%, in average of 2.91%) is in all granites lowest than K<sub>2</sub>O content (between 4.43% and 5.56%, in average of 4.87%). In addition, the % of Na<sub>2</sub>O<sub>c</sub> was calculated, which is obtained by the equation:

 $Na_2O_e$  (%) =  $Na_2O$  (%) + 0.658 ×  $K_2O$  (%)

# 3.2 Petrographic analysis of aggregates

The main information obtained in the petrographic characterization of the aggregates, namely the identification of potentially reactive forms of silica as well as of the minerals that can contribute with alkalis is shown in Table 3

The petrographic analysis of aggregates allows the classification of the aggregates GR1, GR2, GR18 and GR26 as alkali-reactivity uncertain (Class II), while GR3 and GR17 are very unlikely to be alkali-reactive (Class I) [8].

With regard to the constituent minerals, it is possible to verify that the GR1, GR3 and GR26 have the highest content of plagioclase (mineral supplier of Na), while the GR1, GR2 and GR26 show the larger K-feldspar content (mineral supplier of K). Other minerals that can contribute with K, are muscovite and biotite, which have the higher content in GR 17 and GR1, respectively. However, these minerals are less alterable than feldspars.

#### 3.3 Alkalis released by aggregates

The results of alkalis released by the aggregates tested are presented in Figures 1 to 4, and summarized in Table 4.

# Influence of particle size

The results obtained, namely with granites GR1, GR2 and GR3 (Figure 1), show that the reduction in the particle size of the aggregates enhances its alkalis release.

However, despite the higher extraction obtained in the fraction 0-2 mm, it may be more realistic to test aggregates with a particle size distribution closer to that used in mortar or concrete mixes. The comparison of the results obtained with granites GR17, GR18 and GR26 in "M" and F3 fractions (Table 4) confirms the previous conclusion.

Another important observation is that all the tested aggregates have a higher extraction of Na than K, independently of the alkaline solution employed, although this difference is less significant in lime-saturated solution.

#### Influence of type of extraction solution and the presence of calcium hydroxide

The influence of the type of extraction solution was evaluated using F3 fraction and by immersing all aggregates in different alkaline solutions (Table 1).

From Figure 2 it can be seen that  $KOH+Ca(OH)_2$  sat. or  $NaOH+Ca(OH)_2$  sat. solutions extract more alkalis than lime-saturated solution. This result was confirmed in all tested aggregates (Table 4).

The addition of lime-saturated solution to the KOH or NaOH solutions (Figure 3) seems to contribute to increase the alkali extraction, respectively the sodium and potassium ions. In terms of the effect of the concentration of the alkaline solution, the results obtained are not unanimous (Figure 3), i.e., the Na release in KOH solutions is higher with the lower alkaline solution (0.7 M), while in the case of K release the most concentrated solution of NaOH (1M) was the more efficient. Anyway, a greater extraction of Na than K is also visible in all extraction solutions tested.

#### Influence of mineralogy

In order to evaluate if the mineral composition of the granitic aggregates could influence the alkalis release, the granites were tested using the F3 fraction in KOH 1M +Ca(OH)<sub>2</sub> sat. and NaOH 1M+Ca(OH)<sub>2</sub> sat. solutions (Figure 4).

The results obtained show again the same trend, i.e., higher extraction of Na than K. However, in terms of Na release at 455 days two distinct groups of data are observed: one where the extraction is about 0.05% (GR17, GR18 and GR26) and the other about 0.13% (GR1, GR2 and GR3). Concerning the amount of K released, the results obtained are very similar (about 0.01%) between all tested granitic aggregates.

# 4 DISCUSSION

The results obtained confirm previous investigations about the liberation of alkalis by certain minerals present in aggregates in alkaline media. This study shows also that the reduction of the particle size of aggregates enhances the release of alkalis. This behavior can be explained by the increase in the specific surface that is produced in the crushing process, which means that the dissolution reactions occur faster by increasing the area available for reaction [14,15,16].

All granitic aggregates tested are still releasing alkalis after 455 days independently of the alkaline solution or concentration used. However, the alkaline solution type employed affects the

magnitude of alkalis extracted, being the lime-saturated solution much less effective (average of 0.015% as Na<sub>2</sub>O<sub>e</sub>) than the sodium or potassium solutions (average of 0.123% as Na<sub>2</sub>O<sub>e</sub>). This behavior can be related to K-Na ion exchange processes, and also to the possible formation of precipitates of calcium–aluminum–silicates (CASH), calcium–silicates (CSH) and calcium–aluminates (CAH) which likely entrap some alkalis [17].

Regarding the alkaline concentration effect, the KOH 0.7M solution was the most extractive medium for sodium, while for potassium the NaOH 1.0M concentration was more efficient, both with addition of Ca(OH)<sub>2</sub>. It will be necessary to wait for stabilization of the release of alkalis [18,19] to better assess this effect.

For the six granites tested higher Na extraction has been found, 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. This behavior can be related to the lowest ionic radius of sodium (0.95Å), in comparison to the ionic radius of potassium which is much higher (1.33Å), which facilitates its substitution by calcium ions (with ionic radius 0.99Å) [18]. This fact, associated with other possible risk factors for ASR, could indicate that aggregates containing Na minerals will be those at higher susceptibility in terms of alkalis release.

Taking into account on a total aggregate content of  $1850 \text{ kg/m}^3$  used in concrete, the amounts of alkalis that could be supplied to the concrete pore solution by the granitic aggregates are very significantly. Considering, for example, aggregates with particle size of 4.75-9.5 mm the values of alkalis released are on average of 1.88 kg/m<sup>3</sup> (Table 4). These values correspond to 1.68% of total alkalis that these aggregates have in their constitution. It is noteworthy that this amount of alkalis released exceeds the water-soluble alkali content normally liberated during the cement hydration (1.0 kg/m<sup>3</sup> Na<sub>2</sub>O<sub>c</sub>) [17].

# 5 CONCLUSIONS

The work carried out evidences that very significant amounts of alkalis can be supplied with time by aggregates in alkaline environment. This has particular importance in aggregates containing alkali minerals in their composition, such as feldspar-rich ones, which are the main constituents of the igneous rocks, particularly granitic aggregates, which are largely used in concrete structures.

The alkalis release evaluation is dependent on the mineralogy and particle size of aggregate tested, 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 solutions of KOH and NaOH both with addition of Ca(OH)<sub>2</sub>.

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 present significantly contents of reactive silica constituents, and can justify the long-term reactivity presented by this type of aggregates in several concrete structures.

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Particle size	"F1" fraction	"F3" fraction	"F5" fraction	"M" fraction
Solution	(0-2 mm)	(4.75–9.5 mm)	(12.5–20 mm)	(0-4.75 mm)
	GR1	GR1	GR1	-
	GR2	GR2	GR2	-
(1) $C_{\alpha}(OH)_{\alpha}$ saturated	GR3	GR3	GR3	-
(1) Ca(OTI) <sub>2</sub> saturated	-	GR17	-	-
	-	GR18	-	-
	-	GR26	-	-
	GR1	GR1	GR1	-
	GR2	GR2	GR2	-
(2) $C_{\alpha}(OH)_{\alpha}$ sat + N $_{\alpha}OH$ 1M	GR3	GR3	GR3	-
(2) Ca(O(1)) 2 Sat. + IVaO(1) IWI	-	GR17	-	GR17
	-	GR18	-	GR18
	-	GR26	-	GR26
	GR1	GR1	GR1	-
	GR2	GR2	GR2	-
(3) $C_2(OH)_2$ sat + KOH 1M	GR3	GR3	GR3	-
$(5)$ Ca $(011)_2$ sat. $+$ ROTT IN	-	GR17	-	GR17
	-	GR18	-	GR18
	-	GR26	-	GR26
(4) Ca(OH) <sub>2</sub> sat. + NaOH 0.7M	-	-	-	GR26
(5) Ca(OH) <sub>2</sub> sat. + KOH 0.7M	-	-	-	GR26
(6) NaOH 0.7M	-	-	-	GR26
(7) KOH 0.7M	-	-	-	GR26

TABLE 1: Test conditions used to evaluate the alkali contribution by the granitic aggregates [6,13].

TABLE 2: Chemical analysis of the granitic aggregates (wt%) [6, 13].

main elements (%)	GR1	GR2	GR3	GR17	GR18	GR26
SiO <sub>2</sub>	67.05	72.76	71.17	70.92	70.12	71.70
Al <sub>2</sub> O <sub>3</sub>	14.87	14.13	14.37	14.10	14.98	14.72
Fe <sub>2</sub> O <sub>3</sub>	3.74	1.58	1.81	2.92	2.61	2.40
MnO	0.05	0.03	0.04	0.04	0.04	0.04
MgO	1.18	0.48	0.56	0.51	0.69	0.56
CaO	1.90	0.57	0.90	1.04	0.89	0.76
Na <sub>2</sub> O	2.71	2.81	2.88	2.57	3.05	3.47
K <sub>2</sub> O	5.09	4.43	4.80	5.56	4.50	4.84
TiO <sub>2</sub>	0.74	0.21	0.25	0.77	0.31	0.29
$P_2O_5$	0.36	0.33	0.29	0.64	0.24	0.32
LOI	0.88	1.63	1.57	1.20	2.29	0.80
$Na_2O_e$ (%) calculated	6.06	5.72	6.04	6.23	6.01	6.65

		[0,15].				
Samples identification						
	GR1	GR2	GR3	GR17	GR18	GR26
petrographic characterization						
Boasting siling forms $(< 100 \text{ µm})^{\dagger}$	2.4%	21.7%	1.0%	1.5%	4.8%	2.0%
Reactive sinea forms (< 100 µm)*	Class II	Class II	Class I	Class I	Class II	Class II
Undulatory extinction angle (°) of	16.0°	20.0°	18.0°	15.0°	23.0°	11.0°
quartz crystals*	moderate	moderate	moderate	moderate	moderate	weak
minerals						
Quartz	25.1	12.5	28.8	28.4	29.9	28.2
Microcrystalline quartz	2.4	21.7	1.0	1.5	4.8	2.0
Plagioclase	30.5	26.1	36.0	19.0	23.6	30.1
K-feldspar	26.8	26.3	20.4	25.0	18.3	28.8
Muscovite	3.8	9.7	9.5	17.4	14.9	3.5
Biotite + chlorite	10.2	3.1	3.8	6.6	7.6	6.8
Others	1.2	0.6	0.5	2.1	0.9	0.6
Total	100	100	100	100	100	100

TABLE 3: Petrographic analysis and mineralogical composition obtained by point-counting method (vol%) of the granitic aggregates [6,13]

 Note: ‡ Reactivity class: Class I - Potentially reactive silica < 2%; Class II - Potentially reactive silica > 2% (RILEM AAR1.1, 2013 [8]; LNEC E461, 2007[9])

 \*Undulatory extinction: Weak =  $[0^\circ - 14^\circ]$ ; Moderate =  $[15^\circ - 24^\circ]$ ; Strong =  $[25^\circ - 35^\circ]$ ;

TABLE 4: Alkalis release by granitic aggregates (grain size of 4.75 – 9.5 mm) at 455 days of immersion in lime-saturated and
alkaline solutions.

						Alkal	lis released					
		lime-saturated solution						alkaline solutions				
		Ca(OH) <sub>2</sub> sat.				NaOH or KOH 1M + Ca(OH) <sub>2</sub> sat.						
aggregate	size (mm)	Na2O (%)	K2O (%)	Na2Oe (%)	(% total)ª	(kg/m <sup>3</sup> ) <sup>b</sup>	Na2O (%)	K2O (%)	Na <sub>2</sub> O <sub>e</sub> (%)	(% total)ª	(kg/m³) <sup>b</sup>	
GR1	0-2	0.020	0.007	0.024	0.40	0.45	0.172	0.030	0.192	3.17	3.55	
	4.75-9.5	0.007	0.002	0.008	0.13	0.15	0.124	0.011	0.131	2.16	2.43	
	12.5-20	0.004	0.001	0.005	0.08	0.09	0.104	0.007	0.109	1.79	2.01	
	0-2	0.030	0.008	0.035	0.62	0.65	0.168	0.033	0.190	3.32	3.51	
GR2	4.75-9.5	0.007	0.001	0.007	0.13	0.14	0.133	0.017	0.144	2.52	2.67	
	12.5-20	0.006	0.001	0.007	0.12	0.13	0.097	0.011	0.104	1.82	1.92	
	0-2	0.039	0.009	0.045	0.74	0.83	0.186	0.030	0.206	3.41	3.81	
GR3	4.75-9.5	0.007	0.003	0.010	0.16	0.18	0.144	0.014	0.153	2.54	2.84	
	12.5-20	0.005	0.001	0.005	0.09	0.10	0.138	0.009	0.144	2.38	2.66	
GR17	4.75-9.5	0.008	0.004	0.010	0.16	0.19	0.052	0.016	0.063	1.00	1.16	
	0-4.75	-	-	-	-	-	0.055	0.014	0.065	1.04	1.20	
C D ( )	4.75-9.5	0.006	0.003	0.008	0.13	0.15	0.048	0.013	0.057	0.95	1.05	
GKIð	0-4.75	-	-	-	-	-	0.153	0.013	0.161	2.68	2.98	
CDAC	4.75-9.5	0.008	0.003	0.010	0.15	0.19	0.051	0.013	0.059	0.89	1.10	
GR20	0-4.75	-	-	-	-	-	0.059	0.030	0.079	1.19	1.46	
							alkaline solutions					
					NaOH or KOH 0.7M + Ca(OH)2 sat.							
GR26	0-4.75	-	-	-	-	-	0.115	0.016	0.126	1.89	2.32	
						<i>alkaline solutions</i> NaOH or KOH 0.7M						
GR26	0-4.75	-	-	-	-	-	0.090	0.014	0.100	1.50	1.84	
	F1	0.030	0.008	0.035	0.59	0.64	0.175	0.031	0.196	3.30	3.62	
	F3	0.007	0.003	0.009	0.14	0.17	0.092	0.014	0.101	1.68	1.88	
average	F5	0.005	0.001	0.006	0.10	0.11	0.113	0.009	0.119	2.00	2.20	
	M	-	-	-	-	-	0.094	0.017	0.106	1.66	1.96	

Note: " Percentage of the total alkalis (Na2O2) in the aggregate; b Based on a total concrete aggregate content of 1850 kg/m<sup>3</sup>.



FIGURE 1: Influence of particle size with GR3 aggregate - alkalis released (% Na<sub>2</sub>O and % K<sub>2</sub>O) with time in alkaline solutions.



FIGURE 2: Influence of extraction solution ("F3"fraction of GR26 aggregate) - alkalis released (% Na<sub>2</sub>O and % K<sub>2</sub>O) with time in lime-saturated solution and alkaline solutions.



FIGURE 3: Influence of alkaline concentration and Ca(OH)<sub>2</sub> presence ("M" fraction of GR26 aggregate) - alkalis released (% Na<sub>2</sub>O and % K<sub>2</sub>O) with time in alkaline solutions.



FIGURE 4: Influence of aggregate mineralogy - alkalis released (%  $\rm Na_2O$  and %  $\rm K_2O)$  with time in alkaline solutions.