# TO BE OR NOT TO BE... ALKALI-REACTIVE. A CHALLENGE FOR THE PETROGRAPHIC METHOD

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#### Abstract

The mechanisms involved in alkali-aggregate reaction and the characteristics of reactivity of some aggregates are not completely understood, although tens of thousands of mortar and concrete prisms have been tested in the laboratory and field exposure sites have been installed in a number of countries. Aggregates are the least well understood component in the alkali-aggregate reactions and, surely, the most difficult to assess. This happens not just because the materials used as aggregates are widely variable in origin, geological history, composition, texture and degree of alteration, but also because rocks that behave as innocuous in certain areas of the world appear to perform as reactive elsewhere. In consequence, the petrographic assessment of the aggregates is commonly not enough to classify an aggregate as innocuous or potentially reactive.

In this paper questions are raised concerning slow reactive rock types, including suggestions about the possible forms of silica present and their identification.

Keywords: slow reactive aggregates, strained quartz, petrography, complementary methods

### 1 INTRODUCTION

Rocks of all types are used in the manufacture of concrete. The main requirements regarding the aggregates are related to physical and chemical properties as stated in national and international standards such as EN 12620 [1]. However, in what concerns alkali-aggregate reactions (AAR), this standard includes just a reference to the phenomenon. The reports CEN CR 1901:1995 [2] and CEN TR 16349:2012 [3] can be used as guidelines for the prevention of AAR but these documents always forward the final decision on the classification of an aggregate as innocuous or potentially reactive to the regional experience and local standards.

According to Oberholster [4] and the RILEM recommendation AAR-0 (Nixon and Sims [5]) as well as stated in national standards and recommendations (e.g. [6];[7]), when an aggregate comes from a source for which no service record or previous test results are available, its alkali-reactivity potential must be determined in the laboratory. There are lists of potentially reactive aggregates but it can be concluded that some rocks that have not yet been identified as potentially reactive may have a deleterious behaviour in some areas of the world whereas other rocks listed as reactive may perform as innocuous somewhere else. In this context, one specific type of rock cannot be said to always perform as innocuous in concrete.

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Petrographic examination of aggregates can have two different roles regarding aggregate assessment: (1) in the methodology presented in AAR-0 [5], it is the first and quickest test to be applied when the aggregate is delivered to the laboratory, in order to gain information on its composition and character and as first assessment of its potential reactivity, which can be assisted with the work published in RILEM AAR-1.2 [8]; (2) as a routine procedure with local aggregates, which might include the quantification of constituents. In slow reactive rocks microcrystalline quartz is the most frequent component. Alaejos and Lanza [9] studied the effect of different crystal sizes of quartz (0–10  $\mu$ m; 10–60  $\mu$ m, 60–130  $\mu$ m) on the reactivity of aggregates by correlating the results of the petrographic analysis with the results of mortar bar tests and proposed a unique limit for all of them applied to their weighted sum (Equivalent Reactive Quartz, ERQ).

The second phase of assessment corresponds to the performance of mortar or concrete expansion tests that develop in complexity, in time needed to have results and usually also in costs. These tests are divided in mortar bar and concrete prism tests and are used to confirm, or to clarify, the conclusions of the petrographic method. The concrete prism tests are also applied to test different concrete compositions in order obtain the best mixture to prevent ASR.

The main challenge of this cascade of tests is that very often the expansion tests provide results that are not in agreement with the results from the petrographic analysis. Moreover, some aggregates that are classified as non-reactive in the mortar bar test perform as reactive in the concrete prism tests, and the opposite as well [10].

Lindgard et al [11] suggest a classification of the aggregates based on the rate of reaction. Therefore, the aggregates are "fast to normal reactive" (5-20 years), "slow/late reactive" (15-20 years) or "non-reactive". While the petrographic method is able to reliably identify fast/normal reactive aggregates, namely when they contain opal, the limits between non-reactive and slow/late reactive are in most cases based on the experience and expertise of the petrographer.

The present paper refers to four examples of aggregates for which petrography showed to be insufficient for the correct classification of potential reactivity. These aggregates would be classified as innocuous by petrography but have shown reactivity in service performance or in laboratory tests. For such aggregates, supplementary methods should be used to identify reactive forms of silica.

### 2 MATERIALS AND METHODS

### 2.1 Materials

For the development of this study different materials were used:

- Block samples of rocks collected in quarries (2 granites and 1 amphibolite);
- Concrete prims previously submitted to the AAR-4.1 expansion test [5] (the same samples as above: 2 granites and 1 amphibolite);
- Pieces of concrete from a deteriorated structure (a sample containing basalt was selected).

Polished thin sections of  $25 \times 45 \times 0.030$  mm were manufactured by manual processes from progressive grinding to the final polishing.

#### 2.2 Methods for assessment and analysis

The polished thin sections were observed under a LEICA DM750P petrographic microscope using plane polarized light (PPL) and crossed polarized light (XPL). The polished thin-sections were observed under a LEICA DM750P petrographic microscope using plane (PPL) and crossed polarized light (XPL). Modal mineral contents in vol% of the granites and the amphibolite were determined using a Pelcon point-counting device. The description of thin-sections included mineral modal content, structure, and texture/fabric. Petrography of concrete prisms and fragments included the characterization of the coarse and fine aggregates and the identification of cracks and reaction product from deleterious alkali-silica reaction (ASR). Selected amphibolite particles and from light-colored veinlets therein were manually pulverized in an agate mortar and pestle under acetone, for rapid determination of mineral content by powder diffraction – XRD.

Polished thin sections were carbon coated in a Baltec CED 030 evaporator device. The analyses were performed at 15 kV in a JEOL JSM-6400 scanning electron microscope (SEM) equipped with an Oxford INCA X-sight EDS detector.

The Equivalent Reactive Quartz (ERQ) was calculated by the expression ERQ vol.% = vol.% HR Qz (<10  $\mu$ m) + 0.31 vol.% R Qz (10–60  $\mu$ m), according to Alaejos and Lanza [9]. In the expression reactive quartz is designated as R Qz and highly reactive quartz as HR Qz.

For the evaluation of potential reactivity with AAR-4.1 concrete prism test, the criterion in [5] was followed, namely expansion of 0.030% at 15 weeks.

### 3 RESULTS

Table 1 summarizes the main lithological characteristics of the aggregates as well as ASR features observed by optical thin-section petrography.

Fine to medium-grained granitic rocks were selected. Both rocks contain a low but variable content of microcrystalline quartz due to subgraining observed in the boundaries of larger crystals. Besides quartz, K-feldspar and plagioclase, the rocks also contain muscovite, biotite, chlorite, apatite, tourmaline and opaque minerals. Alteration of the K-feldspars and plagioclase is visible in both samples, more intense in the plagioclase crystals than in K-feldspar. The quartz crystals have straight to indented borders, exhibit undulatory extinction and strain lamellae. Point-counting was performed in order to quantify the microcrystalline quartz (< 60  $\mu$ m as explained above).

The AAR-4.1 expansion test classified both aggregates as potentially reactive. The concrete petrography of the prisms after the conclusion of the tests show that alkali-silica gel occurs partially filling some voids in the cement paste (Figure 1). There are also thin rims of gel in the cement/aggregate interfaces, while rare cracks are visible crosscutting particles of the fine fraction of the aggregates.

The amphibolite is mainly composed of amphibole, containing also plagioclase, epidote, titanite, chlorite, apatite and calcite. Feldspars are altered showing a "cloudy" appearance which prevents the correct identification of the minerals. There are portions of the rock exhibiting shear, with subgraining and fragmentation of the crystals. Under cross polarized light (XPL) microcrystalline agglomerates of grains are observed which seem to correspond to quartz and feldspar. There are abundant veinlets containing quartz (with comb texture), amphibole, chlorite, calcite and alteration minerals.

The petrographic examination of the thin sections produced from the concrete prisms after the completion of the AAR-4.1 expansion test confirms the occurrence of alkali-silica gel in cracks that crosscut the aggregate particles and extend to the cement paste (Figure 2).

Due to the very fine-grained material in the "cloudy" areas and in the shear bands the thin sections were studied using scanning electron microscope coupled with X-ray microanalysis (SEM/EDS). This aimed to determine the composition of the gel and to confirm the presence of microcrystalline quartz. It was confirmed that cracks are lined by alkali-silica gel. However, no pure silica is present close to the crack (Figure 3). EDS spot analyses from different areas within the aggregate particles confirm that quartz is scarce and limited to inclusions dispersed within in the amphibole grains, as verified by element mapping (Figure 4). The maps of the "cloudy" areas and of the shear bands demonstrate that they are composed of albite with anorthite lamella, but do not contain free silica. XRD of bulk rock showed that quartz occurs just as a trace component and that albite (not quartz) is the main component of the veinlets. Albite may release alkalis in concrete chemical conditions.

The basalt (local nomenclature) is mainly composed of plagioclase and pyroxene with brownish "cloudy areas" in the interstitial spaces. Reliable point-counting was precluded by the very fine granularity of the rock. The rock contains abundant circular amygdales filled with chlorite and euhedral crystals of zeolite (Figure 5), occasionally also quartz crystals of 60-100  $\mu$ m in size.

The piece of concrete studied originates from a damaged structure. Concrete petrography allowed the identification of manifestations of ASR: cracks with gel crosscut the aggregate particles and extend to the cement paste. The wider crack containing gel occurs in the interface between an aggregate particle and the cement paste. The occurrence of ASR manifestations is quite surprising due to the scarce pure silica minerals visible under optical microscope. Therefore, SEM/EDS analyses were performed in order to detect pure silica that could explain the reactivity of the basalt. Figure 6 shows the results obtained. It was confirmed that chlorite fills the amygdales as well as crystals of zeolite. Pure silica was identified in the amygdales but also in the matrix of the rock, revealing a much higher content of  $SiO_2$  than anticipated by the optical microscopy examination.

## 4 DISCUSSION

At the present state of knowledge it seems that the main challenge both for petrography and for the laboratory expansion tests is the identification of slow reactive aggregates of which some examples have been published. Oberholster [12] refers to serious difficulties for petrography in the assessment of rocks such as granite, granite gneiss, quartzite and quartz-arenite, greywacke, hornfels, argillite, phyllite and mylonite, which may contain cryptocrystalline to microcrystalline quartz, and features associated with deformation of quartz causing crystal lattice irregularities. Granite and granite gneiss were listed as alkali-reactive rocks in Canada in Rogers [13] and, in the list of reactive Australian aggregates, Shayan et al. [14] identified recrystallized granitic rocks from New South Wales as reactive as well as various types of granitic rocks and metadolerite from Western Australia.

In the present paper two groups of samples were analysed. One group corresponds to rocks containing slightly strained quartz for which the features causing reactivity are not clear. In this group of rocks, feldspars are considered to be potential sources of alkalis. In previous studies of granitic aggregates it was concluded that also the results of expansion tests depend on the method used [10] [15] and therefore the explanation of the components involved and the mechanism of reaction with these aggregates needs further study. In this context, the definition of microcrystalline and cryptocrystalline quartz is a matter that deserves discussion. It is an aspect that shows particular interest for slow reactive aggregates and should be revised in the light of the results of laboratory tests being performed on slow reactive rocks. It is known that deformation processes produce undulatory extinction, strain lamellae, serrated boundaries, bulging and also subgraining, which has gained attention in the last decades (e.g.[16]).

Since 1992, when Grattan-Bellew [17] found that the solubility of quartz is highly increased by grain sizes less than 100  $\mu$ m due to the increased surface area, that limit was assumed as the threshold for microcrystalline and 10  $\mu$ m for cryptocrystalline quartz. Alaejos and Lanza [9] suggested a refinement for the classification regarding the crystal sizes and establish the ERQ. The authors also present a summary of the limits established in different countries for the content of cryptocrystalline quartz, including opal and chalcedony, and also on the limits of rocks containing microcrystalline quartz. The limits for the microcrystalline quartz can vary as much as 2% in Portuguese standards, 5% for the Washington State Department of Transportation and 20% in Norway. The content of cryptocrystalline (+opal and chalcedony) quartz seems to be more consensual as the threshold is of 1 to 5%. It is worth mentioning that the content of microcrystalline and cryptocrystalline quartz in the rocks studied for the present paper is for any of these criteria considered too low to produce expansive reactions, which is contradictory to the AAR-4.1 results.

In addition, the correlation of ASR with the size of the crystals of quartz is not consensual. Šťastná et al [18] analysed fine to medium-grained metamorphic aggregates and found a positive correlation between potential reactivity and quartz content, area, perimeter and equivalent diameter but not with specific surface area and with shape factor. The higher ASR potential was also related to undulatory extinction and subgraining which are accompanied by increased dislocation density in quartz. This is in agreement with the conclusions of Tiecher et al [19], in the study of Brazilian deformed rocks, that grains of quartz with strained texture, namely having marked undulatory extinction and deformation bands, dissolve more easily and are highly prone to developing ASR. Also, Locati et al. [20] analysed orthogneisses with variable deformation degree and found that the reactivity of quartz-bearing rocks depended mainly on their textural and microstructural characteristics. These conclusions suggest the need of a re-definition of the characteristics and sizes of crystals that can be involved in ASR, at least for some types of rocks.

The second group of rocks includes those in which the granularity is very fine and the identification of the reactive forms of silica cannot accurately be made under an optical microscope. Work developed on volcanic rocks considers the volcanic glass as potentially reactive when containing more than 65% SiO<sub>2</sub> (e.g. [21];[22]). Tiecher et al. [23] studied the mesostasis in the interstitial spaces of volcanic rocks and concluded that this component, which is associated with the occurrence of ASR, was not amorphous but contained quartz, K-feldspar and clay minerals. In the basalt studied, no volcanic glass was identified but cryptocrystalline material was visible using the optical microscope. SEM/EDS identified quartz (or a polymorph of silica) not only in the amygdales but also in the interstitial spaces. The importance of supplementary methods was also highlighted by Shrimer [24] who reported the difficulties in identifying the reactivity of a diabase from Guyana by petrographic methods. For that diabase SEM and X-ray microanalysis were essential in the identification of very fine-grained quartz.

Regarding the amphibolite, the explanation of reactivity seems to be the most difficult. The fact that quartz just occurs as dispersed inclusions, raises the discussion about how the interstitial fluids access these inclusions. The observation by SEM demonstrates that the rock has abundant cracks and that the interstitial spaces contain silicate minerals. The intense cracking, mainly in the shear bands, should be a preferential open path to facilitate the access of fluids. However, the veinlets do not contain quartz, as expected from the petrographic analysis and the content of silica in the rock seems to be too low to justify the expansion obtained. Moreover, the analyses performed close to the cracks show that there is no pure silica in the vicinity that could be involved in ASR. The role of deformation, alteration and of silicate minerals might have to be revisited to explain the reactivity.

### 5 CONCLUSIONS

Although a lot of work has been developed since 1940, a lot is still to be done in what concerns the assessment of the potential reactivity of aggregates for which there is no experience. The fact that some types of reactive aggregates are still not identified as such by the petrographic method deserves the attention of experts. Furthermore, for some types of rocks there are no well stablished correlations between the petrographic analysis and the results of laboratory expansion tests, nor between the different expansion tests. In this paper just four examples of surprising reactive aggregates were presented. For the sample of basalt the examination under SEM/EDS served to clarify the presence of forms of silica. However the granitic rocks and the amphibolite, with quite low contents of microcrystalline silica, are disquieting aggregates for which the petrographic methods seem to fail. For this type of slow reactive aggregate, further research needs to be carried out in order to understand which components are in the origin of the reaction, namely by the detailed characterization of the silicates present in the rock.

### AKNOWLEDGMENTS

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Sample reference	Description	Undulatory extinction (°)	ERQ (vol%)	Expansion (%) in AAR-4.1 at 15 weeks	Cracks and gel in the prism tests
Granite (GR20)	Fine to medium-grained granite. Micro- to cryptocrystalline quartz in inclusions and myrmekites. Some micro-cracks and subgraining.	19.0	1.31	0.04	Gel in interfaces and in voids. Cracks in the cement paste.
Granite (GR24)	Fine to medium-grained granite. Micro- to cryptocrystalline quartz in inclusions. Locally, subgraining.	20.0	1.35	0.06	Gel in voids. Crack cutting an aggregate particle.
Amphibolite	Medium-grained metamorphic rock. Micro- to cryptocrystalline in shear zones.	not observed	1.40	0.05	Cracks with gel cross the aggregate particles and extend to the cement paste
Basalt	Intergranular texture. Secondary quartz in the amygdales.	not observed	-	Field concrete	Cracks with gel cross the aggregate or form rims around aggregate.

TABLE 1: Petrographic characteristics of the aggregates and of the concrete prisms tested according to RILEM AAR-4.1.

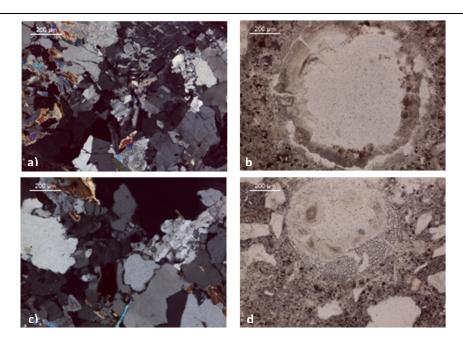


FIGURE 1: Characteristics of the granitic aggregates: a) and b) GR20; c) and d) GR24. a) and c) are examples of the textures of the rocks (XPL); b) and d) show gel partially filling voids of the concrete prisms after being submitted to RILEM AAR-4.1 expansion test (PPL).

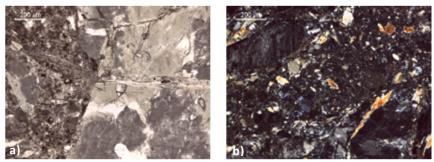


FIGURE 2: Amphibolite: a) crack crossing the aggregate particle and extending to the cement paste; b) shear band showing cryptocrystalline minerals (in the centre), feldspar blasts (with patchy extinction), grains of epidote (Berlin bleu) and elongate grains of amphibole (orange) (XPL).

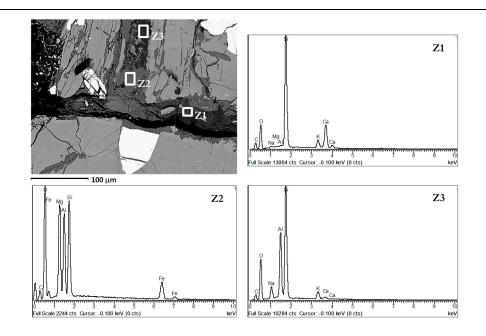


FIGURE 3: Image obtained by SEM (backscatter electron image mode – BSE) of a particle of amphibolite crossed by a crack. EDS spectra confirm the presence of alkali-silica gel (Z1), the existence of chlorite (Z2) and that a silicate mineral (alkali feldspar) (Z3) occurs in the interstitial "cloudy" areas observed under optical microscope. No pure silica was identified in this field of view.

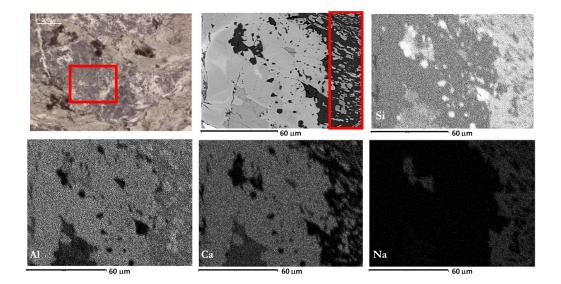


FIGURE 4: Photomicrograph in PPL, SEM image and element mapping of the amphibolite. Quartz is present as small inclusions and in the interstices between amphibole grains, as in the outlined red rectangle of the "cloudy" areas observed under optical microscope (PPL). A similar area outlined on the right hand side of the SEM image in the top-centre panel is mainly composed of Si, Al and Na. The lighter elongate grains contain Si, Al and Ca.

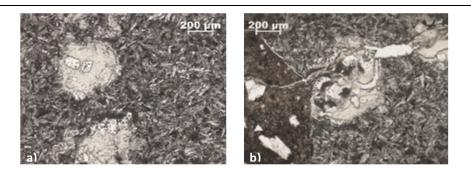


FIGURE 5: Characteristics of the basalt: a) dominant texture of the rock with amygdales containing chlorite and zeolites; b) crack with gel crossing a particle of aggregate. Both images in PPL.

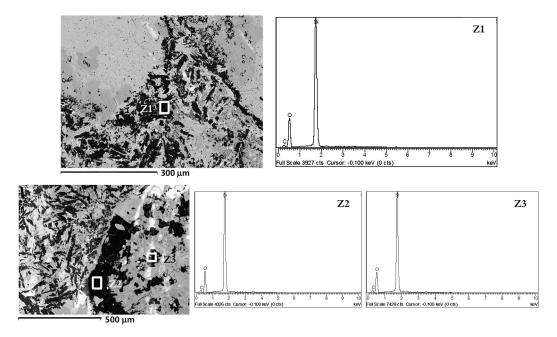


FIGURE 6: Images obtained by SEM-BSE and EDS spectra confirming the occurrence of forms of silica in the interstitial spaces of the plagioclase crystals (above) and in the amygdales (below).