PETROGRAPHIC CHARACTERIZATION OF GRANITIC AGGREGATES. COMPARISON WITH THE RESULTS FROM LABORATORY TESTS

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Abstract

Granitic rocks are widely used as aggregates for concrete in Portugal. Although this type of rock is known in some countries to be non-potentially reactive to alkalis, there are reported cases of alkali-silica reaction related to granitic aggregates.

As defined by the Portuguese specification LNEC E 461, the petrographic analysis is the first to be carried out when aggregates of unknown performance need to be characterized. The characterization is based on the determination of the percentage of reactive forms of silica in order to classify the aggregate as innocuous or as potentially reactive, according to classes similar to those defined in RILEM AAR-1. This assessment is followed by the mortar bar test (ASTM C 1260 or RILEM AAR-2) when the aggregate is classified in Class II or III.

The local experience is that some of the granitic aggregates classified as innocuous by RILEM AAR-2 show a field performance different from that predicted by the tests. Therefore, a research project is being carried out in which systematic concrete prism tests (RILEM AAR-3 and RILEM AAR-4.1) are used to validate the results obtained by petrography and to establish which laboratory test(s) is(are) the most accurate for the assessment of the alkali reactivity of granites.

In the present work, the first results of the research are presented, aiming to contribute to the reformulation of the national specification and to establish a possible correlation among the results from different methods.

Keywords: granitic aggregates, petrographic characterization, expansion tests

Introduction

It is of upmost importance to define which are the laboratory tests, following the petrographic analyses, that best mirror the field performance properties of granitic aggregates in concrete. A lot of work has been done to achieve this goal, but it has not been totally clarifying. The next paragraphs summarize the studies developed so far.

In spite of some countries considering granitic aggregates as being innocuous to alkalis (RILEM AAR-1, 2003), a number of reports have been published worldwide taking this statement as not being general. Under some circumstances, granites may react with alkalis causing a slow deleterious reaction in concrete structures (Cortelezzi *et al.*, 1990). Several facts have been pointed out as being responsible for the reactivity of granitic rocks and a number of methods to predict the behaviour of this type of rock have been proposed.

In 1973, Gogte reported that the potential alkali-reactivity of granitic rocks was a consequence of the amount of strained quartz. Dolar-Mantuani (1981, 1983) used the angle of undulatory extinction to identify alkali-reactive quartz, assuming this angle to be a measure of the imperfections in the crystal lattice. Grattan-Bellew (1986) stated that the presence of microcrystalline quartz in granitic rocks containing strained quartz was the key factor which made these rocks susceptible to alkalis. Kerrick and Hooton (1992), working in Massachussets (USA), and Shayan (1993), working in Western Australia, added that reactivity was related to the extent of deformation and microcrystalline quartz formation in the rock, in which the grain boundaries of recrystallised quartz played a major role. According to Kerrick and Hooton, 1992, the susceptibility to alkalis of granitic rocks might be increased by the presence of deformation in feldspars and in cleavage planes of muscovite and biotite crystals, besides the large amount of microcrystalline and subgranulated quartz. Thomson and Grattan-Bellew (1993) and Thomson et al. (1994) argued that the most reactive component appeared to be the microcrystalline quartz that had undergone significant subgrain development, but not complete recrystallization. West (1994) claimed that undulatory extinction of quartz grains should only be used as a possible indicator rather than a diagnostic feature of alkali-silica reaction (ASR). Wigum (1995a, 1995b) studied, among other rocks, granites and granitic gneisses calculating the average surface area of quartz through grain size measurement. This new approach made it possible to quantify the contents of microcrystalline quartz. The grain size, the grain surface area and, often, the amount of microcrystalline quartz showed a good correlation with the 14 days expansion in the accelerated mortar bar test (ASTM C 1260 or RILEM AAR-2). Wigum et al. (2000) demonstrated the importance of grain size reduction of quartz in the enhancement of reactivity by increasing the total surface area of quartz boundaries available for reaction. Michel et al. (2003) considered that the formation of microcrystalline quartz grains correlated with an increase in the grain boundaries contact with the interstitial solution, contributing to ASR. In 2004, Broekmans published a paper summarizing the structural properties of guartz and their potential role for ASR. Batic and Sota (2004) studied granitic rocks from Argentina and showed the effectiveness of the ASTM C 1293 concrete prism test method and its results consistency with field performance. In the same year, Yan et al. (2004) added that the presence of deformation features, namely dislocations in feldspars and micas, besides quartz, were responsible for the initiation of ASR. In 2006, in the Petrographic Atlas developed under the scope of the PARTNER Project (Lorenzi et al., 2006), it was referred that also myrmekite might enhance the potential for reaction. Shayan (2007) showed the inability of ASTM C 1260 to detect slowly reactive aggregates of granitic nature which exhibited gneissic features in the form of foliation and presence of microcrystalline quartz. Wenk et al. (2008), using neutron diffraction texture analysis in deformed granitic rocks, showed that the preferred orientation of biotite might be used to estimate the deformation state of rocks containing this mineral and quartz, and hence their susceptibility to ASR. Once again the key role of grain size was emphasised. Shayan et al. (2008) claimed that the RILEM Concrete Prism Test at 60°C (RILEM AAR-4) overcame the deficiencies of RILEM Concrete Prism Test at 38°C (RILEM AAR-3) for slowly reactive aggregates, such as deformed granite, which contained microcrystalline quartz as the reactive component. In 2010, Locati et al., in their study of orthogneisses, concluded that the use of different tools to evaluate the potential alkali reactivity of the rocks in concrete was a good strategy compared to using isolated tools, which could result in misleading decisions. Velasco-Torres et al. (2010) compared two granitic aggregates from Spanish Dams. The main difference between the aggregates was the reactive component (microcrystalline quartz versus microcracked quartz). They concluded that granite could develop either a rapid or a slow alkalisilica reaction depending on the deleterious component involved. The main mechanism of formation and accomodation of gel was associated with microcracks rather than with subgrain boundaries.

In Portugal, large amounts of granitic aggregates are produced every day. The huge abundance of this material contributed and still contributes to the building of major structures. The awarenes of the problems caused by ASR, in Portugal, only began in the early 1990's and, since then, a variety of cases have been investigated by Fernandes (2005, 2009), Fernandes *et al.* (2004, 2007), Santos Silva (2005), Santos Silva *et al.* (2000, 2008), Castro (2008) and Castro *et al.* (2009). At the moment, a national project – IMPROVE (Improvement of performance of aggregates in the inhibition of alkali-aggregate reactions in concrete) is being carried out with the goal to accurately evaluate the conditions to minimise the occurence of alkali-aggregate reaction in several lithologies, mainly granitic rocks.

Materials and methods

Three types of crushed granitic aggregates, from Portuguese quarries, were sampled and studied, assessing their petrographic characteristics and their laboratory performance when applied as aggregates in mortar and concrete. It was of extreme importance to check sample heterogeneities which might occur in the quarries. To keep the producers' request for confidentiality of the quarry locations, the aggregates have been denoted A, B and C.

In order to predict the field performance of the granitic aggregates and to compare results, petrographic characterization (RILEM AAR-1, 2003) and expansion tests, namely ASTM C 1260 (2007) accelerated mortar bar test, RILEM AAR-3 (2000) and RILEM AAR-4.1 (2011) concrete prism tests were carried out.

Granitic aggregates

The three granites studied are of Hercynian origin (Ferreira et al., 1987).

Aggregate A is composed of medium to coarse-grained, porphyritic, monzonitic granite. It is a late-tectonic granite relative to D3 Hercynian deformation phase (intra-Westphalian in age).

Aggregate B is composed of medium-grained, porphyritic, monzonitic granite. It is a late to post-tectonic granite.

Aggregate C is composed of medium to coarse-grained, porphyritic, two-mica granite. It is a syntectonic granite.

Petrographic characterization

Since each of the studied crushed rocks has uniform characteristics, representative thin-sections of the aggregate particles from the three quarries were produced and studied under a Nikon Eclipse E 400 POL

polarizing microscope. According to Lindgård and Haugen (2006), this procedure is named "whole rock petro". To complement the petrographic examination with quantitative values, a PELCON automatic point-counter was attached to the polarizing microscope. The point-counting procedure was carried out by traverses in regular increments in two directions to form a virtual orthogonal grid, covering the whole thin-section. During the point-counting, the constituents were identified and thus the reactivity assessment of the individual point which was directly under the crosshairs. A minimum of 1000 points were counted (excluding points falling on the resin) in each of the thin sections. The evaluation was made according to RILEM AAR-1 (2003) and to Portuguese specification LNEC E 461 (2007). Similar categories of aggregates are defined by both RILEM AAR-1 (2003) and LNEC E 461 (2007): Class I – innocuous (reactive silica < 2%); Class II – ambiguous; Class III – potentially alkali-reactive. The assessment of reactivity to alkalis was based on local experience with granitic aggregates namely with the deformation evidences of the quartz crystals (recrystallized quartz) and on their dimensions (identification of microcrystalline quartz <100 μ m, Grattan-Bellew, 1992). Goticular and myrmekitic quartz were also included in the potentially reactive forms of silica.

Expansion tests

For aggregates classified in Class II or III, the assessment is followed by expansion tests, starting with the accelerated mortar-bar test.

The aggregates were crushed, sieved and weighed according to the requirements of ASTM C 1260 (80°C), 2007 accelerated mortar-bar test, RILEM AAR-3 (38°C), 2000 and RILEM AAR-4.1 (60°C), 2011 concrete prism tests.

Results and discussion

The results and the discussion of the petrographic characterization and of the expansion tests are presented in the next two subsections.

Petrographic characterization

The results of the point-counting are displayed in Table 1. Aggregate A shows an inequigranular texture with well developed K-feldspar phenocryts. Plagioclase, quartz, K-feldspar, muscovite and biotite are the main minerals. The accessory minerals are represented by zircon, apatite, and alusite and opaque minerals. As secondary phases, chlorite, rutile, clay minerals, muscovite, calcite and epidote were identified.

Aggregate B shows the same texture as aggregate A with plagioclase, K-feldspar, quartz, biotite and muscovite as the main minerals. Apatite, zircon, sillimanite, opaque minerals and fluorite are the accessory minerals. Chlorite, rutile, clay minerals, microcrystalline muscovite and epidote were identified as secondary phases.

Aggregate C, besides an inequigranular texture with K-feldspar phenocryts, exhibits a preferential orientation of minerals. The main minerals are represented by K-feldspar, plagioclase, quartz, muscovite and biotite. The accessory minerals are represented by tourmaline, apatite, zircon and opaque minerals. The secondary phases are represented by chlorite, rutile, clay minerals and muscovite.

Mineral	Aggregate A	Aggregate B	Aggregate C
Plagioclase	36.0	30.5	26.1
Quartz	28.8	25.1	12.5
Potassium Feldspar	20.4	26.8	26.3
Muscovite	9.5	3.8	9.7
Biotite + Chlorite	3.8	10.2	3.1
Recrystallized and/or goticular, and/or myrmekitic quartz	1.0	2.4	21.7
Accessory minerals	0.5	1.2	0.66
Total	100.0	100.0	100.0

Table 1: Results of the point-counting method (values in %).

Regarding the characteristics of the quartz crystals, the petrographic analysis revealed that aggregate A contains strained quartz crystals showing undulatory extinction ($\sim 18^{\circ}$) (Figure 1A1). Quartz is also present as myrmekite in plagioclase and in goticular form in K-feldspar. Quartz, along with K-feldspar and plagioclase crystals, are frequently microcracked (Figure 1A2).

The examination of aggregate B showed features similar to aggregate A: in this case, the angle of undulatory extinction is about 16° and myrmekite and goticular quartz are also common (Figure 1B1 and 1B2).

Aggregate C, besides featuring quartz with more evident undulatory extinction (~20°) and deformation lamellae, exhibits a quite substantial amount of elongated, microcrystalline and sub-granulated quartz (Figure

1C1). Goticular forms of quartz are present in K-feldspars and plagioclases. Besides quartz, deformation is also confirmed by the presence of sheared phyllosilicates (Figure 1C2) and plagioclases and by preferential orientation of the minerals.



Figure 1: Aggregate A: undulatory extinction in quartz crystals (A1); presence of microcracks in a zoned plagioclase crystal (A2). Aggregate B: goticular quartz (B1); myrmekites (B2). Aggregate C: existence of preferential orientation of deformed quartz (C1); strained crystals of muscovite (C2). All images are taken in crossed-polarized light.

According, strictly, to the percentage of potentially reactive features, it can be said that only aggregate A can be classified as innocuous to alkalis by petrographic characterization (LNEC E 461, 2007). On the contrary, aggregate B and, especially, aggregate C present values of the potentially reactive features above 2%, which classify them as potentially-reactive.

Aggregate C shows a much higher proportion of reactive quartz features (21.7%) than aggregates A and B. This observation contributes to support the fact that syn-tectonic, two-mica granites are more likely potentially reactive to alkalis than the younger granites (Castro, 2008; Castro *et al.*, 2009). Also, according to Hills and Corvalán (1964), the difference in intensity of the undulatory extinction in quartz grains can be interpreted as the result of the degree of tectonic deformation of the granitic rocks of different ages present in a single folded belt affected by successive orogenic events. Quartz undulatory extinction is more intensely developed in older rocks, decreasing in intensity in more recent rocks. In fact, aggregate C is older than aggregates A and B and exhibits a stronger undulatory extinction. However, the occurrence of undulatory extinction was not considered for the assessment of the potential reactivity, as explained before.

Expansion tests

Although it would not be necessary to perform the expansion tests on aggregate A according to the Portuguese specification LNEC E 461 (2007), the same was carried out in order to enable a comparison between all the tests and to check their suitability.

When submitted to ASTM C 1260 accelerated mortar bar test, aggregates A, B and C showed 14 days expansion values of 0.02%, 0.01% and 0.02%, respectively. Expansions of less than 0.10% after 14 days distinguish a non-reactive from a reactive aggregate (Lindgård *et al.*, 2010).With the aim to confirm the results, the tests were carried on for another 14 days. According to the expansion results, all aggregates were considered as being non-reactive (Figure 2). By comparing these results with the ones obtained by Shayan (2007) for gneissic granitic aggregate with strained and microcrystalline quartz, aggregates A, B and C show much lower expansion values, since at 28 days, 0.20% of expansion is far from being reached. It should be highlighted that the aggregates are different from the ones of the present study, showing similarities just with aggregate C. The slowly reactive aggregates studied by Shayan (2007) show, at 28 days, values very close to 0.20%. Although

ASTM C 1260 is widely used for the assessment of aggregates for concrete purposes, it is still not well recognized that its limits correspond to the reactivity of some of the slowly reactive aggregates, leading to their incorrect classification as non-reactive or uncertain (Shayan, 2007). On the other hand, Barisone and Restivo (2000) and Lu et al. (2006) showed that the crushing of rocks to obtain very fine aggregates can destroy the original microstructure and thus under-estimate the alkali reactivity of the rocks in accelerated mortar bar tests. Santos-Silva and Gonçalves (2006) and Santos-Silva et al. (2008) have already considered the accelerated mortar-bar test as inappropriate to evaluate the alkali-reactivity of Portuguese granitic aggregates.



Figure 2: Expansion behavior for aggregates A, B and C in the ASTM C-1260 mortar bar test.

The results achieved so far by the AAR-3 concrete prism test at 38°C are related just to 6 months, half of the time considered as a minimum for any final conclusions to be drawn. All three concrete samples present a similar behavior. From the 140th day, the samples display a significant increase in the concrete prism expansion. After 196 days, aggregate C concrete prism exhibits the highest expansion value (0.03%), followed by aggregates A (0.02%) and aggregate B (0.01%) (Figure 3). Expansions of less than 0.05% after 1 year classify the aggregate as non-reactive (Lindgård *et al.*, 2010).Therefore, it seems appropriate to wait for the completion of this test to draw any further conclusion.



Figure 3: Expansion behavior for aggregates A, B and C in the AAR-3 (38°C) concrete prism test after 196 days.

Although in the Portuguese specification the limit of 0,02% is still accepted, according to Lindgård *et al.* (2010), expansions of less than 0.03% after 20 weeks in AAR 4.1 indicate that the aggregate can be regarded as non-reactive. The aggregates performance during the 20 weeks long AAR-4.1 concrete prism test at 60°C classified aggregates A and C as being reactive (Figure 4), with aggregate C displaying the highest final

expansion (0.07%). Expansion values reached by Shayan *et al.* (2008) for gneissic granites show, at 20 weeks, values close to 0.08% which are similar to the final value obtained by aggregate C. The same authors obtained for a granite, at 20 weeks, a value close to 0.04% which is consistent with the values found for aggregates A. In the PARTNER Project a granitic sand was tested and classified as non-reactive by the AAR-4 test as well as in the AAR-3 test. In Lindgård *et al.* (2010) it is referred that AAR-2 and AAR-4 seem to be the most effective RILEM methods in the assessment of slowly reactive aggregates. However, the slowly reactive aggregates tested are not of granitic rocks, which makes it difficult to establish a comparison with the present samples.

Till now only AAR-4.1 concrete prism test was able to detect reactivity and confirm some of the results that were observed by the petrographic characterization, namely for aggregate C. However, taking into account the expansion values achieved by AAR-3, so far, and by AAR-4.1 concrete prism tests, aggregate A always presents higher expansion than aggregate B. This fact contradicts the results obtained by the petrographic characterization that resulted in aggregate A being classified as innocuous. The explanation for this fact can be the frequently observed microcracks in aggregate A which, according to Velasco-Torres *et al.* (2010), are the main cause of expansive gel formation and accommodation.



Figure 4: Expansion behavior for aggregates A, B and C in the AAR-4 (60°C) concrete prism test.

Conclusions

The tests performed in the granitic samples lead to the conclusion that the ASTM C-1260 mortar bar test shows a poor correlation with the petrographic characterization of the three aggregate A, B and C. The AAR-4.1 (60°C) concrete prism test is showing to better mirror the expected reactivity assessed by petrographic methods. Although the AAR-3 (38°C) concrete prism test is assumed to be the laboratory test which best reproduces the field conditions, the duration until now does not permit to draw any final conclusions from the results obtained. It should also be kept in mind that crushing certain types of aggregates for laboratory tests may change some of their characteristics (microstructure) and therefore the grading actually used in the concrete structures should preferably also be used as such in the laboratory tests. This means that concrete prism tests should be privileged to reproduce field conditions and further experiments need to be done to study the accuracy of the AAR-4.1 (60°C).

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