REACTIVITY STUDY OF BRAZILIAN AGGREGATES THROUGH SILICA DISSOLUTION ANALYSIS

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Abstract

Quartz is a stable mineral under normal exposure conditions, but when exposed to high alkalinity it becomes prone to dissolution and to the alkali-aggregate reaction. The present study aims to assess the relationship between the degree of deformation of quartz grains and the silica dissolution potential of that mineral. To this end, two rocks -- known to be reactive in Brazilian concrete structures -- and an innocuous rock, were selected to undergo different methods of aggregate reactivity as well as silica dissolution analyses. The results showed that the rocks predominantly formed by quartz grains with a marked undulatory extinction and featuring deformation bands are more prone to silica dissolution and to the alkali-aggregate reaction.

Keywords: quartz, Brazilian rocks, chemical method, silica dissolution.

¶ 1 INTRODUCTION

Silica dissolution is closely related to the development of the alkali-silica reaction (ASR) in concrete structures. Generally speaking, the silica that causes ASR originates from aggregates (rocks and / or sands) and, of the silicates that constitute aggregates; quartz is in most cases the one responsible for the reaction.

Several cases of concrete structures affected by the ASR are related to the employment of aggregates of metamorphic origin, comprising highly strained quartz. Marfil and Maiza [1] verified the occurrence of the ASR in paving in Argentina (Buenos Aires and Bahia Blanca), in which migmatite and quartzite were used. Broekmans [2] describes a case of reactivity of mylonites in Norway. Leemann and Holzer [3] proved that two Swiss metamorphic aggregates – a gneiss and a shale – are reactive to alkalis.

In Brazil, there are also various examples of concrete structures that employed metamorphic rocks, containing highly strained quartz, which developed ASR. Examples include the Apolônio Sales Hydroelectric Plant (HP), in Moxotó, built with granite-gneiss; the Joanes II HP, built with gneiss; the Furnas HP, built with quartzite [4]; the foundations of some buildings in the metropolitan area of Recife [5] and the Governador Paulo Guerra Bridge, which employed mylonites [6].

Yet, despite their marked deformation and consequent high potential to develop ASR, aggregates of a metamorphic origin are considered 'slow' when it comes to developing the reaction [3, 5, 7-9]. On occasion, these aggregates will even be considered innocuous or with little reactive potential in accelerated reactivity tests [5, 10]. What occurs with metamorphic aggregates is a reduction in the size of the quartz grains during the metamorphism processes, enabling the surface available to the reaction to increase. Yet, since the newly formed grains are not always strained, they may take longer to react [2].

Thus, based on the aforementioned explanations, the present study aims to relate the reactive potential of aggregates with the degree of deformation of quartz grains occurring in rocks.

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2 MATERIALS AND METHODS

2.1 Materials

Rocks

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The present study involved two rocks comprising quartz grains with different degrees of deformation, and which had been used as aggregate material in concrete structures that eventually got affected by the ASR in Brazil. In addition, an analysis was made of a rock containing straineddeformed quartz grains but having innocuous behavior both in the laboratory and in the field. Table 1 presents the rocks selected for this study.

The petrographic analysis showed that the chosen rocks have the following characteristics:

a) QZITE (Figure 1(a)): granoblastic structure; fine grains. It basically comprises quartz (94%) and micas (6%). The quartz appears highly strained, with serrated grains, indicating these have not yet been recrystalized. The size of the grains varies from 0.05mm to 0.3mm. The micas (muscovite) appear in the shape of very thin and lengthy flakes and measure 0.3mm;

b) MRR (Figure 1 (b)): a fine-grained rock with mylonitic texture; comprising quartz (15%), K-feldspar (34%), plagioclase (13%) and micas (38%). The grains of quartz vary in size, ranging from 0.01mm to 1.2mm, with a prevalence of smaller grains which are grouped in bands and are highly strained, featuring subgrains which in turn are also clustered. The micas also form clusters and measure 0.05mm. The K-feldspars (in which microcline predominates) are highly deformed and pertitized, featuring undulatory extinction and alteration towards micas, with an average size of 2.4mm. The plagioclase measures approximately 1mm and also features undulatory extinction, yet of a lower intensity than the K-feldspar. Formations of pressure shadows can be seen in the regions were strains are more intense, and comprise quartz and micas of low crystallinity;

c) GNRR (Figure 1 (c)): a rock with a phaneritic texture comprising quartz (24%), K-feldspar (24%), plagioclase (37%) and biotite (15%). The quartz grains are not very strained; few have undulatory extinction, which is of mild intensity. Their average size is 1.2mm. The K-feldspars appeared altered to carbonates and micas, featuring pertitization, and size varying between 2.5mm and 5mm. The plagioclase has an average size of 1.2mm. There is a tendency to the formation of biotite bands; these have an incipient alteration towards muscovite and their average size is 1.2mm.

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Cement

An ASTM type-1 cement was employed in this study when conducting the accelerated mortar bar test [11-12]. This cement meets the standard's requirements with regard to alkali content (0.89%), fineness (4,920 cm^2/g), autoclave expansion (0.02%) and no addition whatsoever.

2.2 Methods for assessment and analysis

Accelerated mortar bar test

The accelerated mortar bar test was performed in conformity with the Brazilian standard, NBR 15577-4/2008 [11], which evolved out of the ASTM C 1260/07 [12], yet with different expansion limits as it is based on the behavior of local aggregates.

The test provides recommendations for the classification of aggregate reactivity potential based on expansion at 30 days of mortar bars with a width of 25mm and length of 285mm, immersed in a NaOH solution with 1M concentration at 80°C. The aggregate/cement prescribed by the standard is 2:25:1 and a water/cement ratio of 0.47.

Based on the average of expansions in the three test specimens, modeled with the aggregate to be analyzed and the standard cement, aggregates are classified as potentially reactive when their expansion is higher than 0.19% at 30 days of mortar age [13]. Lower expansions, according to the aforementioned standard, denote potentially innocuous behavior.

Chemical method of ASTM C 289/2007

The chemical method was performed according to ASTM C 289/2007 [14]. The test employed rock fractions finer than 0.30mm and coarser than 0.15mm, and was performed in triplicate with 25g samples. These were stored in stainless steel containers with 25mL of NaOH solution, with 1N concentration, at 80°C (one stainless steel container was used as a blank test). The method prescribed by ASTM C 289/2007 [14] requires an analysis of the amount of dissolved silica and alkalinity reduction in the alkaline solution after 24h of exposure to the aggregates. However, due to criticism by several authors of this method's reliability [7, 15-16], since many aggregates known to be reactive produce false negative results, in addition to the analysis at 24h, longer periods of exposure to the alkaline solution were proposed, namely 72h and 168h. The chemical method from NF P 18-594 [25] also analyses dissolved silica after 24h, i.e. 48h and 72hs.

At the end of the proposed periods the samples were filtered. Part of the resulting solution was used to determine the dissolved silica (mass), and another part to assess the alkalinity reduction (pH evaluation through titration).

Visible spectrophotometer method

This test was performed following the Brazilian method NBR 9848/2004 [17], which is often used for chemical analysis of water. This standard specifies the test method for determining silica (SiO₂) in liquid caustic soda, by means of a visible spectrophotometer using ammonium molybdate.

The method determines the coloring resulting from a silicomolybdic complex, with a pH close to 1, formed through visible spectrophotometry, and a silica concentration proportional to the existing silica. A 680nm wavelength was used in the equipment.

To perform the analyses with the visible spetrophotometer, quartz grains extracted from the rock matrices were used. The aim of this analysis was to verify the influence of quartz deformation on the ASR, excluding the interference of other silicates that are known to contribute both to silica dissolution and with alkalis in the ASR. Thus, the quartz grains extracted from the rocks were immersed in a NaOH solution with 1M concentration at 80°C for 3 days. After this period, the samples were filtered and the resulting solution was used in the analysis by the aforesaid method.

Mineral separation was done in rock samples with a grain size below 0.15mm. Firstly, the micas were separated magnetically by means of a Frantz Isodynamic Magnetic Separator. Then, the separation between quartz and feldspars (K-feldspar and plagioclase) was performed through the difference in density between them. In order to evaluate the efficacy of the method used for separating the quartz from the other minerals it was employed mineral quantification by X-ray diffraction data using the Rietveld Method. Results have shown a percentage of 95% for quartz, 4.5% for feldspars and 0.5% for micas in the samples.

Petrography analysis

The petrography analyses of the rocks along with the quantitative modal analysis were performed using thin slides (30 μ m) on an optical transmitted light microscope. The main aim of this analysis was to characterize and quantify the constituent minerals of the rocks, with an emphasis on the texture of the quartz grains.

In order to investigate the influence of the degree of quartz deformation occurring in the aggregates for the ASR to develop, a methodology was used to classify the different degrees of grain deformation, as described in Table 2 and illustrated in Figure 2.

3 RESULTS

QZITE and MRR rocks were employed in Brazilian concrete structures affected by the ASR, hence proven to be reactive in the field. Through the accelerated mortar bar test (NBR 15577/2008; ASTM C 1260/07) it was verified that these rocks also proved to have a reactive behavior in the laboratory, while the GNRR rock, which was innocuous in the field, proved innocuous in the expansion test, thus corroborating its behavior in the field. Figure 3 shows the average length variation measured over time for the rocks analyzed.

In order to relate the rocks' silica dissolution potential with the degree of deformation of quartz grains occurring in each of them, tests prescribed by ASTM C 289/2007 (chemical method) and NBR 9848/2004 (visible spectrophotometer method) were performed. Figure 3 shows the graph that relates the amount of dissolved silica and the alkalinity reduction for the purposes of classifying the reactive potential of the aggregates.

As described in the section 2.2, some variations were performed in the method proposed by ASTM C 289/2007. In addition to the analysis with 24h exposure of the aggregates to the alkaline solution, their behavior was also studied at 72h and 168h. The graph in Figure 4 shows that the increase in exposure time to the alkaline solution for the QZITE and GNRR rocks resulted in an increase in silica dissolution and, consequently, in alkalinity reduction. However, for the MRR aggregate, there was an increase in the amount of dissolved silica as time passed, but alkalinity reduction was lower after 72h of exposure, and started to increase again at 168h.

Thus, what was generally noted through the aggregate's reactivity assessment by the chemical method of ASTM C 289/2007 was that the tested variations did not prove effective in better assessing the aggregates, since the QZITE rock, proven to be reactive in the field, was classified as reactive when exposed for 24h (time set by the aforementioned standard) but at 72h and 168h it becomes potentially reactive. Furthermore, the MRR rock, which was also responsible for unleashing the ASR in the field, did not prove to be reactive in any of the conditions tested; the GNRR rock, in turn, is innocuous in the field, and was the only one that proved innocuous also after the chemical method.

Therefore, an alternative approach was sought to assess silica dissolution in the selected samples, using the visible spectrophotometer method. As described in item 2.2, this method was employed only with quartz grains extracted from the rocks in order to avoid interference from the other existing minerals. The results obtained can be seen in Figure 5.

Looking at Figure 5, it can be derived that rocks that were reactive in the field (QZITE and MRR) featured the same amount of dissolved silica when tested by the visible spectrophometer method ($21.2\mu g/mL$), while the innocuous sample had much lower dissolution ($0.6\mu g/mL$). Thus, the method proved to be coherent and appears as a promising tool to analyze the silica dissolution of rocks and the possible relationship with reactive potential.

In order to correlate the texture characteristics of the quartz grains occurring in the rocks that were prone to developing the ASR, the methodology presented in Table 1 (see section 2.1) was used. Dollar-Mantuani [18] proposed a correlation of the angle of undulatory extinction of the quartz with its potential reactivity. According to the author, grains with undulatory extinction angle greater than 25° are potentially reactive and smaller than 15° denote innocuous quartz. However, Dollar-Mantuani [18] emphasizes that this measure is not completely accurate in conventional optical petrography; thus, when the measure of undulatory extinction angle is used to define the reactivity of the aggregate, an uncertainty up to 60% is possible to occur. Thus, in the current study, it was quantified different degrees of deformation of quartz through the modal quantification, the results shown in Table 3 were obtained.

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Some aspects related to the data on Table 3 should be highlighted. First, it can be noted that degree 2 of deformation is predominant in the rocks that are reactive in the field (QZITE and MRR). Considering the results presented by Tiecher [19], grains with intense undulatory extinction, forming deformation grains inside the particles (as is the case of degree 2), are more easily attacked by alkaline hydroxides.

The speed with which the ASR developed in the structures incorporating the QZITE and MRR aggregates indicates that the higher the number of degree-2 quartz grains, the faster the occurrence of the ASR in the field, since the structure built with the QZITE rock was affected by the ASR after 13 years [20] and the structure with MRR, after 12 years [5]. In the accelerated test, it was also verified that the QZITE expanded more rapidly and intensely than the MRR, the same occurring in the dissolution analyses, however, it is important to be care with accelerated test. Yet, it is important to highlight that there are other facts intervening in favor of the ASR in these structures, such as humidity and the concrete's alkali content, among others, which are not being taken into account in the present study.

It is also relevant to individually analyze the distribution of quartz grains, according to the different degrees of deformation, in the reactive and innocuous rocks in the field. In QZITE, as shown by Table 3, there is an overwhelming number of quartz grains of deformation degree 2. Yet, this rock also has a large number of subgrains (degree 3), which reflects how intense the rock's deformation is.

Rocks MRR and GNRR originate in the same region (Recife/PE – Brazil), but have very distinctive characteristics. MRR has a great quantity of degree-2 quartz, that is, appears markedly strained, featuring deformation bands. In the GNRR rock, on the other hand, there is a prevalence of degree-0, deformation-free grains. Considering the percentage of degree-2 grains quantified for GNRR and taking into account that no ASR was observed in the structures in which this rock was employed, it can be suggested that values lower than 13% of degree-2 have a great possibility to cause minor damage from ASR. Anyway, it would be necessary to collect more field data correlating the use specific of the GNRR rock and concrete performance related to ASR, according to uncertainness presented on Andrade et al. [6].

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4 DISCUSSION

Throughout the years, the ASR has been related to the deformation of grains of quartz occurring in rocks, and is often identified by the undulatory extinction of the grains. However, as some authors have been reporting, the more deformed rocks do not always result in higher ASR intensity. Monteiro et al. [21] correlated the deformation of metamorphic aggregates to the alkali-silica reaction and verified that in the intensely strained grains the undulatory extinction may disappear, suggesting that other parameters need to be assessed.

In the present study, five levels of quartz grain deformation were observed. These occur due to the formation of displacement walls in the quartz grains, increasing the stresses to which the rock was submitted and which can, for instance, transform a granite (a plutonic igneous rock) into a mylonite (a metamorphic rock). Passhier and Trow [22] explains that a crystal has a certain internal energy, which is minimal when the crystal is free of displacements (perfect crystal – flawless). When the crystal is strained, there is an increase in displacements and in internal energy, which alters the distance between the atoms. This energy boost is proportional to the increase in the length of the displacements by volume of crystalline material, and is also known as displacement density. According to Wenk et al. [23], the continuous increase in the displacement of the crystal leads to a clustering of the networks, known as subgrain walls which, as time goes by, completely break away from the crystal of origin, forming new recovered grains, that is, without displacements.

The results obtained in this study suggest that quartz grains with intense undulatory extinction, and with deformation bands (degree 2), are more easily dissolved by the reaction with the alkaline hydroxides (see table 3). This happens because the displacement walls appearing in the degree-2 grains (which are identified

by the deformation bands in optical microscopy) weaken some zones and facilitate the penetration of alkaline hydroxides, leading to the occurrence of the ASR. However, another quartz grains can also contribute to silica dissolution, but with lower intensity.

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5 CONCLUSIONS

The results obtained in the present study showed that two rocks used in concretes affected by the ASR in Brazil also have a behavior considered potentially reactive in analyses performed through the accelerated mortar bar test.

The assessment of silica dissolution in these rocks showed that the most commonly used method for this purpose, prescribed by the ASTM C 289/2007, is not entirely reliable in the classification of the reactive potential of aggregates, since one of the rocks analyzed yielded innocuous behavior in this test, even though it had proved to be reactive both in the field and in the laboratory. Nor did the proposed increase in exposure time of the samples to the alkaline solution in this same test proved to be effective in classifying the reactivity of the aggregates analyzed.

On the other hand, the silica dissolution analyses performed under the Brazilian method of the NBR 9878/2004 proved quite coherent, making this a very promising method for this type of assessment. Yet, a higher number of samples and tests need to be undertaken for this method to be more reliably employed and also for a relationship between the amount of dissolved silica and the classification of aggregate reactivity to be established.

Combining the expansion and silica dissolution analyses with the assessment of the degrees of deformation of quartz grains occurring in rocks, it was verified that grains featuring a rather strained texture, with marked undulatory extinction under optical microscopy, and having deformation bands, dissolve more easily. This occurs because these grains have fragile zones, easily attacked by the alkaline hydroxides during the ASR. Therefore, it is possible to conclude that rocks featuring a high number of grains with the aforementioned characteristics are highly prone to developing ASR.

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TABLES:

TABLE 1: Rocks selected which had been used in structures with/without ASR									
Rock	Abbreviation used	Origin	Structure	ASR					
				Yes	No				
Quartzite	QZITE	Passos/Minas Gerais	Furnas HP	х					
Mylonite	MRR	Recife/Pernambuco	Foundations	х					
Granite	GNRR	Recife/Pernambuco	Foundations		х				

TABLE 2: Classification proposed to characterize quartz deformation degrees						
Deformation degree	Description					
Degree 0 Quartz is deformation-free	Quartz grain is free of defects, with deformation. This grain extinguished in a homogeneous manner as the plate of the optical microscope is rotated, showing that its crystalline reticule is free of defects (displacements).					
Degree 1 Quartz with mild undulatory extinction	First stage in the deformation process. The quartz grain features undulatory extinction, and is not extinguished in a homogeneous manner, retaining lighter and darker zones. This optical characteristic of the mineral reflects some deformation in the crystalline reticule.					
Degree 2 Quartz with marked undulatory extinction, forming deformation bands	Quartz deformation increases. Undulatory extinction is more marked, creating well defined zones inside the crystal (displacement walls start to form). These zones are called deformation bands and are regions where the crystalline reticule is deformed, thus weakening chemical links.					
Degree 3 Quartz with marked undulatory extinction, forming subgrains	Growing grain deformation, widening deformation bands, resulting from increased defects in the mineral's crystalline reticule, creating arched regions where the chemical links tend to break more easily (well defined subgrain walls inside the grain of origin).					
Degree 4 Recrystallized quartz	Final stage of the deformation process. The subgrains are completely individualized and form new grains, without defects, in a process called recrystallization. The recrystallized grains have the same characteristics occurring in the first stage; they are grains without deformation of the crystalline reticule, but of a smaller size					

TABLE 3: Comparison between the different degrees of quartz deformation in rocks										
	Quantity of grains (%)									
Samples	Degree 0	Degree 1	Degree 2	Degree 3	Degree 4					
	Quartz without deformation	Quartz with mild undulatory extinction	Quartz with marked undulatory extinction,forming deformation bands	Quartz with marked undulatory extinction,with subgrain formation	Quartz recrystallized from subgrain					
QZITE	-	2.8	48.4	36.4	12.4					
MRR	-	1.3	57.4	27.7	13.6					
GNRR	37.1	21.3	13.1	10.8	18.0					

FIGURES:



FIGURE 1: Micrographic images obtained through optical microscopy (transmitted light), under crossed Nicols. 2.5x enlargement. (a) quartzite (QZITE); (b) mylonite (MRR); (c) granite (GNRR)



FIGURE 2: Micrographic images obtained by optical microscopy (transmitted light), under crossed Nicols, showing the deformation degrees of quartz. (a) degree 0; (b) degree 1; (c) degree 2; (d) degree 3; (e) degree 4



FIGURE 3: Expansions in the accelerated mortar bar test (NBR 15577-4; ASTM C 1260)



FIGURE 4: Relationship between the amount of dissolved silica and alkalinity reduction of the aggregates submitted to the ASTM C 289/2007 test



FIGURE 5: Amount of dissolved silica from quartz of the aggregates submitted to the stectrophotometer method (NBR 9878)