Pergamon

Cement and Concrete Research 31 (2001) 1449–1454

CEMENT AND CONCRETE RESEARCH

Diagnosis of the alkali-silica reactivity potential by means of digital image analysis of aggregate thin sections

D. García del Amo[a,]*, B. Calvo Pérez[b]

[a]Department of Analytical Sciences, Open University (UNED), Senda del Rey 9 Madrid 28040, Spain
[b]Department of Geological Engineering, Polytechnic University of Madrid, Madrid, Spain

Received 19 July 2000; accepted 22 June 2001

Abstract

Alkali-silica reaction (ASR) occurs between certain aggregate types and the alkalis in the pore solution of concrete and forms a silica gel. In the presence of free moisture, the gel will expand and cause cracking and differential movements in structures, as well as other deleterious effects such as reductions in freeze-thaw durability and in strengths properties of concrete. The microstructural characteristics of various Spanish alkali-reactive and potentially reactive rock types, used as concrete aggregate, and other Spanish rock types, with a possible potential reactivity, have been studied and quantified with petrographic techniques and methods of digital analysis of microscopic and macroscopic images. The quartz reactivity index (QRI) values have been obtained by multiplying the percentage of quartz in the aggregate by the specific surface area, which is characteristic of this mineral phase. These results have been related to alkali reactivity as measured by an accelerated mortar bar test. The study has demonstrated that the reduction of grain size in quartz produced by the metamorphic processes greatly enhances alkali reactivity by increasing the surface area of quartz grains available for the reaction.

Keywords: Alkali-aggregate reaction; Image analysis; Surface area; Grain size; Metamorphic quartz

1. Introduction

The durability of concrete can be affected by the type of aggregate that is used in its composition. The alkali-silica reaction (ASR) is a solid–liquid heterogeneous chemical reaction in which the solid phase is the aggregate and the liquid phase is an interstitial alkaline solution of concrete [1]. The result of this reaction is an alkali-calcium-silica gel capable of swelling by the absorption of water and exerting considerable pressures on its surroundings [2]. Within the ASRs, the study of the slow ASR (SASR) has suffered from insufficient international attention as opposed to the rapid reaction produced by opal and pseudocrystalline varieties of silica, particularly in modelling of ASR mechanism.

The subject of establishing relations among the microstructural properties of the potentially reactive aggregates and their expansion capacity in the presence of alkalis of the concrete pore solution has been researched in the last few years. Smith and Dunham [3] suggest that the reactivity of quartz with undulatory extinction is a function of a number of features of the quartz grains: undulatory extinction angle, texture and grain size. Kerrik and Hooton [4] showed that besides the effect of microcrystalline quartz, the reactivity of certain mylonites depended upon the degree of foliation (schistosity) in the rock. Some cataclastic rocks (especially mylonites) are found in structures in several areas exhibiting deleterious expansion due to AAR [5].

Thomson and Grattan-Bellew [6] point out that the main reactive component in deformed metamorphic rocks appears to be the microcrystalline quartz produced by a significant development of subgranulation but not complete recrystallisation. Shayan [7] reported that the alkali reactivity of deformed (metamorphosed) granitic rocks was related to the presence of microcrystalline quartz that resulted from recrystallisation of strained quartz. The first step in the search of a quantified relation between the size of the quartz grain of the rock and the expansiveness of that rock when used as an aggregate was presented by Wigum [8]. However, the work methodology is too slow and tedious.

* Corresponding author. Tel.: +34-91-398-72-85; fax: +34-91-398-66-97.

E-mail address: dgarcia@ccia.uned.es (D. García del Amo).

PII: S0008-8846(01)00586-5

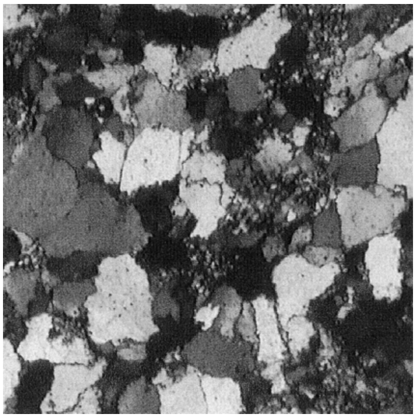

Fig. 1. Digital microscopic image (crossed Nicols).

The aim of this paper is to investigate the mineralogical variable, which controls the alkali-aggregate reaction, by means of petrographic techniques and digital analysis methods.

2. Materials and methods

The aggregates used for this study were typical aggregates of deteriorated concrete from Spanish dams (codes E, S, U and M) and aggregate of Spanish cement companies that was suspected of possible reactivity (code T).

In addition to traditional petrography, the calculation of quantitative parameters, such as percentage of quartz, its area, perimeter and specific surface area, was carried out by means of digital analysis of images (DAI) on a microscopic scale, as well as macroscopic when the grain size allowed it. The equipment used for taking images consisted of a HP Scan Jet 4C-T scanner for hand samples and a Leitz Laborlux 12 POL with a Javelin Chromachip IV CCD (752×582) pixels and a video camera for taking microscopic images. The process of digital analysis was carried out with the Q500MC equipment of Leica Cambridge, which incorporated the digitising card, the software for digital processing and the computer that coordinated all the system.

For the calculation of the percentages of the different mineral phases, digital images taken at true scale by means of a scanner were used. On the subject of the multispectrally segmented images, binary images were obtained where the percentage measurements corresponding to each mineral component were carried out.

To obtain images of the rock texture, the most appropriate technique is the polarisation optical microscope. However, the discrimination of fine quartz grains in microcrystalline rocks presents great difficulties in automatic segmentation methods because, in these images, the mineral grains do not stand out clearly from the background. Besides, added problems such as the same interference colour for different mineral grains, presence of very altered minerals, inclusions, etc. are also present. In order to avoid these problems we have resorted to two methods: either dyeing when this was sufficient and possible or selective choice of image of monomineralised images when the ultimate objective was exclusively the textural study of quartz present in the aggregate and not its quantification.

The quantified textural parameters are the percentage mineral phase by volume and the specific surface area of quartz. The percentage of the mineral phase is probably the classic parameter of quantification of mineral phases. Its calculation is well known from more than a century ago and is based on the principle that the percentage of a mineral phase in the image plane is the same as the percentage in the volume of that phase.

$$V_{\mathrm{v}} = \frac{\sum a(\mathrm{obj})}{\sum a(\mathrm{ref})} \tag{1}$$

where a(obj) is object area and a(ref) is reference area (Eq. (1)).

In the case of image analysis, the value of V_{v} of each mineral phase is given by Eq. (2):

$$V_{\mathrm{v}} = \frac{\sum \mathrm{pixels(phase)}}{\sum \mathrm{pixels(total)}} \tag{2}$$

The quantification of the boundary of each mineral phase in the rock is important since these zones characterise the textural and reactive properties of quartz. In the rock volume, a boundary corresponds to an area, which separates mineral grains. The parameter, which measures the surface area of a particular mineral grain per unit of its volume, is the specific surface area A_{v} and its unit is area/volume (L^{-1}).The Specific surface area is a concept, which is referred to in three-dimensional measurements, its true value for a mineral grain being (grain surface area)/(volume that it occupies). However, given that the measurements in the digital analysis are carried out in two dimensions, it is assumed that the value of the specific surface area will

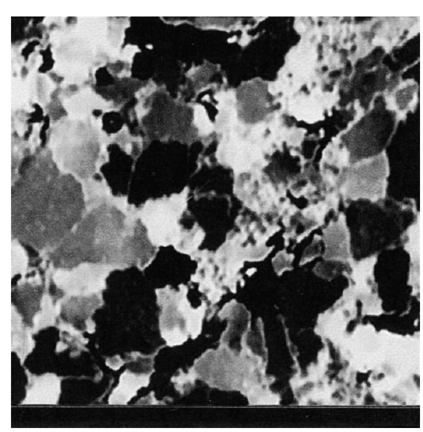

Fig. 2. Maximum intensity image.

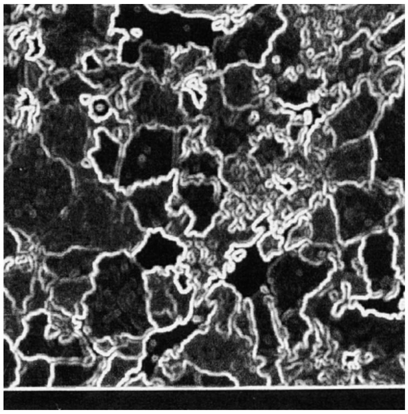

Fig. 3. Gradient image.

approach the value obtained by Underwood [9] for measurements in two dimensions:

$$A_v = \frac{4}{\pi}\frac{\sum B_i(\mathrm{obj})}{\sum A_i(\mathrm{ref})} \quad (3)$$

where B_i (object) is the length of the edge of the boundary grain and A_i (reference) is the area of the mineral grain. (The $4/\mu$ is introduced by stereologic principles.)

In order to obtain the value of specific surface area of a phase (in this case, quartz), A_i (reference) is determined as a reference area for the image dealt with (resolution of the pixel × area in pixels). The resolution of the pixel will vary with the magnification used in the petrographic microscope. Therefore, in the images taken with an objective of 2.5 magnification, the area of the image will be $(R\times512)\times(R\times512)$ ($R=3.32$ μm, the area of the image is 2889456.03 μm^2) and in those taken with an objective of 5 magnification, the area of the image will be $(R\times512)\times(R\times512)$ ($R=1.66$ μm, the area of the image is 722364.006 μm^2).

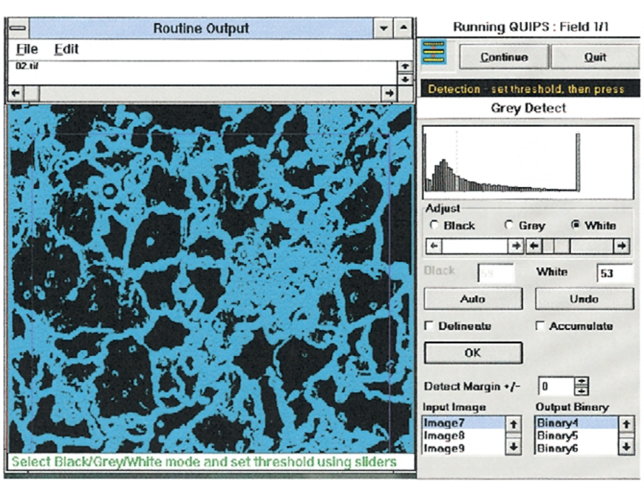

Fig. 4. First gradient binary image.

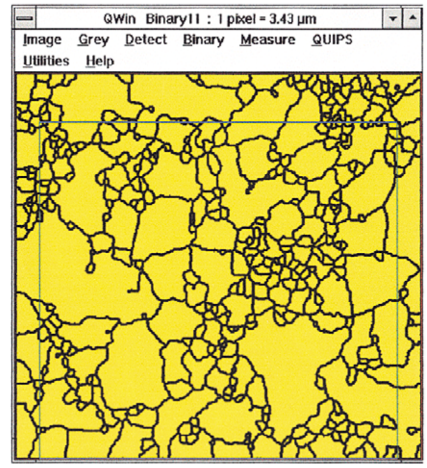

Fig. 5. Binary gradient image following skeletonization and dilation.

The perimeter and area parameters are obtained directly through standard operations of image analysis: the perimeter comes determined as the total edge length of the object calculated from the horizontal and vertical projections (number of horizontal and vertical chords in the object) with a discount factor for the number of corners and the object area as the total number of pixels detected within the object. In the case of the specific surface area, Eq. (3) has been introduced to calculate parameter A_v, which is of interest to the user. A_v is obtained through standard operations of image analysis.

Thus, the process followed for the characterisation of the microscopic samples of the selected rocks has been the following:

a. Images were taken in a 5 × 4 mesh on a thin section or predetermined (images of only quartz), according to the case, using a camera connected to the microscope. The chosen magnifications depend on the grain size of each rock, following the recommendations of Pirard [10]:

> The image has to be four times larger than the largest grain size of the crystals present.
> The smallest crystals have to be greater than 5 × 5 pixels (276 μm^2 at 2.5 × and 69 μm^2 at 5 × ; × : objective magnification).

Table 1
Data of characterisation of the quartz of the aggregates through digital analysis of images

Aggregate code	% Quartz	Specific surface area, quartz phase (μm^{-1})	% Quartz × specific surface area, quartz phase
E	43	0.051	2.193
U1	0.12	0.005	0.0006
U2	39.3	0.005	0.1965
M	86.5	0.033	2.8545
T	100	0.052	5.2

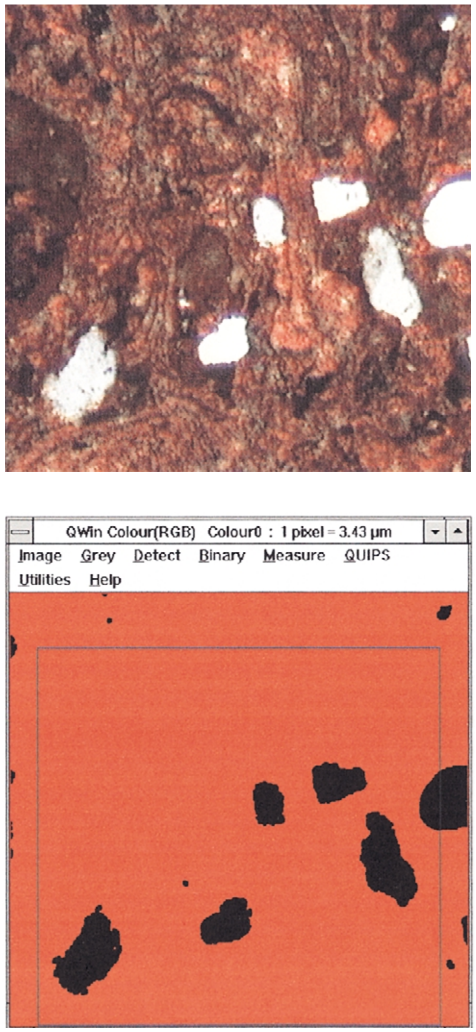

Fig. 6. (top) Digital microscopic image of quartz grains in dyeing calcite matrix. (bottom) Binary image to mask the crossed Nicols image.

b. For digital imaging of each lithological group, a working routine, with a program of digital analysis, was established in such a way that it could deal with many images (20 images per thin section). The programmed steps in the routine were:

1. Acquisition of the image and transformation to digital form (equally spaced pixels, which contain discrete grey level values; Fig. 1).
2. Image enhancement processes: consist of a collection of techniques that improve the visual appearance of an image to a form better suited for analysis by a machine. The result is an image with better visual contrast (Fig. 2).
3. Edge enhancement by gradient convolution: a grey level operation in which the output depends upon rate of change in grey level (Fig. 3).
4. Detection of the boundary: a grey value is selected and transferred into a binary image level threshold (Fig. 4).

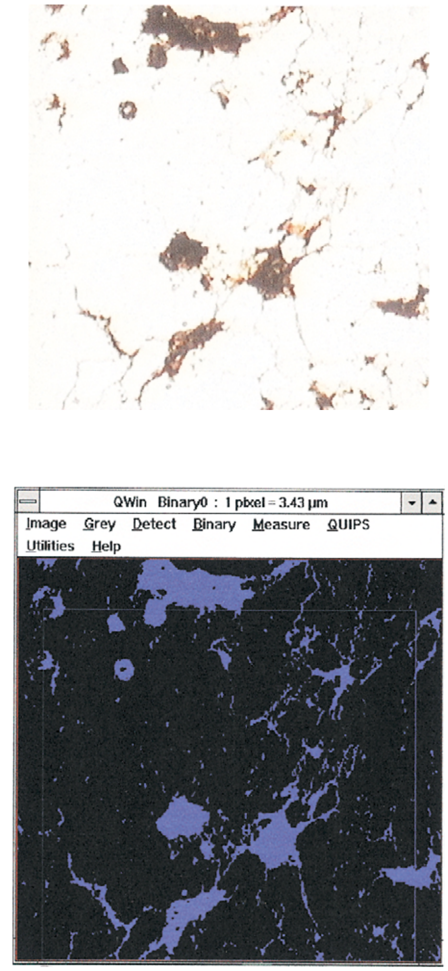

Fig. 7. (top) Digital microscopic image of quartz grains with iron oxides. (bottom) Binary image to mask the crossed Nicols image.

5. Morphological transformations of the gradient binary image (SKELETON, PRUNE, DILATE Y SKIZ; Fig. 5).
6. Carrying out the measurements.

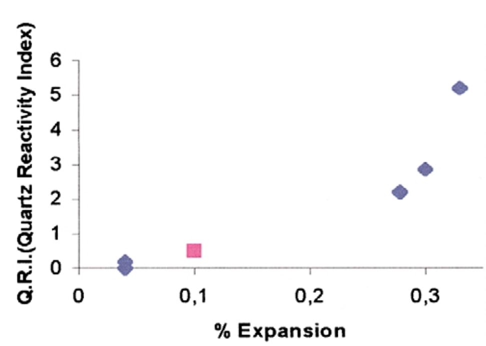

Fig. 8. Relation between the QRI of aggregates and 14-day mortar bar expansion of the same aggregates when tested according to ASTM C-1260.

Table 2
Results of the test for ultra-accelerated expansion of graduated test tubes of mortar in the aggregate problem

Aggregate code	Expansion for 14 days (%)	Expansion for 28 days (%)	Evaluation according to EHE-99 specifications (UNE 146 508 EX/98): 14 days [< 0.10% (nonreactive); 0.10–0.20% (follow-up for 28 days); > 0.20% (reactive)]	Evaluation according to EHE-99 specifications (UNE 146 508 EX/98): 28 days [0.10–0.20% (doubtful); > 0.20% (reactive)]
E	0.28	0.52	Reactive	Reactive
S	0.06	0.12	Nonreactive	Doubtful
U	0.04	0.09	Nonreactive	Nonreactive
M	0.30	0.53	Reactive	Reactive
T	0.33	0.55	Reactive	Reactive

Evaluation of the reactivity according to Spanish specifications.

c. The data files of the 20 images analysed for each thin section are exported to a calculation program in order to handle the data statistically. The quartz reactivity index (QRI) was calculated by multiplying the percentage of quartz in the aggregate by the specific surface area A_v [Eq. (3)].

3. Results and discussion

The results of the statistical treatment of the data for the 20 images analysed for each thin section are shown in Table 1. The constituent rocks of the selected aggregates are metamorphic rocks and/or contain forms of strained quartz. The deformation follows tectonic activities, developed in the region or locally, which lead to ductile or fragile deformation. These processes are interpreted as an increase of the chemical potential and of the instability of the mineral (quartz) by intercrystalline, microfissures, reduction of the grain size or increase of the specific surface area, except in cases in which further intense recrystallisation (posttectonic or postdeformation) has occurred such that the crystalline morphology has been rebalanced and the chemical potential reduced.

Conventional techniques of mineralogical quantification used nowadays, such as point counting, X-ray diffraction analysis with standards, etc., are much slower and more tedious than the proposed digital analysis technique. The digital analysis at microscopic level by means of the polarising microscope requires a previous treatment for discrimination of the quartz phase through selective techniques of staining or by means of selective search for monomineralised phases. This is because quartz, under parallel Nicol prisms, could be confused with various minerals, particularly with the feldspars (similar colour and relief). Also, under crossed Nicol prisms, quartz presents some interference colours, which are very similar to those of other minerals. When the matrix in which quartz grains are found is susceptible to staining (for example, calcite in aggregate code U), it is viewed after the staining process with parallel Nicol prisms and the staining colour detected is converted to a binary image. The binary image of the stained section is subtracted from the crossed Nicols image, leaving only the image of quartz grains (Fig. 6).

If the quartz mineral phase shares paragenesis with coloured minerals (for example, iron oxides in aggregate code M), the procedure is identical to the previous case, using the proper natural colour of the minerals (Fig. 7). When the aggregate entirely consists of the quartz phase (aggregate code T), the whole image is used for the analysis. In cases in which the mineral surrounding the quartz phase is not easily distinguished, it is necessary to select images taken under crossed Nicols in which quartz is the only phase.

A quick and reliable test for the assessment of the potential alkali-aggregate reactivity is the accelerated mortar bars test [11]. Table 2 lists the QRI values and mortar expansion test results for the aggregates studied. An exponential relationship was noted when the 14-day mortar bar expansion results were plotted against the QRI (Fig. 8).

Based on the relationship between QRI and expansion, it appears that aggregates with QRI values exceeding 0.5 would cause mortar bar expansions exceeding the 0.1% limit of the test method, indicating alkali reactivity of those aggregates. The highest values of mortar bar expansion correspond to aggregates originating from rocks subjected to a dynamic metamorphism. For the final adjustment of the curve that relates these parameters, the potential reactivity of these grains or subgrains of quartz has to be compared to that of other grains of quartz of similar size but originating from other types of processes (cataclastic, gneiss and blastomylonite).

This correlation is based on very limited data and the pattern may change if many more aggregates are included in the analysis. Therefore, it would be necessary to test a much larger range of rocks in order to develop a more reliable correlation before suggesting that the petrographic image analysis can replace the accelerated test.

4. Conclusions

Although the prevention of the reaction depends on many considerations, the main one is the evaluation of the aggregate composition and of its behaviour in the concrete prior to the selection of the final aggregate. This constitutes one of the key points relative to the future stability of the concrete structure.

ASR of slow character appears to be associated with microcrystalline quartz, in particular with the appearance and development of subgranulation. The value of the percentage of quartz in the aggregate, multiplied by the specific surface area, called the QRI appears to be closely related to its expansiveness. Aggregates with values greater than 0.5 would exceed the mortar bar expansion limit allowed in ASTM C1260. However, a larger range of rock types needs to be tested to confirm this correspondence.

Acknowledgments

This work has been funded by UPM (Politecnic University of Madrid) (I + D multidisciplinary program).

References

[1] L.S. Dent Glasser, N. Kataoka, The chemistry of alkali-aggregate reaction, Proc. 5th Conf. Alkali-Aggregate Reaction, Cape Town, 1981, Paper S 252/23.
[2] S. Diamond, ASR — another look at mechanisms, Proc. 8th Conf. Alkali-Aggregate Reaction, Elsevier, London, 1989, pp. 83–94.
[3] A.S. Smith, A.C. Dunham, Alkali-silica reaction in concrete: A survey of undulatory extinction of quartz in granites and sandstones. Transport Research Laboratory (TRL). Contractor Report, 291. Department of Transport, 1992.
[4] D.M. Kerrick, R.Dd. Hooton, ASR of concrete aggregate quarried from a fault zone: Results and petrographic interpretation of accelerated mortar bar test, Cem. Concr. Res. 22 (1992) 949–960.
[5] V. Jensen, Diagnosis of Norwegian concrete damaged by alkali aggregate reaction of slow/late type, Proc. Fourth Euroseminar on Microsc. App. Build. Mat. SP (Swedish National Testing, Sweden), 1993: 15.
[6] M.L. Thomson, P.E. Grattan-Bellew, Anatomy of a porphyroblastic schist: Alkali-silica reactivity, Eng. Geol. 35 (1993) 81–91.
[7] A. Shayan, Alkali-reactivity of deformed granitic rocks: A case study, Cem. Concr. Res. 23 (25) (1992) 1229–1236.
[8] B.J. Wigum, A classification of Norwegian cataclastic for alkali-reactivity, Proc. 10th Int. Conf. Alkali-Aggregate Reaction in Concrete, CSIRO, Division of Building Construction and Engineering, Melbourne, Australia, 758–766.
[9] E.E. Underwood, Quantitative Stereology, Addison Wesley, USA and Canada, 1970, pp. 23–47.
[10] E. Pirard, Colour Image Analysis in Mineralography COM-IMA. Short Course "Modern Approaches to Ore and Environmental Mineralogy", Porto, September, 1997.
[11] ASTM, Potential alkali reactivity of aggregates (Mortar-bar method). ASTM C1260, 1994.