

PARTNER Project post documentation study – condition assessment of field exposure site cubes (Part II – results of microstructural analyses)

Isabel Fernandes ⁽¹⁾, Andreas Leemann ⁽²⁾, Benoit Fournier ⁽³⁾, Esperanza Menéndez ⁽⁴⁾, Jan Lindgård ⁽⁵⁾, Ingmar Borchers ⁽⁶⁾, João Custódio ⁽⁷⁾

(1) *Department of Geology, University of Lisbon, Lisbon, Portugal, mifernandes@fc.ul.pt*

(2) *Swiss Federal Laboratories for Materials Testing and Research (Empa), Dübendorf, Switzerland, andreas.leemann@empa.ch*

(3) *Laval University, Québec City, Canada, benoit.fournier@ggl.ulaval.ca*

(4) *Institute Eduardo Torroja of Construction Science (IETcc-CSIC), Madrid, Spain, emm@ietcc.csic.es*

(5) *SINTEF, Trondheim, Norway, jan.lindgard@sintef.no*

(6) *VDZ gGmbH, Düsseldorf, Germany, ingmar.borchers@vdz-online.de*

(7) *National Laboratory for Civil Engineering (LNEC), Lisbon, Portugal, jcustodio@lnec.pt*

Extended Abstract

The PARTNER project was developed from 2002 to 2006 aiming to establish a unified test procedure for evaluating the potential alkali-reactivity of aggregates in Europe. In the scope of the project, 100 concrete cubes 300×300×300 mm³ in size (two from each mixture) using 13 aggregate combinations, as coarse and fine fractions, reactive coarse aggregate with a non-reactive fine or non-reactive coarse with reactive fine fraction, were produced to be exposed outdoors at eight locations across Europe. A cement content of 440 kg/m³ and a water-to-cement-ratio (w/c) of 0.50 were used for concrete production. The cement (CEM I 42.5 R) had a Na₂O-eqv. of 1.26 mass-%. In 2017, cores were extracted from 21 selected cubes for further investigation within the framework of RILEM TC 258-AAA.

The present work aims at relating the microstructural characteristics of the ASR-affected concrete to the block expansion recorded over the years. It investigates possible effects of aggregate type and exposure conditions on the degree of deterioration and on the composition of the ASR products formed. Damage Rating Index (DRI), on eighteen cubes, is used to assess concrete damage through quantification of ASR-related features. Optical microscopy, carried out on the twenty-one cubes, extends on the capabilities of the DRI by providing an increased resolution used for a qualitative damage assessment (QDA). Scanning electron microscopy (SEM), combined with energy-dispersive X-ray spectroscopy (EDS), is used to determine the chemical composition of ASR products formed within the different reactive aggregates. Raman microscopy is applied to further characterize the crystalline ASR products.

DRI is a microscopic petrographic method performed with the use of a stereo-binocular microscope (about 15x magnification) where damage features generally associated with ASR are counted through a 100 mm² grid drawn on the surface of a polished section of concrete. The number of counts corresponding to each type of petrographic features (i.e. cracking within the aggregate particles, with/without reaction products) is then multiplied by selected weighting factors.

The analysis performed under petrographic microscope allowed the definition of the cracking pattern, the identification of the aggregates/phases involved in ASR and to visualize the texture of the ASR products formed. This qualitative analysis was carried out in line with the five classes established for DRI, namely negligible, marginal, moderate, high and very high, based on the frequency and size of the deterioration features: cracks in the cement paste, cracks crossing coarse aggregate particles, cracks cutting fine aggregate particles, de-bonded aggregate particles, presence of ASR products in the cracks, in interfaces and in voids. The classes 1 to 5 were obtained qualitatively by assessing five sectors in each thin section and assuming a percentage of damage to the respective field of view. The final class results from the sum in the frequency of the features listed above.

SEM was used for analysis in the high vacuum mode with an acceleration voltage of 12 kV, a spot size of 4.5 and a beam current of 90-100 µA. A detector (80 mm²) and Energy software with ZAF correction were used for the EDS analysis. In every studied concrete, the ASR product was analyzed in 4-6 individual aggregate particles using a total of between 110 and 300 point analyses per concrete.

A Raman microscope was used for spectra acquisition. The laser wavelength was 532 nm, operating power 20 mW and lens magnification 50x. The Raman spectra give a "fingerprint" to identify ASR products of identical structure. Additionally, some of the main bands can be assigned to specific structural units.

All cubes with highly reactive aggregates reached DRI values > 1800, while moderately reactive aggregates showed DRI values in the range 700 to 1400. Even the cubes incorporating the non-reactive limestone displayed damage resulting in a DRI of 703 and 834, which leads to a damage classification of "very high". The main contribution to the damage is by cracks in the cement paste and, to a lesser degree, by cracks in the aggregate particles. The explanation for the observed cracking are traces of highly reactive chert, as identified by SEM in the otherwise non-reactive aggregate.

Optical microscopy reveals that crack patterns differ from one aggregate type to another, depending for example on the presence or absence of foliation or cleavage. Cracks are much more frequent in very fine-grained lithology but are wider in coarse-grained aggregates. Ettringite formation is abundant in cracks within the cement paste and in open interfaces between aggregate particles and cement paste of the majority of concrete mixtures; however, this was not considered in the damage assessment. Formation of ettringite was attributed to exposure to high temperatures caused by solar radiation. Cracks in the aggregate particles are completely or partly filled with ASR products. Approaching the edge of the aggregate particle, there is a transition from crystalline ASR products to structure-less ASR products.

The composition of the ASR products formed in aggregate particles vary relatively little in spite of the large differences in the composition of the aggregates. The climate at the exposure site has limited impact, as for a specific aggregate the composition of the ASR products is somewhat similar for the samples stored in different locations. The variations of the atomic Ca/Si-ratio are smaller than that of the atomic (Na+K)/Si- and Na/K-ratios. Depending on the feldspar present, this not only leads to an increased availability of alkalis but to a dominance of either sodium or potassium. However, the small variations in composition of the ASR products indicate that they are a stable phase in the chemical environment present in the interior of the aggregate. The values correspond to those reported in other studies that focused on the ASR products formed in aggregate particles. In contrast to the ASR products present in aggregate particles, ASR products extending into the cement paste take up calcium and release alkalis, leading to a great variation in composition.

The Raman spectra show that most bands are identical, independently of aggregate types and exposure site locations. The majority of the crystalline ASR products formed have the same crystal structure. However, in a few cases the Raman spectra clearly indicate the presence of shlykovite. The concrete showing the presence of shlykovite correspond to blocks exposed to very high temperatures in summertime. Concrete exposed to sun radiation may reach a temperature up to 60°C, which enables the formation of shlykovite.

The following conclusions can be drawn from the results of this study:

- DRI shows a weak relation to cube expansion ($R^2 = 0.61$). The differences in the ratio between cracks in aggregate particles and cracks in the cement paste indicate aggregate-specific cracking characteristics.
- QDA performed on thin sections correlates better with cube expansion ($R^2 = 0.81$).
- Crack characteristics are aggregate-specific and depend for example on the presence or absence of foliation or cleavage.
- The amount of ASR products formed displays no direct relation to cube expansion.
- The chemical composition of ASR products formed in aggregate particles is not influenced by the type of aggregate or by the climate of the exposure site.
- The Raman spectra of the majority of crystalline ASR products formed in the cubes are identical to those formed in concrete structures. However, in some of the cubes stored in warmer climate, minor amounts of crystalline ASR product show identical spectra those of crystalline products formed in the CPT and to synthesized shlykovite.

Keywords: PARTNER project; RILEM; concrete expansion; field exposure sites; microstructural analysis.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the financial support of FCT through the projects UIDB/50019/2020 – IDL, IF/00595/2015/CP1326/CT0001 and the Norwegian research project 236661 (mainly funded by the Norwegian Research Council).

This article was submitted for publication in a scientific journal.