A ROBUST TESTING PROTOCOL FOR THE ASSESSMENT OF ASR REACTIVITY OF CONCRETE

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Abstract

A number of ASR testing methods have been proposed over years but are generally found to be lacking. The use of blended cements makes the picture more complicated, as it was widely shown that mechanical and chemical properties of the concrete influence ASR behaviour.

The Swiss Standard SIA 2042 performance test is analysed here and some inconsistencies are highlighted. In parallel, some inputs for a new protocol are proposed, based upon the acquired experience of the authors' laboratory and the research community. The new procedure aims to respect the chemical and mechanical properties of the tested concrete through the storage of samples in a solution matching the pore solution of the mix and a curing of the samples during 28 days before testing.

Preliminary results are promising, showing a constant expansion rate over more than 400 days. The parameters of the test procedure have to be optimised and results have to be further compared with other test procedures in order to propose a robust testing protocol for ASR performance testing of blended cement concretes.

Keywords: ASR assessment, aggregates, pore solution

1 INTRODUCTION

Since Stanton's landmark paper in 1940 [1] for the California road administration, the alkali-silica reaction has been observed worldwide. This reaction is the first non-reinforcement-related cause of concrete degradation in the world and is of particular concern for owners and managers of large mass concrete structures such as dams. As the reaction is extremely slow in the field - indeed, it can take up to 30 years to develop - the development of testing methods has focussed on accelerating the reaction. Factors which are necessary for the reaction to occur are well understood: the availability of water as the main medium for transport as well as its role as reactant, the availability of alkali ions in sufficient quantities, and of course the reactive aggregates subject to the test. Several factors can change the kinetic of the reaction:

- alkali concentration [2-3]
- temperature [4-5]
- aggregate particle size distribution (PSD) [6-7-8]
- Hydration degree of the sample
- geometry of the sample

As it is now known, the wrong use of these parameters can lead to several false positives or false negative in the assessment of ASR reactivity. All these factors must be considered for the design of a test, but the goals of the test will further define how the parameters are chosen. Further, the hydration degree of the sample can be a concern in terms of representativity of field conditions.

The validation of predictions of a concrete reactivity, other than waiting for the field outcome, must rely on chemical and mechanical understanding of the reaction. There has been considerable progress in recent years in understanding the chemical and micro-mechanical underpinning of the reaction, and the qualitative aspects of the results from accelerated tests can now be better understood.

The normative testing method used in Switzerland (SIA M 2042) for the prediction of ASR performance of concretes was developed on the base of old assessment methods (AFNOR NF P 18-454). After 24 hours, concrete prisms (7x7x28 cm) are conditioned at 60°C in sealed containers over water (RH 100%) during at least 5 months. The procedure uses a vapour cure which may cause accelerated leaching.

Concretes of the SIA M 2042 are cast with additional alkali in their mixing water to try to palliate the effect of leaching. This may not be suitable in the case of mixes containing SCMs as the latter are activated by the alkalis [10].

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Concrete prisms are put in reactor after only 24 hours of curing. This may beget two main error sources:

- Concrete with blended cements have considerable changes in their pore solution composition known to have an important effect on ASR control [11, 12, 18] compared to standard OPC concrete used as the testing procedure was developed: the pore solution of the blended paste is not at equilibrium at the start of the test.
- Creep is an important relaxation factor in concrete [13] and results in affected structures to be able to undergo significant deformations with only little damage at the material level. Pastes with SCMs may have different mechanical properties than OPC pastes [14], and should, beyond the chemical effects of reduced reaction, exhibit different expansions simply from their different modulus. The mechanical properties of the blended paste are not developed at the start of the test.

Based on the upon mentioned criteria and on the experience of results obtained with the SIA M 2042 performance tests, we can say that the reliability of the test method seems to be bad, at least for recent concrete with blended cements. The formation of a plateau systematically in the range of about 100 to 200 days give strong suspicions of leaching of the system.

This study shows the effects of the mentioned parameters (conditioning environment and temperature, curing time) through the modification of the SIA M 2042 performance test. The study aims to find a reliable and robust ASR assessment method. The obtained results still have to be confirmed with several aggregates and blended cements.

2 MATERIALS AND METHODS

2.1 General

The aim of the experimental method is to point out every parameter leading to unreliable expansion results, and to propose a robust testing procedure for the assessment of alkali reactivity of concrete. This tests will able to compare the effects of:

- temperature
- testing conditioning
- curing time

Over this batches, the following tests were done:

- Expansion measurements
- SEM microstructure analysis (reacted fraction estimation)
- Measurement of the ionic concentration at different times of the conditioning solutions.

2.2 Materials and mix designs

Concrete sample preparation

The samples (7x7x28 cm) are cast following the SIA M 2042 procedure. The aggregates are reactive aggregates from the Italian Alps (gneiss, micaschist). Following the procedure, additional sodium hydroxide has to be added to the batch water to reach $0.8 \text{ Na}_2\text{O}_{eq}$ concentration.

Additional samples (4x4x16 cm) were cast to realize microscopic analyses. Sealed bottles were filled to extract pore solution.

The used cement is a Portland cement, type CEM I 42.5R (Holcim PUR 4).

2.3 Methods for assessment and analysis

Expansion tests

The different studied systems are resumed in the table 1.

- 9006-9008: following the SIA M 2042 procedure (cooled during 24 hours at 20°C before measuring)
- 9000-9002: following the SIA M 2042 procedure but at 38°C instead of 60°C
- 8006-8008: following the SIA M 2042 procedure but cured at 20°C during 28 days before testing instead of 24 hours (20°C, 95%RH, covered to avoid water leaching)
- 4006-4008: cured 28 days at 20°C and then immersed in 2 litres of a pore solution similar soak solution at 60°C. The pore solution analyses is described in the next paragraph.
- 4000-4002: cured 28 days at 20°C and then immersed in 2 litres of a pore solution similar soak solution at 38°C.

Pore solution extraction and analysis

A batch of concrete was cast in 200 ml sealed containers and stored at 20 °C during 28 days. They were pressed in a pore solution extraction device in order to extract the pore solution (following [9]): The sealed sample is unmoulded and put into the extraction device (5 cm diameter piston in a metallic cell and a container at the bottom to retrieve solution). The sample is loaded with a speed of 100N/min until 1500N.

The load is maintained during 10 minutes. The ionic concentration of the retrieved solution was analysed by inductive coupled plasma (ICP).

ICP measurement of the bottom water of the vapour system reactors (series 9000) at several times were also realised to get information about the evolution of the leaching of concrete samples.

Scanning electron microscopy (SEM)

The mortars samples were cut at different times, immersed in isopropanol, to remove water and stop the hydration. They were then dried and impregnated with an epoxy resin. The samples were mechanically polished down to 1 μ m. A high-resolution montage of 144 pictures at a magnification of 400 × was acquired to analyse a representative amount of aggregates, corresponding approximately to 350 pieces. The amount of degradation in the aggregates were visually compared. Quantification of the reacted fraction following the procedure proposed by Ben Haha et al. [10] are in progress.

3 RESULTS

Expansion tests

Expansion curves for the various systems are shown in Fig. 1. It is seen that:

- the standard SIA M 2042 testing procedure (serie 9006-9008) leads to a plateau after about 200 days as often observed.
- The system cured during 28 days and put in the same conditions as the SIA M 2042 procedure (serie 8006-8008) shows no significant expansion until 600 days.
- The system at 38°C and put in the same conditions as the SIA M 2042 (serie 9000-9002) shows no expansion until 450 days.
- The system cured during 28 days and put in the pore solution similar solution at 60°C (serie 4006-4008) present an expansion at 420 days 3 times higher than the 9006-9008 serie at the same time. Moreover, the expansion rate is constant over the entire measurement period (50 to 430 days, rate: about 0.24 m/mm/month).
- The system cured during 28 days and put in the pore solution similar solution at 38°C (serie 4000-4002) starts to expand at approximately 250 days and seems to have a low but constant expansion rate until 420 days.

Pore solution analysis

The results of the concrete pore solution after 28 days at 20°C are presented in the table 2. These results were used to create the conditioning solution of the serie 4000 for sodium and potassium concentration.

Water analyses of vapour curing systems (serie 9000)

The results of ICP analyses of the reactors water are resumed in the table 3. The figure 2 shows the leaching observed for both vapour systems at 38°C and 60°C. After 520 days, about 88% of the sodium ions and 35% of the potassium ions present at the origin in the concrete prisms are leached. Surprisingly, 38°C systems leach about as much alkalis as at 60°C. This could be due to the fact that leaching is mainly present during the cooling and heating process of measurement protocol. Condensation phenomenon may leach importantly during rapid temperature changes.

Scanning electron microscopy

Figure 3 and 4 show the difference of degradation of reactive aggregates between vapour systems at 60°C ((a) serie 9006-9008, SIA 2042) and pore solution systems at 60°C ((b) Serie 4006-4008). The amount of reacted fraction is clearly higher in the serie 4006-4008. It shows that the vapour system stops to react despite the presence of reactive phases in aggregates.

4 **DISCUSSION**

The combination of expansion curves, solution analyses and microscopic analysis of the microstructure, could show that the Swiss standard ASR performance test SIA M 2042 is unreliable for ASR assessment. This procedure is subject to intense leaching of its pore solution that leads to an expansion plateau before the reaction of the entire reactive phases of aggregates, as compared by SEM with the concretes in pore solutions. The observed expansion plateau corresponds to a drastic change in the pore solution of the concrete due to the leaching of alkalis. It can be extrapolated that in case of concretes with blended cements, the pore solution - that was widely shown to be one of the key parameter for the control of ASR – will not be preserved during the test duration. This will mislead the diagnostic of ASR behaviour.

The proposed protocol (serie 4000) is original notably in the choice of the alkali concentration of the curing solution. This concentration has been chosen to be close to the cement pore solution. It is not possible to have a solution which is exactly the same as it is variable through the hydration process of the cement. To minimise the effect of this variation, we used the results of Lothenbach and colleagues [17] as well as these of Chappex and Scrivener [18], who showed that the concentration largely stabilises after one month of cure. Two types of alkalis are present in the cement pore solution, sodium and potassium. These are known to have different effects in terms of the measured expansion [19], however, their interactions, if any, are not clear. In case of aluminium rich pore solution of certain blended systems, aluminium could also be added to the testing solution. This is at the time further investigated.

This protocol also has the advantage to respect the mechanical properties of the paste, by curing the samples during 28 days at 20°C before testing. As the expansion of ASR is largely due to the opening of cracks in the microstructure due to the pressure involved by the formation of reaction product, the test protocol should reproduce the relative strengths of the phases, aggregate and paste, as they are in the field. This means in particular that tests where the reaction of the aggregate is very rapid may develop unrealistic damage patterns if the paste has not hardened sufficiently when samples are put into the reactor. A mechanistic simulation of such kinetic effects can be found in [13]. It is expected that this factor be particularly important in the case of blends which develop strength slower than PC.

As already well known, expansion is temperature dependent. It was also observed in this study that systems at 38°C expand less than systems at 60°C.

5 CONCLUSIONS

In this study, some problems with the Swiss standard M 2042 performance testing procedure could be identified. It could be shown that the evolution of the pore solution composition (leaching), is one of the main reason for the flattening of the expansion after only few weeks. A new testing procedure is in course of development and was proposed. The values chosen for this test method are justified using the current knowledge in terms of the chemistry of the reaction as well as the micro-mechanics. It aims to respect the mechanical and chemical properties of pastes (especially for recent blended cements). This testing procedure consist of the following protocol:

- Pore solution extraction and analysis of the concrete cured during 28 days at 20°C in sealed containers.
- Samples preparation following the SIA 2042 procedure
- In parallel, curing of concrete samples (7x7x28 mm) during 28 days at 20°C in 95% RH (covered to avoid leaching due to water drops)
- After 28 days, start of the measurements and accelerated conditions for ASR: concrete samples are put in soak solutions similar to the analysed pore solutions at 28 days at 38° and 60°C (2 different protocols)
- The right temperature (between 38°C and 60°C) still has to be defined.

This promising procedure has to be further validated with a number of different aggregates, fast- and slow-reacting alike and several blended cements.

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TABLE 1: Overview of the studied systems.

Temperature	38°C		60°C		
Curing time	1d	28d	1d	28d	
Over water	9000-9002		9006-9008 (SIA M 2042)	8006-8008	
In pore solution (2L)		4000-4002		4006-4008	

TABLE 2: Concentration of the concrete pore solution after 28 days at 20°C (sealed bottles).

	Na ⁺ [mmol/l]	K+ [mmol/l]
Pore solution (28 d at 20°C)	275	152

TABLE 3: Alkali concentration of the water at the bottom of the reactors (vapour systems) after 150 and 530 days.

sample	Al mmol/1		K mmol/1		Na mmol/L	
	150 d	530 d	150 d	530 d	150 d	530 d
9000 - 9002	0.18	0	5.81	36.57	101.78	196.17
9006 - 9008	0.25	0.22	19.18	41.95	84.82	194.43



FIGURE 1: Expansion over time of the studied system (cf. table 1).



FIGURE 2: Leaching of the concrete prisms in vapour systems (serie 9000) at 38°C and 60°C.



(a)





(c) FIGURE 3: 3 a) Reactive concrete sample after 360 days in vapour curing at 60°C (9006-9008). Yellow zones: picture b) reactive sand with low reacted fraction, no cracks. picture c) reactive sand with low reacted fraction, no cracks.





(c) FIGURE 4: Reactive concrete sample after 360 days in pore solution curing at 60°C (4006-4008), Yellow zones: picture b) reactive sands with high reacted fraction, crack trough the paste and some gel accumulated in a pore. picture c) two reactive sands: high reacted fraction, presence of cracks. Some reaction product is visible in the cracks (few gel because of the polishing process: non-calcified gel is easily removed).