

Impact of storage conditions on expansion due to ASR

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Abstract

A number of ASR testing methods have been proposed over the 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.

Swiss SIA 2042 and American ASTM C1293-18 standards are presented down below, and some inconsistencies are highlighted. In order to improve actual used methods, different storage conditions are presented, based upon the acquired experience of the authors' laboratory and the research community. The aim is 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 600 days. The parameters of the test procedure need to be optimised. Results have to be further compared with other test procedures in order to have a better overview of the impact of storage conditions on expansion due to ASR.

Keywords: aggregates; ASR testing regime; pore solution

1. INTRODUCTION

Since Stanton's landmark paper published 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 known: 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. So, all following factors can have an impact on the kinetics of the reaction:

- alkali concentration [1][2]
- temperature [3][4]
- aggregate particle size distribution (PSD) [5][6][7]
- hydration degree of the sample
- geometry of the sample

As it is now well known, the improper use of these parameters can lead to several false positives or false negatives in the assessment of ASR reactivity. All these factors have to be considered for the test design, but in any case, it's the purpose of the test to further define how the parameters should be 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's reactivity, other than waiting for the field outcome, must only rely on chemical and mechanical understanding of the reaction.

There has been considerable progress over 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). The aim

of these standards is to control the expansion of a boosted concrete (0.8% Na₂O_{eq} by mass of cement for SIA 2042 and 1.25% Na₂O_{eq} for ASTM C1293-6).

For the Swiss standard, after 24 hours, concrete prisms (7x7x28 cm) are conditioned at 60°C in sealed containers over water (RH 100%) for at least 5 months. An expansion of 0.02% after 5 months or 0.03% after 1 year are defined as the limit of the concrete reactivity. American ASTM C1293 standards following the same procedure. After 24 hours, cross section 75 mm² prisms are stored at 38°C. For these specimens, the expansion limit is 0.04%.

The actual procedure uses a vapour cure which may cause accelerated leaching.

Both standards require that concrete be casted with additional alkali in their mixing water to try to palliate this effect of leaching. In the case of mixes containing SCMs, because the latter are activated by the alkali, this may not be suitable [9].

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

- Concrete with blended cements have considerable changes in their pore solution composition – well known to have an important effect on ASR control [10][11][12] – compared to standard OPC concrete used when the testing procedure was developed: the pore solution of the blended paste is not at equilibrium when the tests start.

- Creep is an important relaxation factor in concrete [13] and results in affected structures to be able to undergo significant deformations with only little material level damages. 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 due to their different modulus.

The mechanical properties of the blended paste are not developed before starting the test.

Based on the aforementioned criteria and on the experience of results obtained with the SIA M 2042 performance tests, we can say that the test method seems to be unreliable, 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 system leaching.

This study shows the effects of the mentioned parameters (conditioning environment, temperature, and curing time).

The study aims is to have a better overview of the impact of storage conditions on expansion due to ASR.

Of course, the obtained results still have to be confirmed with blended cements.

2. MATERIALS AND METHODS

2.1 General

The aim of this study is to have a better overview of the impact of storage conditions on expansion due to ASR. These tests will able to compare the effects of:

- temperature
- testing conditioning
- curing time

Over these 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 or ASTM C1293 procedure. The chosen 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 Na₂O_{eq} concentration. Because the main goal of this study is to observe the effect of the storage conditions, we decided to not add NaOH in any mixes. With this choice, we will be able to observe the behaviour of the real mix design in various storage conditions.

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). The mix design use for fabrication of concrete is given in table 1.1.

Table 1.1: Mix design with Dmax 22.4 mm

Cement (CEM I 42R)	410	[kg/m ³]
Water	185	[l/m ³]
W/C	0.45	[-]
Aggregates 0/22.4	1770	[kg/m ³]

2.3 Methods for assessment and analysis

Expansion tests

The different studied systems are resumed in the table 2.2.

Table 2.2: Overview of the studied systems

	60°C (T60)		38°C (T38)	
	1 d. cure (C1)	28 d. cure (C28)	1 d. cure (C1)	28 d. cure (C28)
Water Vapour (WV)	WV-C1-T60	WV-C28-T60	WV-C1-T38	WV-C28-T38
Pore solution (PS)	PS-C1-T60	PS-C28-T60	PS-C1-T38	PS-C28-T38

- SIA M 2042 procedure (cooled during 24 hours at 20°C before measuring)
- Temperature as request by ASTM C1293-06 procedure (38°C)
- 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)
 - o Cured 28 days at 20°C and then immersed in 2 litres of a simplified pore solution similar soak solution at 60°C. Extraction and analysis method of the pore solution are described in the next paragraphs.
 - o Cured 28 days at 20°C and then immersed in 2 litres of a simplified pore solution similar soak solution at 38°C.

Pore solution extraction and analysis

14 days before initial test, the amount of concrete, defined by the Swiss standard, is cast and 6 plastic bottles (ø 55 mm, height 90 mm) are filled. After 28 days, pore solution is extracted using "Cold Water Extraction method's (CWE)" [8]. This method can be summarized in five main steps:

- Measure porosity of three first samples;
- Crush 3 samples;
- Mix 1:1 ratio of powder and de-ionized water for 15 minutes;
- Extract solution and analyse it by inductively coupled plasma optical emission spectrometry (ICP-OES);
- Create a simplified pore solution.

ICP-OES analysis on deionised water during Swiss standard test

ICP-OES measurements are done on the water placed under the sample tested in the Swiss standard storage conditions. The aim is to get information about the evolution of the leaching of concrete samples.

Scanning electron microscopy (SEM)

The mortar samples are cut at different times depending of swelling observed on expansion samples. They are immersed in isopropanol to remove water and stop the hydration. Thereafter, they are dried and impregnated with an epoxy resin. Subsequently, these samples are mechanically polished down to 1 μm . A high-resolution stitching of 144 pictures at a magnification of 400 \times is realized in order to observe the most representative stitching of aggregates, more or less 350 pieces per analysis. The amount of degradation in the aggregates is visually compared. Quantification of the reacted fraction following the procedure proposed by Ben Haha et al. [10] is in progress.

3. RESULTS

Expansion tests

Expansion curves for the various systems are shown in Fig. 3.1 to 3.5. It is seen that:

Figure 3.1:

- For samples stored in water vapour condition at 60°C, expansion leads to a plateau after about 200 days. All aggregates used during this study show this same expansion behaviour.
- All mix designs tested present expansion above the limit after 5 months, defined by the standard ($> 0.3 \text{ mm/m}$).

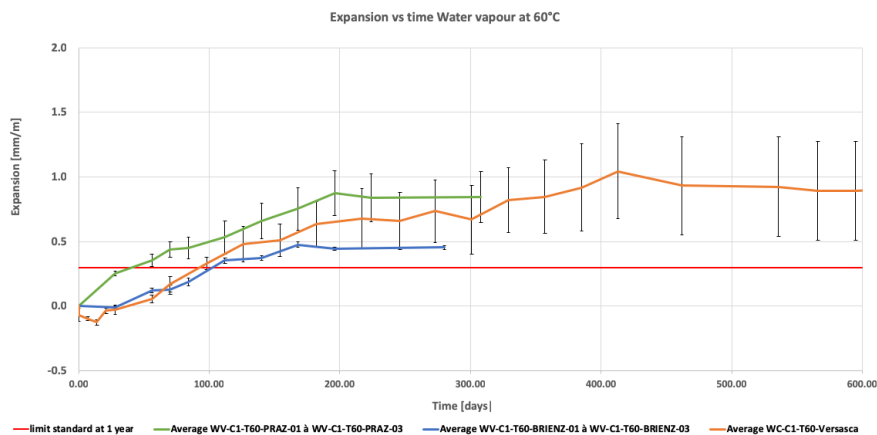


Figure 3.1: Expansion over time following SIA 2042 standard - 3 different aggregates

Figure 3.2:

- Alkalis appeared in the deionised water placed in the container, under the samples (water vapour storage condition at 38°C and 60°C). This can be attributed to leaching of alkalis from the concrete prisms.
- The alkali leaching curve is similar to that of the expansion measurement of samples are stored in water vapour at 60°C.

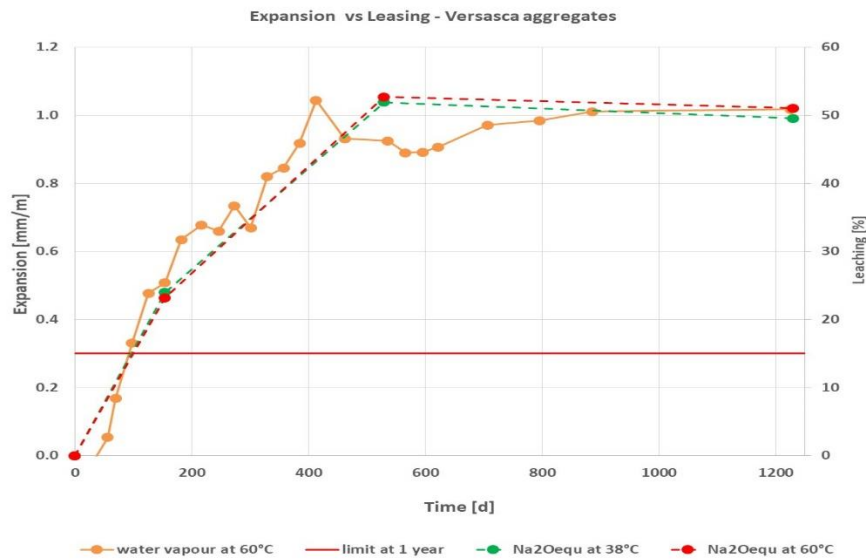


Figure 3.2: Leaching of the concrete prisms in vapour systems 60°C (SIA 2042).

Figure 3.3:

- Expansion results obtained when samples are placed in water vapour condition at 38°C, following ASTM C1293-18 standard, are comparable to the ones achieved using Swiss method (water vapour at 60°C).
- Only one mix design is rejected by this method (Praz aggregates).

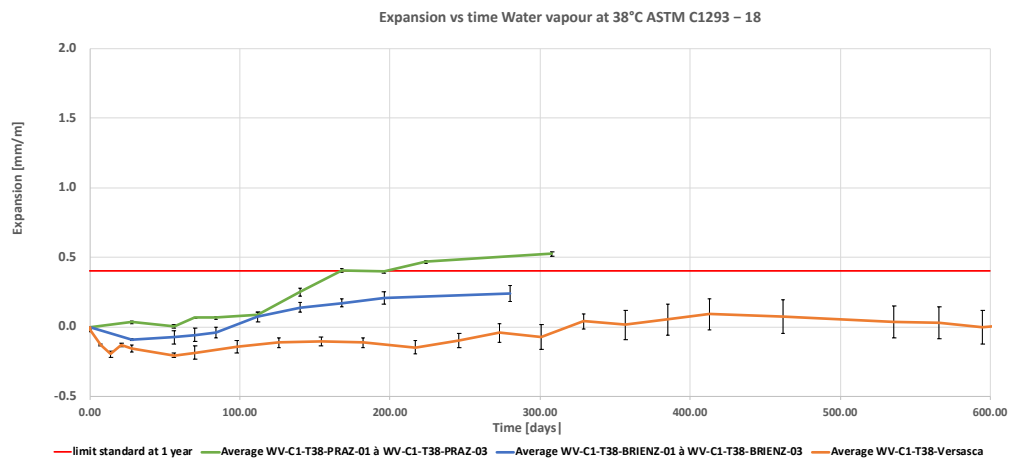


Figure 3.3: Expansion over time following ASTM C1293-18 standard - 3 different aggregates

Figure 3.4:

- Continuous expansion for all mix design (no formation of plateau).
- The beginning of expansion is observed before samples which are stored in water vapour storage condition at 38°C.

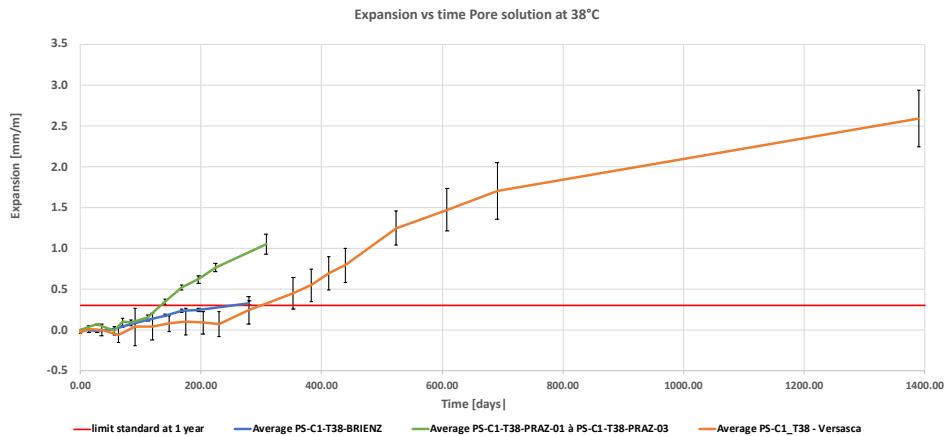


Figure 3.4: Expansion over time of samples store in pore solution after 1 day cure - 3 different aggregates

Figure 3.5:

- Samples placed in pore solution after curing for 28 days start to expand later than specimens stored in pore solution after 1 day curing. We can consider a delaying effect, around 100 days, due to curing time.
- The behaviour of both expansions is similar.

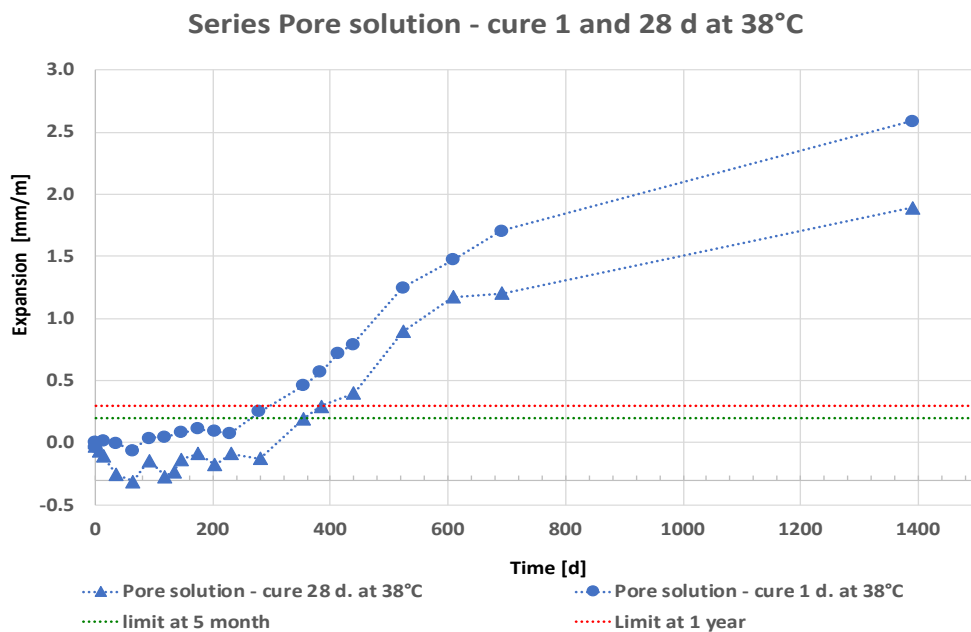


Figure 3.5: Expansion over time of samples store in pore solution after 1 or 28 days cure

Pore solution analysis

The results of the concrete pore solution analysis after 28 days at 20°C are presented in Table 3.1. These results are used to create a series of conditioning solutions stored in simplified pore solution (only sodium and potassium concentration is taking into account).

Table 3.1: 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

Water analyses of vapour curing systems

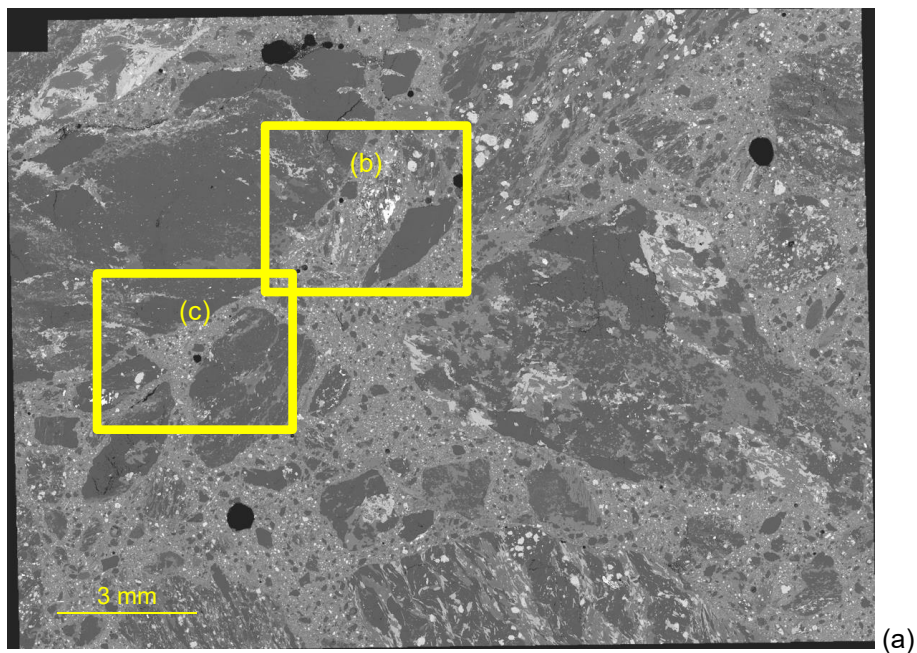
At the beginning of the test, no alkalis were present in the water. Table 3.2 resumes the results of ICP analyses of the reactors water at different ages. On Figure 2.2 we already observed the leaching for both vapour systems at 38°C and 60°C. After 1220 days, about 40% of sodium ions and 35% of potassium ions, which present initially in the concrete prisms, are leached. As presented in Figure 2.2, both evolutions of leaching seem similar, independent to storage temperature. This could be explained by the fact that leaching is mainly present during the cooling process of the measurement protocol. Condensation phenomenon may leach importantly during rapid temperature changes [16].

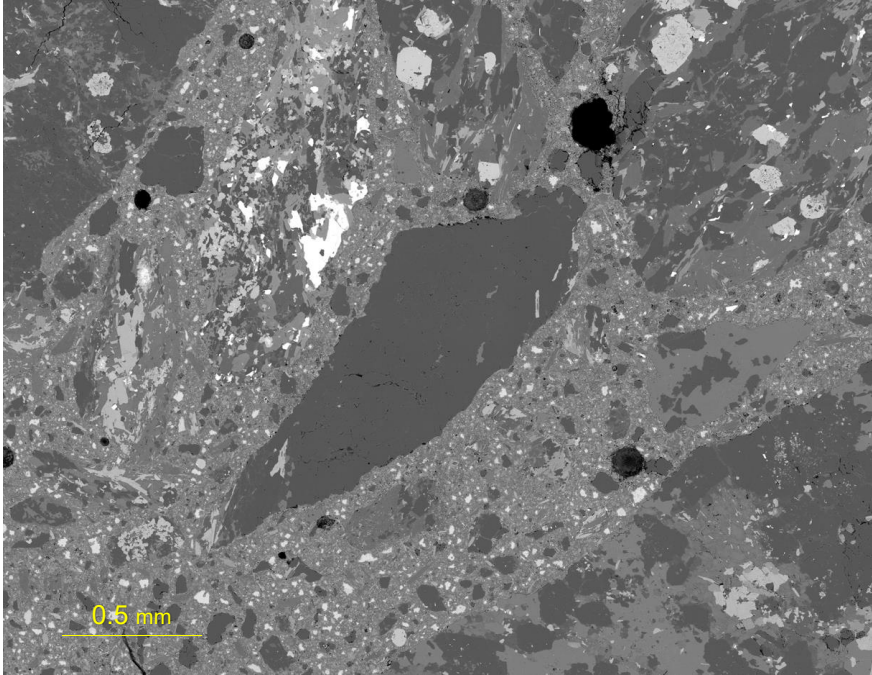
Table 3.2: Alkali concentration of the water at the bottom of the reactors (vapour systems) after 150 and 530 days

sample	Al mmol/l			K mmol/l			Na mmol/L		
	150 d	530 d	1230 d	150 d	530 d	1230 d	150 d	530 d	1230 d
SIA 2042 38°C	0.18	0	0	5.81	36.57	38.37	101.78	196.17	199.22
SIA 2042 60°C	0.25	0.22	.007	19.18	41.95	43.99	84.82	194.43	196.61

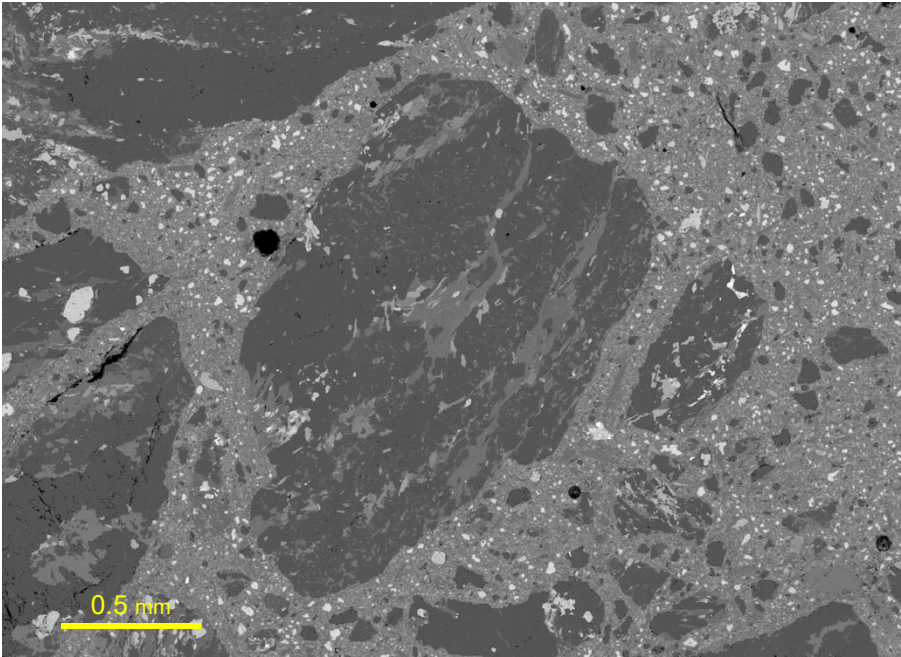
Scanning electron microscopy

Figure 3.6 and 3.7 show the difference in degradation of reactive aggregates between vapour systems at 60°C ((a) standard SIA 2042) and pore solution systems at 60°C (b). The amount of reacted fraction is clearly higher when samples are stored in pore solution. It shows that the vapour system stops to react despite the presence of reactive phases in aggregates.



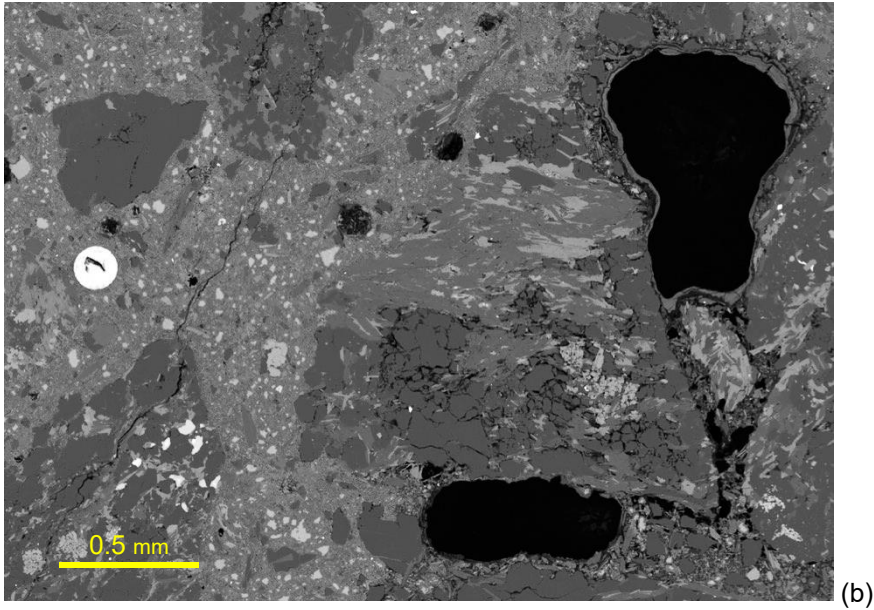
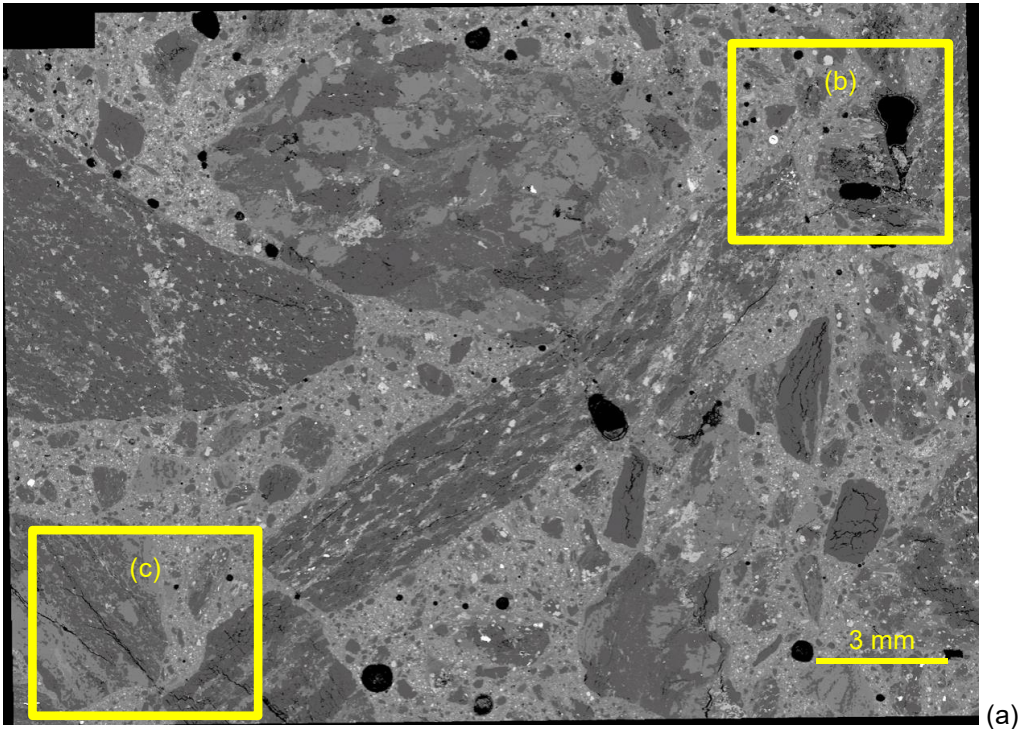


(b)



(c)

Figure 3.6: a) Reactive concrete sample after 360 days in vapour curing at 60°C, 1 day cure at 20°C, HE 100%. Yellow zones: picture; b) reactive sand with low reacted fraction, no cracks. Picture; c) reactive sand with low reacted fraction, no cracks.



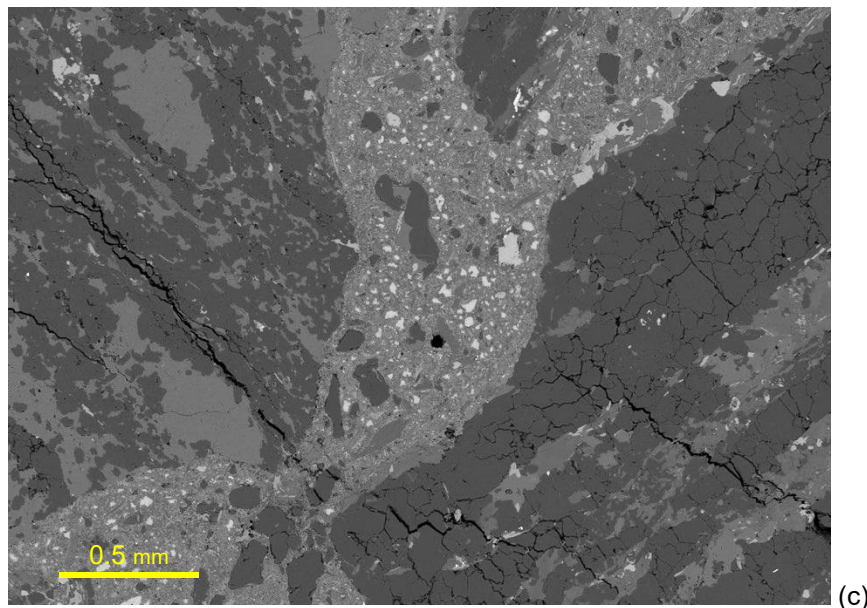


Figure 3.7: Reactive concrete sample after 360 days in pore solution curing at 60°C, 1 day cure at 20°C, RH 100%, Yellow zones: picture; b) reactive sands with high reacted fraction, crack trough the paste and some gel accumulated in a pore. Picture; c) two reactive sands: high reacted fraction, presence of cracks. Some reaction product is visible in the cracks (less gel because of the polishing process: non-calcified gel is easily removed).

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 and the American standard Concrete Prism Test C1293-18 are unreliable for ASR assessment. These procedures are subject to intense leaching of their pore solution, leading 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 parameters for the control of ASR – will not be preserved during the test duration. This will mislead the diagnostic of ASR behaviour.

Some improvements have been proposed to mitigate the leaching effect as much as possible. For example, one improvement includes wrapping samples in a wet paper by water [9]. This method, however, cannot guarantee control over a constant humidity. As a result, in order to be considered reliable, this method should be further improved.

Storing samples in simplified pore solution is original because is able to guarantee a constant amount of alkali during all the testing period. The concentration of alkalis has been chosen to be as close as possible to the one in the cement pore solution. As the matter of a fact, it is not possible to store the sample in a solution which is exactly the same as it is variable throughout the hydration process of the cement. To minimise the effect of this variation, we used the results of Lothenbach and colleagues [9] as well as those of Chappex and Scrivener [10], 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 [11]; 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.

As well known, curing has an effect on mechanical properties. Waiting until 28 days cure at 20°C has the advantage to respect the improvement of the mechanical properties of the paste. 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 method used should reproduce the relative strengths of the phases, aggregates and pastes, as they are in the field. This means, in particular, that tests, where the reaction of the aggregate is very rapid, unrealistic damage patterns may develop 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.

The comparison between expansion measured with samples stored in pore solution after 1 or 28 days cure at 20°C, RH 100%, shows a delaying effect, around 100 days. It's also important to notice the similar kinetics of expansion for both systems. For different mix designs tested, curing has an impact on the maximum expansion measure (2.6 [mm/m] when 1 day cure and 1.9 [mm/m] when 28 days) but shapes of curves are analogous.

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

The main goal of accelerated test in laboratory is to predict the behaviour of the concrete before using it on field. Most of the time, huge differences are observed between field and laboratory results [12][13]. That's why a good understanding and a precise control of all influential reaction parameters is primordial.

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 reasons for the flattening of the expansion curve after only few weeks. A new testing procedure is in course of development. The values chosen to develop this test method can be 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 consists of the following protocol:

- Pore solution extraction and analysis of the concrete cured during 1 days at 20°C in sealed containers.
- Sample preparation following the SIA 2042 procedure
- In parallel, curing of concrete samples (7x7x28 mm) during 1 days at 20°C in 95% RH (covered to avoid leaching due to water drops)
- After 1 day, start of the measurements and accelerated conditions for ASR: concrete samples are put in soak solutions similar to the analysed pore solutions at 1 days at 38° or 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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