ASR - PERFORMANCE TESTING: MAIN FINDINGS IN THE NORWEGIAN COIN PROJECT

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Abstract

Work is going on in several countries to try to develop a reliable performance testing concept for ASR. This topic was also focused on in the Norwegian R&D project "COIN" (2007-2014). The work was closely linked to the work within RILEM TC 219-ACS (2006-2014).

This paper gives a summary of the main findings in the COIN program. The main aim of part I (PhD study) was to document the effect of various curing – and storage conditions on the concrete internal moisture state, the extent of alkali leaching and ASR related expansion. The study documented that the main parameter controlling the ASR expansion measured in the laboratory is extent of alkali leaching.

Based on the findings in part I, a comprehensive follow-up study (part II) has been performed. 20 different concrete mixtures, including six aggregate types and four cement types, have been performance tested according to the most promising test procedures in part I. Moreover, two field exposure sites have been established in order to document the laboratory/field correlation, one in Norway (at SINTEF) and one in Portugal (at LNEC).

Keywords: Alkali-Silica Reaction (ASR), performance testing, alkali leaching, moisture state, field exposure sites

1   INTRODUCTION

1.1   Background

To be able to utilize alkali–silica reactive aggregates for the production of durable concretes, the effects of various measures, in particular different types and proportions of supplementary cementitious materials (SCMs), must be correctly identified by accelerated laboratory performance tests. Several performance tests have been used worldwide for about 20 years. In 2006, Thomas et al. [1] provided a critical evaluation of different ASR performance test methods. The authors concluded that none of the currently available or commonly used test methods meet all the criteria for an ideal performance test. For example, in the case of the most frequently used concrete performance test ASTM C1293-08b [2], the addition of alkalis is required for acceleration purposes; however, the fact that alkalis are leached out of the prisms during exposure in the humid environment, thus leading to reduced prism expansion, is a serious shortcoming of the test. Such alkali leaching will not happen for most concrete structures, except in the surface layer. Another drawback is the duration of the test (1-2 years).

During the last decade, research has been performed in several countries with the aim of improving current ASR performance test methods and develop alternative tests. For example, the RILEM TC 219-ACS (2007-2014) was working on a performance testing concept aiming to develop one or more reliable ASR concrete performance test methods for use worldwide. This work is continued in RILEM TC 258-AAA (2014-2019). In the latter RILEM TC, the main aim of WP1 (Work Package) is to develop one or more reliable ASR concrete performance test methods that might cover several applications/areas, ranging from combination of various aggregates with a standard CEM I binder up to the "ultimate goal" to document the potential alkali reactivity of any concrete mix design ("job mix"). One of the most important aims of WP3 is to establish a reliable procedure for assessing the extent of alkalis that might be released from various types of

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aggregates and supplied to the concrete pore solution. Validation of these results in concrete is also included. Hence, it is crucial to link the results from the accelerated laboratory testing to field behavior - main aim of WP2. Since only national ASR test methods are available in Europe today, RILEM TC 258-AAA aims to give a basis to establish AAR regulations in those countries that lack such, to contribute to improve current national AAR regulations and, hopefully, to give a basis to establish future common AAR test procedures and regulations in larger regions.

1.2   Part I (PhD study)

Whether or not concrete prism tests developed for the assessment of alkali-silica reactivity of aggregates is suitable for general ASR performance testing of concrete has been evaluated. The work was part of the Norwegian COIN program (2007-2014; www.coinweb.com). Since the study was performed in close co-operation with the "Performance testing" task group of RILEM TC 219-ACS, the draft RILEM aggregate concrete prism tests (CPTs) formed the basis for the laboratory program.

As a foundation for the experimental work, an introductory study focused on the following:
• Summary and assessment of the main findings in the EU "PARTNER" project (2002–2006) [3].
• Summary and discussion of the experience gained from extensive performance testing in Norway [4].
• A comprehensive literature review, with the main objective of assessing how various parameters might influence the laboratory/field correlation with respect to ASR performance testing. This work was performed in close co-operation with RILEM TC 219-ACS. In total, 12 authors contributed to the COIN report [5]. Based on this report, a summary paper was prepared [6].

Grounded on the introductory work, the experimental part of the study focused on the effect of specimen "pre-treatment", "ASR exposure conditions" and prism size on:
• Porosity and internal moisture state of the concrete prisms.
• Concrete transport properties (with respect to mobility of water and ions).
• Alkali leaching (rate and amount) from the concrete prisms during the ASR exposure.
• Concrete prism expansion (rate and ultimate expansion).

Additionally, the effect of water-to-cementitious-materials ratio (w/cm) and type of binder have been assessed. This paper gives an overview of the laboratory program and the comprehensive supplementary testing for documentation. Furthermore, some main results and conclusions are presented.

1.3   Part II (Follow-up study)

Based on the findings in part I, a comprehensive follow-up study (part II) was performed. Twenty (20) different concrete mixtures were prepared, involving aggregates from five sources and three cement types. To document the laboratory/field correlation, two field exposure sites have been established.

The main aims with the follow-up study were to:
• Investigate whether some of the ASR test procedures used in part I are able to produce expansion results that reflect the field behavior, i.e. verify the laboratory/field correlation for these test procedures.
• Document various aggregate/binder combinations ("what is safe to use ?").
• Continuously give input to the work in the RILEM TCs on ASR performance testing.
• Form the basis for any needed revisions of the Norwegian ASR regulations.

This paper gives an overview of the test program, in addition to present some selected results.

2   THE TEST PROGRAMME
2.1   Part I (PhD study)
Materials and mixture proportions

Two CEM I Portland cements, one high alkali and one low alkali, and a blended cement (CEM II/A-V) containing approximately 20% fly ash, were used in the study (Table 1). The aggregates used are defined in the Norwegian ASR regulations [7, 8] as "reference Norwegian aggregates" and consist of a non-reactive gneiss/granitic natural sand from Årdal and an alkali-silica reactive coarse aggregate, a crushed cataclasite from Ottersbo (same as "N1" in the EU "PARTNER" project [3]; reactive minerals are crypto- to micro-crystalline quartz). The aggregates were blended to produce a 60:40 coarse:fine ratio.

Details on the concrete mixtures produced are given in Table 2. The bulk of the testing was carried out on a mixture containing 400 kg/m^3 of Portland cement with a water-to-cement ratio (w/c) of 0.45 ("basis" binder). The two CEM I cements were blended to produce a concrete alkali content of 3.7 kg/m^3 Na$_2$O$_{eq}$. The alkali content was chosen with aim to reach an ultimate expansion of the reference test series lying on the ascending part of the "expansion versus alkali level curve", based on comprehensive testing at SINTEF [4].

To examine the impact of w/c, two additional concrete mixtures were cast with w/c of 0.30 and 0.60. The cement content of these mixtures was modified to achieve the desired workability, but the alkali content of the mixes was maintained at 3.7 kg/m^3 Na$_2$O$_{eq.}$ by appropriate blending of the CEM I cements.

One mixture was produced with a water-to-cementing-materials ratio of w/cm = 0.45 using the blended cement containing about 20% fly ash. The alkali content of this mixture was raised to 9.0 kg/m^3 Na$_2$O$_{eq.}$ (including all alkalis in the clinker and the fly ash, and boosting of the alkali level with 4.0 kg/m^3 Na$_2$O$_{eq.}$ by adding NaOH) to obtain an ultimate expansion of the "fly ash concrete mix" on the ascending part of the "expansion versus alkali level curve".

ASR test procedures – test series

The laboratory test program included in total 60 ASR test series, most of them by use of modified versions of the three draft RILEM aggregate CPTs: AAR-3, 2000 (38°C, wrapped prisms) [9] and AAR-4.1, 2006 (60°C, unwrapped and wrapped procedure) [10]. The technical background for the modifications is given in the literature review paper [6]. Reference ASR test series were also performed according to the Norwegian 38°C CPT [8] and the 38°C ASTM C1293-08b CPT [2]. All these five CPTs were originally designated for testing alkali-silica reactivity of concrete aggregates. The main differences between them are exposure temperature (38°C or 60°C; only RILEM AAR-4.1 uses the highest temperature), prism cross section (70-75x70-75 mm or 100x100 mm; only the Norwegian CPT uses the largest prism cross-section) and use of any wrapping (damp cotton cloth and polyethylene; only used in the CPTs RILEM AAR-3, 2000 and RILEM AAR-4.1 "Alternative", 2006). The "pre-treatment" parameters and/or ASR storage conditions that were varied for the RILEM CPTs in particular are described in detail in Lindgård et al. [11, 12].

During all the testing, only deionized water was used, i.e. as batched water, in the moist cotton cloth wrapping (if any) and in the storage containers. For all standard versions of the CPTs, the readings of weight and length were taken after cooling the prisms for about 16 hours inside their storage container in a room kept at ~ 20°C. All readings in the modified versions of the various concrete prism tests were taken without cooling the prisms.

Supplementary tests

In order to document properties of importance for initiation and progress of ASR, comprehensive supplementary testing was an important part of the study (see section 1.2). After exposure, the extent of internal cracking was also documented by microstructural analyses. All the test procedures included are described in detail in [11], [12] and [13]. The documentation was performed at two points of time: four weeks after starting the ASR exposure (in order to document concrete properties in the early stage of the ASR test) and after ending the ASR exposure.

2.2   Part II (Follow-up study)
Materials and mixture proportions

The following five aggregate types were tested: 1) Non-reactive gneiss/granitic natural sand + coarse aggregate from Årdal in Norway (same sand as in part I); 2) Alkali-reactive crushed, coarse cataclasite from Ottersbo in Norway (same as used in part I); 3) A slowly reactive Norwegian natural sand + coarse aggregate from the South East of Norway (same as "N4" in the EU "PARTNER" project [3]; dominated by gneiss/granite - reactive minerals are crypto- to microcrystalline quartz in sandstones, siltstones and cataclastic rocks); 4) Alkali-reactive crushed, coarse "Spratt" from Canada (silicified limestone; reactive minerals are crypto- to microcrystalline quartz); 5) Highly reactive crushed, coarse German river gravel from Upper Rhine (containing limestone with impurities, marl, sandstone and a little content of cataclastic rocks; used as "reference" reactive aggregate in some German ASR tests; reactive minerals are micro- to cryptocrystalline quartz).

Three of the cement types included were the same as used in part I (Table 1). Additionally, a CEM I with very low alkali content and a German CEM III/B cement (incl. 68 % slag) were used (Table 1). The overall mixture proportions of the 20 different concrete mixtures prepared are shown in Table 3. For all mixtures, w/c or w/cm was 0.45. In total, 113 single ASR performance test series were performed.

ASR test procedures – supplementary tests

For each of the concrete mixtures, five to seven of the modified CPT procedures from part I were included. These involved the three "standard" CPTs, i.e. Norwegian CPT [8], ASTM C1293 [2] and RILEM AAR-4.1 [10] (the latter two included both 1 and 28 days of pre-storage at ambient temperature before exposure). Moreover, a "pilot" CPT procedure used in part I applying a cotton cloth wrapping pre-submerged in an alkali liquid with pH 14.2 (storage at two temperatures; 38°C and 60°C) was included. In addition to expansion and weight increase, rate and extent of alkali leaching was measured for all test series.

After exposure, microstructural analysis (of fluorescent impregnated plane polished sections and thin sections) were performed of selected prisms. Furthermore, corresponding prisms were sent to Université Laval for being analyzed according to the DRI method, [14, 15]. Université Laval will also perform image analysis of the pictures taken in fluorescent light of the plane polished sections, similar as was performed in part I [16]. The aim is to compare results from microstructural analysis, image analysis and DRI analysis.

Establishment of field exposure sites

To document the laboratory/field correlation, two field exposure sites have been established. The test setup is the same as developed within the EU "PARTNER" project [3] using concrete cubes with dimension 300x300x300 mm. From each of the 20 concrete mixtures, two monitored cubes were prepared. One was placed at SINTEF in Trondheim (Norway) and one was placed at LNEC in Lisbon (Portugal).

3   RESULTS
3.1   Part I (PhD study)

The results from the extensive laboratory program are published in two articles, Lindgård et al. [11] and [12], and in the PhD thesis [13]. In this paper, only a few selected results are presented. However, the most important findings and conclusion are discussed in section 4 and 5.

Figure 1a shows expansion versus time for test series with the *"basis" binder* (CEM I, w/c 0.45). The 52-weeks expansion of the 38°C test series varied in the range of 0.17-0.31 %, while the 39 weeks expansion of the 60°C test series varied in the range of 0.04-0.22 %. The critical expansion limit is in the range 0.030-0.050 %. The following main trends were observed:

Exposure temperature 60°C:
• The ASR expansion of the unwrapped prisms was several times greater than that of the wrapped prisms.
• Wrapped prisms with only half of the prescribed water content added to the cotton wrapping (the two upper "wrapped curves") expanded significantly more than the wrapped prisms with more water added.
• The test series submerged in deionized water during the whole exposure period revealed hardly any expansion at all (not included in the figure).

Exposure temperature 38°C:
• All the eight test series with the ASTM C1293 CPT [2] revealed almost identical expansion after 1 year.
• The prisms tested according to the ASTM C1293 CPT (with cross section 70x70 mm) revealed significantly less expansion than the larger Norwegian CPT prisms [8] (with cross section 100x100 mm).
• The Norwegian CPT produced the highest expansions of all the CPT test procedures included in the study.
• After one year of exposure, wrapped prisms always expanded less than unwrapped prisms.

Figure 1b shows expansion versus time for all the test series with the *low w/c binder* (CEM I, w/c 0.30). Compared with the CEM I binders with higher w/c, the following main trends were observed:

Exposure temperature 60°C:
• Test series with w/c 0.30 expanded similarly as corresponding test series with w/c 0.45 and 0.60. This was valid both for wrapped and unwrapped prisms.

Exposure temperature 38°C:
• Dramatically lower expansion was revealed for the test series with w/c 0.30 compared with comparable test series with w/c 0.45 and 0.60.
• At w/c 0.30, the wrapped prisms expanded slightly more than unwrapped prisms, in contrast to what was observed at higher w/c.

The test series with the *fly ash binder* (CEM II/A-V, w/cm 0.45) showed a similar trend as described above for the low w/c (0.30) CEM I binder with respect to expansion behavior at the two exposure temperatures, i.e. significantly higher expansion at 60°C compared with 38°C.

Based on the measured *alkali leaching* (by collecting 20 ml samples from the liquid in the bottom of the storage containers), the remaining internal alkali content after 4 weeks of exposure was calculated for all test series (the alkalis present in the cotton cloth wrapping was also accounted for by removing the wrapping from the extra forth prism that had been prepared for each test series). Figure 2 shows the extreme effect alkali leaching in the first 4 weeks of exposure had on the measured 26 weeks expansion for the prisms exposed to 60°C. The figure includes all wrapped and unwrapped 60°C test series with the CEM I binders, i.e. binders with initial alkali content 3.7 kg/m^3 Na$_2$O$_{eq}$ and w/c either 0.30, 0.45 or 0.60.

3.2 Part II (Follow-up study)

The data from part II are still being assessed. In this paper, only a few selected results are included. More precisely, two examples on the remarkable effect of the test procedure used on the measured prism expansion are presented.

Figure 3 shows the effect of prism cross-section (diameter 70 or 100 mm) on expansion for prisms stored over water in sealed containers at 38°C. The aggregate composition is the same as used in part I (Årdal sand / Ottersbo coarse aggregate). The binders included consist of CEM I cement with nominal alkali content 2.0 or 2.8 kg/m^3 Na$_2$O$_{eq.}$. In addition to the laboratory results, the mean expansion of the corresponding 300x300x300 mm^3 field exposed cubes of the mixture with alkali content 2.8 kg/m^3 Na$_2$O$_{eq}$ is included. The corresponding two cubes with alkali content 2.0 kg/m^3 Na$_2$O$_{eq}$ still show slight shrinkage after 5 years of field exposure. These field cubes, as well as the remaining 36 field cubes prepared, will be monitored in the future with annual measurements (twice a year in Lisbon).

Figure 4 shows the effect of adding alkalis to the cotton cloth wrapping, a "pilot" test procedure used to try to eliminate the alkali leaching, [12] and [13]. For the two test series denoted pH 14.2, the prisms were wrapped with cotton cloth pre-submerged in an alkaline liquid with pH 14.2. The aggregate composition is Årdal sand and Spratt coarse aggregate. The binder is CEM I with alkali content 1.5 kg/m^3 Na$_2$O$_{eq.}$. When exposed to 38°C, the prisms wrapped in the alkali clothing revealed a high expansion, while the remaining 38°C prisms hardly expanded at all. The effect of application of the alkali wrapping was far less at 60°C.

In Table 4, Figure 3 and Figure 4, the measured alkali leaching after 1 year of exposure and at the end of the exposure period is given. The negative numbers given in Table 4 and Figure 4 mean net alkali ingress due to the high alkali content in the wrapping at the start of ASR exposure.

4 DISCUSSION
4.1 Part I (PhD study)

The results clearly show that parameters of importance for the development of ASR are significantly influenced by the specimen "pre-treatment", "ASR exposure conditions" and prism cross-section. Most test conditions included are representative test procedures used in various "commercial" CPTs. The extent of the impact depends on the concrete composition/characteristics, i.e. w/cm and cement type.

Consequently, the conclusion from a concrete performance test will differ depending on the test procedure used. This is evident looking at the expansion curves in Figure 1a and 1b. All the test series included in each of the two figures had identical concrete composition, both with CEM I (OPC) binders with nominal alkali content of 3.7 kg/m^3 Na$_2$O$_{eq}$. One of the most remarkable aspects of these differing results is that one of the testing variants that produced one of the lowest ultimate expansions was recommended by RILEM for a long period as an alternative test method to evaluate the alkali-reactivity of aggregates. *(Comment: That method, RILEM AAR-4.1 Alternative 60°C CPT using prisms wrapped with moist cotton cloth and plastic* [10] *is, however, not recommended by RILEM any longer due to the results of this study. For the same reason, the wrapping procedure is not recommended for the RILEM AAR-3 38°C CPT (2000)* [9] *either. The revised version of RILEM AAR-3 (2016)* [17] *uses unwrapped prisms, i.e. the prism size and the exposure conditions are the same as used in the ASTM C1293 38°C CPT* [2] *(2008)).*

The effect of wrapping the prisms was particularly pronounced for prisms exposed to 60°C, where the ultimate expansion of unwrapped prisms was up to five times higher than for corresponding wrapped prisms (Figure 1). The main reason for the tremendous reduction in the ASR expansion when the prisms were wrapped is the high rate of alkali leaching in the early stages of the test (Figure 2). After 4 weeks of exposure, the reduction in the alkali content varied from about 9-20 % (depending on details in the test procedure). The corresponding alkali leaching from unwrapped prisms varied in the range of 3-5 %. In fact, the rate of alkali leaching during the first weeks of exposure was the parameter found to have the highest impact on the development of ASR expansion. When exposed to 60°C, it completely controlled the prism expansion. One reason for this enormous impact is that the nominal alkali content in the CEM I concretes was lying on the ascending part of the "expansion versus alkali level curve". Consequently, if you leach out alkalis rapidly during the early ASR exposure, you will lose much of the expansion potential.

Generally, a high fraction of the in-mixed alkalis was leached out of the concrete prisms during the ASR exposure. It varied with test procedure, exposure temperature and cement type (less percentage alkali leaching from the fly ash cement). The impact on the ASR expansion was less when exposed to 38°C, but still very significant. One important finding is that increasing the prism cross-section from diameter 70 mm to 100 mm decreased the rate and amount of alkali leaching considerably; on average, the ultimate alkali leaching was halved. Consequently, the 100 mm prisms produced the highest expansions of all the test methods (see the upper dotted curve in Figure 1a), i.e. use of the larger Norwegian prisms is favored.

The less permeable concretes (i.e. CEM I with w/c 0.30 and CEM II/A-V with w/cm 0.45) revealed a relatively high degree of self-desiccation. Thus, the internal relative humidity (RH) was lowered for the 38°C

test series. Together with the lower rate of diffusion compared with the more open binders (CEM I with w/c 0.45 or 0.60), the lower internal RH contributed to reduce the rate and extent of ASR (Figure 1b). The same reduction in internal RH was not found for the 60°C test series, and thus the expansion was not reduced.

4.2   Part II (Follow-up study)

The positive effect of increased prism cross-section on the ASR expansion was also confirmed in the follow-up study. As expected, this effect was in particular very high for the OPC (CEM I) concrete with alkali content close to the alkali threshold for the coarse alkali-reactive cataclasite aggregate (Figure 3). For this concrete with nominal alkali content 2.8 kg/m³ Na_2O_{eq}, about three times higher expansion was obtained after 1 and 2 years of expansion for the prisms with 100x100 mm² cross-section compared with the prisms with cross-section of 70x70 mm². In fact, the 100 mm prisms still expand after 2 years, while the expansion of the 70 mm prisms has flattened. The corresponding alkali leaching data (Table 4) show a similar difference as found in the PhD study (part I), i.e. the 100 mm prisms leach out about half of the alkalis compared with the 70 mm prisms after 1 year of exposure and more than 2.5 times less during the entire exposure period.

The expansion revealed in field after only 4.5 years of exposure in Lisbon is already almost as high as the ultimate expansion for the 70 mm prisms. This confirms that the alkali leaching in the laboratory for the 70 mm prisms is unacceptably high, leading to an unsatisfactory laboratory/field correlation. This is in agreement with previous findings [1]. Consequently, the above data suggest that an increase in the prism cross-section, e.g. from 70-75 to 100 mm prisms, will strongly increase the reliability of the CPT for performance testing. This is the main reason why RILEM TC 258-AAA recommends use of prisms with cross-section 100x100 mm in the current draft of the 38°C concrete performance test procedure.

The "pilot" test procedure used in part I to try eliminating the alkali leaching by wrapping the prisms with cotton cloth pre-submerged in an alkaline liquid, an approach focused on in Japan [18], is a somewhat promising tool to mitigate alkali leaching during accelerated laboratory testing [12, 13]. However, this approach should be used with care since some alkalis may still leach from the prisms - or inadequate amounts may be added, depending on several factors; prism cross-section, concentration of alkalis (pH) in the liquid, amount of alkaline liquid added to the wrapping, sealing of the wrapped prisms or not, type of binder, nominal alkali content of the binder, concrete permeability, etc. Example: As shown in Figure 4, when testing low alkali systems (CEM I / OPC with nominal alkali content 1.5 kg/m³ Na_2O_{eq}), the alkalis in the "high pH wrapping" may totally control the expansion due to ingress of alkalis, as was the case for the test series exposed to 38°C. For this test series, the internal alkali content was raised with about 90 % (compared with the nominal alkali content) due to ingress of alkalis. For the corresponding 60°C test series, the alkali content increased with about 47 %. However, for this test series the prism expansion was only slightly increased.

Consequently, selecting the proper composition for the alkali wrapping solution is critical for the reliable evaluation of the efficacy of a particular system in controlling ASR expansion. Significant variability between laboratories carrying out performance testing may also be observed if a consolidated approach is not used for selecting the proper alkali wrapping solution, especially considering the wide variety in the type and dosages of cements and SCMs that could be used for ASR prevention within and between countries worldwide.

5   CONCLUSIONS

Part I (PhD study)

The results clearly show that parameters of importance for the development of ASR are significantly influenced by the specimen "pre-treatment", "ASR exposure conditions" and prism cross-section.

- The rate of expansion and ultimate expansion might vary hugely between "commercial" concrete prism tests that are supposed to give similar conclusions with respect to alkali reactivity.
- The parameter that showed to have highest influence on the measured prism expansion was rate and extent of alkali leaching. When exposed to 60°C, it completely controlled the prism expansion.
- Testing of the most "dense" concretes at 60°C produced higher expansion compared with exposure to 38°C, opposite as was found for CEM I binders with w/c of 0.45 or higher. This reason is assumed to be that also the concrete internal moisture state and the transport properties might influence the rate of the ASR expansion when testing such concretes with a relatively high degree of self-desiccation.

More conclusions and recommendations for performance testing are given in the two articles, Lindgård et al. [11] and [12], and in the PhD thesis [13].

Part II (Follow-up study)

It is too early to conclude before assessing all the results from the follow-up study. However, preliminary findings have in general confirmed the positive effect of increased prism cross-section from 70 mm to 100 mm on the ASR expansion (increased) due to reduced rate and extent of alkali leaching. The

impact on expansion was in particular high for the CEM I (OPC) concretes with alkali content close to the alkali threshold. Furthermore, application of an "alkali wrapping procedure" to hinder alkali leaching may be a challenge. When testing very low alkali systems, the alkalis in the "high pH wrapping" may totally control the expansion due to ingress of alkalis.

6   ACKNOWLEDGEMENT

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TABLE 1: Chemical composition (EN 196-2) of the cements (wt%).											
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Na_2O_e[4]	P_2O_5	LOI[1]
High-alkali CEM I	19.6	4.9	3.5	61.0	2.8	3.8	1.11	0.51	1.24	0.15	2.44
Low-alkali CEM I	20.1	4.7	3.3	63.1	2.0	3.4	0.39	0.34	0.60	0.16	2.24
Very low-alkali CEM I[5]	25	2.1	0.3	69	0.7	2.2			0.20		0.7
CEM II/A-V[2]	26.6	8.7	4.2	50.3	2.4	3.3	1.04	0.56	1.25	0.33	1.20
CEM III/B[3,5]	30	11	1	46		2.7			0.78		

[1] Loss-On-Ignition.
[2] Norwegian blended cement containing 21.6 wt% class F fly ash. Manufactured by co-grinding clinker and fly ash.
[3] German slag cement containing 68 wt% slag (ggbfs).
[4] Included all alkalis in the clinker, the fly ash and the slag.
[5] Only used in part II.

TABLE 2: Composition of the four concrete mixes included in part I (PhD study).					
Materials (kg/m³)		Binder composition			
		CEM I, w/c 0.45	CEM I, w/c 0.30	CEM I, w/c 0.60	CEM II/A-V, w/cm 0.45
Cement	High-alkali CEM I	200	60	285	---
	Low alkali CEM I	200	490	30	---
	CEM II/A-V	---	---	---	400
Aggregates (SSD[1])	Årdal 0/4	735	700	755	725
	Ottersbo 4/8	185	175	190	180
	Ottersbo 8/11	365	350	375	360
	Ottersbo 11/16	550	525	565	540
Deionised water (free) (excl. any water in the superplasticizer)		180	165	189	180
NaOH (solids)		---	---	---	5.2
Alkali content (kg Na_2O_{eq} per m³)[2]		3.7	3.7	3.7	9.0
Superplasticizer		If necessary, add until workable and stable concrete (aimed slump 120 mm)			
De-foaming agent		If measured air content is > 3.0 %, add until air content is reduced to < 3.0 %			

[1] Saturated surface dry condition.
[2] Included all alkalis in the clinker, the fly ash, the slag and the superplasticizer. No contribution of alkalis from the aggregates due to any alkali release is included.

TABLE 3: Concrete mix design in part II (the numbers represent the number of mixes performed).				
Aggregate type[1]	Cement type			Total alkali content (kg/m³ Na_2O_{eq})[4]
	CEM I	CEM II-A/V	CEM I / CEM III/B	
Årdal (F/C)	1	--	--	5.5
"N4" (F/C)	1	--	--	5.5
Ottersbo[2] (C)	3	2	1	2.0, 2.8 and 3.7 (CEM I); 5.0 and 6.5 (CEM II-A/V); 4.0[3] (50/50 CEM I/ CEM III/B)
Spratt[2] (C)	3	2	1	1.5, 2.0 and 2.9 (CEM I); 5.0 and 6.5 (CEM II-A/V); 4.0[3] (50/50 CEM I/ CEM III/B)
Upper Rhine[2] (C)	1	2	3	5.5 (CEM I); 5.0 and 6.5 (CEM II-A/V); 4.0[3] and 5.0[3] (50/50 CEM I/ CEM III/B); 3.1 (CEM III/B; 68 % slag)

[1] F=Fine fraction, C=Coarse fraction.
[2] Årdal sand was used as the fine fraction.
[3] 50/50 high alkali CEM I / CEM III/B slag cement (in total 34 % slag).
[4] Incl. all alkalis in clinker, fly ash, slag and any superplasticizer. No contribution of alkalis from the aggregates due to any alkali release is included.

TABLE 4: Extent of alkali leaching from the concrete prisms during the ASR exposure.									
Test series (see Figure 3 and 4)		70mm-2.0 [1] (38°C)	100mm-2.0 [1] (38°C)	70mm-2.8 [1] (38°C)	100mm-2.8 [1] (38°C)	Norw. CPT (100mm, 38°C)	ASTM C1293 (70 mm, 38°C)	RILEM AAR-4.1 (70 mm, 60°C)	38degC, pH 14.2 [5] (70 mm)	60degC, pH 14.2 [5] (70 mm)
1 year of exposure	kg Na₂Oₑq. [2]	0.41	0.23	0.56	0.28	0.09	0.35	0.65	---	---
	% [3]	20	11	20	10	6	23	44	---	---
At the end of the exposure [4]	kg Na₂Oₑq. [2]	1.39	0.52	1.42	0.49	0.39	0.97	0.77	-1.35	-0.71
	% [3]	70	26	51	18	26	65	51	-90	-47

[1] Nominal alkali content (kg $Na_2O_{eq.}$ – see Table 3)

[2] Alkali leaching expressed as kg $Na_2O_{eq.}$

[3] Alkali leaching expressed as % of nominal alkali content; i.e. (alkali leaching / nominal alkali content) x 100 %

[4] The numbers include any alkalis present in the lining inside the storage containers (not included in the numbers after 1 year of exposure)

[5] Net ingress of alkalis (due to the high alkali content in the cotton cloth wrapping at start of the ASR exposure)
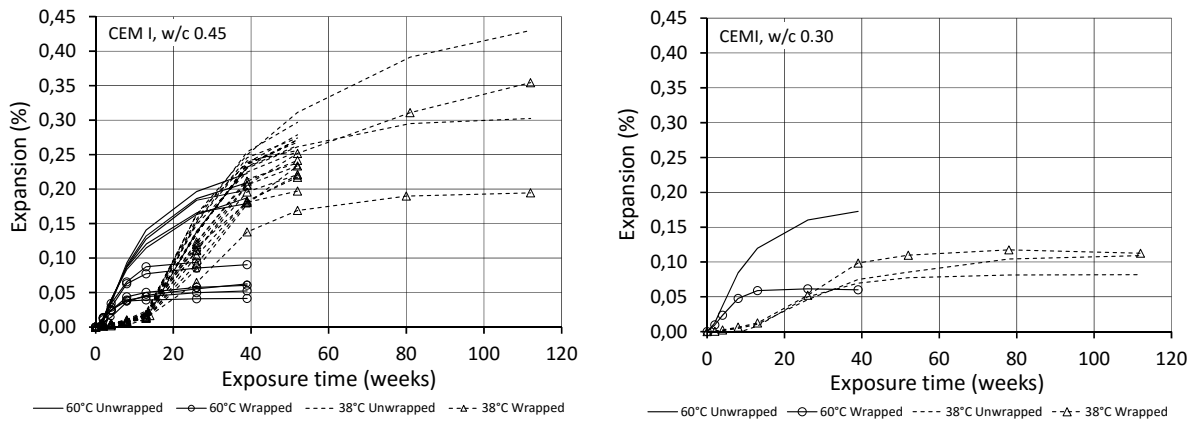

FIGURE 1: Expansion versus time for 32 test series with the "basis" binder (1a-left) and 5 test series with the low w/c binder (1b-right), both with alkali content 3.7 kg per m³ Na_2O_{eq}. The figure includes unwrapped prisms (ASTM C1293-08b 38°C CPT [2], Norwegian 38°C CPT [8] and RILEM AAR-4.1 60°C CPT [10]) as well as wrapped prisms (RILEM AAR-3 38°C CPT [9] and RILEM AAR-4.1 Alt. 60°C CPT [10]) exposed to 100 % RH (i.e. stored on grids over water). The sealed and submerged test series are not included.
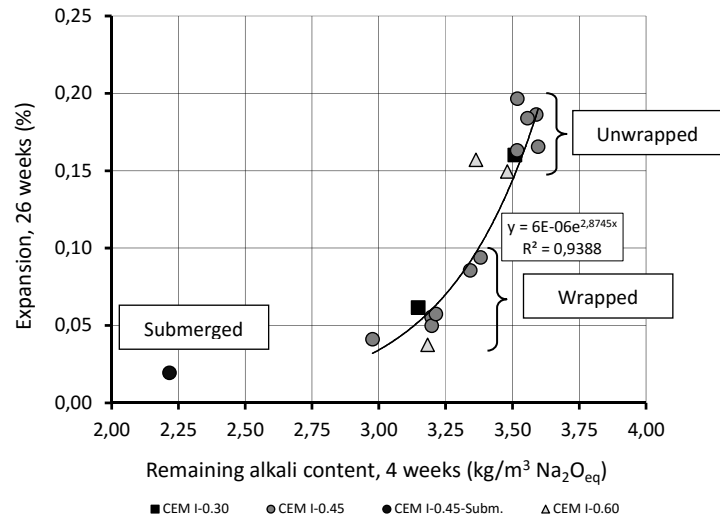

FIGURE 2: Remaining alkali content after 4 weeks of exposure versus expansion at 26 weeks for wrapped and unwrapped AAR-4.1 60°C [10] test series with CEM I binders (w/c either 0.30, 0.45 or 0.60; initial alkali content for all concretes was 3.7 kg/m³ Na_2O_{eq}). The determination coefficient (R^2) is valid for the test series with the "basis" binder (w/c of 0.45), excl. the submerged test series (left point).
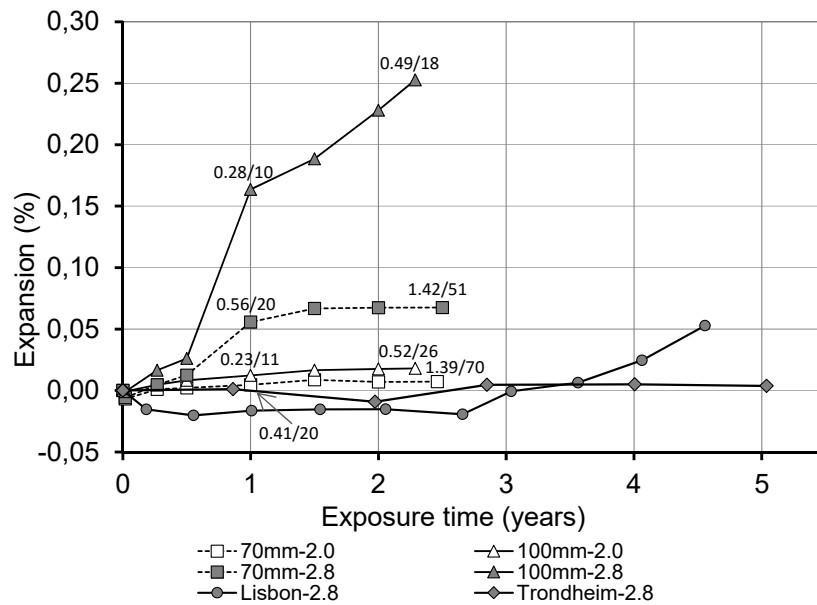
FIGURE 3: Effect of prism cross-section on expansion for prisms stored over water in sealed containers at 38°C. In addition to the prism cross-section (diameter in mm), the legends include the nominal alkali content (2.0 or 2.8) in kg/m³ Na₂O$_{eq.}$ of the CEM I binder. The aggregate composition is the same as used in part I (Årdal sand / Ottersbo coarse aggregate). The figure includes numbers for alkali leaching (Table 4: kg Na₂O$_{eq.}$ / % of nominal alkali content;) after 1 year and after ending the exposure (the latter numbers include alkalis present in the lining inside the storage containers). Furthermore, the mean expansion of the 300x300x300 mm³ field exposed cubes of the mixture with alkali content 2.8 kg/m³ Na₂O$_{eq}$ is included. The corresponding cubes with alkali content 2.0 kg/m³ Na₂O$_{eq}$ still show slightly shrinkage after 5 years of field exposure.
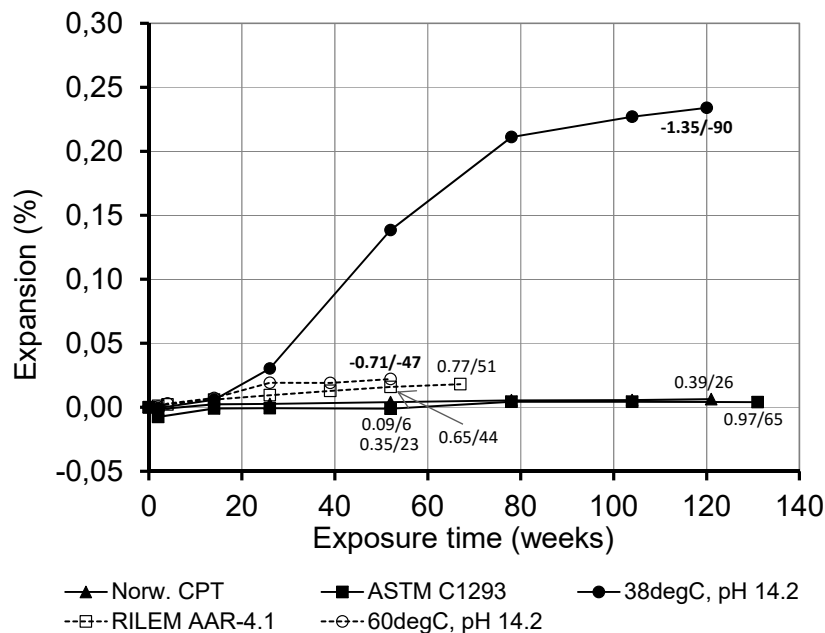

FIGURE 4: Effect of adding alkalis to the cotton cloth wrapping. The concrete is composed of Årdal sand, Spratt coarse aggregate and a CEM I binder with alkali content of 1.5 kg/m³ Na₂O$_{eq}$ (a blend of the very low-alkali CEM I and the low-alkali CEM I cements, see Table 1). For the two test series denoted pH 14.2, the prisms are wrapped with cotton cloth pre-submerged in an alkaline liquid with pH 14.2. The figure includes numbers for alkali leaching (kg Na₂O$_{eq.}$ / % of the nominal alkali content; see Table 4) after 1 year and after ending the exposure (the latter numbers include alkalis present in any lining inside the storage containers).