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ALKALI -SILICA REACTION (ASR) - PERFORMANCE TESTING: INFLUENCE OF SPECIMEN PRE-TREATMENT, EXPOSURE CONDITIONS AND PRISM SIZE ON ALKALI LEACHING AND PRISM EXPANSION

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Abstract

Whether or not concrete prism tests developed for assessment of alkali-silica reactivity of aggregates might be suitable for general ASR performance testing of concrete has been evaluated. This paper discusses how variations in specimen pre-treatment, ASR exposure conditions and prism size influence the rate and amount of alkali leaching and prism expansion, together with a discussion of consequences for ASR test procedures. Furthermore, results from some complementary tests are included.

Generally, a remarkably high proportion of the in-mixed alkalis was leached out of the concrete prisms during the ASR exposure. For prisms exposed to 60°C, the rate and amount of alkali leaching is the main controlling factor for the prism expansion. For less permeable concretes exposed to 38°C, lack of internal moisture and lower rate of diffusion contributes to reduce the rate and extent of ASR expansion (reported in a separate paper).

Keywords: alkali-silica reaction, performance testing, alkali leaching, expansion, microstructural analysis.

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1 INTRODUCTION

1.1 Background

Supplementary cementing materials (SCMs; e.g. silica fume, fly ash, ground granulated blast-furnace slag (ggbs), metakaolin and other pozzolans) control expansion due to alkali-silica reaction (ASR) by binding alkalis and limiting their availability for reaction with alkali-silica reactive aggregates [1]. The efficiency of the SCMs depends on their composition and amount, the nature of the reactive aggregate and the availability of alkali in the concrete. Chappex and Scrivener [2] also showed that the aluminium present in certain SCMs (e.g. metakaolin) may limit the dissolution of silica from reactive aggregates. Consequently, to be able to utilise alkali-silica reactive aggregates for production of durable concretes, the effects of various measures must correctly be identified by accelerated laboratory performance tests (or ideally by relevant long-term field experience). Several such performance tests have been used worldwide for at least 15 years, but the test conditions (e.g. pre-curing, temperature, alkali content, humidity) differ from one test method to another. Thus, the results and conclusions from different test methods may vary widely.

In 2006, Thomas et al. [3] 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. One main problem discovered is that alkalis are leached out of the prisms during exposure in the humid environment and hence reduce the final prism expansion, e.g. as documented for the Canadian 38°C concrete prism test (CPT) [4] (similar to ASTM C1293 [5]). Thus, the authors concluded that this most frequently used CPT world-wide cannot be used to determine the “critical” alkali content for an alkali-reactive aggregate, nor to determine how the level of a SCM required to control expansion varies with the concrete alkali content. The “critical” or “threshold” alkali content is defined here as the lowest amount of alkali that will cause deleterious expansion with a particular aggregate.

Today, research is on-going in several countries with the aim to improve current ASR performance test methods and develop alternative tests. As part of the international harmonisation of ASR performance test methods, the "Performance testing" task group of RILEM TC 219-ACS is working

on a performance testing concept aiming 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 alkali reactivity of any concrete recipe.

1.2 PhD study on ASR

1.2.1. General

The main objective of the PhD study by Jan Lindgård, being part of the Norwegian COIN program (2007-2014, www.coinweb.no), has been to evaluate whether concrete prism tests developed for assessment of alkali-silica reactivity of aggregates might be suitable for general ASR performance testing of concrete. This paper is one of several from the PhD study.

As part of the background study, a comprehensive literature review has recently been performed in close collaboration with the task group "Performance testing" in RILEM TC 219-ACS [6] (all authors of this paper, except one, are members of this RILEM task group). The main objective was to assess how various parameters might influence the laboratory/field correlation with respect to ASR performance testing, either directly or indirectly. The most important findings in the literature survey and recommendations for performance testing have recently been summarised by Lindgård et al. [7]. Additionally, the literature survey identified several issues that need further research in order to develop a reliable performance test procedure.

1.2.2. Parameters focused on in the PhD study

The experimental part of the study has focused on the effect of specimen "pre-treatment" and "ASR exposure conditions" as well as 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 final expansion).

Additionally, the effect of water-to-cementitious-materials ratio (w/cm) and type of binder is assessed.

The specimen "pre-treatment" is defined as the moisture condition during pre-storage and the length of the pre-storage period at ambient temperature (up to the point of the initial (zero) length comparator reading). The "ASR exposure conditions" include various moisture conditions, type of container, use of any wrapping (damp cotton cloth and plastic foil), exposure temperature, length of the storage period and addition of any external alkalis. These conditions as well as prism size varies between various performance test methods used in the different countries.

An extensive laboratory program has been performed, including 58 ASR test series and comprehensive complementary testing for documentation (Section 2.5). The test series cover the variations in test conditions in the most commonly used ASR concrete prism test methods. Additionally, some test series include measures to try to reduce the amount of alkali leaching. A separate paper [8] presents the technical background for the choice of test procedures.

As a basis for the evaluation of alkali leaching, some important findings on this topic in the recently published literature review [7] are summarized in section 1.3.

1.3 Alkali leaching from concrete prisms during the ASR exposure

The problem of alkali leaching from specimens stored over water in sealed containers, leading to reduced prism expansion, was first reported by Blanks and Meissner in 1946 [9]. The authors detected an increasing concentration of alkali ions in the water at the bottom of the containers in which mortar bars were stored, and explained this as water condensing on the surface of the bars and running down the bars into the reservoir below, thereby transporting the alkalis. The mechanism for alkali leaching is further explained by Rivard et al. [10] to be excessive condensation of water on the prism surfaces, leading to an outward diffusion of alkalis from the interior of the concrete.

The following parameters of importance for the rate and amount of alkali leaching are discussed in the literature review [7]:

- Pre-storage conditions (assumed less alkali leaching when pre-stored longer at 20°C due to the higher degree of hydration when exposed to the extreme storage environments).
- Prism size (documented higher fraction of alkali leaching (i.e. higher % of the total alkalis leached out) for specimens of smaller cross-section [3], [11]).
- Use of any wrapping (might reduce alkali leaching [12], or the opposite, decrease the expansion [13], [14]).
- ASR exposure temperature (assumed more alkali leaching at higher exposure temperature [15] due to higher diffusivity).
- Humidity (fog chamber assumed to give more alkali leaching (due to increased condensation of water on the prism surfaces) compared with storage of prisms in a humid container [12], [16]).
- Drying/wetting cycles (does cooling of the prisms prior to length measurements enhance the amount of alkali leaching?).
- Alkali content (amount of alkali leaching assumed to increase with increased concrete alkali level [17]).
- Cement type (could be of high importance [16], [18]).
- w/cm (assumed increased alkali leaching with increasing w/cm due to higher permeability [19], [20]).

In contrast to laboratory testing, where the whole cross-section of the prisms is exposed to alkali leaching, field concrete structures are, according to Rivard et al. [17], mostly not subject to significant alkali leaching (documented by pore solution analysis), probably due to the much higher volume to surface ratio compared with laboratory specimens. Some alkali leaching is, though, expected in the surface layer of concrete structures exposed to moisture. The problem of alkali leaching (leading to reduced prism expansion) is thus a big challenge in accelerated ASR laboratory tests, and is consequently an important issue to focus on in the PhD study.

2 THE TEST PROGRAMME

2.1 General

The PhD laboratory test programme has included four concrete mixtures (Section 2.2) and in total 58 ASR test series, most of them using modified versions of the draft RILEM aggregate concrete prism tests; AAR-3, 2000 (38°C, wrapped prisms) [21] and AAR-4.1, 2006 (60°C, unwrapped and wrapped procedure) [22]. For comparison, six test series with slightly modified versions of the Norwegian 38°C CPT [23] and 12 test series with the ASTM C1293 CPT [5] (38°C, unmodified version) were included. Details for the various CPTs are given in a separate paper [8] (Table 3). An overview of modifications made in this study is given in Section 2.3.

The main reason for incorporating the ASTM C1293 CPT [5] was to establish a link to the comprehensive experience in North America with this method and to document any batch-to-batch variation; 8 concrete batches were needed to cast all the concrete prisms with the "basis" binder and two batches were prepared with the "open" binder (Section 2.2). In a separate paper [8] it was concluded that all the concretes produced are of the desired quality and that test series cast from different concrete mixtures can be compared. Furthermore, it was concluded that the prism expansions can be compared without consideration of the slightly varying, but generally low (<3.0 %) air contents between the test series.

Except for the ASR testing, focus has been on alkali leaching measurements (Section 2.4) and documentation of moisture state in the concrete prisms (reported in a separate paper [8]). Additionally, a comprehensive complementary testing program for documentation of other concrete properties of importance for development of ASR has been performed (Section 2.5).

2.2 Materials and mixture proportions

Two CEM I Portland cements (EN-197-1), one high alkali (1.24 % $\text{Na}_2\text{O}_{\text{eq}}$) and one low alkali (0.60 % $\text{Na}_2\text{O}_{\text{eq}}$), and a CEM II/A-V cement containing 21.6 % of a class F fly ash (co-grinded with the clinker) were used in the study, see Table 1 (*comment: The CEM II/A-V cement normally contains 17-20 %*

fly ash). This type of blended cement has been widely used for years in Norway, partly in order to avoid ASR in combination with alkali-silica reactive aggregates.

A non-reactive natural gneiss/granitic sand from Årdal and an alkali-silica reactive crushed coarse aggregate, a cataclasite with crypto- to microcrystalline quartz from Ottersbo, were used in all mixtures, blended to produce a 60:40 coarse:fine ratio (by mass) - more details are given in [8]. The 14-day expansion of the sand and the coarse aggregate in the RILEM AAR-2 80°C accelerated mortar bar test [24] was measured to be 0.03 % (non-reactive) and 0.30 % (reactive), respectively (prism size 40x40x160 mm).

Details of the concrete mixtures are given in Table 2. Based on a series of considerations, the bulk of the testing was produced on a mixture containing 400 kg/m³ of Portland cement and water-to-cement ratio (w/c) of 0.45 (denoted "basis" binder). The two CEM I cements were blended to produce an alkali content of 3.7 kg/m³ Na₂O_{eq}. The alkali content was chosen (based on previous testing of the aggregates at SINTEF [25]) with aim to reach a final expansion of the reference test series lying on the steep (ascending) part of the "expansion-versus-alkali-level (S-shaped) curve", so that a small loss of alkalis due to alkali leaching would be detectable in terms of reduced expansion. If a high alkali level had been chosen, most of the test series would probably show a rather high expansion (i.e. lie on the plateau of the "expansion-versus-alkali-level curve"), even if significant quantities of alkalis were leached out during the ASR exposure. Then only minor differences in expansion would have been expected between the different test series.

To examine the impact of w/c, two additional concrete mixtures were cast with CEM I cement and w/c of 0.30 and 0.60 (respectively denoted "dense" and "open" binder). The cement contents of these mixtures were modified to achieve the desired workability, but the alkali content of the mixtures was maintained at 3.7 kg/m³ Na₂O_{eq} by appropriate blending of the CEM I cements (Table 2).

Additionally, one mixture was produced with w/cm of 0.45 using the blended fly ash cement (denoted "fly ash" binder, see Table 2). The alkali content of this mixture was raised from 5.0 kg/m³ Na₂O_{eq} (alkalis originating from the blended cement) to 9.0 kg/m³ Na₂O_{eq} by adding NaOH to obtain a final

expansion of the "fly ash" concrete mixture on the steep (ascending) part of the "expansion-versus-alkali-level curve", as well.

2.3 ASR test procedures – test series

2.3.1. Modification of the test procedures

The standard versions of the concrete prism tests have been slightly modified in order to investigate the effects of these modifications. The test procedure for the ASTM C1293 CPT [5] was not modified, apart from the use of prisms with 70x70 mm cross-section and not the prescribed 75x75 mm (*comment: In spite of this, the term "ASTM prisms" is used in this paper*). A summary of the changes is given below. The motivation for the modifications is given in the recently published literature review [7].

During all the testing only de-ionised water has been used as mixing water, in the moist cotton cloth wrapping (if any) and in the storage containers.

For all test series, the moulds were stored at ambient temperature in the laboratory under plastic foil from casting until de-moulding the following day. Furthermore, each prism was always stored vertically in the storage container with the same prism end pointing upwards.

For all standard versions of the CPTs, the mass and length were measured after cooling the prisms for about 16 hours inside their storage container in a room kept at $\sim 20^{\circ}\text{C}$. For these test series, the reference readings were performed at de-moulding (and after the 0.5-h submersion period where used). However, all measurements in the modified versions of the various concrete prism tests were performed without pre-cooling the prisms. To secure accurate measurements, i.e. reduce the influence of any weight loss and temperature variations from reading to reading, a detailed measuring procedure was developed. The reference readings of the "warm" prisms were taken the day after the prisms were exposed to their ASR exposure temperature (see Figure 1). The least reading of the length comparator was 0.001 mm.

The following specimen "pre-treatment" parameters and/or ASR exposure conditions have been varied when modifying the RILEM AAR-3 CPT (2000, [21]) and the RILEM AAR-4.1 CPT (2006, [22]) – see Figure 1 and Table 3 for details and motivation:

- The wrapping procedure (if any) was slightly modified, either by adding only half of the water content prescribed or by removing the prescribed polyethylene bag.
- The length of the "pre-storage" period was varied. The prisms were kept at 20°C until 1, 7 or 28 days after casting before being exposed to the ASR exposure temperature. However, for all test series, the prisms were prepared for final storage (e.g. wrapped) and put into the storage container immediately after de-moulding (and after the 0.5-h submersion period where used) and the initial measurements of weight and length.
- Some prisms were pre-cured for 24h at elevated temperature (60°C) to simulate the curing temperature in a massive concrete structure.
- Some prisms were sealed in epoxy and aluminium foil after de-moulding to avoid any exchange of water with the environment.
- Some prisms were stored submerged in deionised water (to maximize the alkali leaching conditions).
- Some prisms were wrapped with cotton cloth saturated with a basic solution of strength pH 14.2 (1.5 M OH⁻) or 13.2 (0.15 M OH⁻), respectively (instead of the usual de-ionised water), in order to try to reduce the amount of alkali leaching. The lowest pH level corresponds to the typical pH found in water filtered from fresh cement paste with a high alkali Norwegian CEM I after half an hour. The highest pH level corresponds to the calculated pH level in the pore water in the concrete with the "basis" binder after about one month of curing when much of the water has been consumed by hydration resulting in an increase in the concentration of alkali hydroxides in the pore solution. (*Comment: Some may find it strange with a pH above 14, but the pH scale from 0 to 14 is just the common range corresponding to 1M H⁺ and 1M OH⁻, respectively, and is not "limits"*).

2.3.2. Overview of test series

Figure 2 shows the notations used to label the various ASR test series. The full notations give a complete description of the specimen "pre-treatment" and "ASR exposure" of the prisms. However, to simplify, short names are used in most figures and tables when presenting the results.

Tables 4-7 give an overview of all 58 test series included in the test programme.

2.4 Alkali leaching measurements

To avoid any contamination, all the storage containers and any equipment used were washed thoroughly in a mild acid solution and de-ionised water. Additionally, a new lining was always used in each of the 38°C storage containers (no lining was used in any of the 60°C storage containers). Furthermore, "dummy" tests were performed with each type of container used (including any lining, the bottom grid, any separate sealing, any cotton wrapping and any plastic foil). After storing these containers up to one year at 38 or 60°C, no significant quantity of alkalis was measured in samples taken out from the de-ionised water in the bottom of these storage containers. Consequently, any alkalis sampled during the ASR exposure originated from the concrete prisms.

During the ASR exposure, a 20-ml sample was periodically extracted from the bottom of each of the storage containers at the same time that expansion measurements were made. Before sampling, the water in the bottom of each storage container was stirred. The sample was stored in alkali-resistant plastic bottles before being analysed ("dummy" tests documented no alkali supply from the plastic bottles). Simultaneously, the height of the water was measured as basis for calculating the volume of water in each container (for each type of container, several "pilot measurements" were performed in order to prepare a volume vs. height curve). At the end of the ASR exposure period, the total amount of water in the bottom of each of the storage containers was measured by weighing the water, improving the accuracy of the final "container reservoir measurements". The detailed sampling procedure developed in the pilot study is included in the recently published RILEM TC 219-ACS literature review report (Appendix 8) [6]. For the wrapped prisms, sampling the water in the bottom of the containers was only performed in the end of the ASR exposure.

After ending the ASR exposure, the alkali content in any cotton wrapping and in any lining inside the storage containers was also measured. The alkali content in the cotton wrappings was likewise measured after four weeks of exposure (on the "extra prism" made of each test series – see [8]). The linings and the cotton wrappings were cut in smaller pieces, submerged in 1500 ml of de-ionised water in plastic bottles that was shaken once a day for one week before 20-ml samples were extracted after stirring the solution.

The concentration of alkalis, sodium [Na] and potassium [K] (in mg/l), in all the samples collected was analysed by flame atomic absorption spectroscopy (FAAS) of type "SpectrAA-400".

Based on the volume of the concrete prisms stored within each container and the in-mixed alkali content of the different concrete mixes (only alkalis from the cement and the fly ash was included, i.e. the insignificant quantity of alkalis supplied from the minor amount of superplasticizer added to some of the concrete mixes was neglected), the total amount of alkalis leached out from the prisms (i.e. sum of alkalis in the bottom of the containers and alkalis in any wrapping and any lining) was calculated (expressed as kg $\text{Na}_2\text{O}_{\text{eq}}$ per m^3 of concrete and as % of in-mixed alkalis). In these calculations it was assumed that all the three prisms within one storage container leached out the same quantity of alkalis (except for the RILEM AAR-3 CPT, where only one prism was stored inside each container).

2.5 Complementary tests

2.5.1. Overview

In order to document properties of importance for development of ASR, comprehensive complementary testing has been an important part of the study (Section 1.2.2). A separate paper [8] presents and evaluates the results from measurements of concrete porosity, moisture state and transport properties. In addition to the rate and amount of alkali leaching (Section 2.4 and 3.2) and prism expansion (Section 3.3), this paper includes results from visual inspections and microstructural analysis of prisms after the ASR exposure (Section 2.5.2 and 3.4). Some further complementary tests (alkali release from aggregates and dynamic E-modulus) are included in the PhD thesis [26].

Before the laboratory testing started, a "pilot testing" program was carried out in order to develop detailed laboratory procedures to improve the reliability of the measurements.

2.5.2. Visual inspection and microstructural analysis

In order to confirm the presence and amount of ASR after the exposure, microstructural analyses have been performed on 15 selected concrete prisms. These examinations have included analysis of 16 fluorescence impregnated plane polished sections and 25 fluorescence impregnated and polished thin sections, as well as scanning electron microscopy (SEM) analysis of 11 of the thin sections. Additionally, a visual inspection including photo documentation of any surface cracking and precipitation was performed on one prism from each of the 58 test series. As part of this inspection, cut faces of the prism ends were carefully examined to search for any internal gel accumulation in cracks and pores.

The extent of internal cracking in the concrete prisms (Section 3.4.2) was documented in the plane polished sections that cover the whole prism (70x280 mm; two half prisms for the larger Norwegian prisms). In the photos taken in UV-light, the crack patterns appear clearly. By use of image analysis, the crack patterns were analysed to quantify the extent and spread in cracking in the concrete prisms.

Results from the thin section and SEM analyses are included in the PhD thesis [26]. A paper presenting some important findings from all the microstructural analyses was presented at the 14th ICAAR in 2012 [27].

3 RESULTS AND DISCUSSION

3.1 General

Before presenting the detailed results from the study (Section 3.2-3.4), some astonishing expansion results are presented in Figure 3 to demonstrate the substantial impact of the test procedure used on the prism expansion, and consequently on the outcome of a performance test. All the test series included in the figure have identical concrete composition ("basis" binder, Table 2). The figure covers

unwrapped as well as wrapped prisms exposed to 100 % RH and either 38°C or 60°C. Several of the testing variants represent test procedures used in various "commercial" CPTs (Section 2.3.1), i.e. they are by no means extreme. The 52 weeks expansion of the 38°C test series varies in the range of 0.17-0.31 %, while the 39 weeks expansion of the 60°C test series varies in the range of 0.04-0.22 %.

One of the most remarkable aspects of these differing results is that one of the testing variants that produced one of the lowest final 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 (2006) with wrapped prisms [22]) is, however, not recommended by RILEM TC 219-ACS any longer due to the results of this study).*)

3.2 Alkali leaching from concrete prisms

3.2.1. General

The results from the alkali leaching measurements are presented in the following sub-sections. Firstly, the distribution of the alkalis leached out are discussed, i.e. relative quantities found in the water at the bottom of the containers, in the cotton cloth (for wrapped prisms) and in the lining inside the containers. Secondly, the general findings are evaluated, before a more detailed discussion about the effect of prism size, specimen "pre-treatment", exposure temperature and binder type follows. Subsequently, the rate of leaching of Na compared with K is discussed. Finally, the effect of the measures taken to try to reduce the amount of alkali leaching is assessed.

3.2.2. Presentation of results – location of the alkalis leached out

The accumulated amount of alkali leaching is presented in the Figures 4, 6, 7 and 8 expressed as a percentage of the in-mixed alkalis, i.e. 3.7 kg/m³ Na₂O_{eq} for the CEM I binders (w/c ranging from 0.30-0.60, alkalis originating from the cement) and 9.0 kg/m³ Na₂O_{eq} for the "fly ash" binder (CEM II/A-V, w/cm of 0.45, included the alkalis originating from the cement, the fly ash and the added 4.0 kg Na₂O_{eq} NaOH per m³ of concrete) - see Tables 1 and 2. In Figure 5, the alkali leaching from the ASTM prisms is alternatively expressed as kg Na₂O_{eq} per m³ of concrete. However, this way of

presenting the results does not influence the relative differences between the various test series with the CEM I binders. But, due to the higher alkali content in the "fly ash" binder, the relative values between the CEM I test series and the "fly ash" test series is drastically changed. When expressed as a percentage of the in-mixed alkalis, the "fly ash" test series leach considerably less alkalis than the CEM I test series, while the "fly ash" binder leaches most alkalis when expressed as kg Na₂O_{eq} per m³ of concrete (see Section 3.2.7).

For the unwrapped test series (Tables 4-7), the accumulated contents of alkalis measured in the bottom of the various storage containers are shown as continuous lines in the Figures 4-8. Figure 7 (RILEM AAR-4.1) and Figure 8 (RILEM AAR-3) include the alkalis measured in the cotton cloth for the wrapped test series (Tables 3-5) at two ages: four weeks after starting the ASR exposure (measured on the "extra prism") and after ending the ASR exposure (mean of three prisms).

Furthermore, at the end of the ASR exposure of the 38°C test series, the content of alkalis absorbed by the linings used inside the various storage containers has been added to the accumulated content of alkalis measured in the bottom of the storage containers. Moreover, the accuracy of the final "container reservoir measurements" was improved compared with the interim measurements (Section 2.4). These "true" final quantities of alkalis leached out are shown as single data points in the Figures 4 and 5 (ASTM C1293 CPT) and in Figure 6 (Norwegian CPT) at age 52 and 112 weeks. For both CPTs, 25 to 30 % of the total amounts of alkalis leached out from the concrete prisms have been absorbed by the lining (same type of cotton cloth) during the exposure period. Consequently, for these two CPTs the "true" accumulated amount of alkalis leached out throughout the ASR exposure period is significantly higher (up to 25-30 %?) than shown in the Figures 4-6 (since only the final measurements include the alkalis absorbed by the lining).

For the AAR-3 test series (Figure 8), the presented results at age 52 and 112 weeks express the "true" content of alkalis leached out (i.e. sum of alkalis from any wrapping, the container reservoir and the lining). For this CPT, the relative amount of alkalis absorbed by the lining was only about 1/8 to 1/10 of what was found for the test series with the ASTM C1293 CPT and the Norwegian CPT (see above). The main reasons for this is believed to be that the wrapped AAR-3 prisms have a plastic

sheet covering the cotton cloth (only the top and bottom faces are uncovered), the prisms are stored inside a polyethylene bag inside the storage container and a less absorbing lining was used. Consequently, on average about 90 % of the alkalis leached out are trapped in the cotton cloth. The remaining (about 6 %) of the total quantity of alkalis leached out was found in the water in the bottom of the containers. Corresponding numbers for the wrapped AAR-3 test series stored without any polyethylene bag were as follows: about 40 % were trapped in the cotton cloth wrapping, about 55 % were found in the container reservoir and about 5 % were absorbed by the lining.

For the 60°C AAR-4.1 test series, no lining was used. Thus, the accumulated results for the unwrapped test series presented in Figure 7 express the "true" quantity of alkalis leached out throughout the full ASR exposure. Another consequence of not using any lining inside these rectangular shaped steel containers is that the accuracy of the interim volume estimations of the amount of water in the container (based on height measurements) is even better than for the other circular containers in which the lower part of the lining was submerged in the reservoir.

The average distribution of leached alkalis for the wrapped 60°C AAR-4.1 test series was as follows:

- Prisms stored inside a polyethylene bag: 55-65 % of the total amount of alkalis leached out (0.7 kg $\text{Na}_2\text{O}_{\text{eq}}$ per m^3 of concrete) was trapped in the cotton cloth wrapping and 35-45 % were found in the container reservoir.
- Prisms stored without any polyethylene bag: About 25 % of the total amount of alkalis leached out (1.2 kg $\text{Na}_2\text{O}_{\text{eq}}$ per m^3 of concrete) was trapped in the cotton cloth wrapping and about 75 % were found in the container reservoir.

One of the reasons for measuring a relatively higher portion of alkalis in the container reservoir in the 60°C AAR-4.1 containers compared with the 38°C AAR-3 containers might be that most polyethylene bags were broken (leakage in the joints) when exposed to 60°C for some time. Additionally, considerably more water vapour is observed inside the containers stored at 60°C compared with 38°C exposure when opening the containers during the exposure. Thus, the wrapped 60°C prisms are most likely exposed to relatively more "moisture movements" compared with exposure to 38°C.

3.2.3. General evaluation of the alkali leaching results

Figures 4-8 show that considerable quantities of alkalis are leached out of the concrete prisms during the ASR exposure. However, the rate and amount depend strongly on the prism cross-section, specimen "pre-treatment", binder type and exposure conditions. During the first 4 weeks of exposure, alkalis in the range of 3-20 % are leached out, constituting 0.10-0.75 kg Na₂O_{eq} alkalis per m³ of concrete for the CEM I binders (even more for the test series submerged in de-ionised water – see later). At the end of the exposure, from 14-37 % are in total leached out of the 60°C prisms (39 weeks), while corresponding numbers for the 38°C test series are in the range of 10-49 % (112 weeks). The highest number constitutes about 1.8 kg Na₂O_{eq} alkalis per m³ of concrete for the CEM I binders.

Before presenting the detailed results, the following remarks are considered relevant regarding the consistency of the results:

- The accumulated alkali leaching curves are smooth, indicating a satisfactory accuracy of the interim measurements.
- In general, similar ranking is obtained between the various binder types when tested according to different concrete prism tests.
- The distribution of alkalis between various locations inside the storage containers (i.e. in the container reservoir, in the wrapping or lining) is similar for comparable CPTs (Section 3.2.2).
- C.o.v. for the alkali leaching between parallel test series (i.e. repeated test series) and between comparable test series (i.e. with identical binder composition, but slightly different specimen "pre-storage") is acceptable. For the eight ASTM test series with the "basis" binder, the c.o.v. is in the range of 10-20 % (lowest at age 52 weeks). A similar c.o.v. is found for the three Norwegian test series with the "basis" binder. If test series N.1 (pre-cooled before measuring, which has some influence - see later) is excluded, the c.o.v. is reduced to 1-10 % (lowest at age 52 weeks). The c.o.v. for the five unwrapped AAR-4.1 test series with the "basis" binder is in the range of 7-15 % at age 8-39 weeks. If the pre-cooled test series (4.1) is excluded, the c.o.v. is reduced to 1-12 %.
- The total amount of alkali leaching is on a similar level as reported previously by Thomas et al. [3] for the ASTM C-1293 CPT and by Bokern [16] for unwrapped concrete prisms exposed to high humidity at 60°C.

- A very good correlation is found between the amount of alkali leaching at early age and prism expansion, in particular for the test series exposed to 60°C (Section 3.3.7).
- The extent and distribution of internal cracks in the concrete prisms correspond well with the amount of alkali leaching (see further discussion in Section 3.4.2).

(Comment: In addition to measurements of alkalis leaching, it was of interest to measure the remaining alkali content within the prisms. However, the results from the performed "pilot" tests were considered to be too uncertain to be included in this paper.)

As a basis to assess the parameters influencing the rate and amount of alkali leaching, the following general consideration of the mechanisms for alkali leaching is made: Alkali leaching consists of two steps: internal transport of the alkalis (to the prism surface) and external reception of the alkalis ("sink capacity"). The internal transport is dependent on diffusion properties, the distance the alkalis have to diffuse through the concrete pore water (dependent on the prism cross-section) and the "driving force" for alkali leaching (i.e. the difference in alkali concentration in the concrete pore water compared with the alkali concentration in any water (liquid) present on the prism surface [28]). The diffusion properties are primarily controlled by the permeability (dependent on binder type, w/cm and specimen "pre-treatment"), the water content and the exposure temperature. The "driving force" for alkali leaching is influenced by the "sink capacity" (see below) and the concentration of alkalis in the concrete pore water. Rivard et al. [17] has, for example, shown that increased alkali content in the concrete, and consequently higher concentration of alkalis in the concrete pore water, increases the alkali leaching. The concentration of alkalis in the concrete pore water further depends on the binder type and the w/cm, the moisture state and any reduction of the alkali content over time due to binding of alkalis in the ASR gel and/or alkali leaching. The fact that the alkali content in the concrete pore water is reduced with time is assumed to be one of the main reasons for the observed reduced rate of alkali leaching with time for most test series.

The "sink capacity" is dependent on the storage environment. The more water surrounding the prisms, the more leached alkalis can be absorbed by the water. The most extreme storage condition in this respect is prisms totally submerged in de-ionised water that is replaced with new de-ionised water

at every periodic measurement of length and mass of the prisms. Opposite, if only a limited number of water drops condense on the surface of the prisms stored on grids over water and run down the prisms into the water reservoir below, the "sink capacity" might be the limiting factor for alkali leaching. If that is the case, less influence of the parameters influencing the internal transport is expected (e.g. less influence of increased storage temperature, even if higher temperature gives higher mobility of ions and thus higher diffusivity).

3.2.4. Influence of prism size

For all binders, increasing the prism cross-section from 70x70 mm (ASTM size, Figure 4) to 100x100 mm (Norwegian size, Figure 6) decreases the rate and amount of alkali leaching considerably (the cross-sectional area of the latter is double of the former). As a result of this, the final expansion increases substantially (Section 3.3.4 and 3.3.5). For each of the binders, the "alkali leaching ratio" between the two prism sizes is rather constant throughout the full exposure period. On average the ratio is in the range of 1.8-2.5. The surface/volume ratio for the two prism sizes are 0.64 (ASTM) and 0.44 (Norwegian), respectively, giving a relative ratio of 1.45. For test series with equal concrete composition (with assumed similar concentration of alkalis in the concrete pore water) and comparable diffusion properties, it is logical that the rate and amount of alkali leaching decreases when the prism cross-section is increased, since the alkalis have to diffuse a longer distance. The advantage of increasing the prism size is previously reported by several scientists, e.g. Bakker [11], Thomas et al. [3] and Lindgård et al. [25]. An obvious consequence of this finding is that one effective measure to reduce the amount of alkali leaching during performance testing is to increase the prism cross-section.

3.2.5. Influence of cotton cloth wrapping

One important, but somewhat surprising, finding is that from prisms wrapped with a wet cotton cloth considerable quantities of alkalis are leached out during the first 4 weeks of exposure, significantly more than that measured for the unwrapped prisms (Figures 7 and 8). However, later in the exposure period, the rate of alkali leaching from the wrapped prisms is considerably less compared with the

unwrapped prisms (for some of the wrapped prisms exposed to 38°C hardly any more alkalis are leached out beyond 4 weeks of exposure). When exposed to 38°C for 4 weeks, the wrapped AAR-3 prisms with CEM I binders stored inside the prescribed polyethylene bags lose from 10-14 % of alkalis due to leaching (Figure 8) compared with 5-9 % for the unwrapped ASTM prisms of similar size (Figure 4). Corresponding numbers are 11-21 % (wrapped prisms) compared with 30-40 % (unwrapped prisms) after 52 weeks of exposure and 12-25 % (wrapped prisms) compared with 45-49 % (unwrapped prisms) after 112 weeks of exposure.

When exposed to 60°C for 4 weeks, the wrapped AAR-4.1 prisms (stored inside polyethylene bags) with CEM I binders lose 9-15 % of alkalis due to leaching compared with 3-9 % for corresponding unwrapped prisms (Figure 7). After 39 weeks of exposure, the wrapped AAR-4.1 prisms lose 16-32 % of alkalis compared with 26-37 % for unwrapped prisms.

The wrapping procedure is of high importance for the amount of alkali leaching and consequently influences the prism expansions (Section 3.3). When adding only half of the prescribed 80 g of water to the cotton cloth, a significant drop in the early-age alkali leaching (and a corresponding increase in the prism expansion) was observed for the 60°C test series; reduced from 13.0-13.5 % to 8.5-9.5 % (Figure 7). On the other hand, a considerably increased amount of alkali leaching was observed in the end of the exposure period (32 %) compared with comparable test series where the prescribed quantity of water was added to the wrapping (20 %). Reducing the amount of water added to the cotton cloth had less influence on the rate of alkali leaching from the AAR-3 prisms exposed to 38°C.

Furthermore, whether each wrapped prism was stored inside the prescribed polyethylene bag or not also had high influence on the amount of alkali leaching (Figures 7 and 8). When removing the bag, prisms exposed to 60°C for 4 weeks lost as much as 20 % of the in-mixed alkalis, and throughout the full exposure period about 1.5 times more alkalis were leached out from these prisms compared with prisms stored inside a bag. The influence was less at 38°C. Not surprisingly, the 60° test series without the polyethylene bag obtained the lowest expansion of all the wrapped 60°C test series (Section 3.3.4).

The reason for the increased amount of alkali leaching at early age when wrapping the prisms with a wet cotton cloth is believed to be increased "sink capacity" (see Section 3.2.3). For unwrapped prisms, some condensed water is present on the prism surfaces, running down from the top into the reservoir below [9]. These water drops pick up and transport the alkalis leached out from the prisms. For wrapped prisms, when adding the prescribed 80 g of water to the cotton cloth, the cotton wrapping is dripping wet with de-ionised water (some of the water was added on top of the prisms before closing the polyethylene bag – see Table 3). Consequently, the "sink capacity" is high, able to maintain the difference in alkali concentration between the concrete surface and the concrete pore water for a relatively long time, since more water has to reach a certain concentration before the diffusion is slowed down. However, over time, alkalis are gradually accumulated in the cotton cloth wrapping, thus reducing the "driving force" for alkali leaching. Additionally, the alkali concentration in the concrete pore water is decreased with time due to the alkali leaching and binding of alkalis in any ASR gel, e.g. as reported by Rivard et al. [29]. Therefore, the wrapped prisms leach out less alkalis later in the exposure period compared with the unwrapped prisms, where constantly more "pure water drops" are condensing and running down the prism surfaces.

When adding only half of the water to the cotton cloth, the cotton wrapping is not fully wetted and hence a reduced reservoir of de-ionised water is present on the prism surface and in the cotton cloth "pores" (i.e. less "sink capacity"). Consequently, less alkali leaching is measured at early age. In contrast, when removing the polyethylene bag, the wrapped prisms have access to more "moisture movements" as water is condensing and dripping from the underside of the lid down on the top surface of the wrapped prisms (i.e. increased "sink capacity"). The result is increased rate of alkali leaching throughout the full exposure period.

3.2.6. Influence of exposure temperature

In general, elevating the exposure temperature from 38°C to 60°C did not influence the amount of alkali leaching at early age (4 weeks), neither for wrapped nor for unwrapped prisms (Figures 7-9). This is surprising, since the rate of diffusion increases somewhat as the temperature increases, since temperature is a measure of molecular movement. One possible explanation might be that most test

series exposed to 60°C have already developed some ASR gel during the first 4 weeks of exposure. Consequently, some of the alkalis have been bound by the ASR gel and are thus less available for leaching ([28], [29]), i.e. the "driving force" for alkali leaching is reduced for the 60°C test series and thus counteracts the higher diffusion rate compared with the 38°C test series. Another possibility is that the "sink capacity" is limiting the rate of diffusion, not the internal diffusion of ions (see above).

However, a few exceptions were found; for example, the rate of alkali leaching was significantly increased for the 60°C test series submerged in de-ionised water (Figure 7) compared with submerged prisms exposed to 38°C (Figure 8). Similarly, the early-age alkali leaching from the wrapped test series stored without the polyethylene bag increased with the temperature. In both cases, the "sink capacity" is high. Consequently, the rate of diffusion more controls the rate of alkali leaching, and a higher influence of the elevated temperature is thus observed. Furthermore, these latter test series with the highest rate of alkali leaching only develop minor expansion (Section 3.3.4), meaning that less ASR gel is produced at early age. Thus, less alkalis are bound in the ASR gel [29], and consequently a high "driving force" for alkali leaching is maintained.

Fournier et al. [15] also documented a low influence of the exposure temperature on the rate of alkali leaching. They did not find a pronounced increase in the amount of alkali leaching after 3 weeks of exposure of unwrapped prisms when elevating the temperature from 38°C to 60°C (only a tendency to a little more alkali leaching at 60°C).

Throughout the full exposure period, the rate and amount of alkali leaching from the unwrapped AAR-4.1 prisms (60°C) and the unwrapped ASTM prisms (38°C) is comparable for almost all corresponding test series, see Figure 9 (after adding the assumed amount of alkalis absorbed by the lining in the ASTM containers (25-30 % of the total amount of leached alkalis, see Section 3.2.2) to the shown accumulated curves). The main reason for this could be the same as discussed above; the "sink capacity" is limiting the rate of diffusion for these unwrapped prisms, reducing the influence of the increased rate of diffusion with increasing exposure temperature. Only one (minor) exception is observed; for the unwrapped prisms with the CEM I "open" binder (w/c of 0.60), the rate of alkali leaching is somewhat higher at 60°C compared with 38°C in the period beyond 8 weeks of exposure.

In contrast, the temperature dependency for the alkali leaching is more evident (and more as expected) for wrapped prisms in the period beyond 4 weeks of exposure; those exposed to 60°C have a considerably higher rate of alkali leaching compared with those exposed to 38°C (Figures 7 and 8). This is most probably due to a higher "sink capacity" for the wrapped prisms, and thus the diffusion properties are of greater importance for the rate of alkali leaching. Furthermore, the rate of expansion reduces for the wrapped 60°C test series beyond 8 weeks of exposure (Section 3.3.4), i.e. less alkalis are bound in the ASR gel and consequently a higher "driving force" for alkali leaching is maintained.

3.2.7. Influence of binder type

The third somewhat surprising observation is that the rate and amount of alkali leaching is less dependent on the w/c of the CEM I binders than expected (Figures 4-9), despite the huge differences between the measured relative diffusion coefficients of water (RelDs) (reported in a separate paper [8]). However, generally for both exposure temperatures, no good correlation is found between the RelD and the amount of alkali leaching when comparing CEM I test series with different w/c (Figure 10). The reason for this is assumed to be that not only "internal" diffusion properties, but also other parameters (e.g. concentration of alkalis in the pore water and "sink capacity") influence the rate and amount of alkali leaching (discussed further below). Additionally, water (vapour) can move differently than alkalis (that can only diffuse through water-filled pores), i.e. the relative diffusion coefficients measured cannot directly be transferred into diffusion rates of alkalis.

For unwrapped prisms exposed to 38°C, the "open" binder (w/c of 0.60) and the "basis" binder (w/c of 0.45) exhibit a similar degree of alkali leaching throughout the first year of exposure (Figures 4-6), though slightly more for the "open" binder when tested in the Norwegian CPT (Figure 6). The rate of alkali leaching is as expected a little less for the "dense" binder (w/c of 0.30) during the first 6 months of exposure, probably due to the lower rate of diffusion. The lower rate of diffusion is not just due to a more refined pore structure, but also because the limited amount of larger pores (that dominate transport of ions by diffusion) are to a high extent empty in the "dense" binder (which is unsaturated due to self-desiccation). However, relatively more alkalis are leached out from the "dense" 38°C test series with time (*comment: please note that the measurements of alkali leaching from the Norwegian CPT after 1.5*

and about 2 years of exposure (Figure 6) are uncertain due to a rather low water content in the container). Thus, after two years of exposure the total amount of leached alkalis is higher for the test series with the "dense" binder than for the "basis" binder test series (comment: the "open" binder test series were only exposed for one year). This finding is assumed to primarily be connected to differences in the "driving force" for alkali leaching; the lower w/c ratio and the lower evaporable water content in the prisms with the "dense" binder both lead to higher concentration of alkalis in the pore water ([30], [31]), and thus an enhanced "driving force" for alkali leaching. For example, Rivard et al. [29] measured a considerably higher concentration of alkalis in the pore solution expressed from prism with CEM I cement and w/c of 0.40 (about 800 mmol/l after 4 weeks of exposure to 38°C) compared with corresponding prisms with w/c of 0.55 (about 400 mmol). Additionally, the lower amount of alkalis leached out from the "dense" binder test series in the first stage of the ASR exposure and the fact that less ASR-gel is produced in the "dense" binder test series due to a lower extent of ASR (see later), contributes to increase the differences in "driving force" between the "dense" binder test series and the CEM I test series with higher w/c and increased expansion. Furthermore, the slightly increased moisture state of the "dense" binder test series during the ASR exposure [8] will contribute to increase the rate of diffusion with time.

For unwrapped prisms exposed to 60°C, the rate of alkali leaching is significantly higher for the "open" binder compared with the two CEM I binders with lower w/c, which showed comparable alkali leaching throughout the full exposure period (Figures 7 and 9).

For wrapped prisms (Figures 7 and 8), the rate of alkali leaching during the first 4 weeks of exposure seems to be independent of the w/c ratio for both exposure temperatures. However, at the end of the exposure, the wrapped test series with the "open" binder showed the least amount of alkali leaching (no significant alkali leaching from 4 to 39 weeks), while the two CEM I binders with lower w/c still revealed a comparable amount of alkali leaching.

The ratio between alkali leaching from the "fly ash" binder (boosted with NaOH to raise the alkali content from 5.0 to 9.0 kg Na₂O_{eq} per m³ of concrete) and the CEM I binders (with 3.7 kg Na₂O_{eq} alkalis per m³ of concrete) is rather similar for all the concrete prism tests. After two years of

exposure, the total amount of alkali leaching for the "fly ash" test series is in the range of 10-22 %, constituting 0.9-2.0 kg Na₂O_{eq} alkalis per m³ of concrete. As a consequence of the higher alkali content of the "fly ash" binder, the relative alkali leaching ratio between the CEM I test series and the "fly ash" test series "seemingly contradicts" depending on how the results are presented (as a percentage of the total amount or as an absolute measure in kg Na₂O_{eq} alkalis per m³ of concrete).

3.2.8. Rate of leaching of Na compared with K

The Na/K of the alkalis leached out (K recalculated to Na_{eq}) has been calculated after 4 weeks of exposure and at the end of the exposure period in order to assess the following questions:

1. Is the rate of leaching of Na and K comparable throughout the exposure period?
2. Is there any binder dependency?
3. Is the Na/K the same for various exposure conditions?
4. Does the added Na (when boosting with NaOH) leach out in the same rate as the alkalis in the fly ash cement?

The initial Na/K of the various CEM I binders (based on values analysed by our laboratory by FAAS) was as follows: 1.01 ("open" binder), 1.44 ("basis" binder) and 1.85 ("dense" binder). The corresponding Na/K for the "fly ash" binder was 3.37 including the added alkalis (1.19 excluding the added alkalis). The ratio between the Na/K of the alkalis leached out and the Na/K of the various binders is denoted "leached out vs. binder Na/K ratio". A ratio < 1.0 means that relatively less Na than K is leached out compared with the Na/K of the binder. In this comparison, as a simplification it is assumed that all alkalis in the cement minerals are released to the pore water since the degree of hydration is rather high in most binders.

The Na/K of the alkalis leached out is consistent, i.e. for each of the binders the Na/K varies similarly for comparable exposure conditions. However, the Na/K of the alkalis leached out is dependent on binder type, exposure temperature, whether any cotton cloth wrapping is used and on exposure time.

After 4 weeks of exposure to both exposure temperatures, the average "leached out vs. binder Na/K ratio" for unwrapped test series with the "basis" binder and the "dense" binder is in the range of about 0.55 (c.o.v. is in the range of 2.0-2.5 % between test series with identical concrete composition and exposure conditions). The interpretation of this is that less Na than K is leached out in the first weeks of exposure. This is somewhat surprising, since the Na ion is considerably smaller than the K ion. However, the Na ion coordinates more water molecules (6) around itself and interacts more strongly with them due to its stronger polarization power (smaller ion with same charge) compared with the K ion. Thus, a possible explanation of the observed differences in the rate of diffusion between Na ions and K ions is that Na and K "molecules" (that interact with different amounts of water and thus have different "size") are diffusing through the concrete pore water rather than "single" Na and K ions. In free water, the ratio between diffusion of Na⁺ compared with K⁺ is according to a physics handbook [32] reported to be about 0.70.

For the "open" binder, the "leached out vs. binder Na/K ratio" is about 0.75 after 4 weeks of exposure of the unwrapped prisms. The corresponding value for the "fly ash" binder is about 0.70 (when including the alkalis added to boost the alkali level). If assuming that the alkalis originating from the fly ash cement are leached out with a similar Na/K as the "basis" binder (with equal w/cm), the higher "leached out vs. binder Na/K ratio" for the "fly ash" binder compared with the "basis" binder could be due to the fact that the added alkalis are leached out more quickly compared with the alkalis originating from the fly ash cement. This is to be expected as the added alkalis are immediately soluble whereas a significant portion of the alkalis in fly ash are bound in the glass and are not immediately available to the concrete pore solution.

Corresponding "leached out vs. binder Na/K ratios" for wrapped test series (in the range of 0.65-0.95 after 4 weeks of exposure) are always considerably higher than measured for comparable unwrapped test series (0.55-0.75). The difference is highest for the test series exposed to 60°C. When the rate of alkali leaching increases (wrapped prisms leach out considerably more alkalis than unwrapped prisms at early age - see above), it thus seems like relatively more Na is leached out compared with the test series with lower rate of alkali leaching. A similar observation is made for the submerged test series

with the "basis" binder ("leached out vs. binder Na/K ratios" in the range of 0.70-0.80 after 4 weeks of exposure vs. about 0.55 for comparable unwrapped test series).

Beyond the first months of exposure, considerably more Na compared with K is leached out from the concrete prisms with the "basis" binder and the "dense" binder (valid for both exposure temperatures). Consequently, at the end of the exposure period, the "leached out vs. binder Na/K ratio" is in the range of 0.60-0.90 (vs. about 0.55-0.70 after 4 weeks of exposure). The reason for this could partly be that the larger hydrated Na ions (i.e. 6 water molecules surrounding Na^+ moves with it) need longer time than the smaller K ions with less polarized water to diffuse out of the concrete prisms. Another contributing factor could be connected to binding of alkalis in the ASR gel during the ASR exposure. The composition of the ASR gel was analysed by WDS analyses as part of the SEM analyses (see [26]). The average Na/K (both recalculated to $\text{Na}_2\text{O}_{\text{eq}}$) of the ASR gel was 0.80 (mean of 97 analyses). Since the Na/K of the "basis" and "dense" binder is considerably higher (1.44 and 1.85, respectively) than the Na/K in the ASR gel, a surplus of Na might occur in the concrete pore water with time. This could again lead to leaching out of more Na ions with time.

A corresponding increase in the Na/K with time is not observed for unwrapped test series with the "open" binder and the "fly ash" binder exposed to 38°C. Since the Na/K of the "open" binder (1.01) is closer to the mean Na/K of the ASR gel (0.80), this finding supports the theory of a surplus of Na ions with time for the CEM I binders with lower w/c due to alkali binding in the ASR gel (i.e. a considerable less surplus of Na ions will occur for the "open" binder). However, the fact that the Na/K of the "fly ash" binder (included added alkalis) is as high as 3.37, does not support this theory. If so, a surplus of Na ions should have occurred for the "fly ash" binder, and thus enhanced leaching of Na ions with time. On the other hand, only a limited amount of ASR gel is produced in the 38°C test series with the "fly ash" binder (see later). Furthermore, the pozzolanic reaction itself consumes much of the alkalis added as NaOH to the mix water, reducing the expected surplus of Na ions with time.

Contradictory to the unwrapped test series with the "open" binder and the "fly ash" binder exposed to 38°C, the Na/K increases 20-30 % during the ASR exposure for the unwrapped test series exposed to

60°C and the wrapped test series exposed to both temperatures. The reason for this contradiction is not clear. One contributing factor can be that the fly ash reaction product (C-(A)-S-H) might be slightly different when exposed to 60°C compared with exposure to 38°C [33]. On the other side, the "fly ash" test series exposed to 60°C expanded considerably more than those exposed to 38°C, a fact that might support the theory of alkali binding in the ASR gel introduced above.

3.2.9. Influence of other "pre-treatment" conditions

During the 0.5 h submersion period in water after de-moulding (standard procedure for some of the CPTs), 3-4 % of the alkalis mixed in the 70x70x280 mm concrete prisms with the CEM I binder with w/c of 0.45 and 0.60 (constituting up to 0.15 kg Na₂O_{eq} per m³) leached out to the water, most from the prisms with highest w/c (measured on liquid samples collected from the de-ionised water after submerging selected prisms for 0.5 h). Since this submersion period had little effect on the internal moisture state of the prisms beyond the first weeks of exposure [8], it is recommended to skip this submersion sequence from the ASR testing procedures.

Another "pre-treatment" parameter that has been varied is the length of pre-storage at ambient temperature before exposing the prisms to elevated temperature; 1, 7 or 28 days, respectively (Tables 4-6). For most test series exposed to 38°C, the pre-storage length did not significantly influence the alkali leaching properties, neither at early age nor later during the exposure period. However, one exception was noticeable: The wrapped AAR-3 test series pre-stored for 28 days at 20°C (denoted "3.9-W-28d" in Figure 8) leached out considerably more alkalis after two years of exposure than the corresponding test series exposed to 38°C directly after de-moulding (denoted "3.8-W-1d"). A corresponding decrease in expansion was also measured (Section 3.3.4).

For test series exposed to 60°C, no influence on the rate of alkali leaching at early age was observed for the unwrapped prisms when varying the length of pre-storage at ambient temperature. However, pro-longed pre-storage tends to decrease the final amount of alkali leaching from these prisms (about 20 % less for test series pre-stored 28 days compared with the one exposed to elevated temperature at age 1 day). This finding is in contrast to the finding for the wrapped AAR-3 prisms. One explanation

could be that the moist wrapping applied on the AAR-3 prisms directly after de-moulding immediately contributes to leach out alkalis from the prisms, while the unwrapped AAR-4.1 prisms are less prone to alkali leaching during the pro-longed storage at 20°C. The latter prisms are thus allowed to become relatively dense before the ASR exposure and the increased alkali leaching attributed “ASR exposure” begins. However, the measured differences in the total amount of alkalis leached long term did not directly influence the expansion of the AAR-4.1 prisms. One reason could be that also the diffusion properties were influenced by the length of pre-storage at ambient temperature. The RelD was considerably reduced when the length of pre-storage was increased (see [8]), thus it contributes to decrease the expansion (due to reduced moisture and ion mobility, slowing down the ASR process) while the decreased alkali leaching does the opposite.

Pre-cooling the prisms before each measurement (of length and mass) should theoretically increase the rate of alkali leaching, see Section 1.3 and [7]. This study confirms this hypothesis. The influence varies depending on test conditions, but the same tendency is observed for all concrete prisms tests (i.e. for wrapped and unwrapped prisms and for both temperatures, see Figures 6-8 and Tables 4-6); pre-cooling before measuring increase the amount of alkali leaching. The influence is greatest after 4 weeks of exposure (increase of 8-60 %) compared with the end of the exposure period (increase of 4-20 %). The assumed mechanism is that cooling results in drying which again concentrates alkalis near the surface [34] - making them easier to be washed away.

The special "pre-treatment" given to some test series in order to simulate the curing temperature in a massive concrete structure, i.e. pre-curing for 24h at 60°C (Figure 1 and Tables 4 and 5), did not significantly influence the rate of alkali leaching for the test series later exposed to 38°C. With respect to total amount of alkali leaching, the test series later exposed to 60°C performed similarly as the other test series exposed to high temperature directly after de-moulding (i.e. slightly increased alkali leaching compared with longer pre-storage at ambient temperature).

Finally, the extreme exposure condition where the prisms were submersed in de-ionised water from de-moulding in order to maximize the alkali leaching conditions behaved as expected. However, a surprisingly high amount of alkali leaching was measured (probably due to the very high "sink capacity

– see section 3.2.3), with a slightly higher rate for the test series exposed to the highest exposure temperature (Figures 7 and 8). At the end of the exposure period, the measured amount of alkali leaching was as high as 80 % when exposed to 38°C and close to 100 % when exposed to 60°C. Consequently, these test series exhibited minor levels of expansion (Section 3.3.4).

3.2.10. Modifications trying to reduce the alkali leaching

To try to reduce the amount of alkali leaching, some prisms were wrapped with a cotton cloth saturated with a basic solution of strength pH 14.2 or 13.2 (see Table 3 and the background for the selection of the basic solutions in Section 2.3.1). For the test series with pH 14.2 in the wrapping, about 2.7 g of Na₂O_{eq} alkalis was present in the cotton cloth used to wrap each of the prisms (based on calculations), corresponding to about 50 % of the in-mixed alkalis in the CEM I concretes. The measured alkali content in the cotton cloth before wrapping the prisms (measured on "dummy samples" - see procedure in Section 2.4) was practically the same as the calculated value, confirming the consistency of the alkali leaching measurements. For the less alkaline wrapping (pH 13.2), one tenth of the alkali content in the "pH 14.2 wrapping" was present in the cotton cloth, corresponding to only about 5 % of the in-mixed alkalis in the CEM I concretes.

During the first 4 weeks of exposure, the alkali content in the "pH 14.2 wrapping" was reduced from about 2.7 g to about 1.3 g (60°C) and 1.5 g (38°C). At the end of the exposure, the alkali content in these cotton cloth wrappings was even more reduced (to about 0.5 g and 1.1 g, respectively), while the alkali content in the water in the bottom of each storage container correspondingly increased. The movement of moisture inside each of the polyethylene bags in which each wrapped prism was stored was thus able to gradually dilute the alkali concentration in the cotton cloth wrapping during the ASR exposure, most when exposed to 60°C. At the end of the exposure of the "pH 14.2 test series", the sum of alkalis measured in the wrapping of the three prisms within each container and in the container reservoir was a little lower than the alkali content added to the wrappings when preparing the test series. This indicates an uptake of alkalis by the concrete prisms from the "pH 14.2 wrapping" during the exposure period, corresponding to about 15 % (60°C) and 20 % (38°C), respectively, of the in-mixed alkali content of the "basis" binder concrete. However, one cannot rule out that even more

alkalis have been absorbed by the concrete during the first period of the ASR exposure (despite the amount of liquid in the cotton cloth was moderate – see Table 3), before some alkali leaching has occurred later in the exposure period when the concentration of alkalis in the cotton cloth wrapping was gradually reduced.

A result of the gradually reduced alkali content in the "pH 14.2 cotton cloth wrappings" during the ASR exposure is that the preventing effect (i.e. low "driving force" for alkali leaching) is correspondingly gradually reduced. However, the amount of alkali leaching during the first weeks of exposure has proven to be of greater importance for the final prism expansion than the total amount of alkali leaching (Section 3.3.7). It is thus not surprising that a considerable increased prism expansion is measured for these prisms compared with corresponding test series with de-ionised water in the cotton cloth wrapping, in particular when exposed to 60°C (Section 3.3.6).

For the "pH 13.2 wrapping", the alkali content in the cotton cloth wrappings increased considerably from start testing up to 4 weeks of exposure. This confirms that the wrapping with the reduced alkali content is not able to prevent alkali leaching from the concrete prisms during the ASR exposure. As discussed in Section 3.3.6, the effect on the prism expansions is also practically insignificant.

3.3 Prism expansion

3.3.1. General

Any change of length in the period from de-moulding to the reference readings (see Figure 1 and Section 2.3.1), primarily connected to the cement hydration, is discussed separately in Section 3.3.2 (denoted "pre-reference phase"). Of particular importance in this respect is the question; "when to take the reference readings"?

The periodic measurements of length change of the prisms from the reference readings up to exposure time either 39 weeks (60°C test series), 52 weeks or 112 weeks (38°C test series) are presented in the Section 3.3.4-3.3.7. The corresponding mass increase is discussed in a separate paper

[8]. That paper also gives details about the various concrete prism tests (Table 3), included recommended critical expansion limits, ranging from 0.030 % after 20 weeks of exposure for the 60°C RILEM AAR-4.1 CPT [35] up to 0.060 % after two years of exposure for the 38°C Norwegian CPT [36].

In order to document if the measured differences in prism expansion are statistically significant, all the expansion results are treated statistically as described in Section 3.3.3.

3.3.2. Length change in the "pre-reference phase"

Length changes in the "pre-reference phase" (i.e. the period from de-moulding to the reference readings) may be in the form of shrinkage or swelling, depending on the composition of the concrete and the surrounding environment. Generally, continued hydration of concrete cured in water leads to minor swelling/expansion due to absorption of water by the cement hydrates [37]. The magnitude on the swelling might be in the order of 0.005 % or slightly higher according to Aitcin [38]. On the other hand, if the moisture supply to the hydrating cement paste is insufficient, shrinkage occurs due to withdrawal of water from capillary pores caused by the self-desiccation process. These effects are clearly shown by comparing the sealed prisms (Table 3), giving a net shrinkage of -0.011 % during the 6 days of pre-storage at 20°C after de-moulding, with the prisms stored in de-ionised water during the same period giving an expansion of 0.001 %. Both values are for CEM I concrete with w/c ratio of 0.45 ("basis" binder).

Early-age shrinkage of a reversible nature may represent an "error" due to a resulting higher expansion when the prisms are placed in the ASR environment, provided the reference readings are taken after the pre-storage period at ambient temperature. However, if we assume that most of the early phase length changes may be reversible, a net shrinkage in the early phase may represent a "conservative approach", i.e. a phenomenon that is likely to increase the amount of expansion that is attributed to ASR and vice-versa.

The maximum “conservative error” (if we exclude the extreme sealed variant) is 0.006 % (“dense” binder: CEM I, w/c of 0.30, Norwegian prism size). Consequently, if we use an acceptance criterion (critical expansion limit) of 0.040 %, this maximum “conservative error” constitutes about 15 % of the expansion limit (compared with taking the reference readings directly after de-moulding). However, most test series reveal less shrinkage than the “dense” binder test series, which in general reveal the highest shrinkage. The average length change during the 6 days of pre-storage is -0.002 % (when excluding the sealed prisms), i.e. insignificant shrinkage, constituting about 5 % of a critical expansion limit of 0.040 %. We can conclude that the time of the reference readings is of minor importance for the final prisms expansion compared with the huge influence of alkali leaching (Section 3.3.7). Moreover, the time of the reference readings will only influence the conclusion from a performance test if the final expansion is very close to the acceptance criterion.

3.3.3. Statistical treatment of results

In order to evaluate the concrete prism expansion results statistically, arithmetic mean, standard deviation and c.o.v. of all test series were calculated. The multi-specimen c.o.v. for the ASTM C1293 test series ranged between 1 and 12 % at 52 weeks, with a mean of 7 %, i.e. lower than the reported multi-specimen, within-laboratory c.o.v of 12 % [5]. Furthermore, the c.o.v. for the mean expansion of the eight ASTM test series with the “basis” binder (expansion ranging from 0.254 to 0.279 %, Figure 11) was as low as 3.2 %, confirming the consistency of the expansion measurements. In order to test the equality of mean expansion for these eight series, one-way ANOVA was performed. The null hypothesis (H_0) was constructed so that all group means assuming equal to each other. With a significance level of $\alpha=0.05$, the critical f value ($f_{0.05,7,16}$) was computed as 2.6572. The f-value calculated from ANOVA was 0.8871. Given that $f < f_{\text{critical}}$ in this level of significance, there is no strong evidence to reject the null hypothesis. In other words, the difference between the mean of the eight test series were found insignificant with a p-value of 0.5384. Thus, the mean of parallel test series (8 different ASTM test groups) may be considered identical. Consequently, the test series prepared from various batches can be compared (see Section 2.1).

Likewise, all the Norwegian concrete prism test series revealed multi-specimen c.o.v. less than 13% at 52 weeks (mean of 6 %), while the corresponding c.o.v. was less than 12 % for all the 18 RILEM AAR-3 test series (mean of 7 %), except one (18 %). Based on these results, it can be concluded that the precision (repeatability) of the measurements performed with the three CPTs conducted at 38°C is good (average multi-specimen c.o.v. was in the range of 6-7 %).

For comparison, the repeatability in the round robin testing performed in the PARTNER project [39] with the RILEM AAR-3 method (wrapped prisms) was not as good (mean multi-specimen c.o.v. in the range of 14-21 % depending on the final expansion), but was still regarded as satisfactory. The corresponding mean c.o.v. obtained with the RILEM AAR-4.1 Alternative CPT (wrapped prisms) was lower, in the range of 9-16 %.

In the present study, the c.o.v. of the different AAR-4.1 test series was calculated after 13, 26 and 39 weeks of exposure, respectively. Except the sealed test series (with higher c.o.v.), all the c.o.v. values were < 22 % after 13 weeks (mean of 7 %), < 20 % after 26 weeks (mean of 9.5 %) and < 22 % after 39 weeks (mean of 10 %). The precision is thus satisfactory, but not as good as that obtained for the CPTs conducted at 38°C.

Additional statistical analyses, e.g. t-test, ANOVA and f-test, were performed for the evaluation of expansion test results and for the comparison of different test series. The detailed results from these analysis are included in the thesis [26]. In this paper, we only state in the discussion of results whether one test series expand significantly more or less than another.

Furthermore, the statistical analyses showed that the mean of all parallel test series cast in this study was found to be equal to each other at all ages.

3.3.4. "Basis" binder - effect of specimen "pre-treatment", prism size and exposure conditions

As discussed in Section 3.1, Figure 3 demonstrates the substantial impact on the rate of expansion and the final expansion (ranging from 0.04-0.31 % within the first year of exposure) of varying specimen

"pre-treatment", prism size and exposure conditions. The figure covers all unwrapped and wrapped test series with the "basis" binder stored over water in sealed containers exposed to either at 38°C or 60°C (*comment: documented by pilot measurements to give 100 % RH inside the "dummy" storage containers without concrete prisms*), except the test series with the most extreme exposure conditions (sealed and submerged storage), the results of which are included in Figure 12 (see later).

The early age (<13 weeks) rate of expansion for the test series exposed to 60°C is, as expected, considerably higher than those exposed to 38°C. At later ages, the opposite is observed, still as expected based on previous experiences. However, for similar exposure temperature, the "pre-treatment" of the concrete prisms highly influence the rate and level of expansion.

The parameter shown to have the highest impact on the final prism expansion, by reducing it, is the use of any moist cotton cloth wrapping. The reduction is dramatic for exposure to 60°C, but far less pronounced (not statistically significant) for exposure to 38°C (Figure 3). The final expansion of unwrapped prisms exposed to 60°C is up to five times higher than for corresponding wrapped prisms. The increase in expansion for the unwrapped prisms from 13 to 26 weeks is significant (about 0.05 %), while the expansion from 26 to 39 weeks is low (0.015-0.025 %), giving final expansions in the range of 0.18-0.22 %. For all the wrapped prisms exposed to 60°C, the expansion curves almost flatten out after 8-13 weeks of exposure, giving final expansions in the range of 0.04-0.09 %.

When exposed to 38°C, all the "basis" binder test series still expand after one year of exposure, giving 1 year expansions in the range of 0.17-0.25 % for wrapped RILEM AAR-3 prisms (Figure 3) compared with an average expansion of 0.27 % for corresponding unwrapped ASTM prisms (Figure 11). The effect of wrapping on the expansion at 2 years is less clear (see later).

Another parameter that significantly influences the final prism expansion is the prism size. Generally, increased prism cross-section increases the ASR expansion, on average by about 10 % after one year of exposure of the test series with the "basis" binder (Figure 3). The test series with the 100x100x450 mm Norwegian prisms continue to expand throughout two years of exposure. The increase from one

to about two years is about 0.12 %. Corresponding 70x70x280 mm ASTM prisms practically flatten after about 1.5 years of exposure, resulting in about 40 % higher expansion after about 2 years of exposure for the larger Norwegian prisms compared with the smaller ASTM prisms. Note that ASTM C 1293 specifies a prism with a 75x75 mm cross section and one might expect slightly smaller differences in expansion between prisms of this size and the 100x100 mm Norwegian prisms; however, the differences would still be significant.

The main reason for the remarkable reduced prism expansion when either wrapping the prisms or reducing the prisms cross-section is the considerable high impact these parameters have on the rate and amount of alkali leaching (see Section 3.2.4 and 3.2.5). In particular the rate and amount of alkali leaching during the first weeks of exposure have shown to be very important for the development of ASR expansion. The effect is most pronounced for test series exposed to 60°C. Three examples clearly illustrate this finding:

- 1) The early age alkali leaching is significantly reduced when adding only half of the prescribed water content to the cotton cloth wrapping (Figure 7), resulting in an increase in the final expansion by more than 60 % (shown as the two upper "60°C Wrapped" curves in Figure 3; two parallel test series with comparable expansion) compared with the use of the prescribed water content in the cotton cloth;
- 2) About 50 % more alkalis are leached out when removing the polyethylene bag from the wrapped test series exposed to 60°C (compared with use of the polyethylene bag). This test series reveals the lowest expansion of all the test series shown in Figure 3;
- 3) The test series permanently submerged in de-ionised water at 60°C, that leads to leaching of most of the in-mixed alkalis (Figure 7 and Section 3.2.9), hardly shows any expansion at all, see Figure 12. The corresponding test series permanently submerged in de-ionised water at 38°C, and that reveals about 20 % less alkali leaching (Figure 8 and Section 3.2.9), expands slightly more before the expansion curve flattens (Figure 12).

Furthermore, for the wrapped 38°C test series, the final expansion is generally reduced with increasing length of the pre-storage period at 20°C (1, 7 and 29 days, respectively). In particular, the expansion of the two test series exposed for slightly more than 2 years differs remarkably (Figure 3). The test series represented with the upper expansion curve was exposed to 38°C after 1 day, while the test series that

expanded less was stored at ambient temperature for 28 days before being exposed to 38°C. As shown in Figure 8, considerably more alkalis were leached from the latter test series (denoted "3.9-W-28d") than the one that expanded considerably more (denoted "3.8-W-1d"). It should also be mentioned that the "3.9" test series additionally had slightly lower internal moisture content than the more expansive "3.8" test series (discussed in a separate paper [8]), a fact that might have contributed somewhat to the lower measured final expansion. A similar effect of prolonging the pre-storage up to 28 days is not observed for the unwrapped 60°C test series (see Section 3.2.9).

The correlation between alkali leaching and prism expansion is further discussed in Section 3.3.7.

Figure 12 additionally shows the expansion of the two sealed test series. The one stored in the dry 38°C room reveals a little shrinkage (-0.02 %) after one year of exposure. As discussed in a separate paper [8], this test series lost some water during the exposure even though it was sealed. On the other hand, the sealed test series stored at 60°C did increase slightly in weight due to a minor uptake of water through the sealing of epoxy and aluminium foil. Consequently, the prism started to expand slowly, giving a final expansion slightly higher than the wrapped test series (Figure 3).

3.3.5. Other binders - effect of exposure temperature, wrapping and prism size

Figure 13 presents the expansion data for all the test series with the "open" binder (CEM I, w/c of 0.60). In principle, the expansion curves and the internal ranking between the various "open" binder test series are comparable with the "basis" binder test series (Figure 3 and Section 3.3.4). This is not surprising, since the alkali content is identical in both binders (as discussed in the literature review report [7], the ASR expansion primarily seems to be a function of the alkali content of the concrete and to some extent independent of the cement content), the moisture state is sufficient high in the concrete prisms prepared with both binders [8] and the rate and amount of alkali leaching are similar (Section 3.2.7).

Figure 14 shows the expansion data for all the test series with the "dense" binder (CEM I, w/c of 0.30). In principle, the expansion curves for the "dense" binder test series exposed to 60°C are

comparable with (i.e. not statistically different from) the corresponding results obtained for the "basis" and "open" binders with higher w/c (Figures 3 and 13). However, one small deviation observed is that the expansion curve for the wrapped 60°C test series with the "open" binder (Figure 13) flattens earlier and has slightly less (statistically lower) final expansion compared with the two CEM I test series with lower w/c (Figures 3 and 14), even if the amount of alkali leaching is comparable after four weeks of exposure for these wrapped prisms with w/c ratio varying from 0.30 to 0.60.

The rate of expansion as well as the final expansion for the "dense" binder test series exposed to 38°C is, however, dramatically reduced compared with the CEM I test series with higher w/c (Figures 3, 13 and 14). Nevertheless, they all still expand beyond the critical expansion limits. The main reason for this reduction is assumed to be less access to water (i.e. considerably lower internal relative humidity (RH) and evaporable water content), together with the substantially lower relative diffusion coefficient (RelD) for the "dense" binder test series compared with the test series with higher w/c (reported in a separate paper [8]). For the 60°C test series, the reduction in the internal RH and the RelD is less when w/c is lowered to 0.30. Additionally, the increase in internal RH during the ASR exposure (compared with the measured RH, performed at 20°C after cooling the prisms inside polyethylene foil) will be higher for the 60°C test series than for the 38°C test series (discussed in [8]). Thus, a relatively high RH is still obtained internally in the 60°C test series with the "dense" binder, securing sufficient moisture for the ASR to develop.

Another observation made, is that the wrapped "dense" binder test series exposed to 38°C expands somewhat more than the corresponding unwrapped test series in the period after 26 weeks of exposure (Figure 14), while the opposite was found for the CEM I test series with higher w/c (Figure 3 and 13). This finding contradicts that the wrapped test series leach out more alkalis in the first period of the ASR exposure. The main reason for this inconsistency is assumed to be that the internal moisture state is slightly higher in the wrapped "dense" binder test series than in the unwrapped prisms (discussed in [8]).

Furthermore, the expansion curves for the 38°C "dense" binder test series with the Norwegian CPT and the RILEM AAR-3 CPT practically flatten after about 1-1.5 years of exposure (Figure 14), in contrast to the still expanding "basis" binder test series (Figure 3). The ASTM test series flatten for both w/c ratios. Increasing alkali leaching with time (Figure 6 and 8), together with low diffusion coefficient and limited access to water during the ASR exposure (see above) are probably the reasons for these observations.

The "dense" binder test series with the Norwegian CPT still expand more than the test series with the ASTM C-1293 CPT with smaller prism cross-section, primarily due to less alkali leaching throughout the full ASR expansion period. In other words, the effect of the prism cross-section on expansion (see discussion above) is noticeable for all w/c ratios with the CEM I cement, as well as for the "fly ash" binder (see later). The difference in expansion between the two prism sizes, though, seems to increase with increasing w/c. The reason for this is not clear.

The shape of the expansion curves and the internal ranking between the various concrete prism test procedures are comparable for the "fly ash" binder (CEM-II/A-V, w/cm of 0.45, boosted from 5.0 to 9.0 kg/m³ Na₂O_{eq} alkalis – see Figure 15) and the "dense" binder (Figure 14), except that the 38°C "dense" binder test series expand a little more during the first year of exposure before flattening, while the corresponding "fly ash" test series expand more linearly. This analogue behaviour indicates that it is not only the alkali leaching that controls the prism expansion when these two binders are exposed to 38°C, but the internal moisture state and the diffusion properties also play a role. When exposed to 60°C, the internal RH is a little higher during the ASR exposure (compared with 38°C), contributing to the higher measured expansion. One cannot rule out that also other parameters that might influence the development of ASR are slightly different at 60°C compared with 38°C. For example might the fly ash reaction product (C-(A)-S-H) be slightly different [33], the rate of reaction of the fly ash compared with the rate of development of ASR might differ [7] and the pore solution chemistry might differ [7]. An example of the latter was recently presented by Matthias Böhm internally at Verein Deutscher Zementwerke (VDZ) (personal communication with Matthias Böhm): Measurements of alkali content of pore water pressed from cement paste (w/cm 0.50) cured at temperatures ranging from 8-60°C up to one year in tight plastic bottles showed that at 60°C the

amount of alkalis released into the pore water from cement paste with 30 % fly ash content was about 20 % higher than at 40°C.

The "fly ash" test series exposed for either 1 day or 28 days at ambient temperature prior to ASR exposure show comparable expansions for both temperatures (Figure 15). There is, however, a tendency that prolonged storage at 20°C leads to marginally higher expansion during most of the exposure period (only statistically different for the test series exposed to 38°C), despite the fact that the alkali leaching is a little bit higher.

The effect of binder type and exposure temperature on prism expansion for unwrapped test series with the Norwegian 38°C CPT and the 60°C RILEM AAR-4.1 CPT are summarized in Figure 16. The figure clearly shows that the CEM I binders with w/c ranging from 0.30-0.60 expand similarly when exposed to 60°C (documented to be statistically equal at age 26 and 39 weeks), somewhat more than the "fly ash" binder. Also when exposed to 38°C, the "fly ash" test series reveal the least expansion during the first 2 years of exposure. This is remarkable taking into account that the in-mixed alkali content is 9.0 kg/m³ Na₂O_{eq} for the "fly ash" binder (after boosting it from the initial 5.0 kg/m³) compared with only 3.7 kg/m³ for the other CEM I binders. This finding demonstrates the huge effect on the development on ASR of substituting cement by only about 20 % of a class F fly ash. The favourable effect of fly ash with respect to ASR is in accordance with previous findings [1], [25]. *(Comment: Pedersen [40] has previously documented the impact of alkali boosting on the concrete properties (reduced compressive strength). The possible effect of this on the expansion results obtained for the "fly ash" test series is not further evaluated).*

3.3.6. Effect of modifications trying to reduce the alkali leaching

As discussed in Section 3.2.10, the use of a cotton cloth wrapping saturated with a basic solution with pH = 14.2 seems to be able to prevent alkali leaching from the concrete prisms, at least in the first period of exposure, while the solution with pH 13.2 has little influence on the rate of alkali leaching. The effect on the prism expansion of these measures is shown in Figure 17. The effect of wrapping the prisms with cotton cloth saturated with the pH 14.2 solution is remarkable for prisms exposed to

60°C. During the full exposure period, these wrapped prisms expand about 25 % more than the corresponding unwrapped prisms after 26 and 39 weeks of exposure and up to 3.5 times more than the prisms wrapped with de-ionised water. The effect of wetting the cotton cloth with the pH 13.2 solution is negligible, i.e. those wrapped prisms exhibit similar expansion as the prisms wrapped with de-ionised water.

For prisms exposed to 38°C, the effect on the expansion of wrapping the prisms with cotton cloth saturated with the pH 14.2 solution is not statistically significant (but still slightly positive). No effect could be seen when using the pH 13.2 solution. However, the difference between wrapped and unwrapped prisms is generally less pronounced at 38°C (see Section 3.3.4).

Even if the alkali leaching is prevented (or minimized) when applying the pH 14.2 wrapping, the expansion curve for the 60°C practically flattens after about 26 weeks of exposure. This could be due to several reasons, in which reduction of the pH in the pore solution is assumed to be the most important one. The pH is reduced partly due to growth of ASR gel that contains alkalis [28]. Furthermore, Fournier et al. [15] found that the solubility of ettringite increases with increasing exposure temperature, leading to an increase in the concentration of sulphate ions in the pore water and a corresponding decrease in the concentration of OH⁻ ions to maintain electro-neutrality. Consequently, pH is reduced with time when exposed to 60°C. Another influencing factor could be connected to the ASR gel itself. After many ASR cracks have been induced in the concrete prisms, much gel must be produced to fill up and increase the width of these cracks. Thus, the rate of expansion might decrease due to "lack of" ASR gel. Additionally, Bokern [16] showed that the ASR gel is less viscous when the exposure temperature and the water content increase, which is the case for the 60°C prisms. This contributes to reduce the "expanding force" and increase the mobility of the ASR gel.

3.3.7. Correlation between alkali leaching and prism expansion

As discussed previously, alkali leaching has a very significant impact on the expansion of the CEM I concretes with an initial alkali content of 3.7 kg Na₂O_{eq}. For the test series exposed to 60°C, with

sufficient moisture content for ASR to develop and generally a higher relative diffusion coefficient than the 38°C test series [8], the amount of alkali leaching is totally controlling the prism expansion. In particular, the rate of alkali leaching in the first weeks of exposure is of high importance as shown in Figure 18 in which the remaining alkali content after 4 weeks of exposure is plotted against the expansion after 26 weeks. (*Comment: The reason for plotting the remaining alkali content after 4 weeks of exposure is the fact that the alkali leaching from the wrapped test series is only measured at this age and in the end of the exposure – see Section 2.4*). For the "basis" binder (w/c of 0.45), the determination coefficient R^2 for the trend line is 0.94, (excluding the submerged test series where, after just 4 weeks, rapid leaching results in the alkali content of the concrete being reduced to a value below the "alkali threshold" for the aggregate combination used). The impact on expansion of the early-age alkali leaching is similar also for the "open" binder (w/c of 0.60) and the "dense" binder (w/c of 0.30) (Figure 18).

One important reason for the very high impact of the early-age alkali leaching is that the alkali content of the concrete was deliberately selected such that small changes in alkali would have a significant impact on the expansion. This was done by considering the relationship between long-term expansion and alkali content for the reference test series, and selecting an alkali content that lay on the steep, ascending part of the s-shaped curve. Thus, the loss of alkalis at early age has a considerable impact in terms of reduced expansion. If the CEM I concretes had contained a surplus of alkalis (i.e. lying on the plateau of the "expansion versus alkali level curve", instead of the steep part – see Section 2.2), it is expected that alkali leaching would have a much lower impact on expansion. This is probably the reason for the contradictory findings in the RILEM round robin testing with the AAR-4 CPT (unwrapped prisms) and the AAR-4 Alternative CPT (wrapped prisms). The round robin testing program, using various aggregates and a binder with CEM I cement and about 5.5 kg $\text{Na}_2\text{O}_{\text{eq}}$ of alkalis, concluded that the two alternative 60°C aggregate test methods produced comparable expansions [41]. This conclusion is clearly in conflict with the observations from the present study.

The correlation between the total amount of alkali leaching and the final expansion is not particularly good for the prisms exposed to 60°C, most likely because most of the expansion has occurred already after 13-26 weeks of exposure. Furthermore, the prisms revealing the highest rate of alkali leaching show little expansion beyond 8 weeks of exposure.

The early age alkali leaching is also of high importance for the 38°C test series, even though the determination coefficient for the trend line for the "basis" binder test series in Figure 19 ($R^2=0.77$) is not as good as that found for the 60°C test series (Figure 18). The impact on expansion of alkali leaching is similar also for the "open" binder test series exposed to 38°C (Figure 19). In contrast, the "dense" binder (w/c of 0.30) test series do not fit into the same picture. The reason is most likely that the internal moisture state and the diffusion properties also influence expansion, as discussed previously.

Generally, the determination coefficient (R^2) for the trend line between the total amount of alkali leaching and the final expansion is not particularly good for the prisms exposed to 38°C. However, the tendency is that factors that reduce the amount of alkali leaching throughout the expansion period increase the final expansion for the CEM I test series with w/c of 0.45 and 0.60. Two examples illustrating this fact are the positive effect of increased prism cross-section and the different expansion obtained between AAR-3 prisms pre-stored 1 day compared with 28 days at ambient temperature (see Section 3.3.4).

With respect to performance testing, where alkali contents close to the "alkali threshold" normally are used in the concrete mixes, the impact of alkali leaching is regarded to be high. Additionally, the exposure period is normally extended compared with pure aggregate testing. Thus, the total amount of alkali leaching might have even greater influence than found in the present study. Consequently, to be able to mirror what will happen in a real concrete structure with minor or no alkali leaching, minimization of the rate and amount of alkali leaching during laboratory performance testing is crucial.

3.4 Visual inspection and microstructural analysis

3.4.1. Visual inspection

The visual inspections performed on one prism from all series after ending the ASR exposure confirmed the expansion measurements, i.e. those prisms that obtained high expansions also showed the highest extent of surface cracking and internal gel exudation in cracks and pores. The amount of

surface precipitation (assumed to primarily be calcium carbonate resulting from the carbonation of portlandite leached from the concrete) varied dependent on exposure temperature and use of any wrapping. The extent of precipitation was generally higher for unwrapped prisms exposed to 60°C compared with corresponding prisms exposed to 38°C. Hardly any precipitation was visible on the surface of the wrapped prisms after unwrapping them, indicating that any precipitation was absorbed by the cotton cloth.

3.4.2. Plane polished section analysis

3.4.2.1 Internal cracking

The analyses of the 16 fluorescence impregnated plane polished sections (Section 2.5.2) confirmed the results from the expansion measurements as well as the alkali leaching measurements. A huge spread in the extent of internal cracking was observed both internally within most of the prisms and between prisms from various test series. One example of the internal variation of crack intensity is presented in Figure 20, showing a UV-photo of a prism from a 60°C test series with the "open" binder (CEM I, w/c 0.60). The extent of cracking is very low in the bottom part (0-30 mm) and the upper part (80-100 mm) of the prism, while the medium section is more heavily cracked. The main reason for the lower crack intensity in the lower and upper part of the prism is assumed to be higher amount of alkali leaching in these areas. After 39-weeks, a total of 37 % of the in-mixed alkalis leached out during the ASR exposure (Section 3.2.5). This finding is valid for most prisms, except those prisms which showed the least amount of alkali leaching. An example of the latter is presented in Figure 21, showing the lower part of a Norwegian prism with the "basis" binder. In total about 20 % of the in-mixed alkalis leached out during the 112 weeks of ASR exposure. In this prism, the cracks seem to be rather homogenously distributed in the prism.

Corresponding results for the 14 remaining polished sections are presented in the PhD thesis [26] and in a paper presented at the 14th ICAAR in 2012 [27].

3.4.2.2 Measurement of "cracking intensity" by image analysis

At the 14th ICAAR, Lindgård et al. [27] presented a method where image analysis was used to quantify the "cracking intensity" in the concrete prisms, expressed as area-% occupied by cracks in each image (covering the whole prisms). During the transformation of the pictures of the fluorescence impregnated polished sections taken in UV-light to the images analysed, any air voids were removed (if not, these air voids filled with the fluorescence liquid will be miscounted as part of the cracking area). To measure any spread in internal "cracking intensity" over the prisms, each image was divided in crosswise slices of height 25 mm (from the bottom to the top of each prism) or alternatively lengthwise slices of width 14 mm (12.5 mm in the larger Norwegian prisms). The area-% occupied by cracks in each slice was analysed, before the values were normalized to the total area-%.

The measurements of "cracking intensity" over the prism height confirm the findings discussed in Section 3.4.2.1; the upper 25 mm of the prisms exhibited least cracking for most test series, followed by the lower 25 mm of the prisms. Furthermore, in most cases the slices located 25-50 mm from the top of the prisms obtained less cracking than the slices located in correspondent distance from the bottom of the prisms. For some samples with a high amount of alkali leaching (e.g. the prism shown in Figure 20), a larger area of the upper half of the prism had very low "cracking intensity" compared with the mid part, which undoubtedly revealed the highest "cracking intensity" [27].

Also the measurements of "cracking intensity" over the width of the prisms show a "cracking gradient"; the cracking was somewhat less in the outer 10-15 mm of most prisms compared with the interior of the prisms [27]. This indicates that less ASR is taking place in this outer layer, probably due to a higher amount of alkali leaching.

The results from the measurements of total "cracking intensity" (area-%) of all the 16 plane polished sections are presented in Figure 22. In this figure, the cracking intensities, ranging from about 0.3 to 6.7 area-%, are plotted against the measured prism expansions. A very good linear correlation is found ($R^2=0.89$), even though four different binder qualities were used (Table 2), giving concretes with 28 days compressive strength ranging from 44 to 103 MPa. The aggregate composition was, however,

identical in all test series. The good correlation found indicates that the cracks developed due to ASR lead to a corresponding increase in the prism expansion. Or conversely, if a prism expands during the ASR exposure, you will find a corresponding internal crack pattern in the prism.

Furthermore, the promising results indicate that the accuracy of the image analysing technique is sufficiently good to use the method as a tool to analyse the degree of ASR damage in larger concrete samples, at least for post-documentation of the internal cracking in laboratory exposed samples, but probably also for drilled cores taken from real structures. Rivard and Ballivy [42] have also previously found a relatively good correlation between the measured expansion caused by ASR on laboratory concrete prisms and the damage to concrete, as quantified by the Damage Rating Index (DRI) on polished sections prepared from these prisms. If various aggregate types are included in the same diagram, the correlation between prism expansion and internal cracking is assumed to be somewhat poorer (e.g. as is the case for the measurements performed by Rivard and Ballivy [42]).

The thin section and SEM analyses confirmed that ASR was the cause of expansion of the prisms. Further results from these analyses are included in the PhD thesis [26] and the ICAAR 2012 paper [27].

4 CONCLUSIONS AND RECOMMENDATIONS

4.1 Main findings

Based on the comprehensive laboratory study, including 58 test series with modified versions of five concrete prisms tests (CPTs), the following conclusions can be drawn:

- The rate of alkali leaching during the first weeks of exposure is the parameter shown to have highest impact on the prism expansion.
- A modified wrapping procedure developed (cotton cloth added alkalis) might be a promising tool to reduce the amount of alkali leaching during accelerated laboratory testing. Increasing the prism cross-section also decreases the rate and amount of alkali leaching considerably.

- The results clearly show that the amount of alkali leaching and the prism expansion is significantly influenced by the specimen "pre-treatment", "ASR exposure conditions" and prism cross-section. Most test conditions are by no means extreme, but represent test procedures used in various "commercial" CPTs. The extent of the impact depends on the concrete quality, i.e. w/cm and cement type. Consequently, the conclusion from a concrete performance test will differ depending on the test procedure used.
- Generally, a very substantial proportion of the in-mixed alkalis was leached out of the concrete prisms during the ASR exposure; 3-20 % during the first 4 weeks of exposure and 10-50 % at the end of the exposure of the CEM I test series (w/c of 0.30-0.60) with an initial alkali content 3.7 kg Na₂O_{eq} per m³ of concrete. For the test series submerged in de-ionised water, a substantial higher portion of alkalis was leached out of the concrete during exposure.
- A very good correlation is found between the amount of alkali leaching during the first 4 weeks and the final prism expansion, in particular for the test series exposed to 60°C. At this exposure temperature, the rate and amount of alkali leaching is the main controlling factor for the prism expansion. Consequently, to be able to mirror what will happen in a real concrete structure with minor or no alkali leaching, minimization of the rate and amount of alkali leaching during accelerated laboratory performance testing is crucial.
- In principle, the expansion curves and the internal ranking between the various "open" binder test series (CEM I, w/c of 0.60) are comparable with the "basis" binder test series (CEM I, w/c of 0.45) with equal alkali content. This is valid for both exposure temperatures.
- For less permeable concretes (i.e. the "dense" binder (CEM I, w/c of 0.30) and the "fly ash" binder (CEM II/A-V, w/cm of 0.45)) exposed to 38°C, it is not only the alkali leaching that controls the prism expansion, but the internal moisture state and the diffusion properties also play a role [8]. Thus, the two exposure temperatures produce different expansions. When exposed to 60°C, the internal RH is a little higher during the ASR exposure, contributing to the higher measured expansion (compared with 38°C). In a follow-up project [26], the field behaviour of some of these test series will be evaluated: one main aim is to assess which test procedure is best able to foresee the field behaviour of various concrete mixtures.

4.2 Effect of varying the specimen "pre-treatment"

With respect to variations in the specimen "pre-treatment", the main findings are:

- During the 0.5 h submersion period in water after de-moulding (standard procedure for some of the CPTs), 3-4 % of the alkalis mixed in the concrete prisms leached out into the water. Since this submersion period had little effect on the internal moisture state of the prisms beyond the first weeks of exposure, it is recommended to eliminate this submersion sequence from the ASR testing procedures.
- Wrapping of concrete prisms by use of moist cotton cloths and plastic sheets caused leaching of considerable quantities of alkalis during the first 4 weeks, significantly more than measured for the unwrapped prisms. The result was a dramatic reduction of the prisms expansion for the wrapped test series exposed to 60°C, while the effect was far less pronounced (not statistically significant) for the wrapped test series exposed to 38°C. "Traditional" wrapping of concrete prisms with a damp cotton cloth, applied in some test methods primarily with the aim to secure a high moisture content surrounding the prisms, is thus not recommended. *(Comment: This advice is already adopted by RILEM TC 219-ACS based on this study; the Alternative wrapped version of the AAR-4.1 CPT (60°C) is no longer recommended, and the revised version of the RILEM AAR-3 CPT (38°C) use unwrapped prisms similar as the ASTM C 1293 CPT).*
- The wrapping procedure is also of high importance for the amount of alkali leaching, in particular for test series exposed to 60°C. Less water added to the cotton cloth significantly reduced the early age alkali leaching and correspondingly increased the expansion. Opposite, removal of the polyethylene bag increased the alkali leaching and reduced the expansion.
- To try to reduce the amount of alkali leaching, some prisms were wrapped with a cotton cloth saturated with a basic solution of either pH = 14.2 or 13.2. For both exposure temperatures, the measurements indicates a small uptake of alkalis by the concrete prisms from the "pH 14.2 wrapping" during the exposure period, corresponding to about 15 % (60°C) and 20 % (38°C), respectively, of the in-mixed alkali content of the "basis" binder concrete. During the full exposure period, the wrapped prisms exposed to 60°C consequently expanded about 25 % more than the corresponding unwrapped prisms after 26 and 39 weeks of exposure and up to 3.5 times more than the prisms wrapped with de-ionised water. This "modified wrapping

procedure" might thus be a promising tool to reduce the amount of alkali leaching during accelerated laboratory testing (will be followed up [26]).

- The less alkaline wrapping (pH 13.2) was not able to significantly prevent alkali leaching from the concrete prisms during the ASR exposure. Consequently, the effect on the prism expansions was practically negligible.
- With a few exceptions, neither the pre-storage length at ambient temperature nor the "simulated field curing" did significantly influence the alkali leaching properties of the test series exposed to 38°C, neither at early age nor later during the exposure period. Likewise for the test series exposed to 60°C, no general influence on the rate of alkali leaching at early age was observed when varying the length of pre-storage at ambient temperature. However, prolonged pre-storage tends to somewhat decrease the final amount of alkali leaching from unwrapped prisms. However, none of these small differences in amount of alkali leaching directly influenced the expansion of the AAR-4.1 prisms.

4.3 Effect of varying the exposure conditions

With respect to variations in the "exposure conditions", the main findings are:

- In general, elevating the exposure temperature from 38°C to 60°C does not influence the amount of alkali leaching during the first 4 weeks, neither for wrapped nor for unwrapped prisms. Furthermore, throughout the full exposure period, the rate and amount of alkali leaching from the unwrapped AAR-4.1 prisms (60°C) and the unwrapped ASTM prisms (38°C) are comparable for almost all corresponding test series, even though the relative diffusion coefficient is considerably increased with increasing temperature. The main reason for this is assumed to be that the "sink capacity" is the limiting factor, not the rate of diffusion.
- In contrast, the temperature dependency for the alkali leaching is more evident (and more as expected) for wrapped prisms in the period beyond 4 weeks of exposure; those exposed to 60°C have a considerably higher rate of alkali leaching compared with those exposed 38°C, probably due to higher "sink capacity" for wrapped prisms (compared with unwrapped prisms) and thus more influence of differences in diffusion properties.

- For all binders, increasing the prism size cross-section from 70x70 mm (RILEM size) to 100x100 mm (Norwegian size) decreases the rate and amount of alkali leaching considerably. For many test series, the amount of alkali leaching is practically halved. As a result of this, the final expansion increases substantially. An obvious consequence of this finding is that one effective measure to reduce the amount of alkali leaching during performance testing is to increase the prism cross-section.
- Pre-cooling the prisms before every periodic reading of length and mass increases the amount of alkali leaching. The assumed mechanism is that cooling results in drying which again concentrates alkalis near the surface - making them easier to be washed away. However, no significant differences in expansion were found between pre-cooled test series and corresponding test series measured "warm".
- For the Norwegian CPT and the ASTM C 1293 CPT, both using an absorbing lining inside the storage containers in order to maintain a high humidity, 25 to 30 % of the total amounts of alkalis leached out from the concrete prisms have on average been absorbed by the lining (same type of cotton cloth) during the exposure period. The remaining alkalis were found in the water in the bottom of the containers. Thus, it is important to measure all alkalis that have leached out from the concrete prisms.

4.4 Influence of binder composition on alkali leaching

The binder composition, i.e. cement type and w/cm, have different effects on alkali leaching and prism expansion. Thus, the summary part is split into two sub-sections (4.4 and 4.5). With respect to the influence of binder composition on alkali leaching, the main findings are:

- At both exposure temperatures, the rate and amount of alkali leaching are less dependent on the w/c of the CEM I binders than expected, despite the huge differences between the measured relative diffusion coefficients of water vapour (RelD). This demonstrates that not only diffusion properties, but also other parameters, for example concentration of alkalis in the pore water and "sink capacity", influence the rate and amount of alkali leaching.
- For unwrapped prisms exposed to 38°C, the rate of alkali leaching is as expected a little less during the first 6 months of exposure of unwrapped test series with the "dense" binder (CEM

I, w/c of 0.30) compared with corresponding test series with higher w/c (0.45 and 0.60), probably due to the lower rate of diffusion. However, relatively more alkalis are leached out from the "dense" 38°C test series with time, assumed to primarily be caused by higher concentration of alkalis in the pore water and thus a higher "driving force" for alkali leaching.

- For unwrapped prisms exposed to 60°C, the rate of alkali leaching is significantly higher for the "open" binder compared with the two CEM I binders with lower w/c, which exhibit comparable alkali leaching throughout the full exposure period.
- For wrapped prisms, the rate of alkali leaching the first 4 weeks of exposure seems to be independent of the w/c for both exposure temperatures.
- As a consequence of the higher alkali content of the "fly ash" binder (9.0 kg Na₂O_{eq} per m³ of concrete, after boosting it from the initial 5.0 kg/m³), the relative alkali leaching ratio between the CEM I test series (3.7 kg Na₂O_{eq} per m³ of concrete) and the "fly ash" test series depends on how the results are presented. In absolute terms (e.g. in kg/m³ of alkalis) more alkalis are leached from the "fly ash" test series. However, when expressed as a proportion of the initial alkali content, the alkali leaching for the "fly ash" test series is considerably less than that for the CEM I test series.
- The ratio between alkali leaching from the "fly ash" binder and the CEM I binders is similar for all the concrete prism tests.

4.5 Influence of binder composition on prism expansion

With respect to the influence of binder composition (cement type and w/cm) on prism expansion, the main findings are:

- At both temperatures, corresponding test series with the "basis" binder and the "open" binder show comparable expansion (see Section 4.1).
- Similarly, the expansion curves for the "dense" binder test series (w/c of 0.30) exposed to 60°C are comparable with the corresponding results obtained at higher w/c. Furthermore, the "fly ash" binder test series expand almost as much as the CEM I test series at this exposure temperature, in contrast to what is observed at 38°C. One assumed reason for this behaviour is that a higher internal RH is obtained for these less permeable concretes when exposed to 60°C

compared with 38°C (see [8]). One cannot rule out that also other parameters that might influence the development of ASR are slightly different at 60°C compared with 38°C, e.g. the properties of the fly ash reaction product and the pore solution chemistry (see Section 3.3.5).

- When exposed to 38°C, the rate of expansion and the final expansion for the less permeable test series with the "dense" binder and the "fly ash" binder are dramatically reduced relative to 60°C and compared with the CEM I test series with higher w/c exposed to 38°C. This behaviour indicates that it is not only the alkalinity of the pore solution and the alkali leaching that controls the prism expansion when exposing these less permeable binders to 38°C, but also the internal moisture state and the diffusion properties (see [8]). To avoid any "false negative results" during accelerated performance testing, a fixed w/cm of 0.50 could be used (assumed to be conservative) until more research possibly documents that a lower (and more realistic) w/cm is safe to use; i.e. without resulting in a possible lower moisture state in the laboratory prisms compared with real concrete structures exposed to very high humidity.
- Another observation supporting the "lack of moisture theory" is that the wrapped "dense" binder test series exposed to 38°C expands a little more than the corresponding unwrapped test series in the period after 26 weeks of exposure, the opposite to what was found for the CEM I test series with higher w/c. This finding contradicts the observation that the wrapped test series results in more alkalis being leached out during the first period of the ASR exposure. The main reason for this inconsistency is assumed to be that the internal moisture state is slightly higher in the wrapped "dense" binder test series than in the unwrapped prisms (see [8]).
- The "fly ash" test series exposed to either 1 day or 28 days at ambient temperature prior to ASR exposure produce comparable expansions at both exposure temperatures.
- When exposed to 60°C, the CEM I binders expand a little more than the "fly ash" binder. Also when exposed to 38°C, the "fly ash" test series exhibit the least expansion after 2 years of exposure. This is remarkable taking into account that the in-mixed alkali content is 9.0 kg/m³ Na₂O_{eq} for the "fly ash" binder (after boosting it from the initial 5.0 kg/m³) compared with only 3.7 kg/m³ for the other CEM I binders. However, this finding is as expected and demonstrates the favourable effect on the development on ASR of substituting cement by only about 20 % of class F fly ash.

4.6 Complementary testing

The main conclusions from the complementary testing are:

- Measurement of alkali release from the aggregates indicated that only a minor (insignificant) amount of alkalis would be released from the aggregates under the conditions of the tests (further discussed in [26]).
- A new method for the measurement of "cracking intensity" (given as area-% of cracks in an impregnated polished section) based on image analysis has been developed. This method has been used successfully to compare the extent of cracking due to ASR in concretes, both internally within one plane polished section and between different test series.
- The image analyses of the 16 fluorescence impregnated plane polished sections confirm the results from the alkali leaching measurements. The main reason for the lower crack intensity in the outer/upper/lower parts of the prisms compared with the interior is assumed to be the higher amount of alkali leaching in these areas of the prisms.
- A very good linear correlation is found between "cracking intensity" and prism expansion ($R^2=0.89$), and this seems valid for all strength levels tested. This implies that the image analysing technique is sufficiently good to use the method as a tool to analyse the degree of ASR damage in larger concrete samples, at least for post-documentation of the internal cracking in laboratory exposed samples.

4.7 Some recommendations

Collectively, the data presented in this study indicate that the major shortcoming of the various concrete prisms tests is the loss of alkalis from the concrete during exposure. Consequently, the overriding recommendation to RILEM and those involved in the development of performance tests for evaluating the ASR potential of "job mixes" is to develop test procedures that limit, compensate for or, preferably, eliminate alkali leaching during testing. Further details on improvements in test procedures are given in the thesis [26] and in the literature paper [7].

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Table 1. Chemical composition (EN 196-2) of the three cements used in the study.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	Na ₂ O _e	P ₂ O ₅	LOI ¹
High alkali CEM I	19.61	4.87	3.48	61.03	2.83	3.81	1.11	0.51	1.24	0.15	2.44
Low alkali CEM I	20.06	4.67	3.31	63.06	2.01	3.40	0.39	0.34	0.60	0.16	2.24
CEM II/A-V ²	26.61	8.73	4.24	50.34	2.37	3.28	1.04	0.56	1.25	0.33	1.20

¹ Loss-Of-Ignition.

² Blended cement with a class F fly ash content of 21.6 wt%. Manufactured by co-grinding clinker and fly ash. Normally, the content of fly ash is in the range of 17-20 %.

Table 2. Composition of the four concrete mixtures included in the study.

Materials (kg/m ³)		Binder composition			
		CEM I, 0.45 "basis" binder	CEM I, 0.30 "dense" binder	CEM I, 0.60 "open" binder	CEM II/A-V, 0.45 "fly ash" binder
Cement	High alkali CEM I	200	60	285	---
	Low alkali CEM I	200	490	30	---
	CEM II/A-V	---	---	---	400
Aggregates (SSD ¹)	Årdal (gneiss/granite)	0/4	735	700	755
	Ottersbo (cataclasite)	4/8	185	175	190
		8/11	365	350	375
		11/16	550	525	565
Deionised water (free) (excl. any water in the superplasticizer)		180	165	189	180
NaOH (solids)		---	---	---	5.2
Alkali content (kg Na ₂ Oeq per m ³)		3.7	3.7	3.7	9.0
Superplasticizer (SIKA SSP 2000)		If necessary, add until workable and stable concrete (aimed slump 120 mm)			
De-foaming agent (SIKA)		If measured air content is > 3.0 %, add until air content is reduced to < 3.0 %			

¹ Saturated surface dry condition

Table 3. Specimen environment during testing.

Notation	Specimen environment ¹	Comments
A	Three <u>unwrapped</u> prisms stored inside each container	Standard procedure for RILEM AAR-4.1, ASTM C-1293 and the Norwegian CPT
B ³	Each prism was <u>wrapped</u> ² in damp cotton cloth and polyethylene (prism ends not covered). Before wrapping, each cotton cloth was submerged for minimum 10 minutes in 80g deionised water. Excess water not absorbed by the cotton cloth during submersion (~35-45g) was poured on the top surface before sealing the bag. This lead to a “water reservoir” in the bottom of each polyethylene bag, that after four weeks of exposure was measured to be in the range 4-25g for 38°C exposure and 0-2.5g for 60°C exposure	Standard procedure for RILEM AAR-3 and RILEM AAR-4.1 Alternative, including addition of 5ml deionised water on the top surface after every reading
C ³	Equal to “B”, except that each cotton cloth was submerged in half the amount of deionised water (i.e. 40g). All the water was absorbed by the cotton cloth	Motivation: Investigate the importance of the amount of water added to the wrapping
D	Equal to “B”, except that each wrapped prism was <u>not</u> sealed inside a separate <u>polyethylene bag</u> , but was placed on a grid inside the “AAR-3 container”. Neither was 5ml deionised water poured on the top surface at any time	Motivation: Investigate the importance of storing each wrapped prism in a separate polyethylene bag
E	After de-moulding, each prism was coated with <u>epoxy</u> . The next day the prisms were further <u>sealed</u> by packing them in <u>aluminium foil</u> . Further storage in dry containers	Motivation: Try to totally hinder any moisture exchange with the surroundings
F	After de-moulding, the unwrapped prisms were totally <u>submerged</u> in deionised water. After every reading, the water was exchanged with new deionised water	Motivation: Give the prisms the maximum alkali leaching conditions
G ³	Equal to “B”, except each cotton cloth was submerged for minimum 10 minutes in a basic solution with <u>pH 14.2</u> (Na/K-ratio≈1/3) simulating the pH in the pore water of the “standard CEM I binder” after ~28 days of curing. No extra solution was poured on the top surface, beyond the ~60g absorbed by the cotton cloth when it was submerged	Motivation: Investigate if application of a similar pH in the cotton cloth as in the concrete pore water is able to hinder alkali leaching from the concrete prisms
H ³	Equal to “G”, except each cotton cloth was submerged in a basic solution with <u>pH 13.2</u> (Na/K-ratio≈1/3) simulating a less basic pore solution. No extra solution was poured on the top surface, beyond the ~50g absorbed by the cotton cloth when it was submerged	Motivation: Investigate if application of a somewhat lower pH in the cotton cloth compared to the concrete pore water is able to reduce the extent of alkali leaching

¹ In all CPTs, the prisms are stored vertically on grids above water, without being in direct contact with the water. A humid environment close to 100% RH is aimed.

² Each wrapped prism was sealed inside a separate polyethylene bag. 5ml deionised water was poured on the top surface before sealing the bag and after each reading. Each bag was placed in a separate “AAR-3 container” with a tight lid.

³ For 60°C storage, three wrapped prisms were stored in an “AAR-4.1 metal container” instead of single “AAR-3 containers”.

Table 4. Overview of the 18 test series with modified versions of the RILEM AAR-3 38°C CPT (2000) [21].

Test series			Comments
Short name ¹	Full notation ²		
3.1-W-B-0.45	3.1-W-B-0.45-3-	N-7c	Standard RILEM AAR-3 test procedure (<u>wrapped</u> prisms, 7 days pre-storage at 20°C, prisms cooled before every reading)
3.2-W-B-0.45	3.2-W-B-0.45-4-	N-8	As 3.1-W-B-0.45, but readings taken without pre-cooling
3.3-W-C-0.45	3.3-W-C-0.45-4-	N-8	As 3.2-W-B-0.45, but less water in wrapping
3.4-W-B-0.45	3.4-W-B-0.45-4-	S-8	As 3.2-W-B-0.45, but prisms 0.5h submerged after de-moulding
3.5- -E-0.45	3.5- -E-0.45-5-	N-8	Sealed storage (epoxy and aluminium foil) after de-moulding (no water in the bottom of the storage containers)
3.6-W-D-0.45	3.6-W-D-0.45-6-	S-8	As 3.4-W-B-0.45, but no polyethylene bag
3.7-U- A-0.45	3.7-U -A-0.45-6-	S-8	As 3.4-W-B-0.45, but no wrapping (one prism in each container)
3.8-W-B-0.45	3.8-W-B-0.45-3-	S-2	As 3.4-W-B-0.45, but 1 day pre-storage at 20°C
3.9-W-B-0.45	3.9-W-B-0.45-2-	S-29	As 3.4-W-B-0.45, but 28 days pre-storage at 20°C
3.10-W-B-0.45-2	3.10-W-B-0.45- 2-	S-8FT	As 3.4-W-B-0.45, but simulating “field curing temperature” (see Fig. 1)
3.10-W-B-0.45-10	3.10-W-B-0.45-10-	S-8FT	As 3.10-W-B-0.45-2, but repeated test series
3.11-U- F-0.45	3.11-U -F-0.45-5-	S-8	Stored submerged in deionised water after de-moulding
3.12-W-G-0.45	3.12-W-G-0.45-12-	S-8	As 3.4-W-B-0.45, but pH 14.2 in wrapping at start
3.13-W-H-0.45	3.13-W-H-0.45-12-	S-8	As 3.4-W-B-0.45, but pH 13.2 in wrapping at start
3.4-W-B-0.30	3.4-W-B-0.30-7-	S-8	As 3.4-W-B-0.45, but lower w/c ratio
3.4-W-B-0.60	3.4-W-B-0.60-8-	S-8	As 3.4-W-B-0.45, but higher w/c ratio
3.8-W-B-FA-0.45	3.8-W-B-FA-0.45-9-S-2		As 3.8-W-B-0.45, but fly ash binder with boosted alkali level
3.9-W-B-FA-0.45	3.9-W-B-FA-0.45-9-S-29		As 3.9-W-B-0.45, but fly ash binder with boosted alkali level

¹ Used in figures and tables when presenting results.

² See details in Figure 2.

Table 5. Overview of the 22 test series with modified versions of the RILEM AAR-4.1 60°C CPT (2006) [22].

Test series			Comments
Short name ¹	Full notation ²		
4.1-U-A-0.45	4.1-U -A-0.45-1-	S-1c	Standard RILEM AAR-4.1 test procedure (<u>unwrapped</u> prisms, “reactor”, 1 day pre-storage at 20°C, prisms 0.5h submerged after de-moulding and cooled before every reading)
4.2-U-A-0.45	4.2-U -A-0.45-1-	S-2	As 4.1-U-A-0.45, but readings taken without pre-cooling
4.3-U-A-0.45	4.3-U -A-0.45-6-	S-8	As 4.2-U-A-0.45, but 7 days pre-storage at 20°C
4.4-U-A-0.45	4.4-U -A-0.45-2-	S-29	As 4.3-U-A-0.45, but 28 days pre-storage at 20°C
4.5-U-A-0.45	4.5-U -A-0.45-2-	S-8FT	As 4.3-U-A-0.45, but simulating “field curing temperature” (see Fig. 1)
4.6-U-F-0.45	4.6-U -F-0.45-5-	S-8	Stored submerged in deionised water after de-moulding
4.7- -E-0.45	4.7- -E-0.45-5-	N-8	Sealed storage (epoxy and aluminium foil) after de-moulding (no water in the bottom of the storage container)
4.8-W-B-0.45-1	4.8-W-B-0.45-1-	S-2	Standard RILEM AAR-4.1 Alt. test procedure (<u>wrapped</u> prisms ³ , 1 day pre-storage at 20°C), except prisms 0.5h submerged after de-moulding and readings taken without pre-cooling
4.8-W-B-0.45-10	4.8-W-B-0.45-10-	S-2	As 4.8-W-B-0.45-1, but repeated test series
4.9-W-B-0.45	4.9-W-B-0.45-5-	S-8	As 4.8-W-B-0.45-1, but 7 days pre-storage at 20°C
4.10-W-C-0.45-6	4.10-W-C-0.45-6-	N-8	As 4.9-W-B-0.45, but less water in wrapping
4.10-W-C-0.45-12	4.10-W-C-0.45-12-	N-8	As 4.10-W-B-0.45-6, but repeated test series
4.11-W-D-0.45	4.11-W-D-0.45-3-	S-8	As 4.9-W-B-0.45, but no polyethylene bag
4.12-W-G-0.45	4.12-W-G-0.45-12-	S-8	As 4.9-W-B-0.45, but pH 14.2 in wrapping at start
4.13-W-H-0.45	4.13-W-H-0.45-12-	S-8	As 4.9-W-B-0.45, but pH 13.2 in wrapping at start
4.3-U-A-0.30	4.3-U -A-0.30-7-	S-8	As 4.3-U-A-0.45, but lower w/c ratio
4.9-W-B-0.30	4.9-W-B-0.30-7-	S-8	As 4.9-W-B-0.45, but lower w/c ratio
4.3-U-A-0.60-I	4.3-U -A-0.60-8-I	S-8	As 4.3-U-A-0.45, but higher w/c ratio
4.3-U-A-0.60-II	4.3-U -A-0.60-8-II	S-8	As 4.3-U-A-0.60-I, but repeated test series
4.9-W-B-0.60	4.9-W-B-0.60-11-	S-8	As 4.9-W-B-0.45, but higher w/c ratio
4.2-U-A-FA-0.45	4.2-U -A-FA-0.45-9-S-2		As 4.2-U-A-0.45, but fly ash binder with boosted alkali level
4.4-U-A-FA-0.45	4.4-U -A-FA-0.45-9-S-29		As 4.4-U-A-0.45, but fly ash binder with boosted alkali level

¹ Used in figures and tables when presenting results.

² See details in Figure 2.

Table 6. Overview of the 6 test series with modified versions of the Norwegian 38°C CPT [23].

Test series			Comments
Short name ¹	Full notation ²		
N.1-U-0.45	N.1-U- A-0.45-3-	S-1c	<u>Standard Norwegian CPT</u> procedure (1 day pre-storage at 20°C, prisms 0.5h submerged after de-moulding and cooled before every reading)
N.2-U-0.45	N.2-U- A-0.45-1-	S-2	As N.1-U-0.45, but readings taken without pre-cooling
N.3-U-0.45	N.3-U- A-0.45-4-	S-8	As N.2-U-0.45, but 7 days pre-storage at 20°C
N.3-U-0.30	N.3-U- A-0.30-7-	S-8	As N.3-U-0.45, but lower w/c ratio
N.3-U-0.60	N.3-U- A-0.60-8-	S-8	As N.3-U-0.45, but higher w/c ratio
N.3-U-FA-0.45	N.3-U- A-FA-0.45-9-S-8		As N.3-U-0.45, but fly ash binder with boosted alkali level

¹ Used in figures and tables when presenting results.

² See details in Figure 2.

Table 7. Overview of the 12 test series with the ASTM C-1293 38°C CPT [5].

Test series			Comments
Short name ¹	Full notation ²		
ASTM-U-0.45-1	ASTM-U-A-0.45-1-	N-1c	<u>Standard ASTM C-1293</u> test procedure (1 day pre-storage at 20°C, prisms cooled before every reading)
ASTM-U-0.45-2	ASTM-U-A-0.45-2-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-3	ASTM-U-A-0.45-3-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-4	ASTM-U-A-0.45-4-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-5	ASTM-U-A-0.45-5-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-6	ASTM-U-A-0.45-6-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-10	ASTM-U-A-0.45-10-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.45-12	ASTM-U-A-0.45-12-	N-1c	As ASTM-U-0.45-1, but new batch
ASTM-U-0.30-7	ASTM-U-A-0.30-7-	N-1c	As ASTM-U-0.45-1, but lower w/c ratio
ASTM-U-0.60-8	ASTM-U-A-0.60-8-	N-1c	As ASTM-U-0.45-1, but higher w/c ratio
ASTM-U-0.60-11	ASTM-U-A-0.60-11-	N-1c	As ASTM-U-0.60-8, but repeated test series
ASTM-U-FA-0.45-9	ASTM-U-A-FA-0.45-9-	N-1c	As ASTM-U-0.45-1, but fly ash binder with boosted alkali level

¹ Used in figures and tables when presenting results.

² See details in Figure 2.

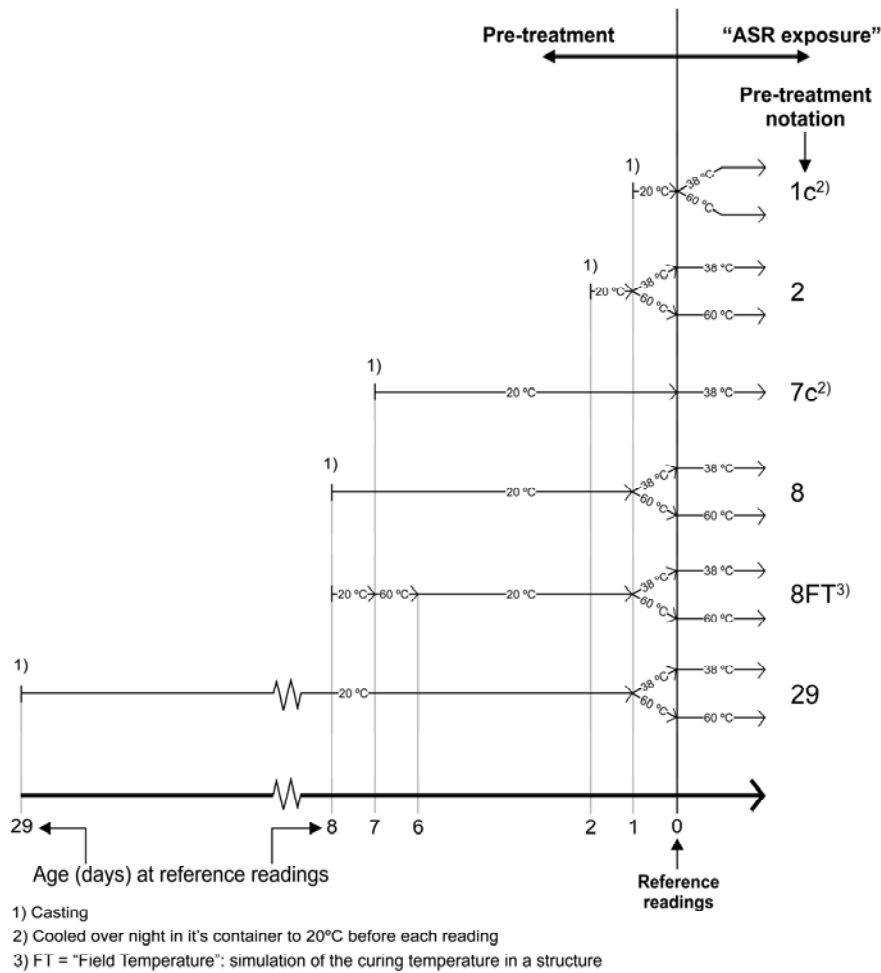


Figure 1 Pre-treatment of the various test series, included notations. For all test series, the prisms were prepared for final storage and put into their storage container immediately after de-moulding (and after the 0.5 h submersion period where used – see Figure 2 and Tables 4-7).

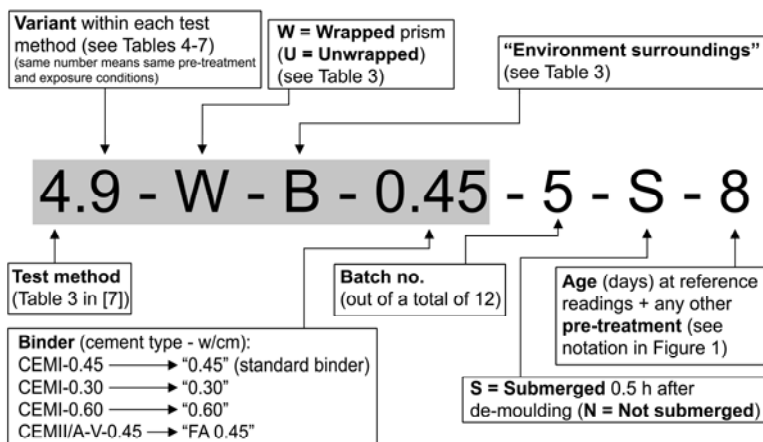


Figure 2 Notations used to name the various test series. The short names given in the Tables 4-7 are marked with grey shadow. For repeated test series, the batch no. is additionally included in the short name.

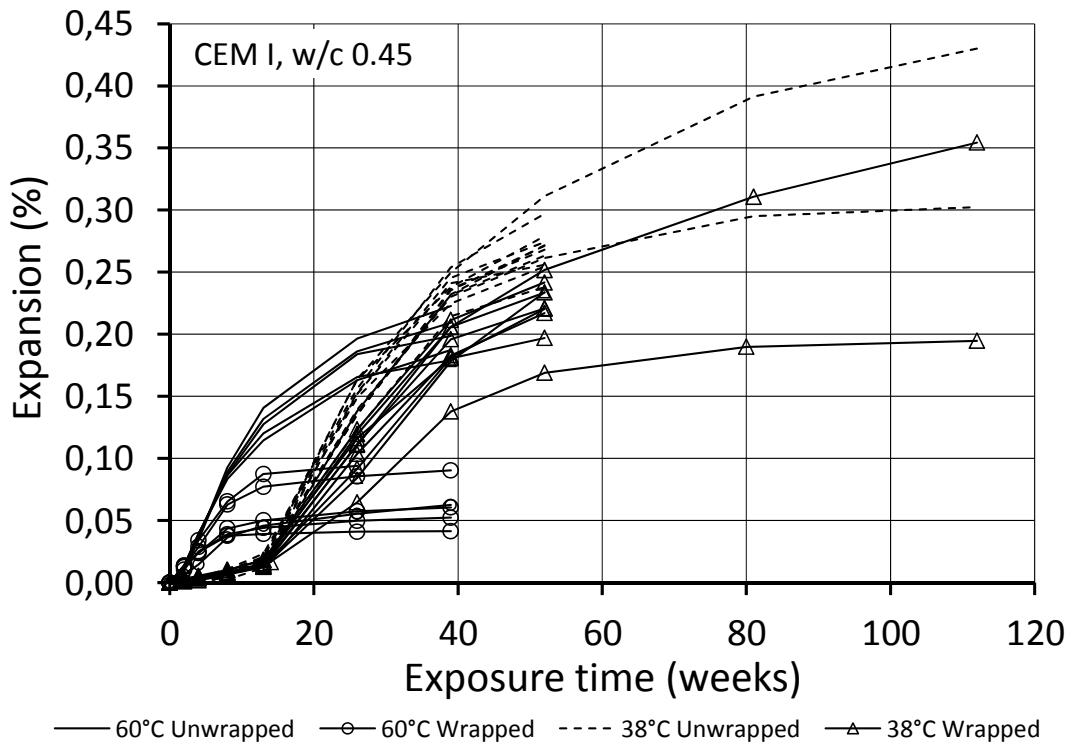


Figure 3 Expansion versus time for 32 test series with the "basis" binder (CEM I, w/c 0.45, $3.7 \text{ kg/m}^3 \text{ Na}_2\text{O}_{\text{eq}}$ alkalis).

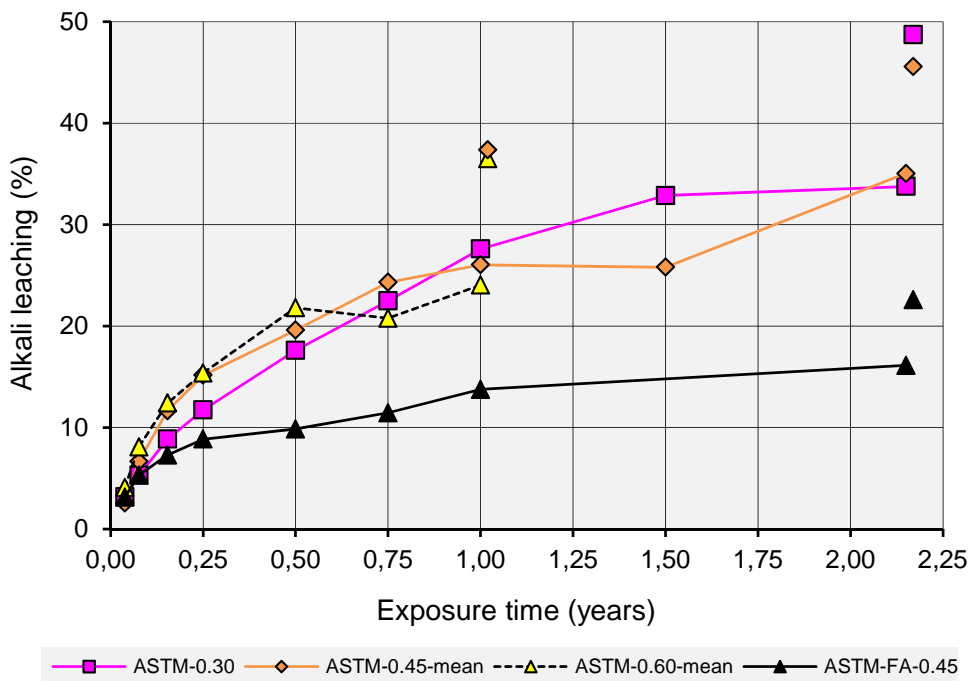


Figure 4 Alkali leaching from the unwrapped ASTM prisms (% of the initial concrete alkali content). The single data points include alkalis absorbed by the lining – added to the accumulated alkali content in the bottom of the storage containers at the end of the exposure period. See Figure 2 and Table 7 for abbreviations.

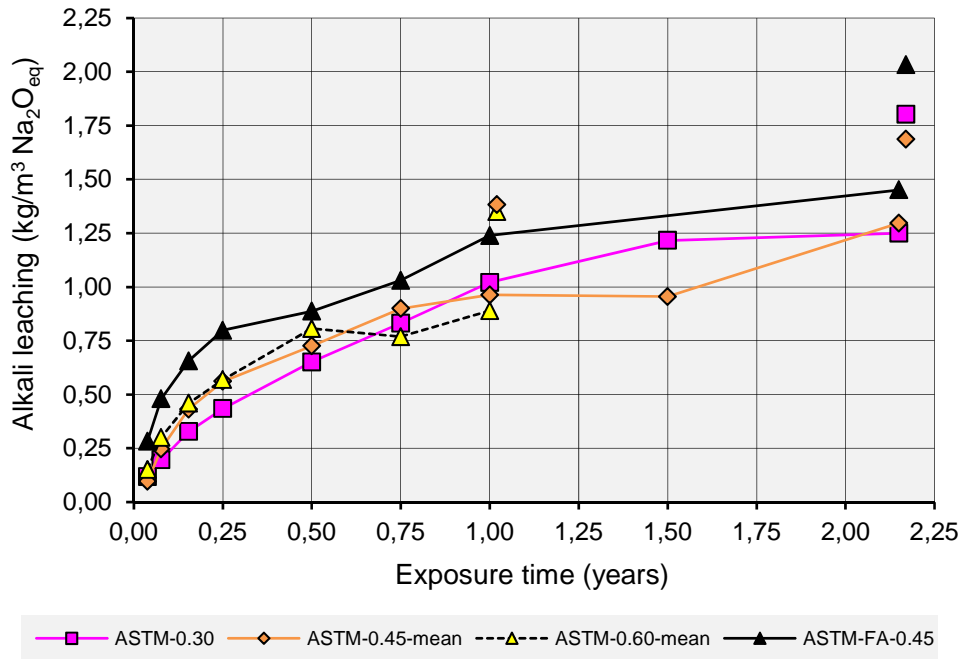


Figure 5 Alkali leaching from the unwrapped ASTM prisms (expressed as $\text{kg/m}^3 \text{Na}_2\text{O}_{\text{eq}}$). The single data points include alkalis absorbed by the lining – added to the accumulated alkali content in the bottom of the storage containers at the end of the exposure period. See Figure 2 and Table 7 for abbreviations.

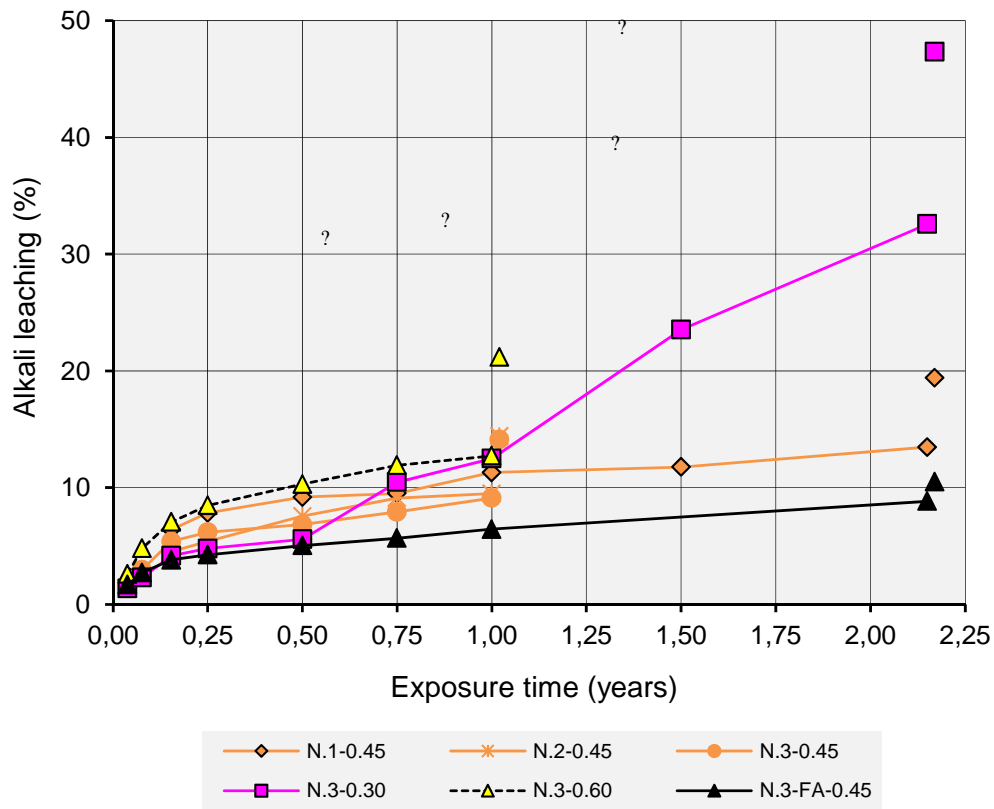


Figure 6 Alkali leaching from the unwrapped Norwegian CPT prisms (% of the initial concrete alkali content). The single data points include alkalis absorbed by the lining – added to the accumulated alkali content in the bottom of the storage containers at the end of the exposure period. See Figure 2 and Table 6 for abbreviations. The four data points with a question mark are uncertain (due to uncertain volume of water).

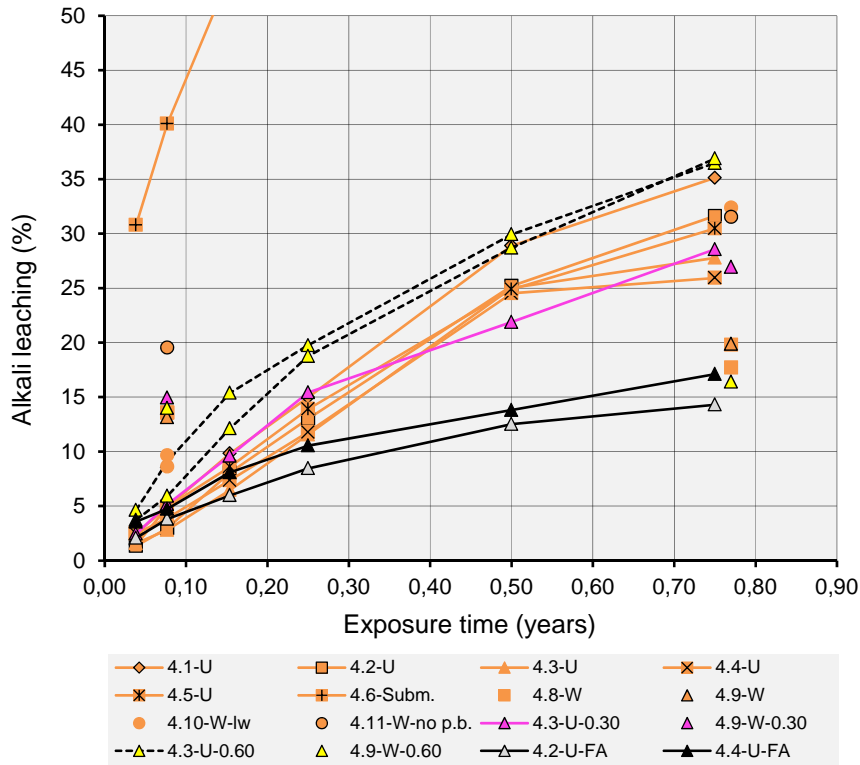


Figure 7 Alkali leaching from the AAR-4.1 prisms (% of the initial concrete alkali content). The single data points represent wrapped prisms (W). The accumulated curves represent unwrapped prisms (U). See Figure 2 and Table 5 for abbreviations. (Remark: Different scaling on the x-axes compared with the Figures 4-6 and 8-9).

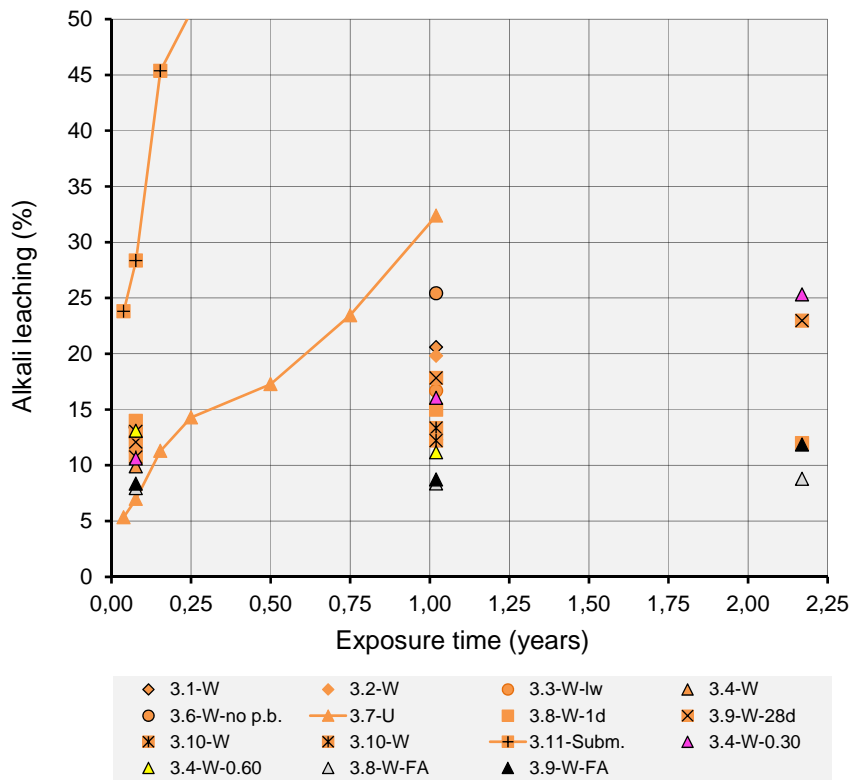


Figure 8 Alkali leaching from the AAR-3 prisms (% of the initial concrete alkali content). The single data points represent wrapped prisms (W). The accumulated curves represent unwrapped prisms (U). See Figure 2 and Table 4 for abbreviations.

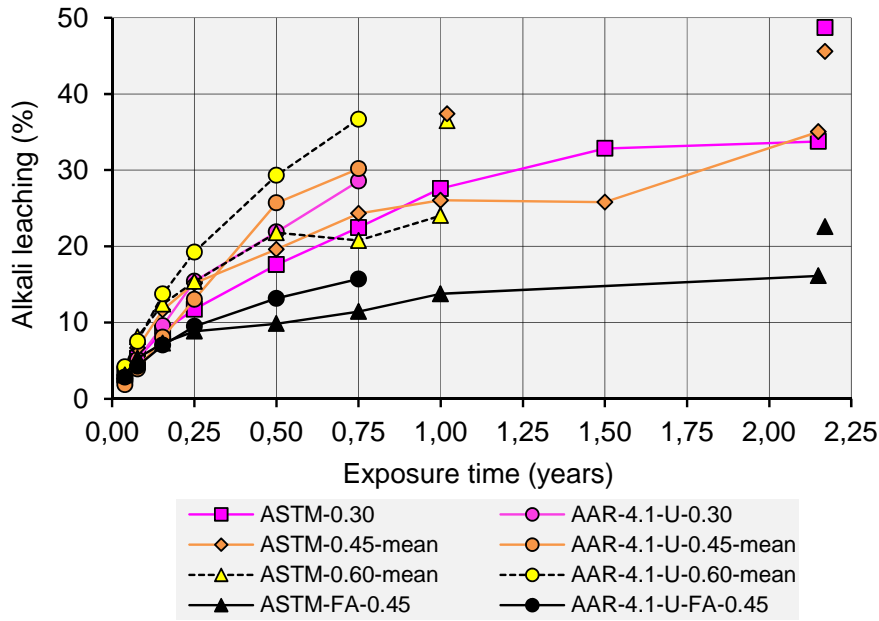


Figure 9 Temperature dependency of alkali leaching; accumulated mean results for the ASTM C 1293 CPT (38°C) and the AAR-4.1 CPT (60°C) – all unwrapped prisms. The alkalis absorbed by the lining in the ASTM CPT are not included, except for the single points at age 52 and 112 weeks (upper points). See Figure 2, Table 5 and Table 7 for abbreviations.

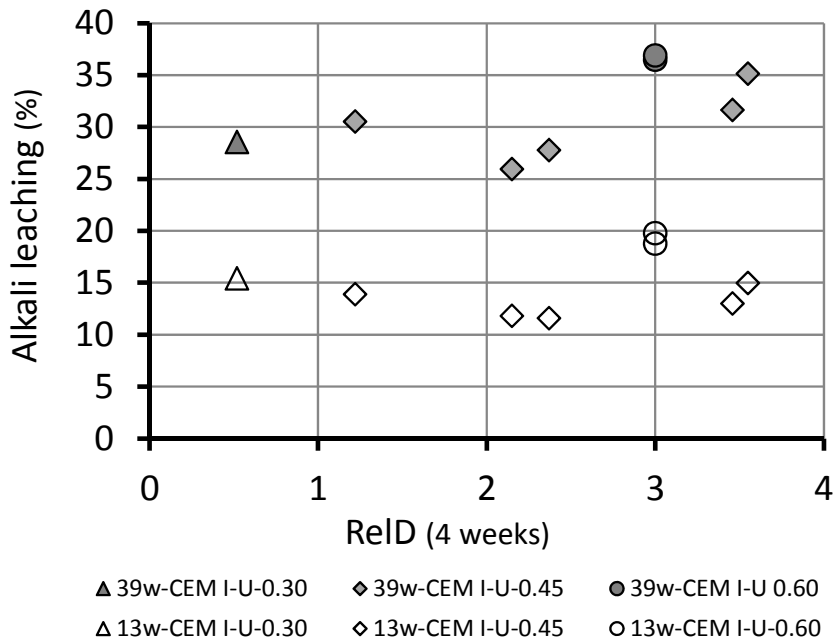


Figure 10 Relative diffusion coefficient (RelD) vs. alkali leaching at two ages (13 and 39 weeks) for unwrapped test series with the RILEM AAR-4.1 60°C CPT. See Figure 2 and Table 5 for abbreviations.

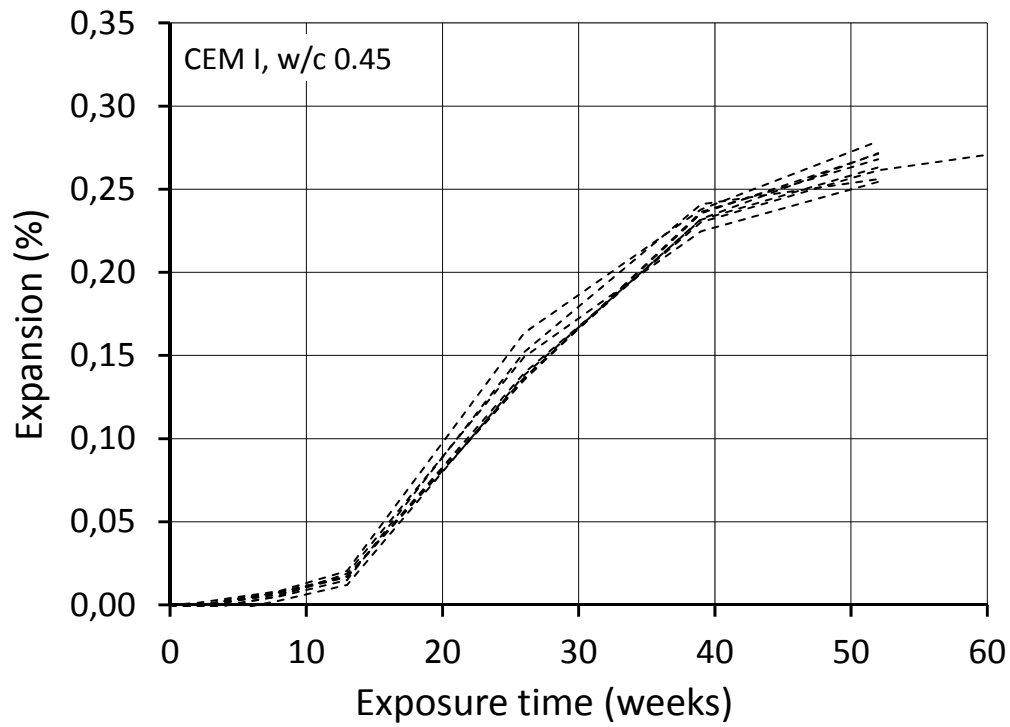


Figure 11 Expansion versus time for the eight ASTM test series with the "basis" binder (CEM I, w/c 0.45, 3.7 kg $\text{Na}_2\text{O}_{\text{eq}}$ alkalis per m^3 of concrete).

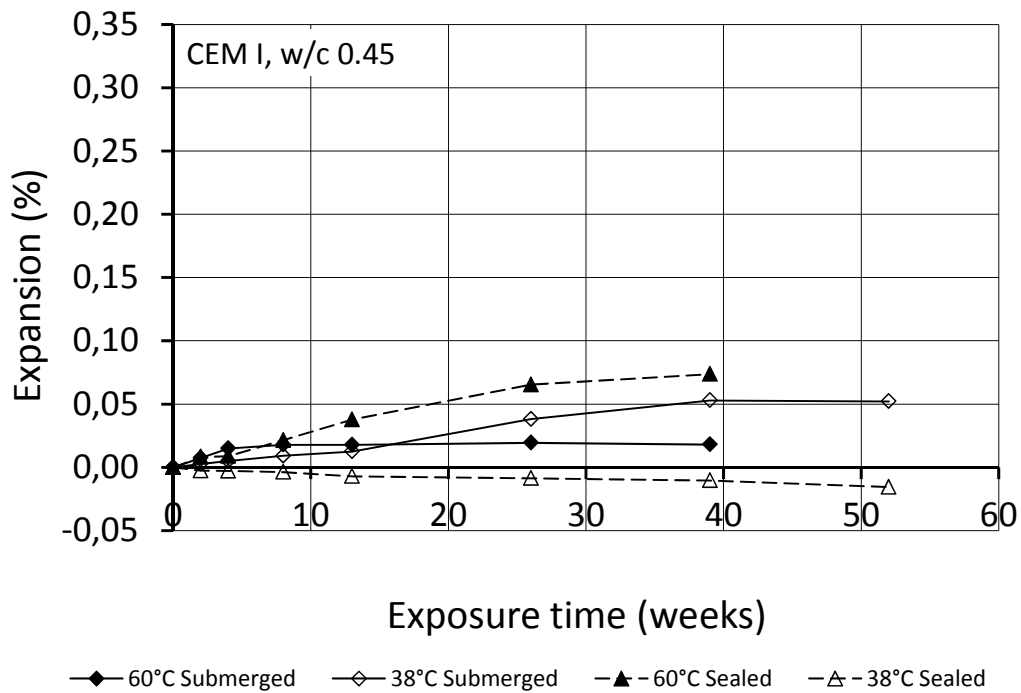


Figure 12 Expansion versus time for four test series with the "basis" binder (CEM I, w/c 0.45, 3.7 kg $\text{Na}_2\text{O}_{\text{eq}}$ alkalis per m^3 of concrete) given "extreme" exposure conditions.

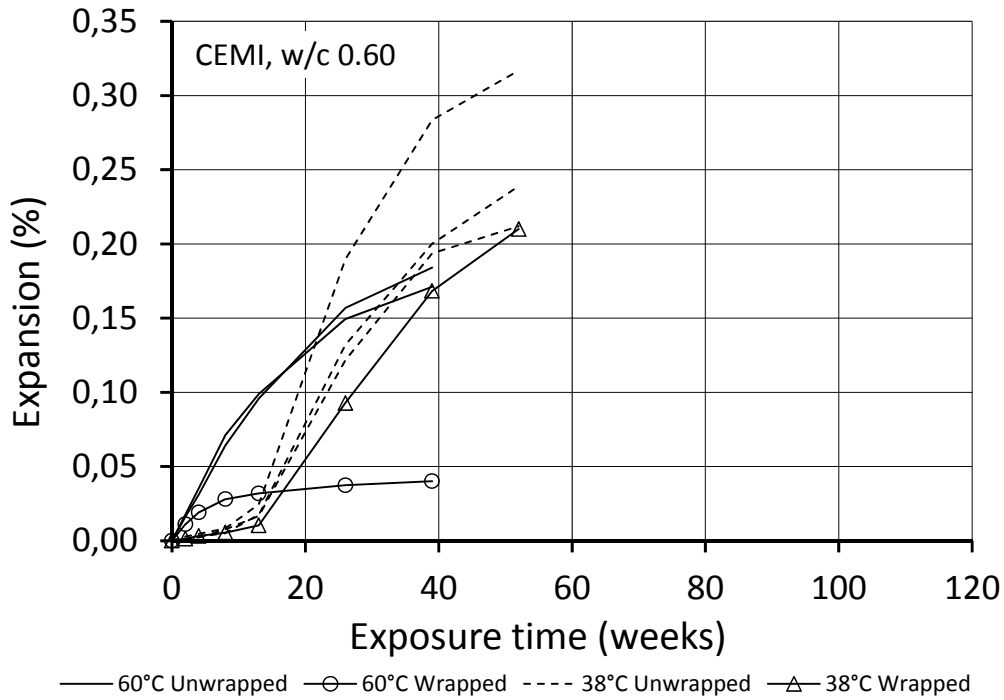


Figure 13 Expansion versus time for the seven test series with the "open" binder (CEM I, w/c 0.60, 3.7 kg Na₂O_{eq} alkalis per m³ of concrete).

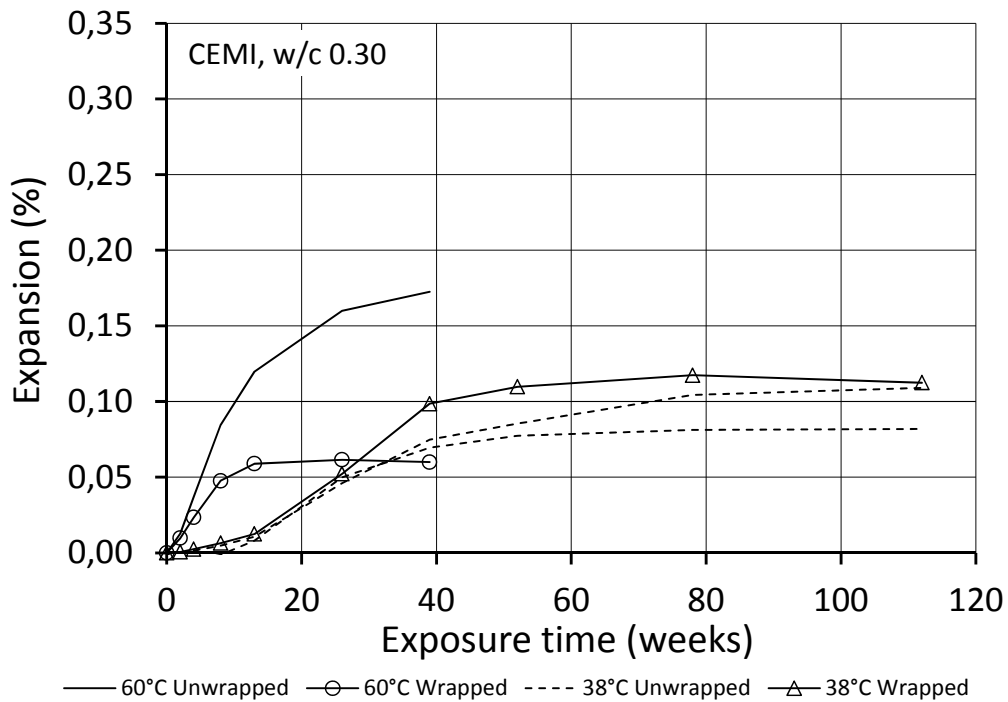


Figure 14 Expansion versus time for the five test series with the "dense" binder (CEM I, w/c 0.30, 3.7 kg Na₂O_{eq} alkalis per m³ of concrete).

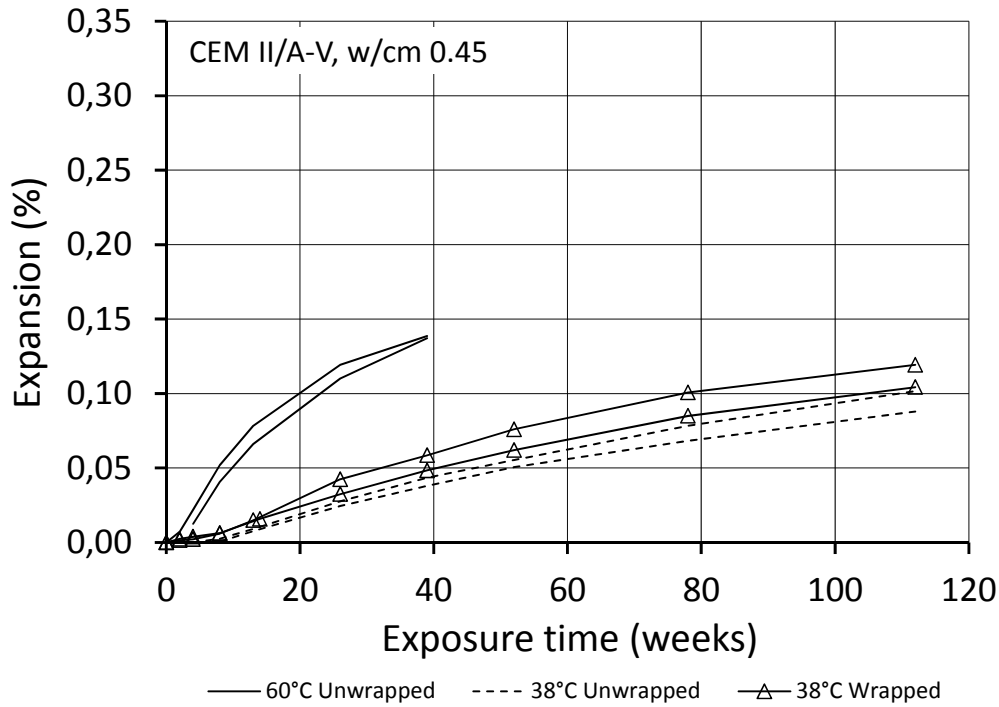


Figure 15 Expansion versus time for the six test series with the "fly ash" binder (CEM II/A-V, w/cm 0.45, boosted from 5.0 to 9.0 kg Na₂O_{eq} alkalis per m³ of concrete).

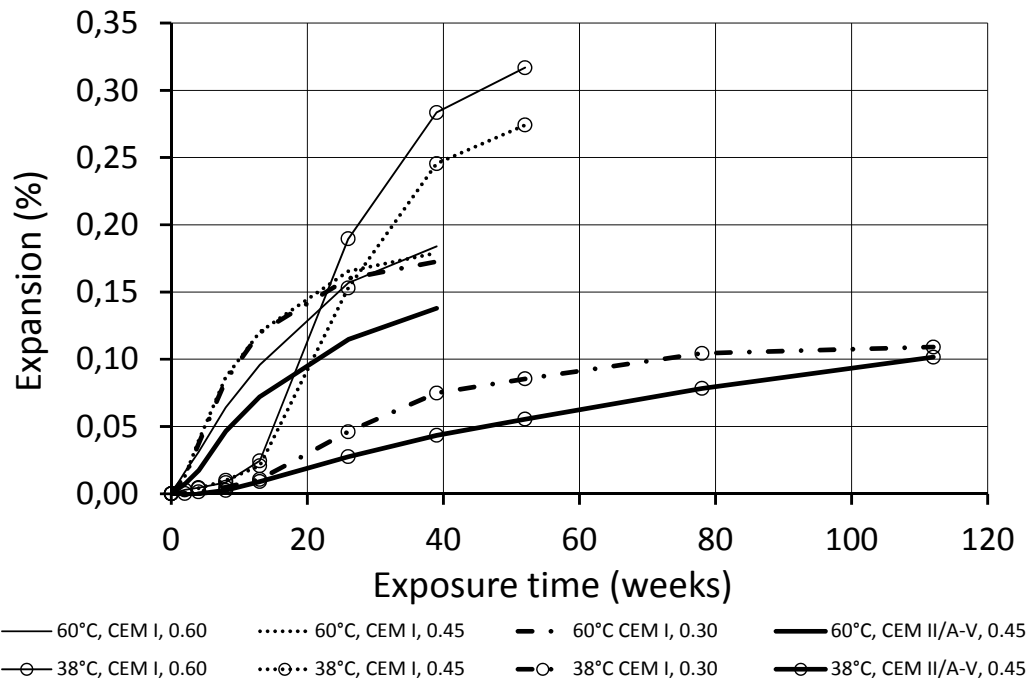


Figure 16 Expansion versus time for unwrapped test series with four different binders (three CEM I binders with 3.7 kg/m³ Na₂O_{eq} alkalis and one "fly ash" binder with 9.0 kg/m³ Na₂O_{eq} alkalis) exposed to either 38°C (Norwegian CPT) or 60°C (AAR-4.1). Except the 60°C test series with the "fly ash" binder (mean of the two test series in Figure 15), the test series were pre-stored 7 days at 20°C before being exposed to elevated temperature.

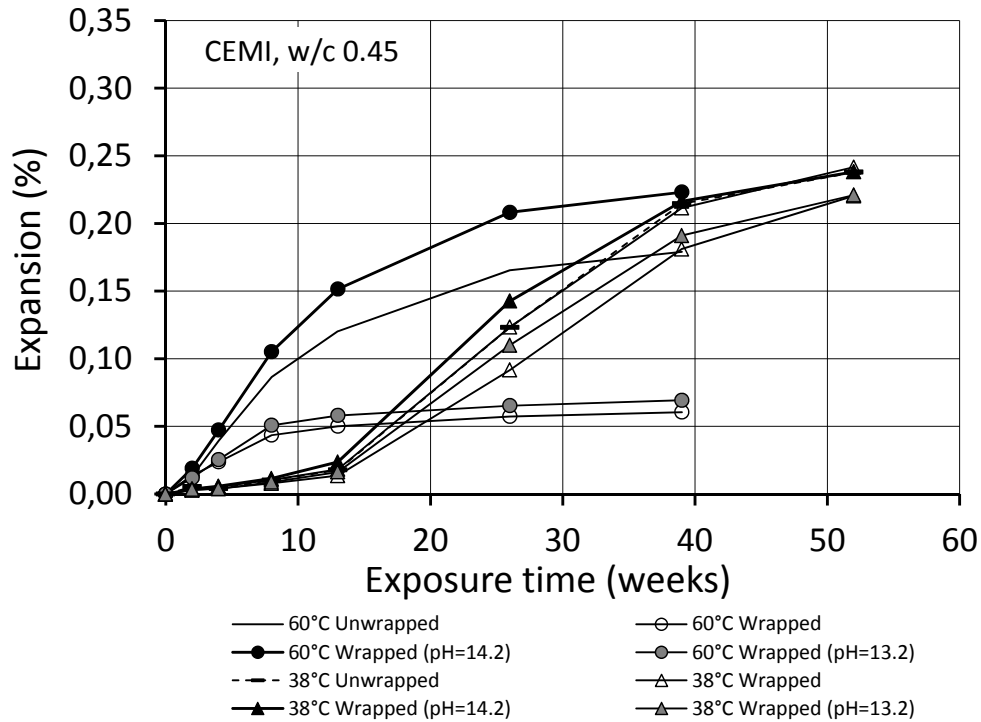


Figure 17 Expansion versus time for various wrapped AAR-3 and AAR-4.1 test series with the "basis" binder (CEM I, w/c 0.45, 3.7 kg/m³ Na₂O_{eq} alkalis). Either de-ionised water (standard procedure) or a solution with pH 13.2 or 14.2 was added to the cotton cloth wrapping. All the eight test series were pre-stored 7 days at 20°C before being exposed to elevated temperature.

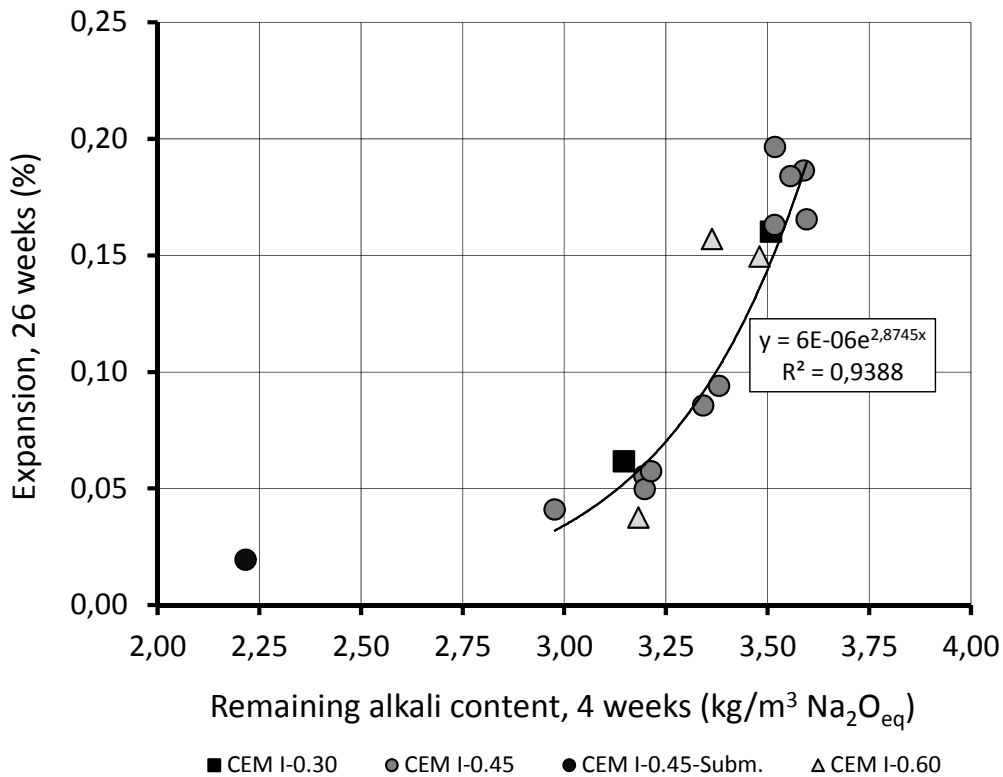


Figure 18 Remaining alkali content after 4 weeks of exposure versus expansion at 26 weeks for wrapped and unwrapped AAR-4.1 60°C test series with CEM I binders (w/c of 0.30-0.60, initial alkali content 3.7 kg/m³ Na₂O_{eq}). The determination coefficient (R²) is valid for the test series with the "basis" binder (w/c of 0.45), excl. the submerged test series.

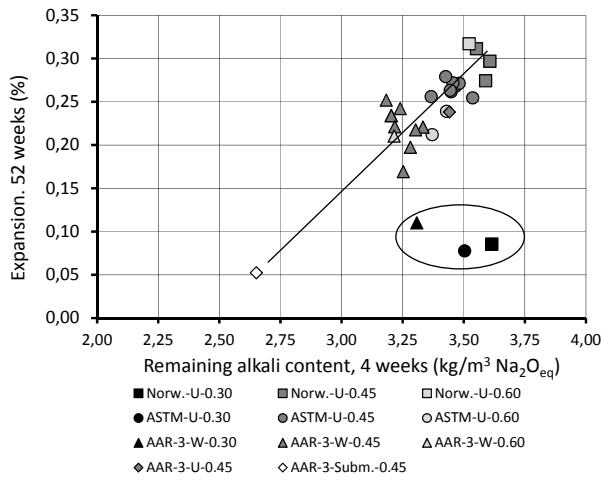


Figure 19 Remaining alkali content after 4 weeks of exposure versus expansion at 52 weeks for wrapped (W) and unwrapped (U) 38°C test series with CEM I binders (w/c of 0.30-0.60, initial alkali content 3.7 kg/m³ Na₂O_{eq}). The determination coefficient (R²) for the trend line for the "basis" binder test series is 0.77.

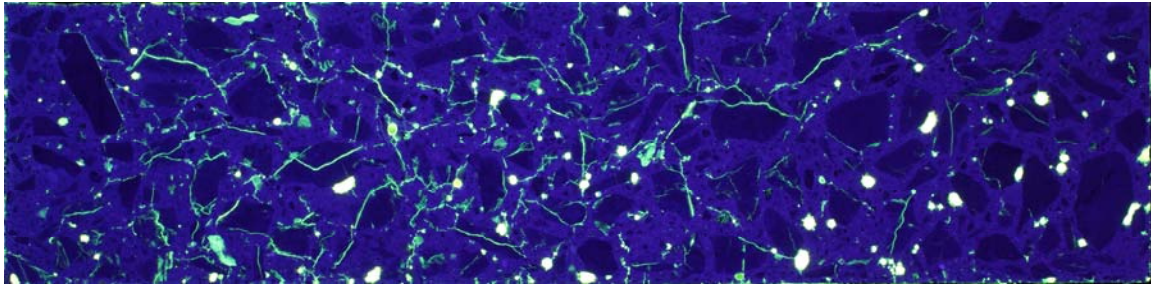


Figure 20 Photo in UV-light of the plane polished section prepared from a prism from test series "4.3-U-A-0.60-I" (see Table 5) with expansion 0.18 %. The bottom of the prism is to the left. Length 280 mm.

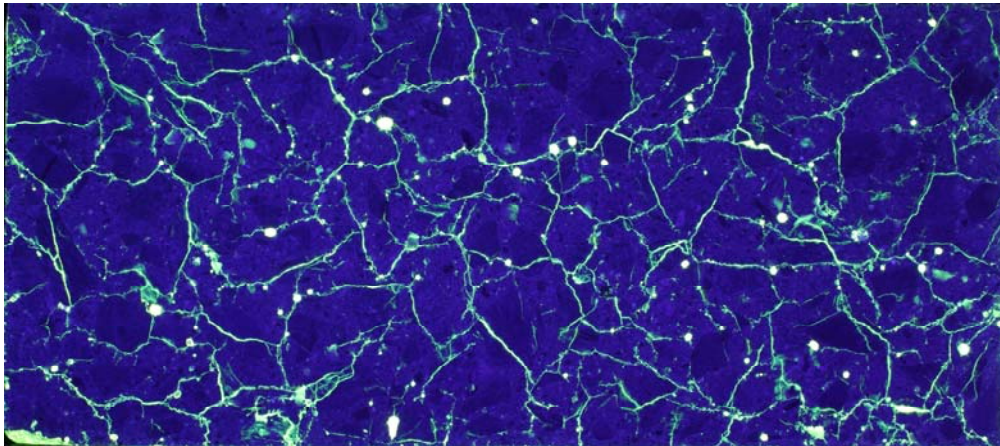


Figure 21 Photo in UV-light of the plane polished section prepared from the lower part of a prism from test series "N.1-U-0.45" (see Table 6) with expansion 0.43 %. The bottom of the prism is to the left. Length 225 mm.

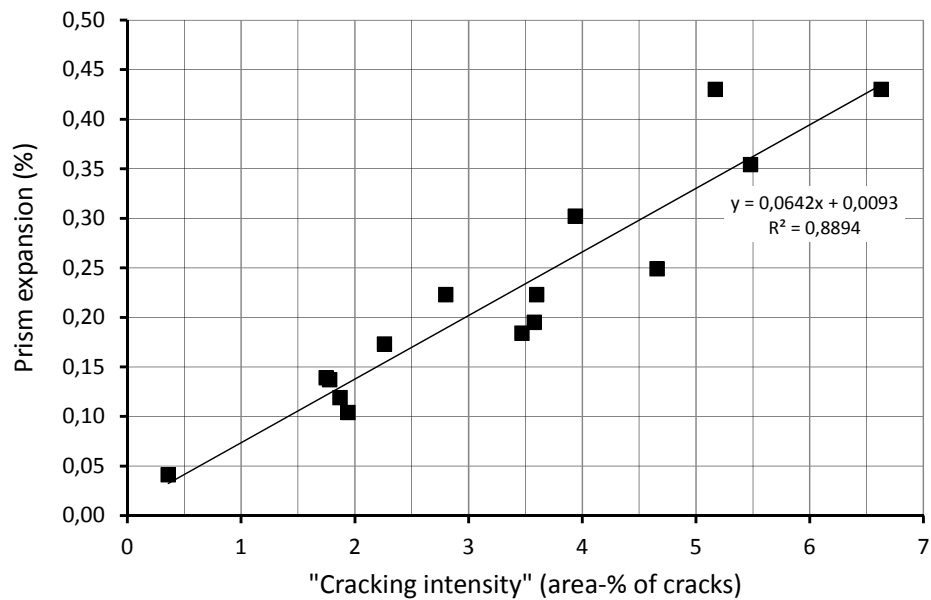


Figure 22 Total "cracking intensity" (area-%) in the 16 plane polished sections plotted against the corresponding prism expansions.