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# Alkali–silica reactions (ASR): Literature review on parameters influencing laboratory performance testing

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ABSTRACT

Utilisation of potentially alkali–silica reactive aggregates requires reliable performance tests to evaluate the alkali–silica reactivity of various aggregate combinations, including their alkali threshold dependence on binder type. Several such performance tests have been used worldwide for more than 15 years, but none of the methods have proven to be reliable for use with all aggregate types and all binders. One of the objectives of RILEM TC 219-ACS (2007–2012) is to develop and validate one or more of such performance tests.

Several parameters may influence the results obtained in an accelerated performance test compared to the field behaviour. Based on a state of the art literature review, this paper discusses which parameters must be considered to be able to develop reliable ASR performance testing methods and provides some tentative recommendations. The internal humidity in the test specimens, the extent of alkali leaching and the storage temperature are of particular importance.

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## 1. Introduction

### 1.1. Background

National regulations for preventing alkali–silica reaction (ASR) in concrete structures are based on various principles that have to take into account a range of material properties and local experience. In order to improve flexibility, extend material selection criteria and optimise concrete mix design, some countries have incorporated the option of performance testing in their provisions. Such options are meant partly to replace technically and commercially restrictive prescriptive requirements by performance-oriented requirements (see e.g. EN 206-1 [1]).

In order to identify a general international ASR performance based testing concept, one of the objectives of RILEM TC 219-ACS 'Alkali–aggregate reactions in Concrete Structures' (2007–2012) is to develop and validate one or more ASR performance tests. In order to maintain the relevance to field structures when testing in the laboratory, it is crucial that the basic conditions do not change to an extent where the correlation of performance ranking under the two different conditions is not maintained. Hence, performance testing in the present context is not about predicting an exact level of deterioration with the selected materials and design, but to determine a relative level of performance with the perception that test results conforming to certain acceptance criteria will assure acceptable field performance.

Setting up a performance test will require both theoretical considerations and practical verification. As a base for the work within RILEM TC 219-ACS a literature survey on influencing parameters has recently been completed [2]. The main objective of the review was to assess which parameters must be considered to be able to develop reliable performance testing methods, i.e. to identify parameters and limitations for accelerating ASR under elevated moisture and temperature conditions. These concerns will be taken into account by the RILEM committee, whereby identified challenges should be dealt with in one of the following ways:

- Research needs (i.e. the performance test will not cover the specific issue until further research has been accomplished).
- Experience based convention for agreed, unified approach.
- The phenomenon discussed can be dealt with within the level of the chosen safety margin.
- Scatter introduced by the specific or collective phenomena of concern can be determined by a precision test.

All authors of this paper are members of the "Performance testing" task group of RILEM TC 219-ACS.

### 1.2. Technical background

Several aggregate types in common use, particularly those with a siliceous composition, may be attacked by the alkaline pore fluid in concrete. This attack, essentially a dissolution reaction, requires a certain level of moisture and alkalis (leading to high pH) within the concrete to take place. During the reaction, a hygroscopic gel is produced. When imbibing water, the gel will swell and thus cause expansion, cracking, and in worst case disruption of the concrete. The deterioration mechanism is denoted alkali–aggregate reaction (AAR) or, more specifically, for siliceous aggregates, alkali–silica reaction (ASR). The less common, so-called alkali–carbonate reaction (ACR) is not discussed in this paper.

Since ASR was recognised as a durability challenge more than 70 years ago by Stanton [3], several comprehensive research projects have focused on test methods for determining the reactivity of aggregates and corresponding acceptance criteria. As part of the international harmonisation of such test methods, the main aim of RILEM TC 106-AAR (1998–2000) and RILEM TC 191-ARP (2001–2006) was to propose and validate test methods for classifying the alkali reactivity of concrete aggregates. The committees have proposed several RILEM aggregate test methods (AAR-1, 2003 [4]; AAR-2, 2000 [5]; AAR-3, 2000 [5]; AAR-4.1, 2006 [6]; AAR-5, 2005 [7]), in addition to recommendations for how to use these test methods and interpret the results (RILEM AAR-0, 2003 [8]) and how to ensure durable non-reactive concrete (RILEM AAR-7.1, 2008 [9]). All the draft RILEM methods have been developed further by RILEM TC 219-ACS (2007–2012) and are planned to be published in a special issue of Materials and Structure during 2012. In USA and Canada, corresponding ASTM and CSA test methods exist (ASTM C 1260-07 [10]; ASTM C1293-08b [11]; ASTM C 295-08 [12]; CSA A23.2-14A-04 [13]; CSA A23.2-25A-09 [14]; CSA A23.2-15A [15]).

In a concrete containing reactive aggregates the potential for a damaging alkali–silica reaction is to a great extent influenced by the composition of the concrete pore solution with its function as a reaction partner for the reactive silica and as a supplier of moisture. In particular the content of alkalis, i.e. sodium ($Na^+$) and potassium ($K^+$), in the concrete pore solution plays a major role for development of ASR. The main contributor of alkalis to the concrete pore solution is usually the cement. In the first place, more $Na^+$ and $K^+$ lead to dissolution of more hydroxyl ions ($OH^-$) from $Ca(OH)_2$ to maintain equilibrium with the increased alkali concentration. For high pH pore solutions and at 20 °C, $[Na^+]+[K^+]\approx[OH^-]$ because the quantity of other ions is insignificant compared to the concentration of alkali ions beyond the first 24 h ([16,17]). The pH of the pore solution will thus increase. This higher alkalinity again leads to dissolution of more reactive silica ($SiO_2$) from alkali–reactive aggregates. Secondly, alkalis will react with the dissolved silica (and calcium) forming alkali–silica gel [18]. As discussed further in Section 3.2.1, the alkali–silica reaction is very similar to pozzolanic reactions.

Thus, the degree of reaction of an aggregate is a function of the alkalinity of the pore solution. For a given aggregate, a critical lower pH-value exists below which the aggregate will not react. Consequently, ASR will be prevented by lowering pH of the pore solution beneath this critical level where the dissolution of alkali-reactive constituents (silica) in the aggregates will be strongly reduced or even prevented, as discussed by Böhm and Baetzner [19]. This pH-value corresponds to a certain alkali threshold that several have reported to exist for initiating and sustaining ASR in concrete [20]. No “absolute” limit is defined, because the critical alkali content largely depends on the aggregate reactivity [21]. For most alkali-reactive aggregates, the alkali threshold when applying CEM I cements in 38 °C concrete prism tests (CPTs) is in the range 3–5 kg $Na_2O_{eq}$ per $m^3$ concrete, but may be lower for some rapidly reactive aggregates. However, due to alkali leaching (see Section 5.4.1) from laboratory exposed samples, the true field alkali threshold may be significantly lower than the limit measured in the laboratory. The majority of existing concrete prism tests apply alkali contents in the range of 5.0–5.5 kg $Na_2O_{eq}$ per $m^3$ when testing the alkali reactivity of aggregates. If the alkalinity is changed during the test, the expansion of the concrete prisms will be influenced.

Internationally, various ways of controlling ASR are suggested (in addition to use of non-reactive aggregates): utilization of low-alkali cement, limiting the alkali content of the concrete, incorporation of supplementary cementitious materials (SCMs; e.g. silica fume, fly ash, ground granulated blastfurnace slag (ggbs), metakaolin) or use of lithium salts. SCMs control expansion due to ASR by binding alkalis and limiting their availability for reaction with alkali–silica reactive aggregates [22]. The efficiency of the SCMs depends on their composition. 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 performance tests (or alternatively by relevant long term field experience). Several such of accelerated laboratory performance tests have been used worldwide for at least 15 years, mainly to evaluate various SCMs and lithium salts (e.g. ASTM C-1293-08b [11] and the Norwegian CPT [23]). In principle two groups of performance test methods exist, one using mortar bars and the other using concrete prisms. However, the test conditions (e.g. temperature, alkali content, humidity) used within these two groups might vary widely from one test method to another. Thus, the results/conclusions from different test methods may vary.

In 2006, Thomas et al. [24] provided a critical evaluation of different test methods. The authors concluded that none of the currently available or commonly used test methods meet all the criteria for an ideal performance test. For example, the main shortcoming of the Canadian 38 °C concrete prism test (CPT) [13] is the duration of the test (2 years) and that the addition of alkalis is required to compensate for alkali leaching effects, i.e. the fact that alkalis are leached out of the prisms during exposure in the humid environment (see Section 5.4.1). Thus, the authors concluded that the method cannot be used to determine the “critical” alkali content for an alkali–reactive aggregate, nor determine how the minimum level of a SCM changes with the concrete alkali content. Thus, research is going on in many countries with the aim to improve current test methods and develop alternative tests.

### 1.2.1. Main challenges

The development of accurate and reliable performance tests for the production of durable concretes is a challenge. Several requirements must be fulfilled, some being somewhat contradictory. On the one hand the test methods should be inexpensive and rapid, calling for extremely accelerated test conditions. On the other hand a performance test should mirror the field performance of the actual concrete for more than 50 years lifetime. Another important requirement is the possibility to test job mixes with identical aggregate and concrete composition that will be used on actual projects. Use of mortar bars is in conflict with this latter requirement.

According to Thomas et al. [24], other important requirements for an ideal performance test for ASR are:

- The test should be capable of evaluating the “critical” alkali contents, i.e. the alkali leaching problem must be solved to avoid the need for a boosted alkali level.
- The test should be capable of assessing all types of SCMs, lithium compounds and combinations of SCM and lithium, with cements of different alkali level.

### 1.2.2. Crucial parameters to ensure a good laboratory/field correlation

As stated by Thomas et al. [24], the only suitable benchmarking of a laboratory performance test is against real concrete structures (if available) or, as a surrogate, against large concrete blocks exposed outdoors and exposed to natural weathering conditions. However, such long-term field experience is available only for a limited number of commercial SCMs, e.g. some class F fly ashes and some slag cements. When developing an accelerated performance test, it is thus crucial theoretically to evaluate fundamental questions in order to ensure a satisfactory laboratory/field correlation. Consequently, the main focus needs to be put on the three parameters known to have the primary influence on the rate and extent of alkali–silica reactions (for a given alkali–reactive aggregate type). These are ([25,26]):

- Humidity
- Alkali content (“controls” the concentration of $OH^-$ in the pore solution)

- Temperature

Additionally, other parameters may influence the laboratory/field correlation, for instance by affecting the humidity or the alkali content of the test samples. This is further discussed in the paper (see Section 1.3).

### 1.3. Assessment of influencing parameters

The main objective of the comprehensive literature review performed within the task group "Performance testing" in RILEM TC 219-ACS, where 12 authors contributed to the report that included about 250 references [2], was to assess how various parameters might influence the laboratory/field correlation with respect to ASR performance testing, either directly or indirectly. More exactly the aim was to evaluate how various aggregate and binder types, the mix design and the laboratory exposure conditions might influence the following important ASR related parameters and thus the laboratory/field correlation:

- Internal humidity of the concrete prisms.
- Composition of the concrete pore solution during testing.
- Properties of hydration products formed during hydration/exposure.
- Aggregate reactivity.
- Type and properties of reaction products, i.e. primarily ASR-gel, formed during exposure.

This paper summarises the main findings in the literature survey. Firstly, precautions when testing various aggregates types (Section 2) and binder types (Section 3) are discussed. Secondly, any influences of mix design parameters (e.g. water-to-cementing-materials ratio (w/cm ratio) and type of any chemical admixtures added) are assessed (Section 4). The last part of the paper (Section 5) evaluates the influence of exposure conditions on ASR expansion, i.e. precuring conditions and storage conditions (incl. addition of any external alkalis). In the concluding part (Section 6), the authors have given some recommendations for what is considered the best approach for performance testing. Finally, some important issues needing further research are summarised (Section 7).

## 2. Aggregate type

Since the alkali–silica reaction was first identified, a great number of rock types have been classified as potentially reactive. There is evidence that apparently similar rock types can vary greatly in reactivity in practice depending on their geological history and geographical location. For several rock types, there are reactive and non-reactive varieties according to differences in the detailed mineralogical composition or texture [4]. There are two generalised classes of siliceous minerals that are known to be expansively reactive with the alkalis in concrete: the metastable types of silica (opal, chalcedony, tridymite, cristobalite) including some disordered forms of quartz, and alumina–silicate glasses mainly in the matrix of intermediate to acid volcanic rocks [27]. Although the classification has a strong regional component, there are minerals and rock types containing minerals generally considered as potentially reactive. Lists are present in literature and usually on standards (e.g. ASTM C294-05 [28]; CSA A23.2-15A [15]; BS 7943, 1999 [29]; RILEM AAR-1, 2003 [4]).

The first step in the assessment of potential alkali reactivity should be the petrographic characterization of the rock types in thin sections under optical microscope ([30,31,32]). In RILEM AAR-1 [4], the list of potentially reactive rock types includes the reference to the countries where deleterious reaction was recognised with each rock. It has been verified that the generic classification of a rock type is not reliable in respect of alkali reactivity. Due to different geological histories, a rock type might be innocuous in one country or region and reactive in another, and therefore the final classification based on the petrographic assessment must follow national or regional experience. The petrographic examination, based on the RILEM AAR-1 [4], allows that an aggregate is classified as very unlikely to be alkali-reactive (Class I), alkali-reactivity uncertain (Class II) or very likely to be alkali-reactive (Class III). The petrographic examination is therefore usually followed by laboratory tests performed on mortars or concretes in order to confirm the results obtained.

### 2.1. Dissolution of silica

Silica is a material which dissolves in strongly acidic or alkaline conditions, and less around neutral pH. The laboratory tests for evaluating the potential reactivity of siliceous aggregates are based on the concept that the free energy of quartz, which determines its solubility, is related to the amount of defects in the lattice and degree of crystallinity [33]. Under ambient conditions, fine-grained amorphous silica dissolves much easier in high-pH solvents than crystalline quartz [34]. The alkali reactivity is affected by a number of factors and is related with the qualities of quartz from different geological environments [35]; e.g. deformed quartz is confirmed to be highly reactive owing to distorted crystal structure and small grain size due to increased surface area.

The presence of alkalis influences the reactivity of aggregates and the extent of the reaction. Therefore, for a specific aggregate, more alkalis available means more expansion, due to a higher concentration of $OH^-$ in the concrete pore solution and thus more silica dissolved. Fournier et al. [36] have shown that "non-reactive" sands can have components which are activated by high alkali content.

### 2.2. Aggregate properties

#### 2.2.1. Mineralogy

The rate at which the rocks containing potentially reactive forms of silica react is variable. In consequence of the results obtained by laboratory tests as well as from field performance, the aggregates might be classified regarding alkali-reactivity as "fast" to "normal" reactive (5 to 20 years), "slow/late" reactive (+15 to 20 years) and "non-reactive" [37]. The terms "highly" or "rapidly" reactive and "low" reactive are also frequently used in the literature. Rocks containing opal are examples of rapidly reactive aggregates for which the accelerated laboratory tests usually give reliable results. By contrast, there are rocks containing strained quartz with strain lamellae for which the field performance shows the occurrence of ASR after some decades and for which some of the laboratory tests (e.g. RILEM AAR-2 [5]) might be ineffective ([38,39]). The reverse may happen with non-reactive aggregates, which may be classified as reactive because the conditions in the test are too severe for some types of aggregates ([30,39]). Ideker et al. [40] and Shayan et al. [39] verified that tests performed with different "non-reactive" natural sands mixed with the same coarse reactive aggregate show different results depending on the test method applied. Also the variation between laboratories was high.

#### 2.2.2. Other aggregate properties

The mineralogy is, however, not the only parameter to consider in the potential reactivity of a rock type. Increased permeability of aggregates with higher porosity may enhance the alkali reactivity, due to easier access to concrete pore fluids ([27,41]). Wenk et al. [42] tested a deformed granitic rock and concluded there is a relationship between the aggregate microstructure and the mortar expansion due to grain size reduction, development of foliation in the rock and dislocation density of quartz, confirming former findings by other authors ([43,44]).

#### 2.2.3. Pessimum

Laboratory tests performed with different types of aggregates have shown that there is not always a linear relationship between the content of potentially reactive constituents and the measured prism expansions. A maximum level of expansion might occur at a particular content of the reactive constituent known as the 'pessimum'. Decreasing levels of expansion will develop for contents of the reactive constituents above or below the pessimum ([45,25,46,47,33,48,4]). Expansion increases with an increase in the amount of reactive constituents up to the pessimum, beyond which it decreases due to the lack of alkalis available for the formation of expansive gel [49]. The pessimum may differ for differing potentially reactive constituents. For fast reactive aggregates, such as those containing opal, maximum expansion occurs for low contents of reactive silica ([49,50]), usually below 10%, whilst for slowly reactive aggregates the percentage will be much higher, even up to 100% (i.e. they do not show a pessimum effect).

The implication when dealing with aggregates showing a possible pessimum is that several concrete mixes have to be performed with different percentages of reactive constituents in order to document the pessimum. It is also important to be able to evaluate such aggregates in a performance test.

Use of a sufficient amount of SCMs has shown to be effective to prevent development of ASR in concrete with aggregates showing a pessimum. However, Buck and Mather [51] showed that some fly ashes when used at a too low replacement level actually caused more expansion, especially with low-alkali cement. They believed it was caused by the additional water-soluble alkalis provided by the fly ash to the system.

### 2.2.4. ASR gel composition

The chemical composition and the texture of alkali– silica gel have been studied by a number of researchers and it is recognised that it varies widely with time and with the location in the concrete. These results are based mainly in qualitative analyses carried out by SEM/EDS ([52,53,54,55,56,57]), and show that gel has high and varying contents of silica, lower and varying contents of calcium and low and relatively constant contents of alkalis, in agreement with other workers ([58,59]). If not extremely low in total alkali content, the $K_2O/Na_2O$ ratio of the cement clinker is normally in the range of 1 (unusually low) to 3, which may be reflected in the gel composition. Any addition of $Na_2O$ or $K_2O$ into the concrete mixture for accelerating the reaction [60] or exposure to external alkalis, e.g. from deicing salts, seawater or in laboratory tests, may also affect this ratio and also the relative content of calcium and alkalis [59].

The formation of ASR products depends on the nature, texture and composition of the aggregate [61], and on whether the aggregate is slowly reactive, rapidly reactive or showing pessimum content [62]. However, the composition of gel seems not to be dependent on the nature of aggregates. Gel with varied composition regarding the Ca content was identified in the same sample, with different composition from one grain to another and also in the same grain ([53,54,63,64]). Calcium content is more prevalent in cracks found in the cement paste [52] than inside aggregates, developing a reverse trend to that of silicon. In cracks, Ca-rich gel is found at larger distance from the coarse aggregates, due to exchanges of alkalis with Ca in the cement paste. It has been verified that expansion does not necessarily increase proportionally to the reaction degree or the amount of gel produced, but it seems to be dependent on Ca content ([52,56,65]).

## 2.3. Grading and size

The influence of aggregate grading on mortar bar or concrete prism expansion has been studied since Stanton [3] in 1940 concluded that the aggregate particles (siliceous magnesium limestone containing opal and chalcedony) in the 170–600 μm range yielded greater expansion than coarser sizes. Diamond and Thaulow [62] tested opal aggregates in the range of 20– 125 μm, and found that the smaller fractions expanded faster than the coarser material, which needed a prolonged exposure, but the total expansions were of the same order. Lu et al. [66] state that within a certain size range, the finer the aggregate particles of argillaceous dolomite limestone, the faster and higher the alkali– silica reaction rate and the expansion.

However, Multon et al. [67] state that the aggregate size causing the highest ASR expansion is dependent on the nature and composition of the aggregate. For rapidly reactive aggregates, it was found that the amount of soluble silica was similar for different particle sizes of a certain aggregate, but the expansion varied for different size fractions, being larger for coarser particles [68]. In an apparently contradictory finding, Zhang et al. [69] concluded that for siliceous aggregates the smaller the particle size, the greater the ASR expansion when aggregate size is within the range of 0.15– 10 mm. They also state that the aggregate grading can affect the expansion of ASR: when there are larger aggregates in the specimen, the expansion is smaller at early ages, but will increase continuously at later ages. Hobbs and Gutteridge [70] concluded that for opaline rocks, expansion increases as the particle size decreases, when the reaction occurs at the surface of the particles. However, when the reaction occurs within the particle, the rate of gel formation will be independent of the particle size.

Barisone and Restivo [71] and Lu et al. [72] showed that the use of very fine aggregates can destroy the original microstructure characteristic of the rocks, and thus under-estimating the alkali reactivity of the rocks in accelerated mortar bar tests (e.g. RILEM AAR-2 [5]).

Wigum and Lindgård [73] state that slowly reactive Norwegian coarse aggregates have proven to be more harmful in the field than fine aggregates. This has been accounted for in the Norwegian regulations [74] by differentiating the critical limits in the Petrographic method and the accelerated mortar bar test [23].

Another parameter to consider, although there is limited literature on the subject, is the shape of the particles. Ramyar et al. [75] found that the angularity of the particles had an effect on the mortar bar expansion for intermediate size fractions, and that the effect of size of the particles was more pronounced in crushed aggregate when compared to rounded gravels of the same aggregate type. Work developed in the UK [60] showed that some greywacke aggregates can give rise to cracking at lower alkali levels than occurs with other aggregates. Therefore, aggregates and aggregate combinations which contain crushed greywacke or crushed greywacke-type aggregates have been classified as highly reactive whilst natural gravel aggregates are considered as having “normal” reactivity.

The accelerated mortar bar test failed to detect the reactivity of glass aggregates at 14-days [76], but the expansions were rising suddenly after an initiation period. This behaviour was not observed in concrete prism tests. The effect is probably due to increased pozzolanicity of filler-sized fine particles at high temperatures during the early testing period. Pedersen [77] also detected that some reactive aggregates showed a similar pozzolanic behaviour when ground to filler size.

The implication to reliably test the ASR performance of aggregates, including size effects, is that the fractions used in structures should preferably also be used in the laboratory tests. It should also be kept in mind that, crushing certain types of aggregates for laboratory testing may change some of their characteristics.

## 2.4. Alkali release

Some aggregate types containing certain minerals, e.g. micas, clay minerals, alkali feldspars, zeolites and volcanic glass may gradually release significant quantities of alkalis, i.e. sodium ($Na^+$) and potassium ($K+$), to the concrete pore water ([78,79,80,81,82]). Temperature has an influence on the extraction of alkalis, as concluded by Lu et al. [83], which varies with the type of rocks as well as with the fineness of the rock particles and the type of solution. Wang et al. [84] report that the factors that influence the maximum alkali release include the types of alkali minerals, the pore solution/aggregate ratio, the pH and the type of alkali ions in pore solution from sources other than the alkaline minerals.

Bérubé et al. [80] tested the extent of alkali release for 17 aggregate types from Canada. Most of these aggregates contributed alkalis in the range 0.45 to 0.70 kg $Na_2O_{eq}$ per $m^3$ of concrete, but the amount varied from about 0.1 to 1.6 kg $Na_2O_{eq}$ alkalis per $m^3$ of concrete dependent on the aggregate type.

The most common tests to evaluate alkali release are based on the immersion of alkali-bearing aggregates in alkaline solutions such as of calcium hydroxide, potassium hydroxide and sodium hydroxide ([85,86,87,81]), whilst the hot-water extraction method uses distilled water [87]. In Bérubé et al. [81] a summary is presented of the procedures used by different authors to evaluate the alkali contribution by aggregates. The task group “Releasable alkalis” in RILEM TC 219-ACS is presently developing a reliable test procedure to measure the extent of alkali release from various aggregate types, including corresponding interpretation criteria.

### 2.5. Lightweight aggregates (LWA)

Lightweight aggregates (LWAs) have been used in several important structures, e.g. in some bridges and oil platforms. Most of these LWAs contain silica, often in a poorly crystalline, glassy condition, making them potentially alkali-reactive. In tests performed at SINTEF, four commonly used LWAs have also developed ASR in the ultra accelerated mortar bar test [5]. However, there is a gap in knowledge internationally whether these and other LWAs may give deleterious ASR in real structures. In a review in 2000, no cases of ASR were found in LWAC [88]. However, instances of ASR in LWAC have later been reported in Japan (PC girders of a bridge and PC sleepers) by Matsuda et al. [89]. There are no international agreed requirements regarding how to test and evaluate the alkali reactivity of a LWA or a LWA Concrete (LWAC). Even though some ASR test methods allow testing of LWAs (e.g. ASTM C-1293-08b [11], where the LWA concrete mixes are designed on a volume basis which is necessary for such low-density aggregates), the interpretation criteria may be questioned. The main reason for this is that most test methods for ASR apply only expansion criteria, which cannot be applied uncritically for LWA, since experiences show that the ASR gel (if developed) initially accumulates in voids in the LWAs and primarily contributes to a weight increase but only a moderate length increase. After the gel has accumulated in and partly filled the voids in the LWAs, the rate of expansion might increase [90]. More research is therefore needed to be able to develop suitable laboratory test procedures and corresponding acceptance criteria for LWA and LWAC. The acceptance criteria should include evaluation of the measured weight increase (since an increased weight of the LWAC may alter structural design parameters), and they also need to be correlated with long time field experiences with use of LWAC in various concrete structures.

## 3. Binder type

Type and amount of various binders (i.e. different cements and SCMs) significantly affect the concrete pore solution alkalinity. The concentration of $Na^+$, $K^+$ and $OH^-$ is dependent on the quantity of sodium and potassium compounds in the anhydrous Portland cement clinker and in the supplementary cementitious materials (SCMs). Any significant change in the pore solution composition caused by a change in the binder type and composition is discussed in this section. Any other contributors of alkalis, e.g. any chemical admixtures (alkali boosting), de-icing salts or any alkalis released from aggregates, are discussed in other sections in the paper.

### 3.1. CEM I-type of clinker

When Portland cement is mixed with water, the alkali sulphates go rapidly into the liquid phase converting to alkali hydroxides, thus increasing the hydroxyl ion concentration. Alkalis locked into the crystal structures of clinker minerals become available as the hydration proceeds [26]. Consequently, the alkali release rate varies from one cement type to another, depending on the distribution of alkalis between rapid-release and slow-release sources, and on the total alkali content in the cement. Since alkali–aggregate reaction proceeds slowly under site exposure conditions, it is possible that most of the cement alkalis are released for reaction at a constant time. Under accelerated conditions in a laboratory performance test, it is important to ensure a rate of alkali release from the binder (more important for blended cements) corresponding to that in the field.

From early mortar bar studies, Hobbs [25] stated that considerably varying expansion results observed for mortars with various cements but with similar total alkali contents ($kg/m^3$) might be attributed to different alkali release rates of cements, variations in sodium/potassium ratio and different rates of strength development.

In order to assess the total content of available alkalis present in cement or concrete, it has become standard practice to express the alkali content in terms of “sodium oxide equivalent”: $Na_2O_{eq} = Na_2O + 0.658\ K_2O$ (in weight percent). Leemann and Lothenbach ([91,92]) stated that concrete mixtures produced with cements having similar $Na_2O_{eq}$ but different K/Na ratios can expand considerably differently in accelerated laboratory tests. However, there are also contradictory findings in the literature [93]. Hou et al. [94] stated that K and Na ions behave similarly in the ASR reaction, but the rate of reaction is higher with K than with Na. In contrast, Borchers and Műller [95] found that Na produced a higher reaction rate in laboratory tests compared to K. Considering these, it is possible to obtain misleading conclusions if two cements having equal sodium oxide equivalent but extreme variations of $Na_2O$ and $K_2O$ levels are assumed to act similarly in a performance test. In other words, if one CEM I cement is used in a performance test in order to determine the critical alkali limit for the aggregate in question, the test result will not necessarily be valid for all types of CEM I cements.

Within the cement paste, the ASR gel becomes richer in calcium with time, releasing alkalis to the pore water ([96,97,98]). This alkali recycling during ASR reveals that the reaction may theoretically continue until all the reactive silica is transformed into alkali–silica gel. The swelling capacity of ASR gel is also related to the calcium ions present in the ASR gel, which depends on the amount of $Ca^{2+}$ available in the pore solution. The latter varies with the type of binder used. It is also known that the process of ASR reduces the alkalinity of pore solution by binding some alkalis in the alkali–silica gel [99]. Then the question arises as to how the time-dependent alkali recycling phenomenon might affect the concrete performance during its service life?

In hardened concrete, the alkalis supplied by the binder (i.e. the cement or any SCM incorporated) may be dissolved in the pore solution, bound by the hydration products or adsorbed either by aggregates or the ASR gel in different amounts [20]. At a given age, the presence of alkalis still bound in unhydrated binders (important especially for SCMs that release alkalis slowly into the system) and the availability of alkalis from alkali releasing aggregates should also be considered.

The type of cement and the type and amount of any SCMs incorporated alter the permeability of concrete, thereby influencing water uptake, leaching of alkalis, the resistivity to drying during exposure and the extent of self-desiccation. This should be taken into account while testing the concrete performance, because field structures might be less affected from some of these parameters when compared with laboratory samples. Consequently, the paper also discusses these parameters comprehensively.

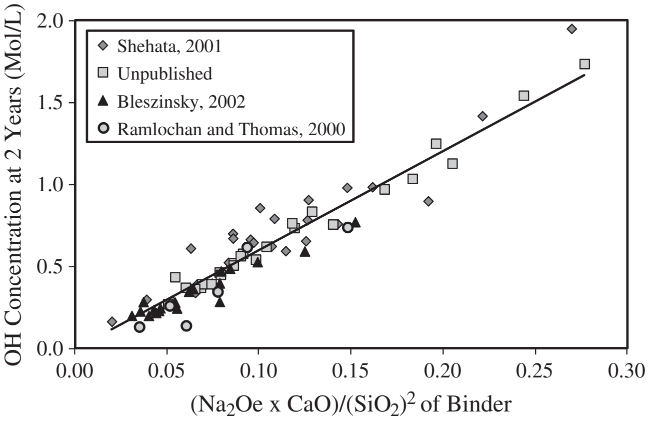

**Fig. 1.** Relationship between pore solution composition and the chemical composition of the binder ([108,109,100,101,22]).

### *3.2. Supplementary cementitious materials (SCMs)*

#### *3.2.1. Role of SCMs in prevention of ASR*

SCMs are known to control ASR expansion mainly by their capability to reduce the alkalinity of the pore solution by binding alkalis in the hydration products. The SCMs that are low in calcium and high in silica are most effective in reducing pore solution alkalinity, thereby ASR expansions. As summarised by Thomas et al. ([100,101]), after the analysis of extracted pore solutions from 79 cement pastes of different binders, a direct linear relationship between the $OH^-$ concentration of the pore solution and the "chemical index" $(Na_2O_{eq} \times CaO)/(SiO_2)^2$ of the binders (Fig. 1) were observed after 2 years of exposure. In other words, SCMs with a high (reactive) silica content and a low amount of CaO and alkalis will be the most effective in terms of lowering the pore solution alkalinity and preventing expansion due to ASR. Since the alkali reactivity of various aggregates varies greatly, no general "safe" lower concentration of hydroxyl ions in the pore solution can be stated. However, in the literature this limit is reported by several authors to lie in the range of 200–300 mmol/l ([102,103,104,20,105,24,106]). These $OH^-$ concentrations correspond to pH-values in the range of approximately 13.3–13.5. There is also evidence that alumina might play in important role in the alkali binding capacity of SCMs [107].

However, the empirical relationship between the 2 years expansion of 132 concrete mixes tested in accordance with ASTM C-1293-08b [11] revealed a different chemical index $[(Na_2O_{eq})^{0.33} \times CaO]/(SiO_2)^2$, see Fig. 2 [22], compared with the empirical relationship derived from the pore solution analyses. The cementing materials used to produce these concretes were the same as those used for the pore solution study discussed above. The reactive coarse aggregate was siliceous limestone (Spratt). According to Thomas [22], the relationship is likely quite different if a different reactive aggregate or, even, a different test method is used. When comparing the two chemical indices, the author concludes that the alkali content of the binder appears to play a less important role in expansions when compared with the pore solution composition owing to leaching of alkalis during the concrete prism test (while no alkali leaching occurred for the cement pastes stored separately in sealed bottles) and this may reduce the apparent importance of the initial alkali content. This effect can be observed when looking at the expansion data for the concrete mixes produced with low-alkali cement. The expansion is lower than that expected based on the chemical composition. However, it is known that the concrete prism test will likely underestimate the expansion with low-alkali cement because of alkali leaching [24].

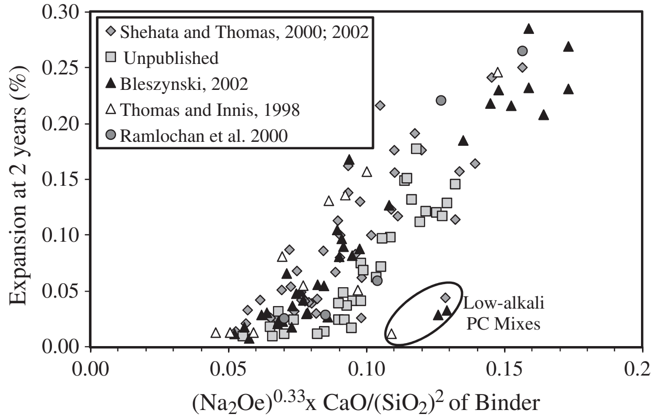

**Fig. 2.** Effect of binder composition on the expansion of concrete containing siliceous limestone ([110,111,108,112,109,100,22]).

Duchesne and Bérubé [113] state that the mean Ca/Si molar ratio ($= CaO/SiO_2$ ratio = C/S ratio) of non-blended samples was 2.0, while this value ranged between 1.24 and 1.46 for blended types independent on the type of SCM. The reduction of pH of the pore solution is mainly attributed to the incorporation of alkalis by low Ca/Si hydration products in the presence of SCMs.

In addition to these main effects, when SCMs are used partially to replace the Portland cement, there is a dilution of the alkalis available from the clinker, a lower rate of alkali release, a decrease in the pH of the pore solution owing to the reduction of $Ca(OH)_2$ in the paste, restricted ingress of water into the concrete caused by reduced permeability and an increase in the resistance to cracking by increasing the strength of concrete [26]. The decreased permeability will also decrease the ion mobility and thus possibly reduce the rate of ASR. Finally, the extent of self-desiccation might also be increased by incorporating SCMs, resulting in a lowered internal RH in the test prisms (unpublished results from Jan Lindgård's PhD study (2007–2012) at NTNU). Thus, at a constant testing temperature, the type, amount and fineness of SCMs and reactivity of aggregates are among the most important factors that control pore solution alkalinity and consequent ASR expansions.

Some authors revealed that ASR is very similar to pozzolanic reactions, pozzolanic reactions proceeding before ASR ([114,94,22]). The reactive silica present in finely-divided SCMs reacts rapidly with the alkali hydroxides in the pore solution forming an alkali–silica gel containing small amounts of calcium. Over time calcium exchanges for alkalis in the gel and the resulting gel will have relatively low Ca/Si ratio when compared with that formed in Portland cement paste. The main difference between the pozzolanic reaction and ASR is not only the characteristics of resulting products (C–S–H formed by the pozzolanic reaction is rigid, whereas ASR gel can imbibe water and swell), but also (owing to the fineness of SCMs) that the products formed through the pozzolanic reaction are homogenously distributed throughout the binder paste instead of accumulating around the weaker Interfacial Transition Zones (ITZ) or cracks within the aggregate as is the case of ASR.

This is mostly important when testing the performance of binders by exposing mortars or concretes to high temperatures; the acceleration of the pozzolanic reaction and ASR might not be at the same level. At high temperatures, the pore structure at early stage is altered, the capillary porosity is decreased and the transport of alkalis and water to the reaction sites is thus hindered. Mortar bar tests also show that, at a given age, the fineness of SCMs affects ASR expansion ([115,116]). These results may be attributed to the acceleration of pozzolanic reactions by increasing SCM fineness. At high temperatures, finer SCMs will react even faster. Thus, in a performance test method the pozzolanic reaction and the ASR should preferably be accelerated to the same extent.

#### *3.2.2. Fly ash (FA)*

In FA-containing binders, the alkali fixation in the resulting reaction products starts at the same time as the pozzolanic reaction, i.e.

after a period of approximately 28 days, which results in a successive reduction of the dissolved alkali content. Low-CaO FA reduces the pore solution alkalinity beyond just dilution [117]. Due to the pozzolanic reaction, the C–S–H phases are low in calcium, i.e. they have a low $CaO/SiO_2$ ratio, and have thus a high alkali binding capacity. The fly ashes that were found to be most effective in reducing the alkalinity of the pore solution expressed from paste samples were also found to be the best for controlling ASR expansion [118].

Sibbick and Page [119] stated that the effectiveness of the FAs in suppression of ASR was dependent on the initial alkali content of the mix and on the alkali content of the FA, but the results of pore solution analysis did not provide a simple explanation for the corresponding expansion data showing that the differences in expansions cannot *only* be explained by changes in the pore solution chemistry. Thomas et al. [120], following an overall survey, differentiate with respect to quality parameters of the SCM: limiting the total alkali content of the ggbs and fly ash to 1.0% and 4.5%, respectively (and some additional limitations), the alkali contribution from the SCM may be assumed to be zero, in spite of some contradicting laboratory conditions test results.

Shayan et al. [121] investigated the long-term results of concrete prisms at various alkali levels. It was shown that the two Australian fly ashes studied were effective in preventing deleterious ASR damage in concretes with alkali contents as high as 7.0 kg $Na_2O_{eq}/m^3$, but they produced only a delaying effect (up to two and six years depending on the type of aggregate) in concretes containing extreme amounts of alkalis; 12.5 kg $Na_2O_{eq}/m^3$. These results indicate that the effectiveness of fly ashes is dependent on the alkalinity of the mixtures as well as the type of the aggregates.

Exposure site studies up to 16–18 years [122] show that fly ash used at replacement levels of 25% and 40% was effective in significantly reducing expansion and cracking with all three flint aggregates at all alkali levels. The authors state that there is no evidence of alkali contribution by the fly ash. It was also indicated that the laboratory concrete prism test expansions did not confirm the field performance of blocks from the same mix. Significantly greater levels of alkalis are required to produce expansion in laboratory-stored concrete prisms compared with field-exposed blocks. Thus, the suitability of present performance tests is questionable due to alkali-leaching problems.

### *3.2.3. Silica fume (SF)*

Being a highly effective pozzolanic material, silica fume (SF) is among the most efficient SCMs for reducing ASR expansions even when used at rather low replacement levels (8–10%). However, the quantity of silica fume needed to prevent ASR is dependent on the aggregate reactivity. Depending on the level of replacement, silica fume decreases the $Na^+$, $K^+$ and $OH^-$ ion concentrations in the pore solutions of cement pastes and concretes due to binding of alkalis. Silica fume inclusion thus increases the $Na^+$ and $K^+$ content of C–S–H of the hydrated cement paste [123].

Silica fume rapidly binds alkalis probably due to a reaction very similar to ASR [111]. Thus, the alkali concentration in the pore solution decreases within the first two days of hydration [117]. The alkali–silica gel at the border of the silica grain reacts with available calcium to form C–S–H phases that have a low Ca/Si ratio. Most alkalis are bound by the alkali–silica gel in the silica fume particles and low-calcium C–S–H phases. At later ages the alkali–silica gel reacts with calcium, and alkalis will be released into the pore solution and increase the alkalinity of the pore solution after 28 days up to 2 or 3 years [111]. Alkali recycling starts after a fixation phase, at least part of the alkalis eventually become available for alkali–silica reaction.

As a consequence, performance testing of silica fume containing concretes need a prolonged testing time in order to detect the possible increased alkali level with time. However, a challenge is that more alkalis will be leached out of the test prisms with time, reducing the alkali level in the concrete pore solution. Due to its extreme fineness, the pozzolanic reaction rate of silica fume is higher than that of other SCMs (e.g. fly ash).

### *3.2.4. Ground granulated blastfurnace slag (ggbs)*

Similar to other SCMs, hydration products of slag (ggbs)-incorporating cementitious systems have decreased Ca/Si ratios, ranging between 1.55 and 1.79 [114]. The extent of alkali release is much less than that of clinker, and is almost independent of the alkali content of the ggbs. The alkalinity of the pore solution of ggbs containing cements is mainly attributed to the reduced clinker content of the cement. In cements with ggbs, alkalis are mainly absorbed by the C–S–H phases. Up to 40% ggbs, the Ca/Si ratio of the C–S–H phases and therefore the sorption properties are similar to that of OPC [117].

Arano and Kawamura [124] stated that at the early stages of ASR, the amount and composition of the gel produced does not seem to be affected by ggbs addition; however, decreased expansions may be due to the decreased mobility of ions and reduced $OH^-$ concentration of the pore solution. Hester et al. [125] observed that 50% replacement of Portland cement with ggbs significantly reduced the expansion of concrete in laboratory expansion tests. The authors indicate that the alkali level of the ggbs was not a contributory factor at this replacement level.

However, Zhao et al. [126] analysed the pore solution chemistry of mortar samples and suggest that the effect of ggbs is to produce a delaying effect by changing the gel composition for a temporary period.

Bleszynski et al. [127] investigated the ASR performance of ternary and binary mixtures incorporating ggbs by using concrete prism test and outdoor exposure site studies. Binary mixtures contained 35% and 50% ggbs replaced with Portland cement, respectively. Concrete prism tests revealed that the mixtures with blastfurnace slag were also capable of limiting expansion to below the CSA threshold 0.04% at 2 years. However, at a replacement level of 35%, the prisms still showed an increasing expansion trend beyond two years. A ternary blend mixture (3.8% SF and 25% ggbs) showed the most effective measure against ASR expansion in field studies.

Studies on the mechanism of ggbs in reducing ASR expansions are still far from elucidating the role of slag in controlling ASR expansion. Regardless, numerous field and laboratory studies confirm the efficiency of ggbs in elimination damaging expansion at replacement levels of 50% or more.

### *3.2.5. Other SCMs*

Some other SCMs have been found to be effective in reducing ASR expansions, e.g. metakaolin and other calcined clays, rice husk ash, zeolites and siliceous fillers. Incorporation of 20% metakaolin was found significantly to reduce the long-term $OH^-$, $Na^+$, and $K^+$ ion concentrations in pore solutions [109]. Burned and ground rice husk ash becomes quite pozzolanic (similar to microsilica), owing to its amorphous silica content and high surface area. The pozzolanic reaction depletes the CH content of tricalcium silicate pastes to about 1% at 28 days, and the hydration product C–S–H has Ca/Si ratio of about 1.3 [128]. Zeolites are found to be effective in reducing the alkali–aggregate reactions. Naiqian and Tingyu [129] explain the effectiveness of zeolites in reducing ASR as the decrease of pore solution alkalinity in concrete through ion exchange and pozzolanic reaction.

Pozzolanic behaviour of certain rock fillers may also mitigate the alkali–silica reaction, as discussed by Pedersen [77] and Pedersen et al. [130]. Examples of highly reactive materials being very effective pozzolans when crushed down to fines (finer than 63 μm) are Icelandic rhyolite and crushed bottle glass. These materials have a distinct amorphous silica phase. Fines from slowly reactive materials, such as Norwegian cataclasite and mylonite are not pozzolanic at normal curing temperatures, because the silica phase in these slowly reactive materials is well crystalline.

## 4. Mix design

### 4.1. Water/binder ratio

### 4.1.1. Pore solution alkalinity

By decreasing w/cm ratio the hydration products tend to become more homogeneous and contain less crystalline hydrates, particularly portlandite. At very low w/cm ratios, some of the portlandite may occur in nanometre dimensions rather than in well-crystallised form ([131,132]). Decreased w/cm ratio will lead to increased $OH^-$ concentration in the pore solution and vice versa [133]. With decreasing w/cm ratio, the pH increases and thus the dissolution of silica increases. Additionally, the release of alkalis from aggregates increases due to the increased solubility of alkali-minerals at high pH. On the other hand, in a dense paste (low w/cm ratio), transport and ingress of water or solutions, respectively, is reduced as well as the release of alkalis from aggregates [84].

### 4.1.2. Self-desiccation– relative humidity

*4.1.2.1. The role of water in the alkali– silica reaction.* Moisture is generally accepted to be one of the main factors affecting ASR. Water is important as a transport media for ions. The role of water is also important in the expansion stage. The overall expansion and cracking of concrete is basically caused by sorption of water by the alkali– silica gel, which in turn swells and thereby causes damage.

The water content in ASR-affected structures is normally expressed as relative humidity (RH), which is a measure of the thermodynamic state of the pore water. However, measurement of RH is notoriously very difficult and uncertain, particularly in the field. The critical limit for developing ASR is reported to lie in the range of 80– 90% RH depending on several factors, as discussed by Larive et al. [134].

*4.1.2.2. Mechanisms causing self-desiccation.* The hydration process of cement gives a reduction in the overall volume of the paste. This is due to the fact that the reaction products (i.e. C– S– H gel and CH) have a smaller volume than that of the original reactants (cement + water). This phenomenon is referred to as chemical shrinkage, and has some major effects:

1) It causes autogenous shrinkage, which is a volume contraction of the total concrete body. In the plastic phase, the chemical shrinkage equals the autogenous shrinkage.
2) In the hardening phase, the chemical shrinkage results in empty pores within the concrete. These pores will remain empty if no water is supplied from the surroundings. This leads to a lowering of the RH in the concrete, a phenomenon called self-desiccation. Generally, the extent of self-desiccation increases with decreasing w/cm ratio.
3) When water is gradually consumed during the hydration process and the chemical shrinkage pores are left empty, the remaining water will be in a state of "tension stress". This is the mechanism explaining the autogenous shrinkage in the hardening state.

*4.1.2.3. Practical implications.* For practical purposes the effects of self-desiccation might become important for concretes with w/cm ≤ 0.45. At low w/cm ratios this effect is large and may reduce the RH even below 80% over a period of time, provided there is no water supply from the surroundings. Consequently, a minimum limit should be considered for the w/cm. If such test limitations are not introduced, the internal RH in laboratory test prisms might be lower than in structures exposed to water in service. This could lead to incorrect test conclusions, i.e. some potentially alkali-reactive mixes could be classified as non-reactive based on performance testing because of the lack of water. A suggestion for such a minimum limit could be w/cm ≥ 0.40— in other words, performance tests should not be conducted at w/cm less than 0.40. However, the type of binder, in particular the type and amount of any SCM used, will influence the extent of self-desiccation. More research is thus needed as basis to agree on a possible lower w/cm limit for performance testing.

Additionally, the aggregate porosity and the aggregate moisture state at the time of mixing might significantly influence the RH within the concrete. If rather porous (≥0.8%) normal density pre-wetted aggregates are used, they may theoretically totally counteract the effect of self-desiccation. This is due to supply of water from the aggregates to the cement paste during the curing period ([135,136,137]). As a consequence, it might be conservative to use pre-wetted aggregates in laboratory performance testing. Conversely, if dry porous aggregates are used, this will likely exacerbate self-desiccation.

The shrinkage due to self-desiccation of a concrete with w/cm ratio 0.35 might be in the order of 0.01% after one week curing [138], and in extreme cases up to 0.02% [139]. Consequently, it may significantly influence the measured prism length in the early age, in particular the reference readings if the concrete prisms are prolonged pre-cured at 20 °C for e.g. one week as in the RILEM AAR-3 concrete prism test [5]. One important question needs further research or at least detailed and informed discussion within RILEM TC 219-ACS to achieve consensus: *What is the most correct "reference length" to apply in ASR expansion testing; the length after de-moulding, the shortest length after some shrinkage has occurred or the length after a pre-curing period*? The magnitude of the irreversible shrinkage will also influence the reference length, as will the internal prism temperature during the reference readings.

### 4.1.3. Transport properties

Increasing w/cm ratio will result in a higher and more continuous porosity (more capillary pores), and consequently internal transport processes will be accelerated, the rate of alkali leaching will increase and water or possibly other solutions will penetrate more easily ([140,141]). All these mechanisms might influence the rate and extent of ASR during laboratory performance testing, calling for similar concrete quality to be used in laboratory performance testing as in the actual field structures. If deviations are necessary, laboratory testing should aim to give conservative results.

Several authors, e.g. Stark [142] and Sellevold [143], have shown that moisture fluctuations in the field basically take place in the outer layer (some centimetres thick) of the concrete. The depth of the influenced zone will decrease with decreasing w/cm ratio, as shown by Yang et al. [144], as the result of a reduced permeability. As a consequence, the residual concrete mix water, depending on the w/cm ratio (influencing the extent of self-desiccation), rather than ambient wetting and drying, determines the prevailing moisture content in the interior of massive concrete structures, as discussed by Stark [145]. For such structures the extent of self-desiccation, mainly controlled by the w/cm ratio, may govern the interior RH level of the concrete. Also during laboratory testing, there is a probability for development of a moisture profile through the prism cross-section, with lowest RH in the mid part, in particular if the size of the concrete specimens is rather large (≥100 mm cross-section) combined with a rather low w/cm ratio (≤0.40).

The type of binder will also influence the permeability of the concrete, and thus the permeability related ASR mechanisms, i.e. internal transport processes, alkali leaching, water uptake and sensitivity to drying during exposure and measuring in the laboratory. Furthermore, increased permeability in aggregates may enhance the alkali reactivity due to easier access to concrete pore fluids [41].

### 4.2. Binder content

Unless alkalis are added during mixing, the cement content directly controls the alkali content of concrete mixes and, hence, the rate and amount of expansion due to ASR. However, changing the cement content can also modify the water/binder ratio as discussed above and this can influence transport properties, including alkali leaching, the concentration of ions in the pore solution, and self-desiccation. Differences in the cement-to-aggregate ratio, within the range usually encountered with typical concrete mixes, are unlikely to have a significant impact on the outcome of the test unless an aggregate exhibits a pronounced pessimum behaviour.

### 4.3. Alkali boosting

The alkali content of the concrete is a critical factor in determining both the rate and amount of expansion induced by ASR. Fig. 3 (produced from unpublished data from the Building Research Establishment, U.K.) shows the expansion of concrete prisms as a function of the alkali content of the concrete, for concrete with a range of cement contents (and w/cm), cement alkali levels and with and without alkali boosting (by the addition of $K_2SO_4$ to the mix water in this case). The data indicate that expansion is primarily a function of the alkali content and to some extent independent of the cement content, the alkali content of the cement and whether or not the alkali content was boosted.

The alkali content of the concrete is often boosted to ensure that there are sufficient alkalis present to identify reactive aggregates and to compensate for alkali leaching. However, extensive alkali boosting is not recommended for performance testing as it masks the critical role of the alkali content of the job mixture. Other concerns regarding alkali boosting include the following:

- The effect of alkali boosting on expansion also depends on the type of aggregate [146].
- The alkali compound added might influence the behaviour of the concrete [147].
- For a binder with 7.5% silica fume, Pedersen [77] documented that alkali boosting significantly increased the concrete permeability and reduced the compressive strength up to one year of standard curing. The capillary porosity was also increased, but less pronounced.
- The addition of alkalis may accelerate the release of alkalis from certain aggregates ([81,148,83,84]).
- Also the type of the alkali ions ($Na^+$ or $K^+$) and the source influence the release of the alkalis.
- The addition of alkalis may change the K/Na ratio, which could impact the expansion ([91,92]).
- The increased pH will reduce the concentration of calcium in the pore solution and some $Ca^{2+}$ is required for the formation of swelling gels ([149,133,150]).
- Experiences with mortar bar tests at the FIB (unpublished data— information received from Colin Giebson) show clearly that alkali boosting mortars with cements (CEM I) different in $Na_2O_{eq}$ to the same alkali level by adding NaOH does not result in the same expansion. This finding appears to contradict the observations at BRE shown in Fig. 3.

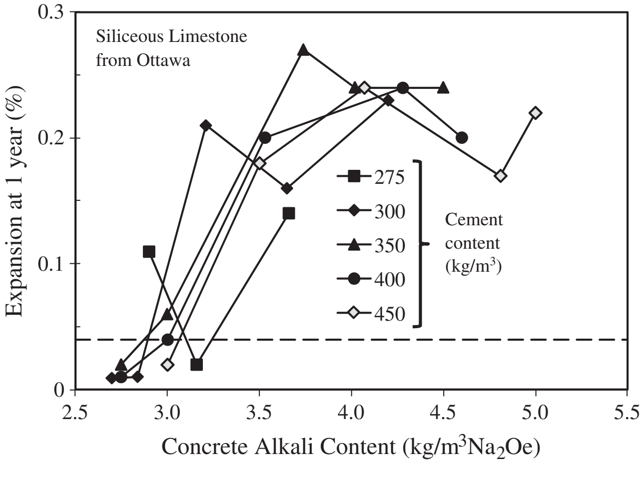

**Fig. 3.** Expansion of concrete prisms as a function of alkali content (produced from unpublished data from the Building Research Establishment, U.K.). The dashed line represents the critical 1 year expansion limit.

The effect of extensive alkali boosting is currently subject to further research within RILEM TC 219-ACS.

### 4.4. Chemical admixtures

There are no indications in the literature that chemical admixtures that are added either to modify the workability or the set behaviour of the concrete either in the laboratory or the field significantly influence ASR, unless the admixtures contain significant quantities of alkalis including sodium, potassium and lithium. The latest generation of admixtures normally do not contribute to the alkali content of concrete, but an exception is still shotcrete accelerators ([151,152]), available in both high and low alkali level versions.

The use of air-entraining admixtures is unlikely to have a direct effect on ASR, but the presence of an air-void system may impact the amount of expansion. There is some conflicting evidence in the literature regarding the role of air content on ASR expansion. Whereas it is generally agreed that air will not prevent ASR expansion, there is some evidence that it can reduce or delay expansion with some aggregates by accommodating ASR gel ([153,154]). However, there are some other findings in the literature that air entrainment is not beneficial in reducing ASR expansions ([60], [155]). It is recommended that the air content of the performance test is the same as that intended for the job mixture. However, alternatively it may be recommended to use lower air content in the laboratory test prisms, since this is a conservative approach.

Lithium-containing admixtures are effective in controlling expansion with some aggregates. Feng et al. [156] summarised the findings in the literature about the effect of lithium salts on the reaction products formed, and stated that the efficiency of lithium salts in suppressing the ASR expansions depends on the nature and reactivity of the aggregate, the form of lithium, the amount of alkalis in the pore solution and the dosage of lithium salt added (lithium to alkali molar ratio). It is essential that the lithium to alkalis (sodium and potassium) ratio, $[Li]/[Na+K]$, in the performance test is equal to that of the job mixture being tested.

## 5. Exposure conditions

### 5.1. Pre-storage conditions

The "pre-storage period" is defined as the period from casting of the concrete prisms up to the point of the initial (zero) length comparator readings. The "pre-storage conditions", i.e. the storage conditions during the pre-storage period, vary for different concrete prism tests. After casting, most test methods describe storage of the moulds at 18–23 °C and minimum 90–95% RH in the surroundings, while other describe more humid storage of the moulds, e.g. in a fog room with 100% RH. After de-moulding the day after casting, some test methods describe direct exposure of the prisms to the actual storage temperature, e.g. ASTM C-1293-08b [11]. Other methods describe 0.5 h submergence of the prisms in water after de-moulding, before further preparation for final storage. Finally, the length of the pre-storage period at 18–23 °C normally varies from 1 day (e.g. as in ASTM C-1293-08b [11] and RILEM AAR-4.1 [6]) to 7 days (e.g. as in RILEM AAR-3 [5]). For performance testing, some laboratories use an even more prolonged pre-storage period, up to 28 days, dependent on the type of binder [157].

Possible influences of the variations in pre-storage conditions and ASR exposure conditions on vital “ASR parameters” (i.e. prism internal humidity, composition of the concrete pore solution, aggregate reactivity, properties of hydration products formed and properties of any reaction products formed) and thus on the outcome of a performance test are discussed in this section.

### 5.2. ASR exposure conditions

The result of a performance test is strongly dependent on the ASR storage conditions and thus on the initiation and progress of ASR. The following parameters are evaluated and discussed:

- Moisture conditions
- Type of container
- Prism size
- Wrapping (if any)
- Storage temperature
- Storage period
- Any external alkalis added

### 5.3. Internal humidity

During laboratory performance testing, the internal moisture content within the concrete prisms is aimed to be very high, i.e. higher than in many real concrete structures. Thus, the prisms should be subjected to “worst-case humidity conditions”. The pre-storage and storage conditions might also significantly influence the interior RH of the prisms.

The following parameters may increase the influence of self-desiccation when using a relatively low w/cm ratio, and thus contribute to maintain a “rather low” internal RH in the concrete prisms: prism size (worse when increased), micro climate in the storage containers (worse the lower the RH is inside the containers), length of the pre-storage period (reduced internal RH if prolonged storage period due to a higher degree of hydration), permeability (less water uptake if low) and storage temperature (the degree of influence may vary dependent on the micro climate in the storage container). Any possibility for drying of the prisms due to evaporation, e.g. if they are pre-stored in a climate with less than 100% RH in the air, will also naturally influence the prism interior RH.

The curing temperature might influence the concrete porosity and permeability. For an OPC, a higher curing temperature in the early age will normally lead to a coarser porosity and consequently an increased permeability, as reported by Kjellsen et al. [158], Kjellsen and Detwiler [159] and Lothenbach et al. [160]. Somewhat contradictory, Schmidt et al. [161] found that the total capillary porosity measured after three months on concrete samples cured at 20, 40 and 60 °C, respectively, decreased with increasing storage temperature. The influence of curing temperature was most pronounced for the concrete containing 30% fly ash compared with the OPC concrete. However, in these tests all concretes were presumably pre-cured at approximately 20 °C until de-moulding (not stated specifically in their study), i.e. in the early hydration period the curing temperature was equal. This implies that their measured lower capillary porosity at elevated temperature primarily reflects a higher degree of hydration and that more fly ash reacts earlier at elevated temperature.

In general, a concrete subjected to prolonged pre-curing period at 20 °C will have a lower permeability when starting the ASR test compared with a concrete exposed to the ASR storage conditions directly after de-moulding. The time at which the temperature is elevated and the magnitude of the elevated temperature (normally in the range 38 to 60 °C) will consequently influence the concrete water uptake, the water transport properties, as well as the drying properties, and thus also the internal concrete moisture content during the accelerated ASR testing.

In order to maintain a high internal RH in the test prisms during the entire test period, the ambient humidity during storage must be very high (≈100%). The micro climate in the storage containers is thus of great importance. Important parameters are size and design of storage containers, type of lining (if any) and application of a watertight seal. Large containers may lead to an inhomogeneous distribution of moisture. For example, the experience gained with the Norwegian 38 °C CPT [23] indicated that when large storage containers holding several concrete prisms were replaced by smaller containers holding only 3 prisms in each, there was a general increase in expansion. Also a Norwegian sandstone, proven to be reactive in the field, showed expansions above the critical limit in the smaller containers, but not in the larger ones [73].

The sorption properties and the internal RH in concrete are to a certain degree dependent on the storage temperature. A raised temperature in part of a concrete sample/structure will lead to increased local vapour pressure. This will initiate moisture transport from warmer to colder regions and, eventually, reduce the local moisture content, and as a consequence lead to a decrease in RH, as discussed by Nilsson [162]. This phenomenon will take place during cooling of concrete prisms overnight, before the prisms are measured the day after. During cooling, moisture will move from the warmer inner part to the colder outer parts of the specimen.

On the other hand, if the moisture content within a concrete is rather constant (as one can assume for small concrete prisms stored over water in a sealed container, at least if the w/cm ratio is not too low), a general increase in the temperature will give rise to a small increase in the internal RH. For example, according to tests reported by Sjöberg et al. [163], the RH within a concrete with w/cm 0.40 and internal RH 90% will increase approximately 0.25% per °C. Thus, an increase of the concrete temperature from 20 °C to 40 °C might increase the internal RH by approximately 5%. The effect decreases with increasing w/cm ratio ([163,162]). The effect is max in the middle RH-range (around 50–60%), and decreases to zero in very dry and in saturated concrete [164]. One consequence of this phenomenon is that the internal RH in concrete prisms will increase with increasing storage temperature, provided there is no change in the concrete internal water content.

The susceptibility to loss of moisture during the exposure period will increase with increasing storage temperature. The extent of any weight loss is controlled by the relative humidity in the surroundings and the type of container. If concrete prisms are stored over water in containers placed in a dry and hot room, as is the case for several concrete prism tests (Norwegian CPT [23], ASTM C1293-08b [11], CSA A23.2-14A-04 [13], RILEM AAR-3 [5]), the risk of drying is high compared to storage in containers placed in a humid environment—e.g. in a reactor, as in the RILEM AAR-4.1 CPT [6]. If the lid is broken or not made watertight, the risk of evaporation of the water in the bottom of the container is significant provided storage in a dry room or an oven. Also the storage period influences the sensitivity to loss of water. For instance, SINTEF have experienced that RILEM AAR-3 containers [5] are particularly vulnerable to drying due to the small amount of water in the bottom of the containers (only 350 ml) combined with a long testing time (one year for aggregate testing).

As a consequence, the test set up and the test procedures must aim to avoid loss of water during storage and measuring. Important parameters in this respect are quality control (e.g. control of the water level and use of watertight lids), strict measuring procedures (measure quickly with as low moisture loss as possible), evaluation if the prisms should be pre-cooled or not before measuring (the prisms will dry during cooling, because moisture will move from the warmer inner part to the colder outer parts) and storage temperature (the higher storage temperature, the more drying during cooling). As a quality control, the mass of prisms should always be measured, evaluated and reported.

As several have experienced, e.g. Lindgård et al. [165], after a possible weight loss in the first one to two weeks, the weight of the prisms normally increases with increasing expansion and with time. However, if the prisms show too low mass increase with time or in extreme cases weight loss over time, this is most likely due to insufficient water present in the system, and consequently the test results might be questioned. Another reason for a decrease in prism weight might be connected to the ambient humidity the first day of curing. Unpublished data from the PARTNER project [37] showed that storage in a fog chamber with 100% RH the first day after casting lead to a higher reference prism weight compared with storage at approximately 95% RH (as required in the method). When testing some non-reactive aggregates in the 60 °C AAR-4.1 reactor [6], the prisms pre-stored in the fog chamber showed a slight weight loss in the end of the test period, while the prisms stored at 95% RH showed a slight weight increase. However, the expansion test results were comparable.

Submerged storage of the prisms will give rise to high internal water content, unless large test specimens and a low w/cm ratio are applied. One consequence of the likely higher moisture content in submerged concrete prisms, compared to most field concrete structures, is development of a less swelling gel due to a reduced viscosity [166]. However, an essential consequence of any submergence in water is extensive leaching of alkalis [167], and such storage is not recommended.

Wrapping of concrete prisms, by use of moist cotton cloths and plastic sheets, are applied in some test methods primarily with the aim to secure a high moisture content surrounding the prisms, but some have also expected less leaching of alkalis from wrapped prisms. On the other hand, wrapping might reduce the access to ambient moisture in the air. If the wrapping effectively hinders contact between the ambient moisture content in the storage container and the prisms (e.g. as in the RILEM AAR-3 CPT [5], where the wrapped prisms are stored in plastic bags), the amount of water added during the wrapping procedure and on top of the prisms at every measuring point of time may have large influence on the internal moisture content within the concrete prisms. Unpublished data from Jan Lindgård's PhD study (2007–2012) at NTNU reveals that wrapping significantly reduces the extent of evaporation during cooling (provided cooling of the prisms before each length reading) and during measuring.

Another important factor is to keep the internal prism temperature constant during all measurements. Several methods state that the maximum allowed variation in the room temperature where the prisms are being stored before and during the measurements is ±2 °C, e.g. RILEM AAR-3 [5]. *But how sensitive is the recorded expansion of a moderately deviating prism temperature at measuring compared with the temperature during the reference readings*? A concrete will expand approximately 0.001% if the temperature increases 1 °C [168]. This means that a 5 °C temperature change corresponds to approximately 0.005% length change. When the critical expansion limit for several concrete prism methods is in the range of 0.030–0.040%, a 5 °C temperature deviation constitutes 1/6 to 1/8 of the critical expansion limit, i.e. the influence of a deviating internal prism temperature might be significant.

An aspect that significantly can influence the recorded reference length of the prisms (being the basis for calculating the expansion) is whether the initial length readings are taken immediately after de-moulding or after 30 min submergence in water. Without submergence, the internal prism temperature may be somewhat higher than 20 °C due to the cement hydration. If the prisms are submerged, the water temperature will control the internal prism temperature. If the quality control in some laboratories is not satisfactory, a temperature variation up to 5 °C is likely to occur.

### *5.4. Composition of the concrete pore solution*

As discussed previously, the content of alkalis (i.e. $Na^+$ and $K^+$) in the concrete pore solution plays a major role in development of ASR. Thus, all conditions during pre-storage and storage that may contribute to change the alkali content in the pore solution, either reduce it (e.g. due to alkali leaching or binding of alkalis) or increase it (e.g. due to alkali release from aggregates or ingress of any external alkalis e.g. from de-icing salts) will consequently influence the rate and extent of ASR during laboratory performance testing.

In several studies, results from pore solution analyses are reported. However, there is no consensus on the procedure on how to extract pore solution, analyse it and interpret the results. In particular for low w/cm ratios (e.g. <0.50), it is very difficult and in many cases impossible to press any pore water from the concrete, even though high pressures are applied. If extremely high pressures are used, there is also a question over whether the pressed pore water is representative for the pore water within the concrete. Some researchers have thus used alternative methods to detect the soluble alkali content in the concrete pore water. One example is the “hot water extraction method” that Berubé et al. [87] used to measure the extent of alkali release from various aggregate types.

#### *5.4.1. Alkali leaching*

The problem of alkali leaching from specimens stored over water in sealed containers was first reported by Blanks and Meissner in 1946 [169]. The authors detected a build up of alkali ions in the water at the bottom of the containers in which mortar bars were stored, and explained this based on water condensing on the surface of the bars and running down the bars into the reservoir below, thereby providing transport of the alkalis. The mechanism for alkali leaching is further explained by Rivard et al. [20] to be excessive condensation of water on the prism surfaces, leading to an outward diffusion of alkalis from the interior of the concrete. The degree of alkali leaching depends strongly on the storage conditions. Additionally, the conditions during pre-storage might significantly influence the rate and extent of alkali leaching. A prolonged pre-storage period at a moderate storage temperature (18–23 °C) is assumed to reduce the early extent of alkali leaching due to a lower permeability when the concrete prisms are exposed to the ASR storage environment. On the other hand, early exposure of the prisms to a very humid environment, e.g. a fog chamber with 100% RH or even more extreme storage conditions, e.g. submerging the prisms in water after de-moulding for a long period, are expected to increase the extent of alkali leaching.

Thomas et al. [24] found that specimen size clearly has a large impact on expansion and this effect can be largely ascribed to more leaching of the alkalis from smaller specimens. In their study they found that the impact of alkali leaching will be less for larger concrete prisms, but is still significant. Three concrete prisms ($75 \cdot 75 \cdot 300$ mm$^3$) containing a reactive siliceous limestone (Spratt) were stored over water at 38 °C in a container. By assuming a constant reservoir volume of 1.8 l and neglecting any alkalis that may wick up the absorbent material lining in the container, it was estimated that approximately 35% of the alkalis originally in the concrete found their way into the water reservoir after 1 year, and as much as 20% after just 90 days. Also tests performed by Bakker [170] and Lindgård [167] showed that the larger the cross-section of a concrete prism, the greater the expansion, which was interpreted as being caused by higher extent of alkali leaching for the smaller specimens. However, even for larger concrete prisms (cross-section $100 \cdot 100$ mm$^2$) alkali leaching cannot be neglected [165].

Rogers and Hooton [171] found that concrete prisms (assumed size $75 \cdot 75$ mm$^2$ cross section) stored in a moist room showed the least expansion, as well as the greatest amount of alkali leaching. Storage in a polyethylene bag resulted in less leaching of alkalis and more expansion. Results of percent change in alkalis at 130 weeks of exposure exhibited;

- 22% decrease for prisms stored at 23 °C in plastic bags with 100 ml of water
- 42% decrease for prisms stored at 38 °C over water in a sealed box
- 63% decrease for prisms stored at 23 °C in a moist room

Fournier et al. [36] studied deviations between the 38 °C concrete prism test and the 60 °C accelerated concrete prism test. They verified that in the 60 °C test, the ultimate expansion was considerably lower, probably due to higher extent of alkali leaching and changes in pore solution composition (more sulphate dissolved—see later). The increased alkali leaching at elevated temperature is expected, since the diffusion increases with increasing temperature.

According to Bokern [166], under extremely humid conditions, like in a fog chamber with temperature 40 °C, intensive alkali leaching occurs. A loss of 20% of the initial soluble alkali content after 28 days and more than 30% after three months is possible for a concrete with OPC and w/cm ratio 0.55. In these tests, 100 mm cubes were used. Comparable values were also reached in the 60 °C RILEM AAR-4.1 CPT [6] after three months storage of the concrete prisms with cross section $70 \cdot 70\ \text{mm}^2$ on grids over water in the small containers inside the reactor.

Submergence of test prisms in water will remove most soluble alkalis from the concrete pore water, thus substantially slow down or stop any potential ASR, as shown e.g. by Lindgård [167].

Wrapping in cotton cloth and plastic has sometimes been applied in order to decrease alkali leaching, but there are also references showing that wrapping may decrease the expansion ([30,172]). Lindgård [167] documented that wrapped concrete prisms stored at 60 °C showed significantly lower expansion than unwrapped prisms due to increased alkali leaching in the early age.

Nixon et al. [173] and Åhs [174] have shown that alkalis can diffuse towards the surface of concrete on wetting and drying. The alkali concentrated regions are generally located in the outermost regions of concrete structures after drying of concrete. Thus, even for concrete containing low alkali cement, local ASR formation might become possible [175]. As a consequence, drying/wetting cycles during cooling of concrete prisms overnight (before each measurement) might transport alkalis to the prism surface and thus enhance the extent of alkali leaching (compared to measuring the prisms without pre-cooling).

Rivard et al. [176] showed by chemical analysis of the water beneath test prisms that the alkalinity reduction of the concrete pore solution with time was mostly associated with alkali leaching. It was shown that for the same reactive mixture, concrete alkali leaching seemed to be greater for the specimens containing higher alkali level ($5.25\ \text{kg/m}^3\ \text{Na}_2\text{O}_{\text{eq}}$) compared with specimens with lower alkali level ($4.00\ \text{kg/m}^3\ \text{Na}_2\text{O}_{\text{eq}}$).

The cement type or binder combination also influences the rate of alkali leaching due, among other things, to the influence on the concrete permeability. According to Bokern [166], concrete made of OPC or cement with ggbs (20%) seems to be particularly vulnerable to alkali leaching compared with cement with silica fume or fly ash. Recent investigations by Schmidt [177] also show (Fig. 4) that the extent of alkali leaching during ASR-testing in a 40 °C fog chamber is influenced by the binder combination. Naturally, this will influence the measured expansions.

In contrast to many laboratory results, pore solutions in field concrete are mostly not subject to alkali leaching, according to Rivard et al. [176], probably due to the higher volume to surface ratio compared with laboratory specimens. The problem of alkali leaching is thus a big challenge in laboratory tests.

### 5.4.2. Alkali release from aggregates

The storage conditions during ASR testing might also influence the rate and extent of alkali release from aggregates. Ideker et al. [178] showed that the contribution of alkalis from a “non-reactive” sand resulted in increased concentration of $K^+$ in the pore solution, elevated pore solution pH and a higher rate of expansion at early age compared with other “non-reactive” sands tested. The difference was most pronounced for the 60 °C CPT compared with the 38 °C CPT.

### 5.4.3. Storage temperature

Fournier et al. [36], Lothenbach et al. [160] and Schmidt et al. [161] have documented that the concentration of sulphates in the pore solution is increased when the storage temperature is elevated, see Fig. 5. Consequently, the concentration of $OH^-$ in the pore solution, and thus the pH, is correspondingly decreased resulting in a lower solubility of $SiO_2$. The cause of the higher concentration of sulphates in the pore solution at elevated temperature is higher solubility of ettringite [36]. If the pre-storage of ASR concrete prisms at approximately 20 °C is prolonged e.g. from 1 day to 7 days, less ettringite will be available in the concrete prisms (more is transformed to mono-sulphate), and probably less ettringite will be dissolved when the ASR test is started (i.e. the temperature is elevated). Consequently, the pH of the concrete pore solution will be increased.

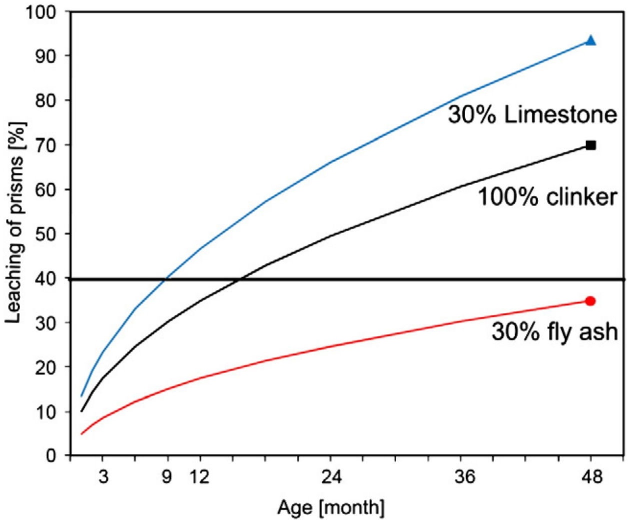

**Fig. 4.** Estimated development of alkali leaching of concrete prisms with different binders, 400 kg/m³ of binder, w/cm = 0.45, storage at 40 °C and 100% RH in a fog chamber [177].

Partly as a result of the drop in $OH^-$ concentration (Fig. 5), the addition of only 10% fly ash to the binder was apparently able to suppress the expansion below the critical expansion limit for a highly reactive aggregate when exposed to 60 °C one day after casting [161]. When pre-stored at 20 °C for at least 28 days before starting the 60 °C CPT, the concrete prisms with 10% fly ash expanded far beyond the critical limit. Also when adding 20% fly ash, the length of the pre-storage period affected the measured prism expansion significantly, but the effect was less pronounced. When adding 30% fly ash, no expansion was revealed for any of the three pre-curing periods applied (1, 28 and 90 days, respectively).

Schmidt et al. [161] believe that the accelerated rate of reaction of the fly ash when exposed to elevated temperature after 1 day also contributes to reduce the expansion of the concrete prisms exposed at early ages (see further discussion later). However, the extent of alkali leaching was not discussed in the paper, but might according to our experience also have influenced the results presented, since the rate of alkali leaching is expected to be highest in the concrete prisms exposed to the ASR storage at earlier ages (i.e. at age 1 day).

### 5.4.4. External alkalis

According to Nixon et al. [179], the introduction of sodium chloride to cement paste, mortar or concrete at the mixing stage, results in an elevation of the hydroxyl ion concentration of the pore solution to a level similar to that produced by a Portland cement with an equivalent alkali level. This will increase the likelihood of damaging ASR in concrete with alkali-reactive aggregates. Correspondingly, various external alkalis may influence the concrete pore solution chemistry. Thus, when testing the influence of various de-icing salts on ASR in a performance test, similar “alkali-conditions” (i.e. identical type and amount of cement, admixtures and any external alkalis; etc.) should preferably be used as will be used in the field.

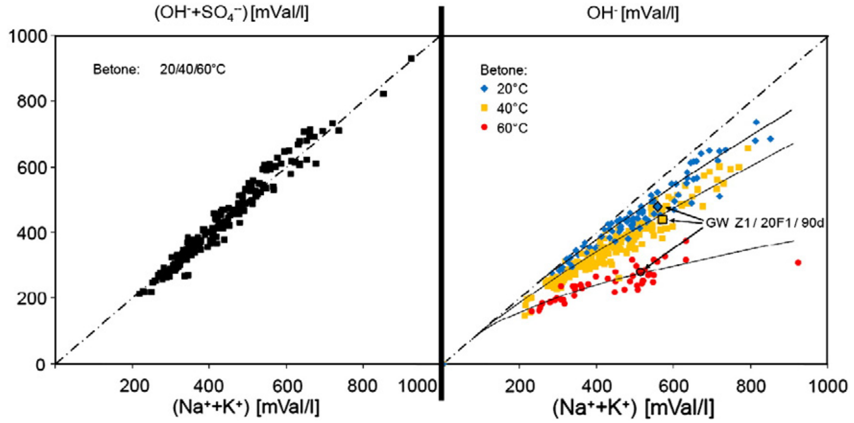

**Fig. 5.** Concentration of anions $OH^-$ and $SO_4^{2-}$ vs. concentration of cations ($Na^+$ and $K^+$) of pore solutions at different temperatures [161].

Additionally, alkali leaching from the test samples during expansion testing should be considered when fixing testing conditions, considering threshold levels or conforming to acceptance criteria.

Information on the influence of external alkalis on the alkali binding capacity of the hydration products formed with OPC, ggbs, FA and SF was not found in the literature study.

### *5.5. Aggregate reactivity*

The solubility of silica species is controlled by pressure, temperature, particle size, pH and by dissolved species in the solute [35]. Dove [180] concluded that the net dissolution rate of quartz in aqueous solutions containing mixtures of cations is dominated by the ions with the stronger surface interaction ($Ba^{2+} > K^+ \approx Na^+ \approx Li^+ \approx Ca^{2+} > Mg^{2+}$).

Regarding the effect of temperature, experiments show that the exposure temperature influences the aggregate reactivity. The solubility of $SiO_2$ increases with the temperature (Fig. 6), but the effect is different for different forms of silica. High exposure temperature also seems to activate some apparently “non-reactive” aggregates [39]. A greater amount of gel is formed and higher expansion is observed at a given time since the reaction rate is accelerated by temperature. However, the temperature should not be analysed in isolation, as it works simultaneously with other factors.

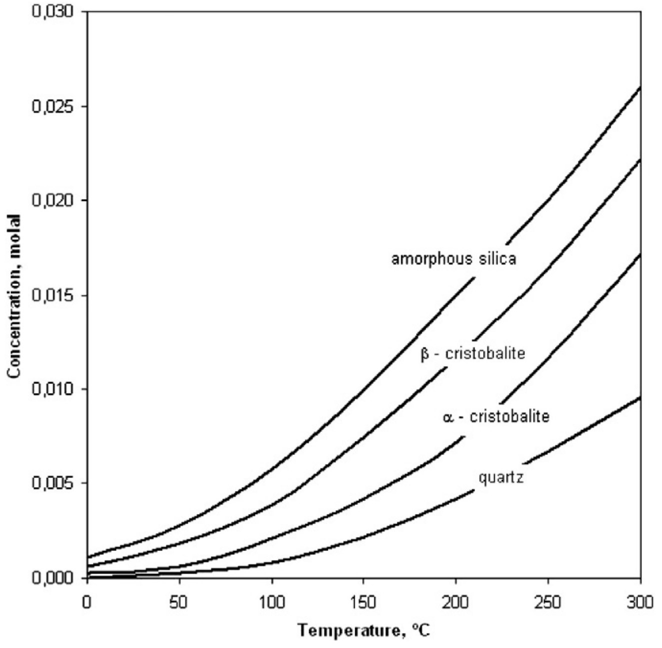

**Fig. 6.** Solubility of polymorphs of silica regarding temperature based on equations from Rimstidt and Barnes [181,182].

The effect of temperature is mirrored in the experimental results of tests with different temperatures. Fournier et al. [146] performed field tests on concrete blocks in two different locations, one in Texas and one in Canada, to study the effect of ambient temperature. Those authors concluded that the expansion is faster and higher for the place with highest exposure temperature, but it depends on the type of aggregate selected. The difference in expansion increases with decreasing reactivity level. Additionally, Iler [183] concluded that with testing at 60 °C, more siliceous material from the aggregate is likely to be dissolved, compared with that at 38 °C or at ambient conditions.

Very fine particles of certain rock types have the capability to react pozzolanicly and hence increase the C–S–H phase, as suggested by Pedersen [77]. An important finding by Pedersen [77] was a significant temperature effect. Some rock fillers that were not pozzolanic at ordinary curing temperature may be highly pozzolanic at a temperature of 80 °C. This fact is of high significance when using test regimes with very high temperatures. Testing of mortar or concrete mixes with significant amounts of fines from alkali-reactive rocks may then give a “false negative” result if tested at very high temperatures. This arises from the fact that the pozzolanic reactivity increases with increasing temperature. Pedersen [77] examined exposure temperatures of 20, 38 and 80 °C, and concluded that methods using 80 °C should not be used for performance testing of real mixes.

### *5.6. Properties of hydration products formed*

The rate of hydration of Portland cement increases with increasing temperature, which is more pronounced at lower degrees of hydration. The composition of C–S–H does not differ from that formed at ambient temperature up to about 50 °C, but beyond this temperature Ca/Si ratios increase moderately. At high temperature curing, the paste might have a higher porosity and/or coarser pore structure, even decreasing the strength at long hydration times [184]. According to the authors' experience, the effect of curing temperature is more pronounced at the early period of hydration.

The effect is even more complicated when SCMs are present in the cementitious system. The average Ca/Si ratio of hydration products of fly ash (FA) increases with increasing storage period and increasing temperature, which results in a decreased alkali sorption capacity [185]. Up to 40% ggbs, the Ca/Si ratio of the C–S–H phases and therefore also the alkali sorption properties are similar to that of OPC [117]. By contrast, for ggbs, higher temperatures may lead to a higher degree of condensation of the silicate anions in the C–S–H phases, and therefore to a lower Ca/Si ratio [186]. Thus, the C–S–H phases formed can absorb more alkalis. Additionally, De Weerdt and Justnes [187] have documented that not only the C–S–H phases are changed when elevating the temperature from 20 °C to 80 °C, but also the aluminium containing phases. However, more hydration products of fly ash are formed at 40 °C and 60 °C than at 8 °C and 20 °C, thus more alkalis can be bound [185]. The two opposite trends indicate that there may be an optimum temperature for the highest alkali binding capacity of FA. Baetzner and Böhm [188] found that, in the presence of FA, the alkali concentration in the pore solution often was lowest at 40 °C. Thus, testing of FA containing concretes at this temperature will not be conservative.

In order to access the alkali reactivity potential of specific concrete compositions in a relatively short time, mortar or concrete samples are often exposed to high temperatures at very early ages. Bokern [166] assumes that ASR takes place within 28 to 56 days in very accelerated laboratory conditions (temperature 60 °C), when maximum alkalinity in the pore solution usually is achieved. Under normal conditions (20 °C), the pozzolanic reaction of FA starts after the age of 28 days [117]. If the concrete with FA is exposed to high temperatures at an early age, alkalis may be bound by the accelerated pozzolanic reaction that takes place before the ASR. This means that the pore solution has a lowered alkali hydroxide content when the ASR is about to start. To reduce this impact on the alkalinity of the pore solution, Bokern [166] thus recommends storing the concrete samples under normal temperatures to allow a normal development of the pore solution composition. This is confirmed by Schmidt et al. [161], where the accelerated concrete prism test at 60 °C is extended by a prestorage period of 28 and 90 days at 20 °C before the samples are tested at 60 °C.

Temperature effects on laboratory test expansions vary with respect to the alkali content of the system. If a fixed amount of alkalis is available, maximum expansions are observed at a pessimum level of temperature (38–40 °C) [189], i.e. the expansions are reduced for lower and higher temperatures. Tests carried out with an unlimited amount of alkalis show that for particular aggregates, total expansion decreases with increasing temperature [190]. The latter case might be connected to the behaviour of calcium hydroxide as the solubility of calcium hydroxide decreases by increasing temperature. For many field structures, the tendency is that the higher temperature, the higher the rate of alkali–silica reaction [173].

Diamond et al. [102] tested opal-containing sealed mortar specimens at 20 °C and 40 °C, respectively. It was observed that the rate of reaction and expansion is higher at 40 °C, however, the ultimate expansion and the percent of alkalis reacted become higher at 20 °C at later ages [99]. This should be kept in mind while evaluating the laboratory versus field expansions.

The implications of the findings discussed above are that the time of starting the ASR exposure as well as the exposure temperature might significantly influence the outcome of a performance test. This is due both to accelerated pozzolanic reactivity if the concrete is exposed to elevated temperature at early ages and to the temperature-dependence of the alkali binding capacity of various SCMs.

### *5.7. Properties of reaction products formed*

#### *5.7.1. ASR-products*

The expansive forces caused by gel depend on the gel composition as well as of the amount of gel present in the concrete [27]. Time seems to have an effect on the development of the products of reaction. With extended exposure time, a larger amount of ASR gel is produced, leading to a higher expansion ([56,191]). The composition of gel evolves with time; initially the gel absorbs water without taking in $Na^+$ and $K^+$ ions [56]. In later stages, due to the evolution of the reactions, the Ca content increases and the gel becomes more viscous and expansive than the original alkali-rich gel. Kawamura et al. [56] also observed that higher temperature results in a greater amount of $Ca^{2+}$ in the gel, assumed to be a pre-requisite for the formation of expansive gel ([57,59,65]). However, there is no general agreement in the literature regarding the influence of the Ca content in the ASR gel on its expansion ([65,192]).

Additionally, the Ca content is reported to be a function of the place where gel occurs inside the concrete; Knudsen and Thaulow [52], Kawamura et al. [56] and Fernandes [64]. Among others, Kawamura and Iwahori [193] and Bokern [166] have shown that an increase in the alkali content in ASR gel (i.e. high $Na_2O/SiO_2$ ratio) decreases the viscosity. Thus, the viscosity of alkali-rich gels may be so low that they cannot produce sufficiently high expansive pressure to crack mortar or concrete specimens, but lead to exudation of ASR gel on the surface of the exposed test samples. This exudation limits the gel content inside the specimens and probably results in reduced expansion. Struble and Diamond [194] measured swelling properties of alkali–silica gels of various $Na_2O/SiO_2$ ratios. Under "free-swelling conditions" they recorded swelling ranging from 0.1 MPa to almost 11 MPa. Gels with $Na_2O/SiO_2$ ratio of 0.33 and less exhibited the lowest swelling pressures.

In field concretes, the alkali content is often limited, while calcium is continuously brought into the pore solution due to portlandite dissolution. Additionally, in laboratory test specimens, the high level of sodium content (resulting from the NaOH enrichment of the mixing water or storage in alkaline solution) leads to increasingly sodium-rich gel with lower viscosity. Furthermore, Bokern [166] showed that the addition of SCMs may hinder deleterious ASR in laboratory tests, but not always in the field, partly because the viscosity of the gel decreases at elevated temperature and at a higher RH level. Thus, in the field, ASR-related expansion can be more intense, but slower.

Regarding properties of ASR gel, it was found that products obtained in laboratory tests are similar to those identified in field concretes (amorphous and crystalline) ([195,196,197]). If so, this fact is important as it means that temperature is probably not as important to the morphology of the alkali–silica gel as it is for the reactivity, rate and amount of gel produced [197]. Davies and Oberholster [198] compared alkali–silica products formed in field concretes with those formed during the 80 °C NBRI test [199]. They showed that, compared to the field, the NBRI test does not modify the naturally occurring process. Nevertheless, during the test they noticed fluid gels exuding into the NaOH solution, in the form of thin filaments. SEM examinations of gel conducted at the completion of such accelerated laboratory tests have revealed that morphologies were very similar to those found in field concretes [63] irrespective of whether NaOH or KOH solutions were used (massive gels, sponge-like texture and rosette-like phase).

By contrast, Fernandes et al. [38] observed that gel formed in mortar bar specimens showed an amorphous structure whilst the gel from old concrete structures is partly crystalline. Gavrilenko et al. [191] compared concrete cores taken from Spanish dams and mortar bars made up with the same aggregates (crushed granitic mylonite and quartzite) by scanning electron microscopy and semi-quantitative analysis of gels performed by EDX. In both cases they found large varieties of gel: compact smooth gel (amorphous), lepispheres and sheet sponge or clot morphology (crystalline/porous). However, field and laboratory reaction products had very different chemical compositions. Gels formed in mortars (alkali-boosted) were highly enriched in Na and poor in Ca, with often more silica than in the field concretes.

The alkali–silica reaction product has initially low fluidity and considerable swelling capacity in the presence of water. There is also evidence, at least under laboratory conditions, that dehydrated gel can be rehydrated and will re-expand when additional water is added to the specimen [134]. However, dried and carbonated gels are unlikely to regain their expansive properties, and they are not soluble in water [99].

Tests on specimens submerged in different salt solutions in ASR storage containers led to the conclusion that the ASR products formed differ in composition. NaOH is the more aggressive and produces a greater amount of gel, but KOH produces more crystalline gel [200]. In the literature, it is reported that the expansion increases until a certain level of alkalis is reached and then decreases for higher alkali contents, concluding that there is a "pessimum" effect of external alkalis (probably related to available silica) [201].

### 5.7.2. Delayed ettringite formation (DEF)

Delayed ettringite formation (DEF) is attributed to high temperature during early age curing [202]. At elevated temperature there is an incongruent dissolution of ettringite with much of the sulphate going into solution and being encapsulated by the rapidly forming C–S–H. During subsequent storage at ambient temperatures the sulphate is slowly released from the C–S–H and ettringite forms at later ages. Under certain conditions this delayed formation of ettringite can lead to expansion and cracking of the concrete. Expansion due to DEF has not been demonstrated for concrete cured below 65 °C, motivating temperature limits in many concrete standards. Hence, it is strongly recommended to consider this curing temperature limit for any ASR performance test.

## 6. Conclusions and recommendations

Based on the literature survey, the authors have provided recommendations for performance testing. These recommendations include precautions when testing various aggregates and binders, important factors to take into account during mix design, as well as possible influences on ASR expansion of various conditions during pre-storage and the ASR exposure.

### 6.1. Precautions dependent on type of aggregate

- When dealing with aggregates showing a possible pessimum, this must be accounted for during performance testing, e.g. by performing several concretes mixes with different percentages of reactive constituents.
- The aggregate fractions used in structures should also be used in the laboratory tests, since the aggregate size causing the highest ASR expansion is dependent on the nature and composition of the aggregate. Concrete prism tests are thus recommended instead of mortar bar tests. It should also be kept in mind that crushing some certain types of aggregates may affect their reactivity.
- Testing and assessment of alkali release from aggregates should preferably be taken into account during performance testing, since the extent of alkali release might vary with the test conditions, e.g. with the exposure temperature. However, the first and urgent step is to agree on a test method for measurement of alkali release representative of that occurring in practice and corresponding interpretation criteria.
- The ASR aggregate test methods and the corresponding interpretation criteria (maximum allowed expansion) have been developed for normal weight aggregates and are not necessarily applicable for lightweight aggregates (oven dry particle density less than 2000 kg/m$^3$) or heavy weight aggregates (oven dry particle density greater than 3500 kg/m$^3$). Also the weight increase should be taken into consideration since experiences show that the ASR gel (if developed) initially accumulates in voids in the LWAs and primarily contributes to a weight increase but only a moderate length increase. After the gel has accumulated in and partly filled the voids in the LWAs, the rate of expansion might increase

### 6.2. Precautions dependent on type of binder

- The same type of OPC cement should be used in performance testing as planned to be used in the structure. The reason is that some concrete mixtures produced with cements having similar $Na_2O_{eq}$ but different K/Na ratios have been observed to expand considerably differently in accelerated laboratory tests. However, there are contradictory findings in the literature about this issue.
- The type of cement and the type and amount of any SCMs incorporated influence the parameters related to ASR, including composition of hydration products, pore water composition and permeability. This should be taken into account when testing the concrete performance, during mix design, pre-curing and exposure, as further discussed below.
- It is recommended to test the actual binder composition in combination with the actual aggregate to be used in the structure. Even though the chemical composition of e.g. two fly ashes is quite similar, their ASR mitigation properties might differ significantly.
- If the aim is to document the ability of a commercial binder (e.g. a fly ash cement) to prevent ASR for a number of aggregate types within a region, a possible alternative approach is to test the binder in combination with an assumed "worst case reference aggregate" (e.g. as described in the Norwegian ASR regulations [74]).
- An acceptable performance test method requires an approach that accelerates the pozzolanic reaction and the ASR to the same extent, since an extensive acceleration of the pozzolanic reaction might lead to less ASR expansion in the laboratory testing (not conservative). This might be of particular interest when testing fly ash containing cements, in which the pozzolanic reaction and thus the alkali binding starts after a period of approximately 28 days when cured at 20 °C.
- Performance testing of silica fume containing concretes needs a prolonged testing time (at least two years) in order to detect a possible increased alkali level with time when the alkali–silica gel continuously reacts with calcium. Thus, at least part of the alkalis becomes available for alkali–silica reaction.

### 6.3. Important factors to take into account during mix design

- The w/cm ratio influences the concrete properties, and thus the outcome of an ASR performance test. Decreasing the w/cm ratio on the one hand might lead to increased ASR expansions (due to increased $OH^-$ concentration in the pore solution), while on the other hand may reduce the ASR expansions (due to a denser paste, and thus slower and less transport and ingress of water or solutions, and due to a higher degree of self-desiccation and thus a reduced internal RH). As a consequence, the authors suggest that the net influence of a reduced w/cm ratio should be investigated further, before executing commercial accelerated laboratory performance testing of concretes with w/cm ratio below 0.40.
- As a conservative approach, pre-saturated aggregates might be used to counteract self-desiccation to a certain extent (might be particularly effective for highly porous aggregates).
- The alkali content of the concrete is often boosted to ensure that there are sufficient alkalis present to identify reactive aggregates and to compensate for alkali leaching. Nonetheless, extensive alkali boosting is not in general recommended for performance testing because it masks the critical role of the alkali content of the job mixture. Additionally, there are several other concerns regarding alkali boosting (e.g. added alkalis might influence the behaviour of the concrete and thus affect the ASR properties). However, there are contradictory findings in the literature on the influence of some alkali boosting, thus calling for more research.
- It is recommended that the entrained air content of the performance test is the same as that intended for the job mixture. Alternatively, one might use lower air content in the laboratory test prisms, since this is a conservative approach. Whereas it is generally agreed that entrained air will not prevent or delay ASR expansion, there is some evidence that it can reduce expansion with some aggregates by accommodating ASR gel.
- In the case of using lithium to reduce the risk of ASR, it is essential that the lithium to alkalis (sodium and potassium) molar ratio, [Li]/[Na + K], in the performance test is equal to that of the job mixture being tested.

### *6.4. Influence of conditions during pre-storage and ASR exposure*

- The "pre-storage conditions" (i.e. moisture conditions during pre-storage and the length of the pre-storage period at ambient temperature) and the ASR exposure conditions (i.e. moisture conditions, type of container, prism size, wrapping (if any), storage temperature, storage period and any external alkalis added) might have significant influence on vital "ASR parameters" (i.e. prism internal humidity, composition of the concrete pore solution, aggregate reactivity, properties of hydration products formed and properties of any reaction products formed) and thus on the outcome of a performance test.
- A laboratory performance test should be designed to subject the prisms to "worst-case humidity conditions", while considering the problems related to increased alkali leaching. As a quality control measure, the mass of prisms should always be measured, evaluated and reported.
- When using a relatively low w/cm ratio, e.g. as in high performance concrete, the self-desiccation will increase and, as a consequence, the internal RH will decrease. Several parameters might increase the influence of self-desiccation (e.g. the prism size and the micro climate in the storage containers) and thus contribute to maintain a "rather low" internal RH in the concrete prisms. A good laboratory performance test should take into account these parameters to avoid a too low internal RH in the concrete prisms.
- It is essential to keep the internal prism temperature constant during all measurements, because the recorded expansion is rather sensitive to a moderately deviating prism temperature at the time of measuring compared with the temperature during the reference readings. The time of reading the prism reference length might also be of importance, in particular for concretes showing some shrinkage in the early age (e.g. when testing binders with high extent of self-desiccation).
- The extent of alkali leaching, one of the biggest challenges during accelerated ASR testing in the laboratory, should be minimised. It is important to account for alkali leaching when drawing conclusions based on a performance test.
- The rate and extent of alkali leaching are heavily influenced by the storage and exposure conditions. The following parameters are of particular importance:
    - ○ Reduced alkali leaching occurs with larger prism size, lower concrete permeability, lower exposure temperature and less moisture condensing on the prism surfaces.
    - ○ Increased alkali leaching occurs when prisms are submerged in water or exposed to extreme moisture conditions (e.g. fog chamber), prisms wrapped in wet cotton cloth or prisms subjected to drying and wetting cycles (e.g. during cooling before measuring).
- To make a performance test more practical, there is a need to find a way to accelerate the reaction, e.g. by elevating the storage temperature. However, exposing the prisms to 60 °C during ASR testing might be questionable due to several reasons:
    - ○ Lack of experience with respect to laboratory-field correlation.
    - ○ Higher rate of silica dissolution and alkali release from aggregates.
    - ○ The concentration of sulphates in the concrete pore water increases with increasing temperature, and thus the concentration of $OH^-$ is reduced correspondingly.
    - ○ The influence of the pre-curing conditions (e.g. length of curing at ambient temperature before starting the ASR test) on the prism expansion might be of higher importance, dependent on the type of binder.
    - ○ The alkali binding capacity is influenced by the exposure temperature.
    - ○ The pozzolanic reactivity of the SCM or the ggbs used might be accelerated significantly, and thus contribute to a poorer laboratory/field correlation.
- To build up experience with ASR testing at 60 °C and collect data for evaluation of the laboratory/field correlation, research laboratories are encouraged to keep on testing various concretes by use of the RILEM AAR-4.1 test method and cast concrete cubes for outdoor exposure. Also the extent of alkali leaching should be documented.
- In a performance test, exposure of the test prisms to temperatures above 60 °C should be avoided. At such high temperatures, some other deterioration mechanisms may occur, e.g. DEF.

## 7. Further research

The literature survey has identified several issues that need further research in order to develop a reliable performance test procedure. Among the most important ones are:

- Among current test procedures, a variant of a 38 °C concrete prism test seems to be the best candidate to be developed as a performance test method. However, the period of testing is a major drawback. More research is thus needed to find a reliable way of accelerating a performance test method to make it more practical.
- Testing and assessment of alkali release from aggregates is a matter of extensive dispute for their use in concrete. The task group "Releasable alkalis" in RILEM TC 219-ACS is presently developing a reliable test procedure for such measurements.
- There is a gap in knowledge internationally whether LWAs might give deleterious ASR in real structures. There are no internationally agreed requirements regarding how to test and evaluate the alkali reactivity of a LWA or a LWAC in the laboratory.
- More research is needed to investigate whether test results obtained with one type of OPC are also valid for other types of OPCs or if each OPC has to be tested separately, because there are contradictory findings in the literature about the influence of the Na/K ratio on the ASR expansion.
- More research is needed to evaluate the effect of alkali boosting with different binder types and/or OPCs with different alkali levels, because some alkali boosting might be needed to compensate for small changes in the cement alkali content.
- The net influence of a reduced w/cm ratio should be investigated further as the basis to agree on a possible lower w/cm limit for performance testing.
- It is of urgent importance to try to reduce the rate and extent of alkali leaching in future performance test methods.
- Further research is necessary to test the performance of special concrete mixtures, e.g. self compacting concrete and fibre-reinforced concrete, because the design considerations (restricted aggregate size, high filler content, inclusion of other ingredients), in addition to physical (e.g., permeability, unit weight) and mechanical (e.g., strength, toughness) properties of these special concretes might differ from ordinary concrete.

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