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Investigations on the feasibility of the CWE method to verify potential alkali release from aggregates in Norwegian field concrete

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Abstract

Concrete elements exposed for 26 years to seawater spray and deicing salts along a road close to Sandnessjøen in Norway were investigated. Concrete cores from 4 elements with different binder compositions (A: 100 % Portland cement (PC), B: 96 % PC and 4 % silica fume (SF), D: 93 % OPC and 7 % SF, and E: 77 % OPC, 20 % fly ash (FA) and 3 % SF) were analysed. Chloride ingress and alkali leaching were qualitatively assessed with µ-XRF elemental mapping. Cold Water Extraction (CWE) was performed to determine the amount of free alkali (Na and K) both on the inner part of the concrete cores which was not affected by leaching, and on the pure aggregate. The potential alkali release from the aggregates was determined using the newly developed RILEM AAR-8 method. This method showed that the used aggregate released less alkalis than the limit for being classified as a low-releaser. Small amounts of alkali (especially Na) were released from the aggregate even by applying the more "gentle" CWE method on milled powder from the aggregates. Hence, care should be taken to check that potential alkali release from aggregates during field exposure is not partly masked by alkalis released during application of CWE in the lab. As the amount of potentially bound alkali in the different hydrated binders is not known, the uncertainty in the share of alkali coming from cement free available in the pore solution complicates potential verification of alkali release further. For future investigations, reference samples with different binders combined with various types of aggregates (non-reactive & non releasing, reactive & non-releasing, high releaser, ...) are planned to better estimate the share of alkali from the binder available in the pore solution as well as the alkali released from aggregates.

Keywords: RILEM AAR-8, aggregate, alkali release, CWE, µ-XRF

1. INTRODUCTION

Alkali-silica reaction (ASR) is one of the most important durability issues in Norway, leading to expansion and extensive cracking of concrete structures. To occur, three essential conditions are needed, i.e. the aggregate has to be reactive usually due to the presence of unstable siliceous phases, the alkali concentration in the concrete pore solution needs to be high, and the concrete structure should be exposed to a high moisture load. Soluble alkalis from Portland cement (PC) account for the highest contribution of alkalis to the concrete's pore solution. Consequently, low alkali cements or addition of supplementary cementitious materials (SCMs) are often used to prevent ASR. However, besides cement, it is also shown that aggregates [1, 2], especially dissolution of certain types of minerals (feldspars in particular) in aggregates [3] can contribute with alkalis to the concrete's pore solution.

On the other hand, alkalis can be bound to a certain extent in the C-S-H gel, and the amount of bound alkalis is shown to increase with decreasing Ca/Si ratio. The Ca/Si ratio of C-S-H can be decreased as result of the addition of pozzolanic SCMs [4]. Additionally, in long-term field exposure alkali ions might also exit the concrete by leaching. Current Norwegian regulations to prevent ASR focus on the alkali content of the concrete and the type of aggregate used, characterised as reactive or non-reactive, normally by a petrographic analysis. Neither the potential alkali release from aggregates and the potential increased alkali uptake in hydrate phases of novel blended cements, nor the potential leaching from the concrete is considered so far in the Norwegian ASR regulations. Especially, the real behaviour

of aggregates with respect to alkali release in field exposure, and consequently the potential contribution of alkalis to the concrete's pore solution, is not yet known and needs further investigations.

2. EXPERIMENTAL

In 1993-1994, the Norwegian Public Roads Administration (NPRA) established a field station in Sandnessjøen for testing of 17 different concrete compositions. One element of each of the compositions was placed along fv. 17 (County Road 17) on the way from Sandnessjøen centre to the Helgeland bridge. One side of the elements was exposed to seawater splash, while the other side was exposed to de-icing salts. In addition, 17 concrete beam elements were placed at a ferry quay to marine tidal exposure [5].

2.1 Materials

After 26 years of exposure, concrete cores of 100 mm diameter were drilled from elements placed along the country road and examined from the side exposed to sea splash. Cores were taken from concretes with 4 different binder compositions including Portland cement (PC), silica fume (SF) and fly ash (FA), i.e. A) 100 % PC, B) 96 % PC + 4% SF, C) 93 % PC + 7 % SF and D) 77 % PC + 3 % SF + 20 % FA (wt% of binder). The concrete composition and the chemical composition of the binders are given in Table 2.1 and Table 2.2.

The fine and coarse aggregate was from glacio-fluvial origin composed of the rock types granite and gneiss collected from Årdal, Norway. The main minerals are plagioclase, K-feldspar, quartz and mica, also including some chlorite, epidote and amphibole [6]. The alkali content in the different binders is very similar in Na₂O eq. (wt% binder) except for concrete D. Though the alkali content in the concrete will differ due to variations in binder content. The lower alkali content in the binder of concrete D is due to the use of low alkali (LA) sulphate resistant (SR) Portland cement.

Material	A	В	D	E
(kg/m³)	(100PC)	(96PC/4SF)	(93PC/7SF)	(77PC/3SF/20FA)
Portland Cement (CEM I) (PC)	326	373	379	307
Silica fume (SF)	0	15.5	30	13.4
Fly Ash (FA)	0	0	0	78
Water (w)	143	123	137	126
Fine aggregate 0/8 mm	995	928	970	1014
Coarse aggregate 8/16 mm	847	904	832	820
Moisture sand 0-8 (m _s)	31	29	31	31
Moisture gravel 8-16 (mg)	4	4	3	3
Air entrainer	0	0.34	0.47	0.59
Superplasticizer	2.56	6.93	4.36	7.94
Paste volume (%)	28.33	28.60	30.75	29.45
Equivalent w/b ¹	0.55	0.39	0.39	0.39
Calculated density ²	2314	2351	2353	2367
Na ₂ O eq	0.15	0.16	0.08	0.16

Table 2.1: Concrete composition of the concretes A, B, D and E [5]

¹ equivalent w/b calculated as (w+m_s+m_g)/(PC+FA+2*SF)

² The density was calculated by summing up the mass for the cement, silica fume, water, sand, gravel and admixtures.

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Oxide (%)	А	В	D	E	Aggregate
	(100PC)	(96PC/4SF)	(93PC/7SF)	(77PC/3SF/20FA)	
CaO	63.2	60.7	60.2	49.4	5.6
SiO ₂	21.5	24.4	26.7	30.5	61.7
Al ₂ O ₃	4.6	4.5	3.4	9.1	14.7
Fe ₂ O ₃	3.5	3.4	4.6	3.5	6.6
MgO	2.1	2.0	1.6	1.8	3.4
SO ₃	3.0	2.9	2.0	2.3	-
K ₂ O	1.0	1.0	0.4	1.0	1.7
Na ₂ O	0.4	0.3	0.2	0.3	3.7
Na ₂ O eq. (wt%/binder)	1.1	1.0	0.5	1.0	4.8

Table 2.2: Chemical composition of the binders in the concretes A, B, D and E, and the used aggregate

2.2 Methods

First, the concrete cores were cut longitudinally into 2 halves with the same orientation. The upper half core (facing towards the top of the concrete element exposed in the field) was used for elemental mapping with micro-X-ray fluorescence (μ -XRF) and the other half was used for cold water extraction (CWE) and subsequent analysis of sodium (Na) and potassium (K) concentration in the solution and pH of the solution [7, 8]. All cores were cut wet, trying to minimise the amount of water. After cutting, the cores were surface dried with a wet cloth, packed in plastic and stored at 5 °C until further analysis.

For μ -XRF investigations no further treatment of the concrete cores was necessary. The concrete cores were aligned with the flat surface oriented upwards in the μ -XRF instrument. Analyses were done with a Bruker Tornado M4 instrument equipped with silver (Ag) X-ray source and two silicon drift detectors for fast acquisition. All measurements were performed under vacuum (20 mbar) and an X-Ray beam focused to 20 μ m spot size. The instrument parameters for elemental mappings were 600 μ A and 50 kV. Large qualitative overview mappings were collected with a 50 μ m distance between each pixel and 2 ms/pixel acquisition time. Besides that, the concrete cores were analysed semi-quantitatively by mapping the concrete cores in adjacent areas of 80 mm width and 10 mm depth from the surface towards the bulk of the cores. These mappings were collected with 25 μ m distance between each pixel and 10 ms/pixel acquisition time for better signal statistics. Applying a phase distinguishing tool in the software, cement paste, and aggregate was differentiated as two different phases and the spectra from the cement paste were analysed separately to evaluate the amount of chloride and alkalis in the paste only. The same procedure as described in [9] was used.

Concrete as well as pure aggregate were used for pore water extraction with cold water extraction (CWE). CWE was performed on the part of the concrete core unaffected by external environment. Hence, a 50 mm part of the half concrete core was cut from the centre which was unaffected by leaching or chloride ingress as proven with μ -XRF mapping. Materials used for CWE were milled down until passing an 80 μ m sieve. The procedure of extracting pore water from the samples is described in detail in [7, 8]. About 20 g of sample and 20 g of water was used for CWE of the samples from concrete cores and the aggregate. The pore water extracted by CWE was analysed for Na and K by flame spectroscopy.

The potential release of alkalis from the Årdal aggregate was also measured applying the RILEM AAR-8 method [10]. The grading is "as is" for a fine (0/4 mm) aggregate, while a coarse aggregate must be crushed and sieved for achieving a specified 0/4 mm grading curve. The principle of testing is extraction of any releasable alkalis from the aggregate using 0.7 M alkali solutions, sodium hydroxide for extracting potassium and potassium hydroxide for extracting sodium. The alkali solutions are saturated with calcium hydroxide. At specific time intervals (2, 6, 13 and 26 weeks) a small sample is collected from each of the solutions and analysed for any alkalis. As described in the guidance document RILEM AAR- Investigations on the feasibility of the CWE method to verify potential alkali release from aggregates in Norwegian field concrete Tobias Danner, Klaartje De Weerdt, Bård Pedersen, Jan Lindgård

0 [11] it is, however, still considered pre-mature to provide guidance on the interpretation and application of AAR-8 findings. The main reason for this is lack of validation of how the outcome of the test may be applied to predict alkali release from the aggregates under field conditions. This verification project on the samples from the Sandnessjøen field station will hopefully together with other investigations contribute to future guidance for interpretation of AAR-8 results.

3. RESULTS AND DICUSSION

3.1 Micro X-Ray Fluorescence (µ-XRF)

The μ -XRF chloride maps are shown in Figure 3.1. The chloride ingress depth was decreasing from concrete A to B to D to E (A > B > D > E) with increasing amount of SCM used in the binder. It should be noted that the pure PC concrete, concrete A, also had a higher w/b ratio (0.55) compared to the other three binders (0.40). The ranking is typically a result of pore refinement in the concrete and hence higher resistance to chloride ingress. A comparison of μ -XRF chloride maps and quantitative chloride profiles of cores from element B and E showed that the chloride ingress depth corresponds well and that the transition to dark blue/black colour in the maps relates to a concentration of ≤ 0.01 % Cl⁻ by weight of concrete. This shows that chloride ingress depth in concrete A was much deeper compared to the other and that concentrations > 0.01 % Cl⁻ by weight of concrete can be found in concrete A even at a depth of 100 mm.



Figure 3.1: µ-XRF chloride ingress map of cores from concrete A, B, D and E over an area of 100 x 100 mm from the surface to the left (sea-splash exposure) towards the bulk of the cores. Intensities expressed as counts per second (cps) are normalized for each picture separately.

In all concrete cores, leaching of alkali metals in the cement paste was observed towards the exposed surface. The results presented in Figure 3.2 are only semi-quantitative with the current measurement conditions, i.e. no reference samples were used for calibration. The results showed leaching of Na within the first 15 mm before the amounts appeared to stabilize to an approximate constant level. For K, the

largest decrease in concentration seemed also to be within the first 15 mm towards the surface. After that, the slope was decreasing but did not appear to flatten out completely until 50 mm from the surface.

The low amount of K in the binder of concrete D (Table 2.2) agrees with measured lower intensities in concrete D by μ -XRF (see Figure 3.2). Concrete A, B and C, on the other hand, could have been expected to be more similar in K content based on their composition, though K levels determined by μ -XRF are considerably different. In the case of Na, all binders seem to have a similar content (Table 2.2), with concrete A having the highest Na content. This is also reflected in the Na content measured with μ -XRF. The density and thus porosity of materials amongst many other factors play an important role for the total output of measured intensities and calculated contents using μ XRF. Further research is needed to explain the absolute differences between the investigated concretes.



Figure 3.2: µ-XRF normalized mass % of K (left) and Na (right) from the sea-splash exposed surface towards the bulk of the core.

3.2 Cold Water Extraction (CWE)

Table 3.1 shows the measured Na and K content and Na₂O_{eq} determined with CWE expressed as weight % of concrete or aggregate. For the concretes about double as much K was measured in the pore solution compared to Na which agrees with the ratio of K/Na in the binders. As expected, the lowest release was measured in concrete D with the low alkali cement. However, the difference between free alkali from concrete D and concrete A was only minor. On the other hand, CWE performed on mortar indicated a standard deviation of approximately 10% determined on triplet samples [8]. There is no apparent correlation between the cement content in the concrete nor the amount of SCM in the binder, and the amount of free alkalis measured by CWE.

(3				
Sample		Nacwe, concrete	K _{CWE} , concrete	Na ₂ O eq CWE, concrete
			wt% of materia	al
	A (100PC)	0.042 (0.038)	0.072 (0.071)	0.113 (0.107)
Concrete	B (96PC/4SF)	0.051 (0.047)	0.100 (0.099)	0.148 (0.142)
	D (93PC/7SF)	0.037 (0.033)	0.067 (0.065)	0.103 (0.097)
	E (77PC/3SF/20FA)	0.047 (0.043)	0.094 (0.093)	0.138 (0.132)
Aggregate	Årdal	0.0048	0.0015	0.008

Table 3.1: Content of free Na and K measured by CWE in wt% of concrete or aggregate, respectively (green numbers: corrected for potential contribution of aggregates during CWE)

The amount of concrete powder used for the CWE analysis was equal for all samples, but there is no information on the potential variation of paste vs. aggregate volume in the samples tested. The higher

release of Na compared to K from the pure aggregate is in line with the chemical composition of the aggregate (Table 2.2).

During CWE of concrete, the aggregates present in the fine ground concrete might also contribute with alkali. To determine the potential contribution of the aggregates during CWE, the Na and K content in wt.% of aggregate (Table 3.1) was multiplied with the wt.% of aggregate in the concrete (see Table 2.1). The results are shown in Table 3.2. The contents measured by CWE (Table 3.1) can then be corrected by subtracting the potential contribution of aggregates for each concrete (green numbers in Table 3.1).

The theoretical maximum contribution of alkalis from the binder (Table 3.3) in the concrete can be calculated based on the binder content (CEM I + FA + SF) in the concrete (Table 2.1) multiplied with the alkali content in the Portland cement. For this, the Na₂O and K₂O content given in Table 2.2 was first recalculated to Na and K content.

Table 3.2: Potential contribution from aggregates to measured content of Na and K in concrete (Table

3.1)

Concrete	wt% aggregates in concrete	Na _{CWE} , aggregate	K _{CWE} , aggregate	Na₂O eq.
		wt% of concrete		
A (100PC)	80	0.0038	0.0012	0.006
B (96PC/4SF)	78	0.0037	0.0012	0.006
D (93PC/7SF)	77	0.0037	0.0012	0.006
E (77PC/3SF/20FA)	77	0.0037	0.0012	0.006

Table 3.3: Potential maximum contribution from binder to measured content of Na and K in concrete
(Table 3.1)

Concrete	wt% of binder (CEM I+FA+SF) in concrete	Na (binder)	K (binder)	Na₂O eq.
		wt% of concrete		
A (100PC)	14	0.041	0.117	0.149
B (96PC/4SF)	17	0.036	0.137	0.157
D (93PC/7SF)	17	0.025	0.063	0.084
E (77PC/3SF/20FA)	17	0.037	0.143	0.162

Figure 3.3 shows the CWE results obtained on concrete, corrected for the alkali released by the aggregates during CWE, compared to the theoretical maximum amount of alkali coming from the binder. For Na, the free Na measured with CWE is, except for concrete A, slightly higher than the theoretical amount of Na coming from binder. This might indicate some contribution of Na from aggregates during field exposure in concrete B, D and E. In case of K, the theoretical amount of K coming from the binder is, except for concrete D, higher than the free K content measured with CWE. This might indicate some contribution of K from aggregate during field exposure in concrete D.

Regarding the impact of the binder on the measured Na and K content with CWE, one would expect that the addition of supplementary cementitious materials (SCM) such as silica fume and fly ash would lower the amount of the available alkali in the pore solution [4]. The reason for this is that SCMs can lead to the formation of calcium silicate hydrate (C-S-H) with a lower Ca/Si ratio compared to plain Portland cement (PC). Lowering of the Ca/Si ratio of C-S-H leads to increased adsorption of alkali metals. Thus, one would expect the highest alkali content for concrete A (pure PC), followed by concrete B (4% SF), and concrete E (20% FA + 3% SF) which are all based on the same type of CEM I (P30). However, as mentioned earlier for the investigated concretes there is no clear correlation between the amount nor type of SCMs added and the Na and K content determined by CWE. In case of concrete D, it has to be taken into account that the CEM I used was a low-alkali cement compared to cement used in the other concretes.



Figure 3.3: Alkalis measured with CWE on concrete and corrected for the potential contribution of aggregates during CWE, and the theoretical maximum amount of alkalis in the binder.

K is not released considerably by the aggregate during CWE (see Table 3.1). Potential K release during CWE will therefore most likely not considerably conceal any release of K in the field in the concrete. The amount of Na released during CWE of the aggregates was higher, i.e about 10% of the measured Na in concrete (see Table 3.1). When analysing concrete powder with CWE, we cannot distinguish between Na released in the pore solution in the field and the Na released during the CWE analysis. Thus, Na release during CWE might mask Na released earlier in the field which makes the results not conclusive regarding potential release of Na from the present aggregates in field. Consequently, it is also important to not only look at the Na₂O_{eq}, but to consider the release of Na and K separately.

3.3 RILEM AAR-8 test

In connection to the second Round Robin Test performed within the RILEM TC 258-AAA, the Årdal (Granite and gneiss) aggregate was tested according to the new RILEM AAR-8 procedure [12]. The Årdal aggregate released 0.025 wt% Na₂O (=0.018 wt.% Na) and 0.022 wt% K₂O (=0.018 wt.% K), equivalent to 0.039 wt.% Na₂O_{eq}, after 26 weeks of exposure [13]. This corresponds to about 0.030 % Na₂O_{eq} by weight of concrete in the different samples. Higher amounts of K and Na were released during AAR-8 compared to CWE (Table 6). Even though a finely ground powder (<80 µm) is used during CWE compared to particles <4mm in case of AAR-8, this is expected due to the treatment with alkaline solution compared to water used during CWE. Moreover, the exposure time is also very different, only minutes for the CWE method and 26 weeks for RILEM AAR-8.

Method	Na	К	Na ₂ O eq.	
	wt % of aggregate			
CWE	0.005	0.002	0.008	
AAR-8	0.018	0.018	0.039	

Table 3.4: Comparison of alkali release from aggregate determined with CWE and RILEM AAR-8

When comparing the AAR-8 results for the Årdal aggregate (0.039 wt.% Na₂Oeq) with the anonymized AAR-8 results from the Norwegian "ARA-2020" project shown in Figure 3.4, Årdal is amongst the lowest releasers of the tested aggregates. In fact, Årdal released less alkalis than the limit given in RILEM AAR-8 for being classified as a low-releaser.

The verification of the alkali release is not straight forward in the case of a low releasing aggregate as the potential release by the aggregate is rather low compared to the alkali coming from the cement. For the A concrete the amount of alkali potentially released by the aggregates is $0.039 \text{ wt.}\% \text{ Na}_2\text{O}_{eq} \text{ x}$ (995 kg sand/m³ concrete + 847 kg gravel/m³ concrete) = $0.73 \text{ kg Na}_2\text{O}_{eq}/\text{m}^3$ concrete, whereas the amount

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of alkali coming from the cement is 1.06 wt.% $Na_2O_{eq} \times 326$ kg cement/m³ concrete = 3.45 kg Na_2O_{eq}/m^3 concrete.



Figure 3.4: Anonymized RILEM AAR-8 results of different types of aggregate from the Norwegian "ARA-2020" project.

Figure 3.5 compares the free Na and K content determined by CWE on concrete corrected for the potential contribution from aggregate during CWE, with the theoretical Na and K content from the binder in addition to the potential contribution of Na and K from the aggregates based on AAR-8.



Figure 3.5: Comparison of the free Na and K content determined by CWE on concrete corrected for the potential contribution from aggregate during CWE (CWE_concrete- aggregate), the theoretical Na and K content from the binder, and the potential contribution of Na and K from the aggregates based on AAR-8.

The sum of the total alkali from the binder (based on chemical composition) and the aggregates (alkalis released during AAR-8) can be regarded as the maximum potential free alkali contribution in the concrete system. The actual free alkali present in the concrete was determined using CWE and corrected for the alkali released by the aggregates during CWE. It can be seen that the free alkali content determined with CWE (Na and K), corrected for the release from aggregates, is consistently lower than the sum of the potential alkali source i.e. binder and AAR-8-aggregate (Figure 3.5). However, in case of Na, considering concrete B, D and E, the free Na content determined with CWE and corrected for the sum of the potential Na sources. Verification of Na release from aggregates might be difficult as parts of the potential alkali release in field can be masked by alkali

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release during CWE, as mentioned before. This seems to be less relevant in the case for K. For K, the potential release of alkali as determined by AAR-8 is (except for concrete D) rather low compared to the contribution from the Portland cement which might also complicate verification.

The uncertainty in the system is the share of alkali coming from the binder which is actually available as free alkali, as part will be taken up by hydrates such as C-S-H, or even by alkali silica gel (in the case of ASR reactive aggregates, but this was not the case for the Årdal aggregate), and part will still be locked into unhydrated cement grains (or unreacted fly ash). This is illustrated in Figure 3.6. If we would assume that 40% of the Na₂O_{eq} coming from the binder is actually available, we can see from Figure 3.6 that the CWE is much higher than the available alkali which is not feasible. If we on the other hand assume that 80% of Na₂O_{eq} originating from the binder is available, the corrected CWE results from the concrete are consistently lower (except concrete D) than the sum of the alkali coming from the binder and the aggregates can be nothing or "everything". This is why reference samples with non-releasing aggregates, e.g. pure limestone are needed to indicate the share of alkali coming from the Portland cement or binder available in the pore solution.



Figure 3.6: Comparison of the alkalis (as Na₂O_{eq}) determined by CWE on concrete corrected for the release by aggregates during CWE (CWE_conc-aggr), a share of 40%, 80% or 100% of the theoretical alkali content from the binder combined with the potential contribution of alkali from the aggregates based on AAR-8.

4. CONCLUSIONS

Concrete elements exposed for 26 years to marine splash water along a road close to Sandnessjøen, Norway were investigated. Concrete cores from 4 elements with different binder compositions (A: 100% OPC, B: 96% OPC and 4% SF, D: 93% OPC and 7 % SF, and E: 77 % OPC, 20 % FA and 3 % SF) were analysed. Chloride ingress and alkali leaching were qualitatively assessed with μ -XRF elemental mapping. Cold water extraction (CWE) was performed on concrete samples and the pure aggregate to calculate potential alkali release from the aggregates in the field.

The main aim of the study was to investigate if CWE and the newly developed AAR-8 method can be applied to verify potential alkali release from aggregates in field exposed concrete samples.

Chloride ingress was decreasing with increasing amount of supplementary cementitious material (SCM). All samples showed alkali leaching in the outer 15 mm compared to the bulk concrete.

During CWE analysis of pure aggregate used in the concrete samples a small amount of Na was released while the release of K was negligible. The Na released during CWE of the pure aggregate might mask parts of the potential release of Na from the aggregates in the concrete core. This shows

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the importance of a separate CWE analysis of the pure aggregate to correct the values obtained on the concrete sample.

The RILEM AAR-8 method showed that the Årdal aggregate released less alkalis than the limit for being classified as a low-releaser. In contrast to the CWE method, the released Na and K from the aggregate during AAR-8 was equal. The maximum alkali contribution from the aggregate as determined by AAR-8 was about 18-35 % of the maximum amount of alkali coming from the binder. Both the dominance of the alkali coming from the binder and the uncertainty in the share of alkali coming from binder being free available in the pore solution complicates potential verification of alkali release.

Overall, the results showed that without reference or calibration samples with inert aggregates (e.g. limestone) it is difficult to estimate the share of alkali release from the binder and thus the potential release from aggregate in field samples. This is for sure the case for low releasing aggregates like the Årdal aggregate investigated.

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