

Factors affecting the alkali release from concrete aggregates

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Abstract

An alkali-silica reactive aggregate (ASR) can be used in concrete mixtures, provided that deleterious ASR is adequately mitigated. The main methods used to mitigate deleterious ASR expansion are either by reducing the alkali-loading from the cement, by use of sufficient levels of cement replacement by appropriate supplementary cementitious materials (SCMs) or doing both, while also preventing the ingress of external alkalis. However, certain aggregates can also contain alkalis that may be released into the pore solution over time thus raising the alkali loading above the ASR threshold and leading to inadequate ASR mitigation. This issue has been studied by others but in this study, it was found that a non-reactive coarse aggregate also released alkalis. This could potentially lead to inadequate ASR mitigation if a concrete mixture made with this coarse aggregate also included a reactive fine aggregate and the ASR threshold alkali loading was exceeded. Two different methods were used to determine the level of alkali release and the effects of varying temperature, storage solution concentration, time, and solution composition were also evaluated.

Keywords: alkali release; aggregate; test methods

1. INTRODUCTION

In some cases, structures built with alkali-silica reactive (ASR) aggregates, but mitigated using low-alkali loadings or by use of supplementary cementitious materials (SCMs), the level of mitigation thought to be sufficient, has been found to be inadequate. While there are several possible reasons for this, one of them is due to increased alkali loading due to release of alkalis from certain alkali-bearing aggregates.

The contribution of alkalis from some aggregates was acknowledged as early as in 1940 by Stanton and has been touched on as a subject by many others since then [1-3]. Alkalis released from aggregates into the pore solution of concrete may be the reason why concretes with low alkali loading and/or made from low-alkali cement still crack in the field [4]. Tests of the water-soluble alkali content of concrete from mass concrete structures have shown much higher alkali contents than expected from the cement used [4], suggesting that some aggregates do release significant amount of alkalis into the pore solution of concrete [5]. This is of concern when low-alkali cement is used as a precaution for ASR and as they mention, can still cause a problem in the field [5].

Constantiner and Diamond [5] tested the pore solution from mortars with extremely low-alkali cements. The mortars were made with feldspar aggregate passing the 36 μ m sieve, an aggregate to cement ratio of 1.0 and water/cement ratio of 0.65 [5]. They showed that three different feldspars all release alkalis to the pore solution when compared to the pore solution of a control mortar made with 100% inert limestone.

The aggregates most likely to release alkalis are feldspars and micas [4-6]. Even glass, such as used in mason jars for sample storage, can release alkalis into the system when in connection with the alkaline environment of concrete or mortar [2]. The amount of alkalis released and the rate of the release are affected by the type of minerals in the aggregates as well as the extent of metamorphic alteration, and aggregate particle size [6].

While various tests have been studied, no test method has been standardized for estimating the extent of alkali increase from an aggregate into the concrete mixture over its service life. The test conditions selected when testing the release of alkalis from the aggregate greatly influence the amount of alkalis released and the rate of release [4]. The main influencing factor in testing the release of alkalis from aggregates is the ratio of solution to aggregates [7], which in concrete is lower than 0.1 but much higher in any of the proposed test methods. It was found that an increase in this ratio by 4 times may increase the alkali released by 40 times [7]. Therefore, the higher ratio used in such tests may overestimate the alkalis released in field concrete where there will be a limited volume of pore solution [7]. Usually, in

alkali-release tests, the storage containers are agitated, which is different from the static environment in concrete. Testing the aggregate in alkali hydroxide solutions showed that even though the rate of alkali release diminished, the aggregates were still releasing alkalis after 1.5 years in alkali solution.

Due to their higher surface area, finer aggregates release more into solution than coarser graded aggregates [6]. The more the aggregate is crushed, the higher quantity of alkalis is released. The authors suggested that particles finer than 1.0 mm might overestimate how much alkali the aggregate will release into the pore solution.

Solution combination and the pH level of the test solution will also affect the release of alkalis greatly [7]. The pH level of a saturated calcium hydroxide solution is around 12.4, which is lower than most concrete pore solutions. Testing in these solutions may therefore underestimate the amount of release from the aggregate [5]. More alkalis are released into supersaturated lime solutions than in distilled water [6]. Even higher amounts are released into alkali hydroxide solutions than in lime solution [4]. Alumina affects the alkali release from the aggregates by forming C₄A-hydrates or hydrogarnet, depending on temperature, leaving the alkalis in solution and available for other reactions [8]. An increase in temperature will also increase the release of alkalis [6].

Similar to the approach used for expressing alkali contents of cement, aggregate alkali contents are expressed as %Na₂O_{eq} by mass of total alkali in aggregate [6]. Berube et al. [9] estimated that Spratt could potentially release 0.22 kg/m³ if used as coarse aggregate and Sudbury 0.51 kg/m³. These evaluations were done using the hot-water extraction method. In another study by the same authors, the estimated release from Sudbury gravel could be >3.08 kg/m³ when using an aggregate content of 1850 kg/m³ and Spratt would be ~0.16 kg/m³ [4]. In comparison, the possible alkali increase from aggregates in the Three-Gorges dam was estimated to be up to 0.98 kg/m³ [6].

The expansion in commonly used ASR test methods, such as the ASTM C1293 concrete prism test, are influenced by variables including alkali leaching from the prisms, and the alkalis contributed from other sources than the cement. The release of alkalis from aggregates with time can affect performance of concrete in the field, but does that have any effect on the results over the 12- or 24-month period of the C1293 test?

2. EXPERIMENTAL

2.1 Materials

2.1.1 Aggregates

Three coarse aggregates, two of them ASR reactive, were tested. Use of well documented aggregates in this study was essential to detect if modified changes to the test methods worked. The two reactive aggregates, Spratt and Sudbury, chosen for this study were supplied by the Ministry of Transportation Ontario (MTO) and have been used in ASR testing for many years. Their level of reactivity and level of mitigation needed is therefore well documented. Table 2.1 shows the specific gravity, water absorption and dry rodded density of the aggregates used in this study. Table 2.2 shows the alkali contents of these aggregates.

Sudbury aggregate: The Sudbury gravel, contains reactive greywacke and argillite from Sudbury, Ontario, Canada [10]. This gravel is considered a slowly reactive aggregate and will cause deterioration in the field if used in combination with 100% high-alkali cements (~1.0%). An aggregate of similar composition has shown satisfactory results in the Lady Evelyn Lake dam, west of Latchford, ON, when used with low-alkali cement (< 0.6%) and low cement content after 25 years in service (now 45 years) [11]. It is estimated from different studies that, to mitigate deleterious ASR, the alkali loading must either be less than 3.3 kg/m³ [11] or that the cement used should have an alkali content of less than 0.75-0.79% (equivalent to an alkali loading of 3.1-3.5 kg/m³) when the safe alkali level was evaluated with the AMBT and the CPT respectively [12]. When using high-alkali cement in combination with SCMs, 15% of low-calcium, low-alkali fly ash should be sufficient to mitigate the expansion from this aggregate [12]. A mineralogically similar crushed coarse aggregate used in the nearby Lower Notch dam in Ontario with high-alkali cement (1.08% Na₂O_{eq}) and 20% fly ash had not shown cracking after 40 years in service (now 50 years) [13]. Lab CPT tests indicate that slag replacements of 35% should also be sufficient to mitigate the expansion [14].

Spratt aggregate: The Spratt (Spratt #3 in this case) aggregate is a highly reactive siliceous limestone [15], from near Ottawa, Ontario. Spratt is a crushed limestone with the reactive silica consisting of about

5% chalcedonic chert [16]. It is one of the most reactive aggregates in Canada and can show deleterious expansion even when used with low-alkali cement, i.e. the recommended maximum alkali loadings for concrete are much lower than loadings recommended for the Sudbury gravel. It has limited field experience in structures, but did cause expansion and cracking in concrete curbs and gutters after 9 years in service in Canada. Since then Spratt aggregate has been used in multiple research projects and therefore the behaviour of this aggregate is well known. The average expansion in the accelerated mortar bar test (AMBT) is 0.339%, well above the 0.10% limit in ASTM standards [16] and predicting the safe alkali loading has been a little troublesome. Using the AMBT and the concrete prism test (CPT), Thomas et al. [12] predicted a safe alkali level for the cement to be 0.65-0.69% respectively. That would convert to an alkali loading of 2.73kg/m³ assuming 420 kg/m³ of cement as required in the CPT. It has, however, been shown that the Spratt aggregate can cause expansion and cracking in field samples with alkali loadings as low as 1.9 kg/m³ [17]. Previously it had been suggested that alkali content below 4.0 kg/m³ would be low enough to prevent deleterious expansion from the aggregate [18] but later the safe alkali loading was lowered to 3.0 kg/m³ [11]. In combination with high-alkali cement, a fly ash replacement of 20% or a 30% slag replacement should be sufficient for mitigation [19, 20]. However, when tested according to the ASTM C1567, 35% slag replacement was not sufficient to mitigate the expansion of this aggregate [14].

Non-reactive aggregate: The non-reactive coarse aggregate was 19 mm crushed dolomitic limestone from Dufferin's Milton quarry near Toronto. This aggregate has been shown to be durable, when used in concrete, for many decades.

Table 2.1: Specific gravity, water absorption and dry rodded density (DRD) of aggregates

Aggregate	Specific Gravity (kg/m ³)	Water Absorption (%)	Dry Rodded Density (kg/m ³)
Spratt	2715	0.55	1570
Sudbury	2742	0.37	1660
Milton 19 mm 2014	2720	1.85	1620
Milton 7 mm 2014	2720	1.85	–

Table 2.2: Alkali and SiO₂ Contents of Aggregates

Aggregate	total Na ₂ O %	total K ₂ O %	Na ₂ Oeq. %	Reference/Data Source	SiO ₂ %
Spratt	0.03	0.1	0.096	Drolet et al. 2017 [21]	8.37
Sudbury	3.01	2.03	4.35	Berube et al. 2002 [4]	– *
Milton 19 mm 2014	0.11	0.15	0.21	From CBM Concrete	1.36
Milton 7 mm 2014	0.18	0.21	0.32	Data from CRH	0.65

* No data available

2.1.2 Chemicals

The alkali solutions were made dissolving reagent grade KOH, NaOH and Ca(OH)₂ in de-ionized water.

2.2 Test Methods

While various test methods have been proposed, there is no published standardized method for measuring the amount of alkalis that aggregates may release into concrete. Two methods have been proposed and these were used in this study to evaluate the three different coarse aggregates.

The first method was suggested by Bérubé and Fournier [21] after their review of methods previously tried. The aggregate to be tested is dried and immersed in two different 1M hydroxide solutions in the ratio of 1 part aggregate to 4 parts of solution. A NaOH solution is used to measure the release of K⁺ and KOH solution is used to measure the release of Na⁺. The containers are stored at 60°C and agitated periodically. Samples of the solution are collected every week for the first month, and then monthly thereafter. The alkali content of a blank solution is subtracted from the alkalis measured at any given age.

The other method is currently in development by RILEM committee TC-258 AAA. The method is similar in basis to the method proposed by Bérubé and Fournier [21] but there are some significant differences in test details. The aggregates are crushed and graded into a fixed grading curve. The 1 part to 4 part ratio of aggregate to solution is the same as used in the other method but the solution concentration is 0.7M. The containers are stored at both 38°C and 60°C and manually agitated once a week. Sampling is done at 2, 6, 13, 26 and 52 weeks and the solutions are chemically analysed. The alkali release is reported in milligrams of alkali ion per liter of solution (i.e. mg K/L or mg Na/L) or in mg alkali ion per kg of aggregate.

The solution samples were analysed with ICP-AES. All samples were diluted with 5% HNO₃ before running the analysis, as the sensor requires a neutral or acidic sample for detection. Prior to each analysis, the equipment was calibrated with standardized calibration solutions that were diluted with distilled water to cover the range of ion concentration measured.

3. TEST RESULTS

The potential release of alkalis from the aggregates used was tested by both (a) the method currently under development by RILEM and (b) the method recommended by Berube et al. (2004). In addition, the effects of varying temperature, storage solution concentration and solution composition were tested. The results are summarized as follows.

Figure 3.1 shows the quantity of K⁺ ions released from the three different aggregates into 1M NaOH solution at 60°C as a function of time. It shows that the alkali release from the aggregates is highly variable, where Spratt limestone released very little alkali into the solution, but the non-reactive limestone released the highest amount. The Sudbury greywacke-argillite aggregate also released a fairly significant amount of alkalis into the solution. There is a noticeable decrease in the K⁺ ion concentration in the beginning of the test which may be due to reactions occurring between the aggregate and solution that deplete the trace amount of K⁺ present in the test solution. This difference in the amount of alkali release between the aggregates is due to geological degradation/weathering of minerals such as feldspar or mica. The Sudbury aggregate is a gravel containing greywacke and argillites that may be releasing alkalis from minerals such as feldspars or micas into solution. The other two aggregates are both limestones but these may contain different but small amounts of alkali-releasing clay or shale minerals which would explain the difference in the amount of alkali released from them (Bérubé & Fournier, 2004).

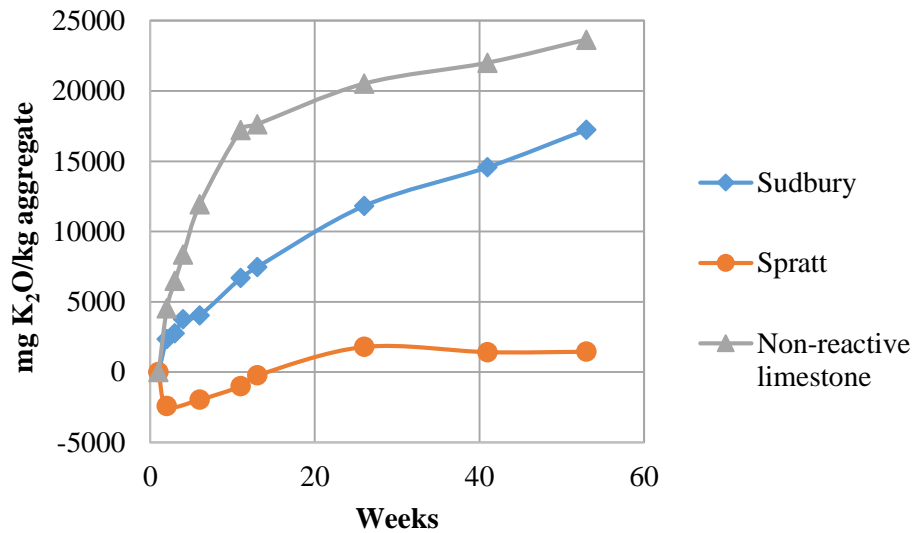


Figure 3.1: Amount of K^+ released from aggregates into 1N NaOH solutions.

Figure 3.2 shows the amount of alkali ions released from the Sudbury aggregate into four different alkaline solutions at $60^\circ C$. Four different containers were prepared for this: two with 1M NaOH solution to measure the release of K^+ and two with 1M KOH solution to measure the release of Na^+ . In two containers, one with KOH and one with NaOH, 3g of $Ca(OH)_2$ was added to provide an oversaturated solution with regard to calcium. The presence of calcium in solution is believed to aid in the release of alkalis from the aggregates and provide a more realistic environment when compared to the pore solution of concrete.

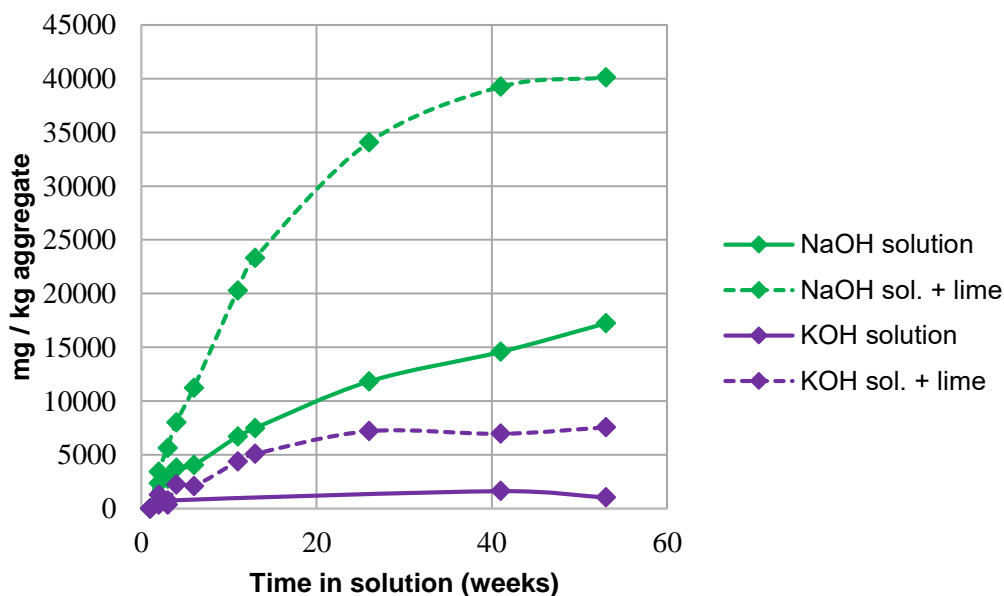


Figure 3.2: Amount of Na^+ (into KOH) and K^+ (into NaOH) ions released from Sudbury aggregate into alkaline solutions with and without $Ca(OH)_2$ addition at $60^\circ C$.

Figure 3.2 clearly shows that adding $Ca(OH)_2$ to the storage solution does increase the amount of alkalis released from the aggregate. The amount of alkalis released more than doubles in both cases. The release of Na^+ after 1 year was 1,020 mg/kg aggregate into a pure KOH solution but 7,550 mg/kg aggregate into a KOH solution with lime addition. The aggregate released 17,230 mg/kg aggregate of K^+ into a pure NaOH solution but with the addition of lime the release was 40,100 mg/kg aggregate. The

release of K^+ is much higher than Na^+ both with and without the lime addition. That is due to the chemical composition of the aggregate and which minerals become unstable in this environment.

Temperature affects the rate and amount of alkalis released from the aggregates. Higher temperatures should accelerate the reaction and shorten the test duration required. The method in development by RILEM requires aggregates to be tested at both 38°C and 60°C. Figure 3.3 shows the K^+ released from both Sudbury and Spratt aggregates immersed in 0.7M NaOH solution at both temperatures. Figure 3.4 shows the release of Na^+ from the same aggregates in 0.7M KOH solution at both temperatures. The Spratt limestone does not release a significant amount of either sodium or potassium. On the other hand, the Sudbury aggregate releases much higher amounts of both alkalis at 38°C than at 60°C. By comparison, at 60°C the Sudbury aggregate releases only a small amount more alkalis than the Spratt aggregate.

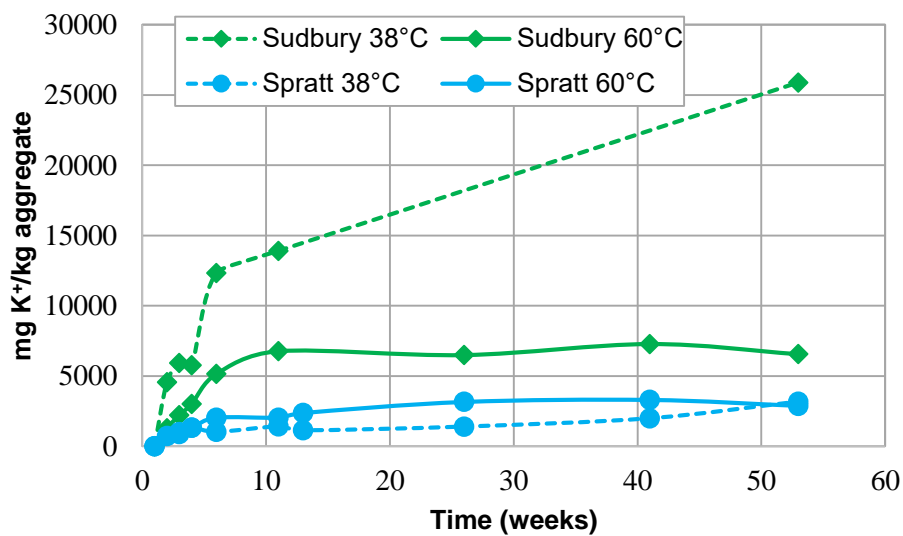


Figure 3.3: Effect of temperature on the release of K^+ from Sudbury and Spratt aggregates into 0.7M NaOH solutions.

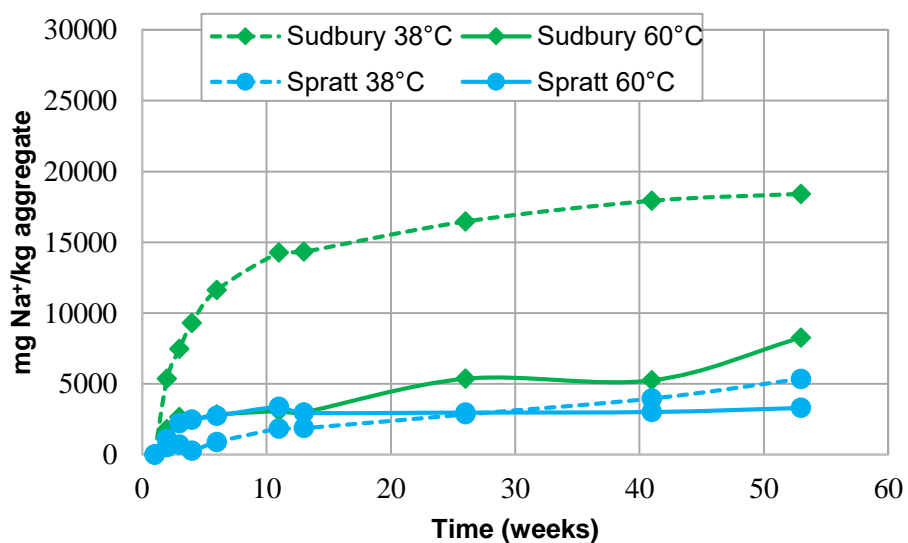


Figure 3.4: Effect of temperature on the release of Na^+ from Sudbury and Spratt aggregates into 0.7M KOH solutions.

As stated earlier, previous research showed that more alkalis were released from aggregates stored in alkaline solutions, such as NaOH and KOH than when stored in water or lime solutions [22]. The solution concentration during the test will also affect the amount of alkalis released. Tests were run with Sudbury and Spratt aggregates at 60°C in both 0.7M and 1M solutions of NaOH and KOH. The results are shown in Figure 3.5 for the release of K⁺ into NaOH solution from Sudbury aggregate is higher in the 1M solution than the 0.7M solution. The Spratt aggregate releases a small amount in either solution and there is little or no increase in the release even if the concentration of the solution is increased. In Figure 3.6, the release of Na⁺ from Sudbury aggregate into KOH solution at 1.0M is higher than in 0.7M solution, but initially the reverse is true for the Spratt Aggregate.

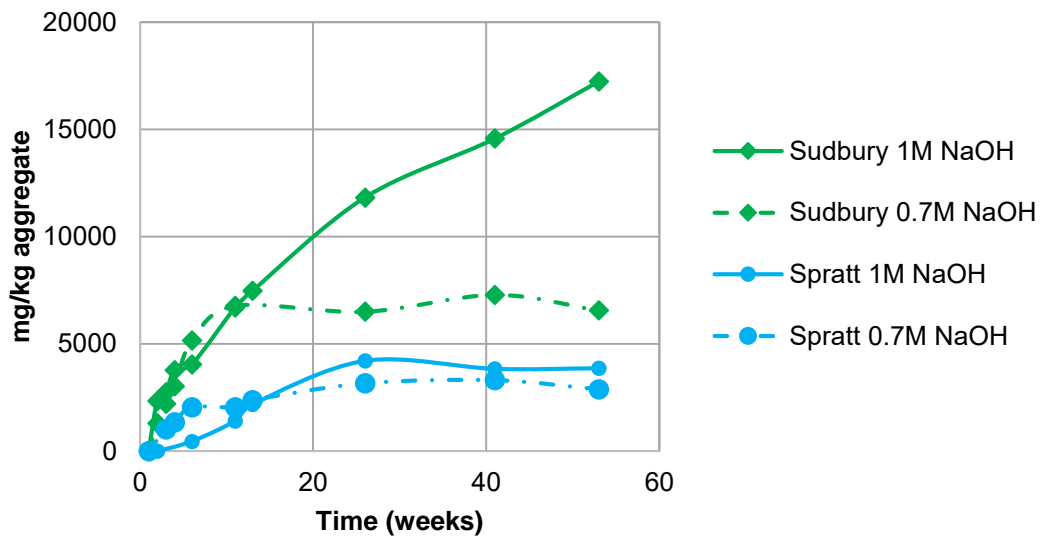


Figure 3.5: Effect of solution concentration on the release of K⁺ from Sudbury and Spratt aggregates.

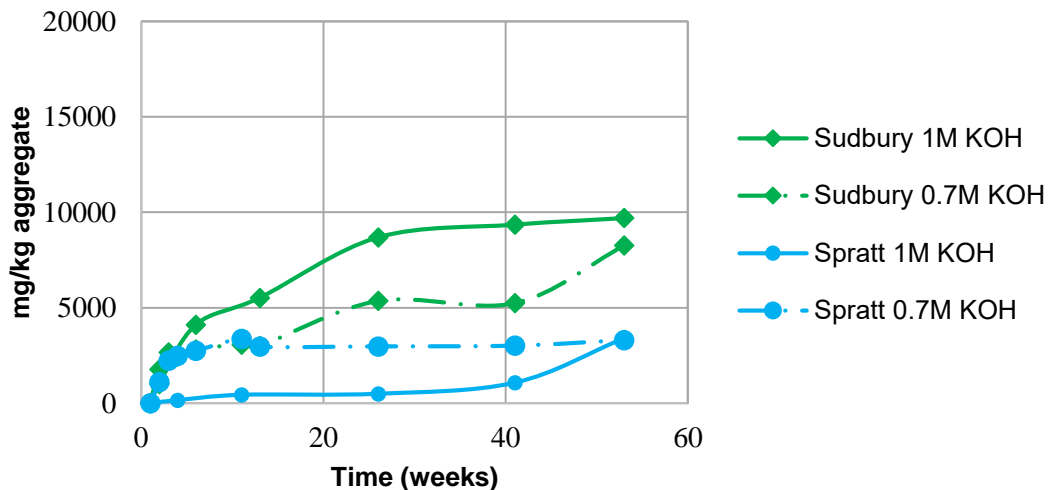


Figure 3.6: Effect of solution concentration on the release of Na⁺ from Sudbury and Spratt aggregates.

4. DISCUSSION

The test results showed that both the Sudbury reactive aggregate and the non-reactive limestone both release alkalis into highly alkaline storage solutions. Since the limestone is non-reactive it does not affect the results of the CPT but, when combined with a reactive fine aggregate, it may result in higher expansion. The concentration of the storage solution, storage temperature and duration all affect the quantity of alkalis released by the aggregates. Additions of $\text{Ca}(\text{OH})_2$ with the alkalis in the storage solution significantly increased the alkali release from the aggregates.

In both of the tests performed, four times the amount of solution was used compared to the weight of aggregate. Periodic agitation of the containers was required to prevent precipitation. Both of these test procedures result in higher amounts of alkalis being released into the solution and are not representative of what happens in the field. The amount of pore solution in concrete is much lower than 4 times the weight of the aggregate used in these tests and the pore solution in concrete is not agitated. As stated earlier, the Lower Notch dam in Northern Ontario was constructed using a coarse aggregate similar to the Sudbury aggregate, and using 20 and 30 % fly ash replacement of a high-alkali cement ($> 1.0\%$), and there are no indications of ASR after 50 years in service [13].

The amount of alkalis released over the two years of the concrete prism test (CPT) are likely not high and it is probable that they do not have a big impact on the results of the test. In field concrete, whether in structures or at exposure sites, this alkali release would be more of a significant variable over long periods of time. The variables of the tests must be standardized, and the results of those tests must be related to the actual release of alkalis from aggregates into concrete in field conditions.

If the aggregate releases alkalis into the pore solution, it would be advisable not to use low-alkali cements or low-alkali loading as the sole preventive method for ASR since the alkali loading might increase in the concrete with time, even if not detected over the test period of the CPT. Using SCMs in combination with low-alkali cement or low-alkali loadings would be advisable as the hydration products of alumino-silicate containing SCMs would continue to bind alkalis, and reduce their concentration in pore solution.

However, when prescriptive levels of SCMs alone are used for ASR mitigation, the cement replacement level of SCM used may need to be increased above those required in ASTM C1778 when aggregates that release alkalis are used.

5. CONCLUSIONS

It is known that some aggregates may release alkalis into the pore solution of the concrete over time. A standardized test is not yet available for detecting this alkali release. As well, numerous factors will affect the quantity of alkalis released from an aggregate during the lifetime of the structure. Different maximum levels of alkali loading that are recommended in standards such as ASTM C1778 that are based on several factors including the aggregate reactivity, exposure conditions and expected service life. Any alkali release from aggregates over time could cause the selected maximum alkali loading to be exceeded, therefore it is recommended, that if there is a possibility that the aggregate to be used will release alkalis over time, the use of appropriate SCMs is recommended in combination with selection of a maximum alkali loading.

Variations of two different test methods were evaluated for estimating the amount of alkalis released from three aggregates and the following is concluded:

- a) Non-reactive aggregates can release alkalis as well as reactive aggregates. This may affect concrete durability if either of the fine or coarse aggregates are marginally reactive and the alkali loading exceeds a critical threshold.
- b) The maximum amount of alkalis released from aggregates into the test solution in the test method currently in development is not necessarily the amount that will be released into the concrete. The volume of aggregate to solution is much higher in concrete than in the test, and no agitation occurs in the concrete as occurs on a regular basis in the laboratory test.
- c) Various test parameters control how much alkalis the aggregate will release. Increasing solution concentration, temperature, and addition of lime to the test solution will increase the amount of alkalis released over time.

- d) Increasing temperature may not necessarily increase the amount of alkalis released. This may be aggregate specific, i.e. the optimum temperature for alkali release from an aggregate may depend on the aggregate's mineralogy and weathering condition
- e) Whether the aggregate releases more Na⁺ or K⁺ will depend on presence of alkali containing minerals such as feldspar or mica.

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