EVALUATION OF THE AVAILABLE ALKALI CONTENT IN CONCRETE THROUGH A MODIFIED HOT-WATER EXTRACTION METHOD

Benoit Fournier[1*], Leandro Sanchez[2], Josée Duchesne[1] and Steve Goyette[1]

[1]Dept. of Geology and Geological Engineering, Université Laval, Quebec, QC, CANADA
[2]Department of Civil Engineering, University of Ottawa, Ottawa, ON, CANADA

Abstract

The "available" alkali content is considered a key parameter in the assessment of the residual expansion of ASR-affected concrete structures. Test methods including pore solution extraction under high pressure, as well as hot-water extraction measured on powdered concrete specimens, were proposed for assessing changes in the concrete pore solution composition over the years.

This paper compares the results of two hot-water alkali extraction methods, i.e. the method originally proposed by Rogers & Hooton (1993) and revisited by Bérubé et al. (1994, 2002), and a modified version called *Espresso method*. The proportion of "available" alkalis obtained by the above two methods are compared for extractions carried out at different ages on concretes of different strengths (25 and 35 MPa) and alkali contents (ranging from 2.7 to 4.6 kg/m^3). The *Espresso method* was found to provide improved reproducibility characteristics, releasing up to 85% of the total alkali content of the concretes tested.

Keywords: Available alkalis, water soluble alkalis, prognosis of ASR, residual expansion

1 INTRODUCTION

1.1 The critical effect of alkalis for ASR development

The development of alkali-silica reaction (ASR) in concrete is a function, amongst other critical parameters, of the pH of the concrete pore solution. Diamond [1] showed that a direct correlation exists between the alkali content of the cement used for concrete manufacturing and the pH of the pore solution, reaching values > 13.8 for high alkali cements (> 0.90% Na_2O_{eq}). Some experiments suggest that ASR expansion in the presence of highly reactive natural aggregates can be minimized when the alkali concentration in the pore solution falls under 0.6N [NaOH+KOH] in the long term [2]; this threshold alkali concentration will actually vary according to the reactivity level and type of reactive aggregate involved [3,4].

On the other hand, with ASR development in concrete, the alkalis are progressively consumed through the formation of secondary reaction products generated within the reactive aggregate particles; however, when cracking due to ASR extends into the cement paste, an ion-exchange process occurs with the calcium ions thus gradually recycling some alkalis to the concrete pore solution [4,5].

Other than portland cements, alkalis may be provided to the concrete pore solution, under certain conditions, from other sources such as supplementary cementing materials (SCMs), aggregates, chemical admixtures, external sources (e.g., seawater and de-icing salts), wash water (if used), etc. [4]. In addition, various processes related to the exposure conditions (contact with fresh water, salt water, wetting-drying cycles, etc.) may contribute at concentrating or diluting the alkali content in some parts of the concrete elements, thus affecting the expansion rates within and between the affected structural components of structures in the field [6]. Alkali leaching has also become a very critical issue that requires special attention in the process of establishing performance tests for the evaluation of the potential alkali-reactivity of concrete aggregates as well as the preventive effect of SCM against ASR expansion [7,8].

Therefore, on the one hand, the determination of the alkali concentration in concrete is critical for establishing the threshold alkali content in concrete for ASR prevention in mixtures incorporating different aggregate types. On the other hand, measuring the "available" alkali content is considered a crucial part of management approaches for determining the potential of future expansion (i.e. *prognosis*) and, therefore, for selecting appropriate measures of ASR mitigation in affected concrete members [9-12]. In the approach proposed by the above authors, the water-soluble alkali content is used as an indicator of the "available" alkali content, in combination with other relevant information (e.g., expansion tests on cores, environmental and stress conditions in the field), for estimating current expansion rate in ASR-affected concrete structure; the higher the water-soluble-alkali content, the higher the value calculated for the current expansion rate.

1.2 Measurement of the alkali content in concrete

The measurement of the alkali content in concrete has long been the source of interest amongst the scientific community. Longuet et al. [13] has proposed a method for extracting the pore solution in concrete

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[*] Correspondence to: benoit.fournier@ggl.ulaval.ca

under pressure, thus allowing to study of its evolution over time and providing new insights in the understanding of the hydration/chemical reactions/processes in concrete. The technique has also been used by many researchers for studying ASR-related mechanisms (e.g. [1,2,14-17]). Other than the fact that this approach requires a somewhat special equipment (extracting cell), which is actually commonly found nowadays in many research laboratories around the world, it was however found difficult to extract pore solution from old concrete or concrete with low water-binder ratios. In order to resolve the above issues, a method was developed by Rogers and Hooton [18], which was further re-evaluated by Bérubé et al. [9,19] who proposed some modifications for improving the accuracy and reliability of the method, as well as to simplify the experimental procedure. Bérubé and Tremblay [20] showed that a good estimate of the concrete pore solution composition may be obtained through the use of an indirect method consisting in determining the active- or water-soluble alkali content of concrete, in a kg/m^3 Na_2O_{eq} basis, by subjecting a representative ground sample of the concrete under study to a hot-water extraction method.

The hot-water alkali extraction method first involves the crushing and grinding of a representative sample of concrete to pass a 150-μm sieve. A ten-gram representative subsample is then obtained through appropriate splitting operation. The sample is then placed in a beaker, soaked in 80 ml of distilled water which is brought to boil for a period of ten minutes. The solution is then left to rest overnight (i.e. about 20 ± 4 hours) and filtered the next morning for recovering the solution that is analysed for its alkali content using appropriate means (e.g. atomic absorption spectroscopy (AAS), inductively coupled plasma atomic emission spectroscopy (ICP-AES), etc.). The results are finally expressed in kg of alkalis per m^3 of concrete, using a measure (or a good estimate) of the concrete density [9].

The need for revisiting the hot-water alkali extraction method proposed by Bérubé et al. [9] came up from the technical personnel carrying out the test procedure. First, it was sometimes found difficult to control the boiling intensity resulting in variability in the extraction rate, as well as potential losses of solution and/or of granular material. Also, solid material (≈ cementation) was found to form in the container during the rest (overnight) period, thus contributing in alkali fixation and reduction of the alkali concentration in the extraction solution. Also, additional testing showed that using a vacuum filtration system resulted in a more efficient and reproducible recovery of the solution than the conventional "gravity" type filtration method.

2 SCOPE OF WORK

This paper reports the results of an investigation aiming at optimising the testing conditions used during the hot-water alkali extraction method. The efficacy and precision of the method proposed by Bérubé et al. [9] is compared to that of a slightly modified procedure (*Espresso* method). In order to do so, the hot-water alkali extraction was carried out on concrete specimens of different strengths (25 and 35 MPa), total alkali contents (ranging from 2.7 to 4.6 kg/m^3) and age.

3 MATERIALS AND METHODS

3.1 Concrete mix designs and specimens for testing

As part of this study, concrete cylinders, 100 by 200 mm in size, were manufactured from 25 and 35 MPa concrete mixtures incorporating a high-purity limestone coarse aggregate and a quartzitic sand (ASTM C109)[21]. Two CSA type GU high-alkali cements were used in combination with the above aggregates. The characteristics of the materials are presented in Tables 1 and 2.

Table 3 gives the proportioning of the various concrete mixtures made in this study; the testing matrix is presented in Table 4. The 25 and 35 MPa concrete mixes AS-1 and AS-4 were made with the CQ cement (0.88% Na_2O_{eq}) and had total alkali contents of 2.73 and 3.22 kg/m^3 Na_2O_{eq}, respectively. The 35 MPa concrete mixes AS-5 and AS-6 were made using the cements CQ (0.88% Na_2O_{eq}) and DC (1.14% Na_2O_{eq}), respectively. The total alkali content of mixture AS-6 was 4.22 kg/m^3 Na_2O_{eq}, while NaOH was added to the mixture water of concrete AS-5 to increase its total alkali content to 4.63 kg/m^3 Na_2O_{eq}.

After 24 hours in their molds, the cylinders were stripped and then stored in a moist-cured room at 23±1°C covered with plastic sheets to protect them from excess moisture (and alkali leaching).

In accordance with the testing matrix presented in Table 4, the water soluble alkali content was determined after 28, 60 and/or 90 days on cylinders from one or several of the mix designs described above.

3.2 Techniques for water soluble alkali determinations

Two methods aiming at determining the "available" alkali content in concrete were compared as part of this study. Both used distilled water as an extraction media. The first method has been proposed for use as part of a management approach for the prognosis of ASR in aging concrete structures [9-12].

In the method proposed by Bérubé et al. [9], a 10.0 ± 0.1 g sample of pulverized concrete is placed in a beaker with 80 ml of distilled water. The solution is brought to boiling for a period of 10 minutes and then let to rest overnight. The solution is then filtered and the volume completed to 100 ml with distilled water. The alkalis in solution are then analysed by appropriate means (e.g. atomic absorption, ICP, etc.).

More recently, a modified version of the above method, the *Espresso* method was developed. In this case, a 10.0 ± 0.1 g subsample is placed in a Büchner funnel with a Whatman no.1 filter paper (Figure 1A). Attached to the trap on the side arm of the filter flask, there is a vacuum which sucks the air out. The boiling distilled water is progressively poured into the Büchner funnel (i.e. over the pulverized concrete sample) until all the solution goes through the specimen. The solution is then left to cool down to room temperature and the volume completed with distilled water to a fixed level using a volumetric flask. The alkalis in solution are then analysed by appropriate means (e.g. AAS, ICP-AES, etc.). The typical laboratory conditions are as follows: temperature 23 ± 2°C, relative humidity $\geq$ 50% (ideally < 65%).

3.3 Concrete sample preparation

The reliability of the hot water alkali extraction method starts with a proper preparation of the sample for testing. As recommended by Bérubé et al. [9], a sample particle size < 150 μm was adopted for testing. The following rigorous concrete sample preparation method has been adopted for this study. A series of 10g representative sample are produced through the progressive grinding of a concrete specimen (cylinder, core, etc.). In order to do so, a 2 kg concrete sample is crushed, ground and pulverized in order to pass the 150 μm sieve. The following procedure is then proposed to prepare the material for testing (note: care should be exercised at all steps to avoid sample contamination):

- The core or cylinder (e.g. 100 x 200 mm) is split in half in the longitudinal direction using a concrete saw.
- The concrete is then broken with a hammer or similar means to produce particles of about 25 mm in size.
- The concrete particles are crushed, using a small jaw crusher or another appropriate mean, by multiple passes, i.e. by reducing gradually the distance between the jaws, in order to avoid producing excessive amounts of dust. The concrete is sieved between each pass to remove the particles < 5-mm, and the operation is repeated until all the starting material is < 5 mm.
- The material (< 5 mm) is split in order to recover a representative 1kg subsample.
- That subsample is then pulverised using a roller crusher, disk pulveriser, or other appropriate means (e.g. figure 1B) until all the material is < 150 μm. Once again, for all methods used, the size reduction should be done gradually and carefully in order to avoid producing excessive amounts of dust. This is done by reducing progressively the distance between the disks, rollers, and by sieving the material over intermediate sieves between each pass until all the material goes through that sieve.
- Two or three subsamples of 10 ± 0.1g are then obtained by splitting. The samples are kept in air-tight bags/containers until ready to be tested in order to avoid carbonation. The rest of the < 150 μm material should also be kept in air-tight bag or container, and in a freezer, as a reserve for potential further testing.

3.4 Parameters evaluated

The two hot-water alkali extraction tests were used to evaluate the available alkali content from concretes of various total alkali contents. The two methods were first compared for similar conditions, and then the effect of the following parameters was evaluated in order to optimize the *Espresso* method: 1) temperature of the extraction solution: 25 and 100°C; 2) volume of the extraction solution: 80 and 300 ml; 3) reproducibility of the test (Bérubé et al. [9] vs *Espresso* methods).

In all cases, a number of subsamples (i.e. 10.0 ± 0.1 g) obtained through the rigorous sample preparation procedure described in the previous section were tested in order to verify and compare the variability of both test procedures. The results presented in the next section are expressed as the percentage of the original (total) concrete alkali content extracted through the hot-water extraction method used.

4 TEST RESULTS

4.1 Effect of the alkali extraction method

Figure 2 compares the results of the hot water alkali extraction from concrete sample AS-4 (35 MPa concrete, alkali content of 3.22 kg/m^3 Na_2O_{eq}) at an age of 28 days. Figure 1A corresponds to the method proposed by Bérubé et al. [9], with the exception that the filtration was carried out under vacuum instead of the conventional "gravity" method. Figure 1B presents the results obtained from the *Espresso* method where 80 ml of boiling water were poured through the ground aggregate material while performing filtration under vacuum (set-up illustrated in Figure 1A). In both cases, after the solution had cooled down to room temperature (i.e. the next morning in the case of the Bérubé et al. [9] method; and within about 60 minutes in the case of the Espresso method), the volume of the solution was completed to 100 ml.

Figure 2 shows that the extraction rate of the original concrete alkali content is similar from one method to the other, with average values of 83.7% (Bérubé et al. [9] method) and 84.6% (*Espresso method*);

however, the coefficient of variation (C.V.) was reduced from 5.4% to 2.1% through the use of the *Espresso* method.

4.2 Optimisation of the Espresso method

Effect of the extraction temperature

Figure 3 compares the results of the hot water alkali extraction using the *Espresso* method for concrete sample AS-5 (35 MPa concrete, alkali content of 4.63 kg/m^3 Na_2O_{eq}; age of 28 days), and with extraction solutions of 25°C and 100°C. In both cases, 80 ml of distilled water was poured through the powdered concrete while vacuum filtration was performed. Upon completion of filtration, the solution was left to rest until reaching 23 ± 1°C and then the volume of the solution was completed to 100 ml with distilled water. The increase in temperature of the extraction solution increased the alkali extraction rate and improved reproducibility, i.e. from average values of 76.1% (25°C; C.V. of 6.3%) to 85.7% (100°C; C.V. of 3.8%).

Effect of the volume of the extraction solution

Figure 4 compares the results of the hot water alkali extraction for concrete sample AS-5 (35 MPa concrete, alkali content of 4.63 kg/m^3 Na_2O_{eq}; age of 28 days) when the *Espresso* method is used with extraction solution (i.e. boiling distilled water) volumes of 80 and 300 ml. In both cases, the distilled water was poured through the powdered concrete while vacuum filtration was performed. Once the solution reached a temperature of 23 ± 1°C, the volumes were completed to 100 and 500 ml (with distilled water) for the extraction solutions of 80 and 300 ml, respectively.

Despite similar average alkali extraction rates (85.0% for 80 ml vs 84.7% for 300 ml), the use of a larger volume of extraction solution resulted in a significant reduction in the variability of the test, i.e. C.V. of 12.8% vs 1.7%. This suggests that a larger volume of solution induces a more constant extraction rate of the alkalis and lower variability from one sample to another. On the other hand, when removing one "outlier" (59%) from the calculations in the case of the 80-ml series, the average extraction rate goes from 85.0% (C.V. of 12.8%) to 87.9% (C.V. of 7.11%), thus suggesting a higher extraction rate with a lower volume of hot water; the exact reason for this result is unknown at this stage and will require further investigations.

Alkali extraction over time

Figures 5 to 7 compare the results of hot water alkali extraction using 300 ml of boiling distilled water in the *Espresso* method. Similar average extraction rates were obtained after 28 days (84.1%), 60 days (84.3%) and 90 days (84.5%) for concrete AS-1 (25 MPa concrete, alkali content of 2.73 kg/m^3 Na_2O_{eq}) (Figure 5). In addition, the C.V. obtained on ten subsamples at each testing periods were 3.81% (28 days), 0.84% (60 days), and 1.94% (90 days), indicating a very good reproducibility of the test results. In the case of the concrete AS-4 (35 MPa concrete, alkali content of 3.22 kg/m^3 Na_2O_{eq}), an average extraction rate of 85.7% was obtained at 28 days (Figure 6). Once again, a very low C.V. of 3.63% was obtained based on 10 subsamples.

The results obtained on concrete AS-6 (35 MPa concrete, alkali content of 4.22 kg/m^3 Na_2O_{eq}) at 28, 60 and 90 days are presented in Figure 7A to 7C. It is interesting to note that the average extraction rates are somewhat similar at 28 days (85.1%, Figure 7A) and 60 days (88.0%, Figure 7B), while a much lower average value (69.6%, Figure 7C) was obtained at 90 days. It is difficult to conclude at this stage that this corresponds to a real behaviour or perhaps related to a wrong set of results since such a drop had not been previously observed on other samples. A much higher average extraction rate was indeed obtained, also at 90 days, from concrete AS-5 (84.1%, Figure 7D). Additional series of tests will be required to verify the behaviour of concrete AS-6 at 90 days. It is worth mentioning that, in general, all the results of testing presented in Figure 7 showed very low variability, with C.V. ranging from 0.65 to 3.17%.

If we consider that the results obtained at 90 days on concrete AS-6 are possibly wrong, and based on the results obtained on concrete AS-1 that showed a constant average extraction rate for concretes from 28 to 90 days of age (Figure 5), it could be assumed that the results of the "right" extraction rates at 90 days for concrete AS-6 would possibly be around 85 to 88%. This suggests that the addition of alkalis (NaOH) to the concrete mixture AS-5 did not result in a significant difference in the alkali extraction rate compared to that obtained for the concrete with alkalis provided by the cement only (i.e. As-6).

Comparison between the Bérubé et al. [9] and the Espresso methods

Figure 8 compares the results of the hot water alkali extraction performed on concrete AS-5 (35 MPa concrete, alkali content of 4.63 kg/m^3 Na_2O_{eq}; age of 28 days) using the Bérubé et al. method (boiling for 10 minutes in 80 ml of distilled water) and the *Espresso* method (300 ml of boiling water passing through the sample). Rejecting the outlier (last subsample) on Figure 8A, an extraction rate of 77.4% was obtained (C.V. 7.0%) for the former method. The extraction rate obtained through the *Espresso* method was 84.7%, with a very low C.V. of 1.7%.

5 DISCUSSION

The testing carried out in this study allowed the development of a modified version of the hot-water alkali extraction method proposed by [9,18]. The procedure starts with a rigorous sample preparation process aiming at producing representative subsamples through progressive crushing/grinding/sieving operations of the material to avoid producing excessive amounts of dust. The final method involves the testing of 10 ± 0.1g (< 150 μm) concrete samples filtered under vacuum on a Büchner funnel with 300 ml of boiling water. The solution is then left to cool down to 23 ± 1°C and the volume completed with distilled water to 500 ml using a volumetric flask. The alkalis in solution are then analysed by appropriate methods (e.g. AAS, ICP-AES).

The *Espresso method* generates an alkali extraction rate (i.e. compared to the original concrete alkali loading) ranging between 80 and 88%, with excellent reproducibility between companion test subsamples. The average alkali extraction results were globally similar from 28 to 90 days for the 25 and 35 MPa concretes of various alkali contents (ranging from 2.73 to 4.63 kg/m^3, Na_2Oeq), with one exception/outlier (AS-6 at 90 days) (Table 5). In the case of laboratory concrete specimens ground to the same fineness and incorporating a non-reactive (and low-alkali) quartzite aggregate, Bérubé et al. [9] reported alkali extraction rates of about 70% up to about 16 weeks, which increased to about 80% at the 32 and 52-week measurements.

Despite the above results suggesting that the *Espresso* method shows reproducible extraction rates from one subsample to another, a number of fundamental questions remain regarding its reliability. For instance, despite the fact that Bérubé and Tremblay [20] reported a good correlation between the results of the hot water alkali extraction method and the pore solution extraction approach (under pressure), it is still largely unclear whether the alkali extraction rates obtained in this study actually correspond to the alkali concentration (and corresponding pH) in the pore solution of "real" concrete subjected to natural environmental conditions. The results obtained in this study suggest that the use of a larger volume of solution (e.g. 300 ml) in the *Espresso* method allows a more constant release/extraction rate of the alkalis, reaching between 80-88% of the total (original) concrete alkali content, while resulting in a lower variability of the results from one subsample to another. Although Bérubé et al. [9] reported that the use of boiling water does not seem to have any influence on the extracted alkali concentration; the results obtained in this study indicated that increasing the temperature of the extraction solution from 25 to 100°C in the *Espresso* method actually resulted in an increase of about 13% in the alkali extraction rate. Considering that Duchesne and Bérubé [22] and Bérubé et al. [10] reported that significant amounts of alkalis from the cement, about 40% in the case of ordinary portland cement concretes, are incorporated in the cement hydrates, this raises the question about the possible extraction of alkalis incorporated in the cement hydration products, which may actually not be "available" to the concrete pore solution under normal environmental conditions.

The evolution of the pore solution composition, as a function of time, in concretes undergoing ASR is also quite complex and several mechanisms play roles that may actually not be reliably highlighted through the hot-water alkali extraction method. In this study, all concretes tested were manufactured with non-reactive and very low-alkali coarse and fine aggregates. This means that alkali contribution by the aggregates, as well as incorporation of alkalis by ASR products can be neglected. It has been suggested in the literature that alkali recycling was occurring when alkali-silica gel was circulating within the cement paste [4,5]. However, it is still unclear which proportion of those alkalis from ASR products is actually recycled, and what proportion of those alkalis, if any, is extracted from those products during the hot-water alkali extraction method that would not occur under natural environmental conditions. Also, studies have reported that alkalis can progressively be released into the concrete pore solution from the fine and/or coarse aggregates, thus contributing at maintaining ASR over extended periods of time [23]. While the proportion of alkalis from different types or aggregates that can be released over time to the pore solution of "real" concretes is still uncertain, Bérubé et al. [9] showed that grinding the aggregate material to < 150 μm can result in alkali released ranging between 0.3 to 1.6 kg/m^3 Na_2O_{eq} during the hot water alkali extraction test, thus requiring correcting the test results accordingly. On the other hand, test methods that are currently used for estimating the long-term alkali contribution from aggregates use different extraction solutions (saturated $Ca(OH)_2$, NaOH, KOH) [25,26], which are thought to represent more realistic conditions since they better mimic the concrete pore solution. The real meaning of the results obtained through those alkali extraction procedures for aggregates and concretes, in terms of prognosis of AAR, still remains to be validated.

Also, when a sample of concrete is extracted from a concrete structure and that sample is allowed to dry before performing the alkali extraction, there may have a detrimental effect on the measurable (water soluble) alkali content in the test compared to the alkalis "available" under normal conditions [24].

6 CONCLUSION

The measurement of the "available" alkali content in concrete can yield interesting information in assessing whether the concrete tested still carries the essential conditions for sustaining ASR reaction and deleterious expansion over time (*prognosis*). The available alkali content in concrete can be obtained by hot-water extraction or by pore solution extraction methods.

Results obtained using the hot-water alkali extraction method proposed by Bérubé et al. [9] were compared to those obtained by a modified procedure called *Espresso* method. The proportion of "available" alkalis obtained by the two methods were compared for extractions carried out at different ages for concretes of different strengths (25 and 35 MPa) and total alkali contents (ranging from 2.7 to 4.6 kg/m^3). The *Espresso method* was found to provide improved reproducibility characteristics, releasing up to 88% of the total alkali content of the concretes tested. However, despite the fact that the hot-water extraction method has the potential of being a quick and reproducible tool for evaluating the effects of alkalis on ASR expansion and damage generation (e.g. alkali profiling within and between structural elements, effects of alkali leaching/concentration on expansion, threshold alkali content for different types of reactive aggregates in real concrete structures, potential alkali contribution by aggregates in field concretes), "fundamental" work is still required to validate the reliability of this type of testing in providing an assessment of the "available" alkali content in "real" concrete exposed to natural environmental conditions.

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TABLE 1: Aggregates used in the study.

Aggregate	Location	Rock Type	Specific gravity	Absorption (%)	Reactivity
Coarse	Newfoundland (Canada)	High-purity limestone	2.68	0.4	NR
Fine	Ottawa Sand (USA)	Quartzitic sand	2.65	---	NR

TABLE 2: Chemical composition of the cements used.

Cement	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Na_2O	K_2O	Na_2O_{eq}	LOI
CQ, % →	18.7	3.8	5.2	60.8	2.3	0.21	1.02	0.88	1.7
DC, % →	20.9	2.9	3.5	61.6	3.6	1.09	0.42	1.14	1.7

TABLE 3: Concrete mix-designs used.

Components	25 MPa	35 MPa
Cement (kg/m^3)	314	370
Sand (kg/m^3)	790	790
Coarse aggregate (kg/m^3)	1029	1029
Water (kg/m^3)	192	174

TABLE 4: Matrix of testing for the hot-water alkali extraction method.

		25 MPa Concrete	35 MPa Concrete		
Mix →		AS-1	AS-4	AS-5	AS-6
Cement and concrete alkali contents		CQ 2.73 kg/m^3	CQ 3.22 kg/m^3	CQ + NaOH 4.63 kg/m^3	DC 4.22 kg/m^3
Extraction method	Bérubé et al. (2005a)		28 days	28 days	
	Espresso	28, 60 & 90 days	28 days	28 & 90 days	28, 60 & 90 days

TABLE 5: Average alkali extraction rates (%) obtained with the *Espresso* method on the various series of subsamples obtained from the different concretes investigated.

	Mix →	25 MPa concrete	35 MPa concretes		
		AS-1	AS-4	AS-5	AS-6
Age (days)	28	84.1	85.7	84.7	85.1
	60	84.3			88.0
	90	84.5		84.1	69.6

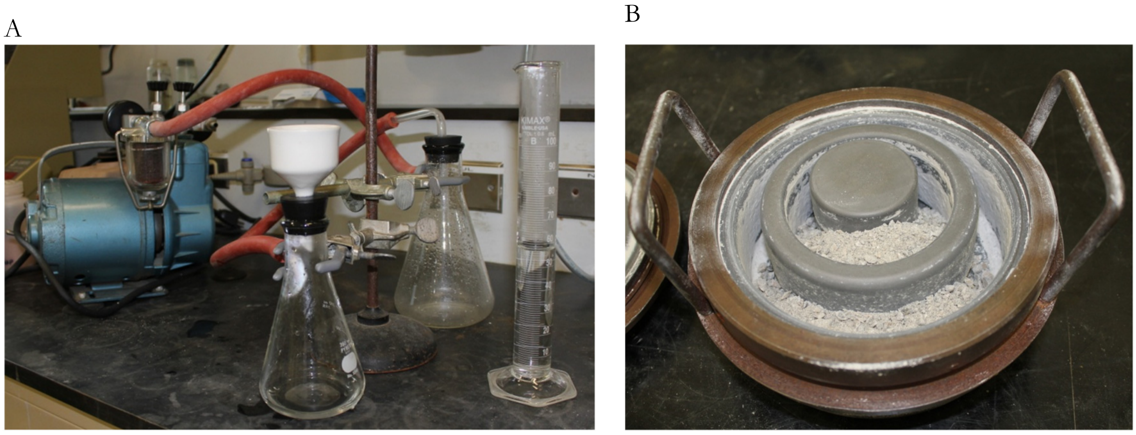


FIGURE 1: A. Set-up used for the hot water alkali extraction using the *Expresso method*. B. Mechanical tool used to pulverize rock/aggregate materials.

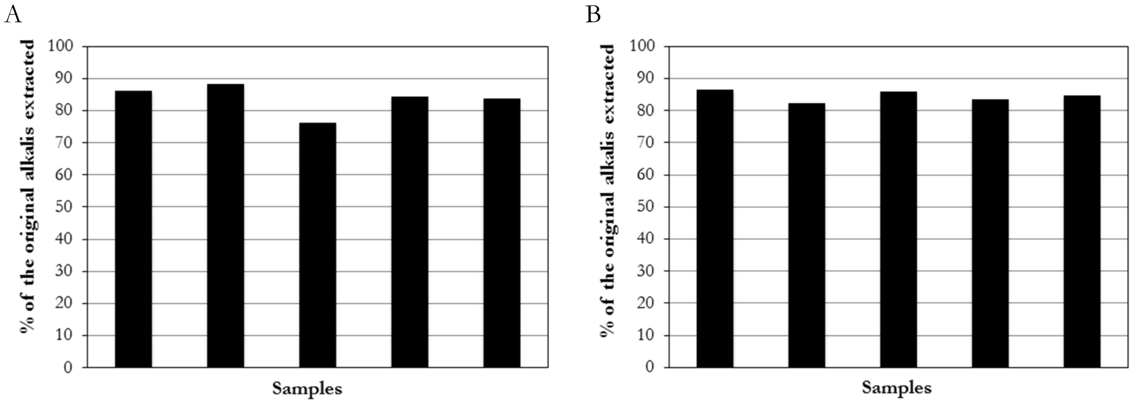


FIGURE 2: Results of hot water alkali extraction testing performed on concrete AS-4 (35 MPa, alkali content of 3.22 kg/m^3 Na_2O_{eq}, age : 28 days). In both cases, the volume of extraction solution is 80 ml. The results are expressed as % of the original concrete alkali content extracted for a number of subsamples. A. Results of tests carried out according to Bérubé et al. [9] method, but using vacuum filtration. B. Results according to the *Espresso* extraction method.

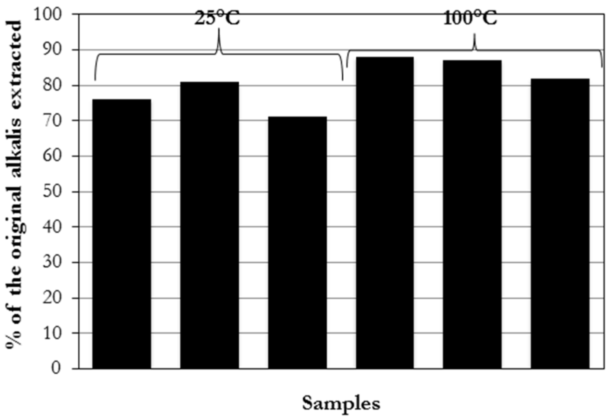


FIGURE 3: Results of hot water alkali extraction testing performed on concrete AS-5 (35 MPa, alkali content of 4.63 kg/m^3 Na_2O_{eq}, age : 28 days) using the *Espresso method* (volume of extraction solution of 80 ml) - comparison for extraction solution (80 ml of distilled water) at 25 or 100^{o}C. The results are expressed as % of the original concrete alkali content extraction for a number of subsamples.

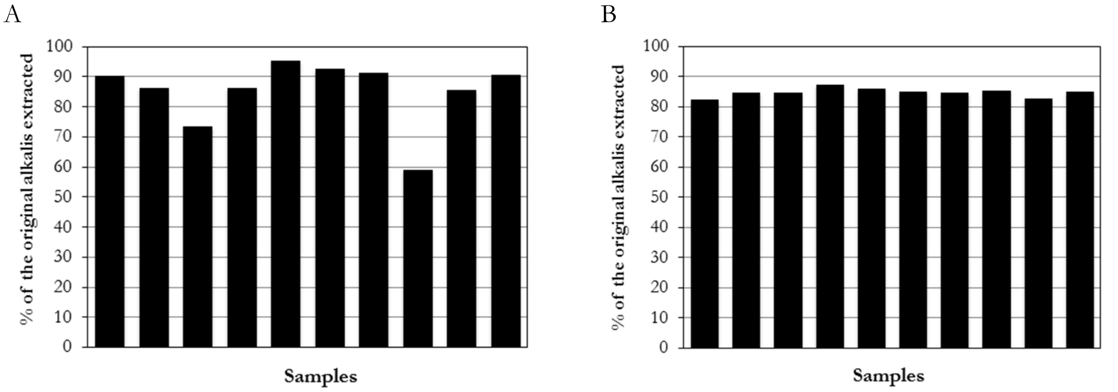
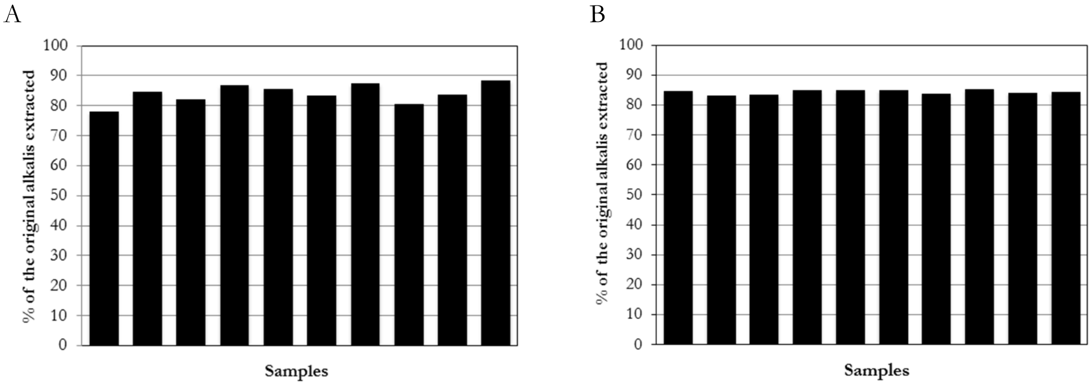


FIGURE 4: Results of hot water alkali extraction testing performed on concrete AS-5 (35 MPa, alkali content of 4.63 kg/m^3 Na_2O_{eq}, age : 28 days) using the *Espresso method* – effect of the volume of the extraction solution. The results are expressed as % of the original concrete alkali content extraction for a number of subsamples. A. Volume of extraction solution (100^{o}C) = 80 ml. B. Volume of extraction solution (100^{o}C) = 300 ml.



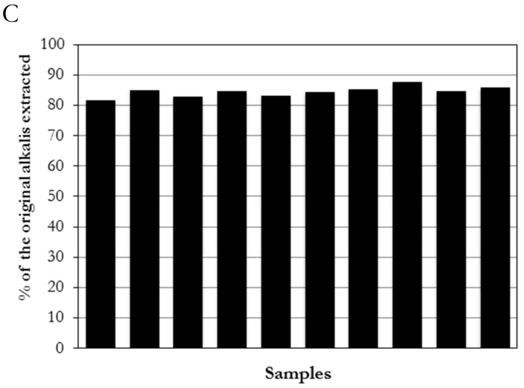


FIGURE 5: Results of hot water alkali extraction testing performed on concrete AS-1 (25 MPa, alkali content of 2.73 kg/m^3 Na_2O_{eq}) using the *Espresso method.* The results are expressed as % of the original concrete alkali content extraction for a number of subsamples. The extractions were carried out using 300 ml of boiling water on the concrete specimens of the following ages: A. 28 days. B. 60 days. C. 90 days.

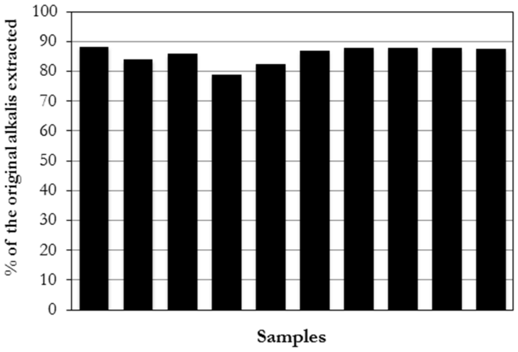


FIGURE 6: Results of hot water alkali extraction testing performed on concrete AS-4 (35 MPa, alkali content of 3.25 kg/m^3 Na_2O_{eq}) using the *Espresso method.* The results are expressed as % of the original concrete alkali content extraction for a number of subsamples. The extraction was carried out using 300 ml of boiling water and on a 28-day old concrete sample.

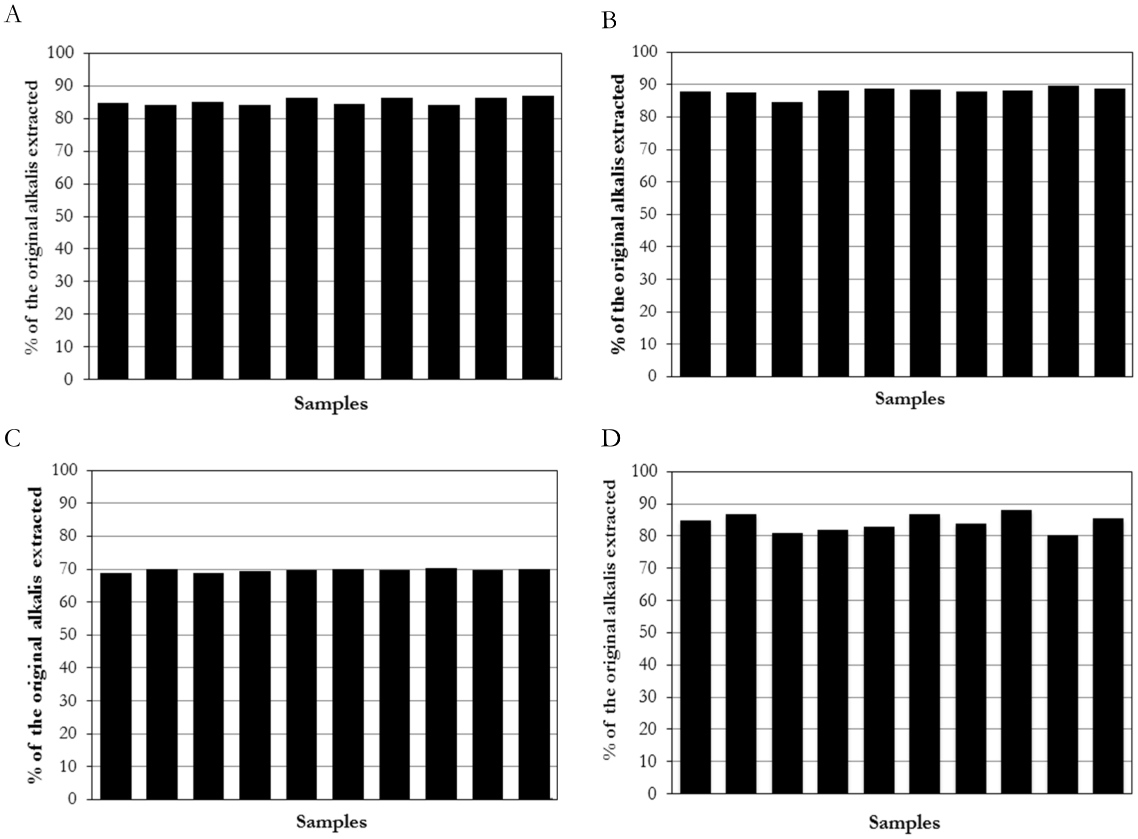


FIGURE 7: Results of hot water alkali extraction testing performed on concrete AS-6 (35 MPa, alkali content of 4.22 kg/m^3 Na_2O_{eq}) (A to C) using the *Espresso method* (300 ml of boiling water). The results are expressed as % of the original concrete alkali content extraction for a number of subsamples. A. Extraction at an age of 28 days. B. Extraction at an age of 60 days. C. Extraction at an age of 90 days. D. Concrete AS-5 (35 MPa, alkali content of 4.63 kg/m^3 Na_2O_{eq}) – Extraction at an age of 90 days using 300 ml of boiling water).

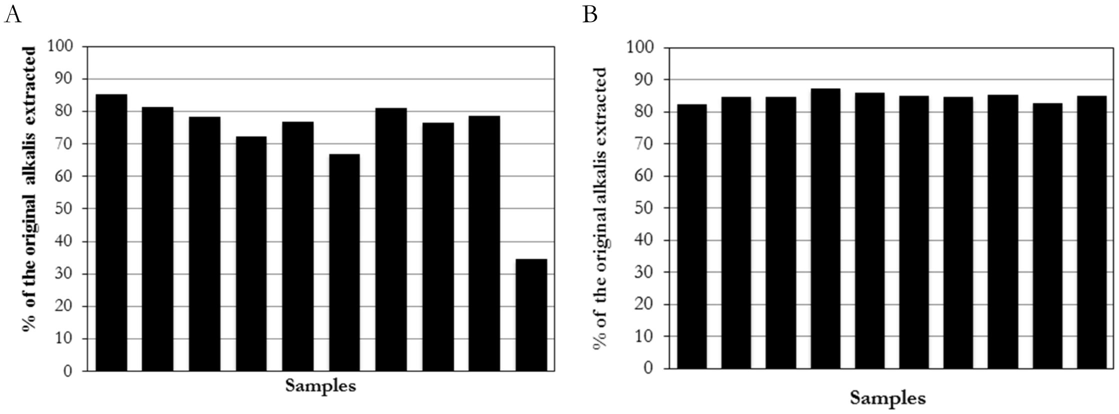


FIGURE 8: Results of hot water alkali extraction testing performed on concrete AS-5 (35 MPa, alkali content of 4.63 kg/m^3 Na_2O_{eq}, 28 days). The results are expressed as % of the original concrete alkali content extraction for a number of subsamples. A. Results of tests carried out according to Bérubé et al. [9] method; the volume of extraction solution is 80 ml (100°C). B. Extraction carried out using the *Espresso method* using 300 ml of boiling water.