Laboratory and field investigations of the influence of sodium chloride on alkali–silica reactivity

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Abstract

Concrete cylinders, 255 mm in diameter, were made with high- and low-alkali cements, a highly alkali–silica-reactive coarse aggregate, and subjected to various conditions at 38 °C: (1) immersion in 3% NaCl solution; (2) immersion in 6% NaCl solution; (3) humid air at 100% RH, and (4) 14-day cycles including 12 days in humid air, 2 days of drying, and 3 h in 6% NaCl solution. After 1 year, a number of cylinders were drilled to obtain dry powder samples from different depths, which were analyzed for total and soluble chloride and for soluble sodium and potassium. Concrete cores were also taken in a number of parapets and abutments, either exposed to deicing salts or not, on which chemical analyses were also performed on slices taken at different depths from the exposed surface. The results obtained suggest that making concrete with a low-alkali content is an effective way to prevent expansion due to alkali–silica reaction even for concretes exposed to seawater or deicing salts; this is attributed to the fact that the OH$^-$ ion concentration in the pore solution, and then the pH, is decreased in the near-surface layers of concrete exposed to sodium chloride, which does not penetrate at depth in concrete.
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1. Introduction

A number of studies suggest that exposure to deicing salt or seawater can initiate or accelerate alkali–silica reactivity (ASR) by supplying additional alkalis to concrete [1–7]. The principal mechanism proposed is pH increase in the concrete pore solution [2,4,8]. However, Kawamura and Takeuchi [5] and Kawamura and Igarashi [9] observed a significant decrease of the OH$^-$ ion concentration in the pore solution of mortar bars stored in NaCl solution compared with control bars immersed in distilled water or stored at 100% RH, despite the fact that the latter expanded less. According to the authors, the Cl$^-$ ions would accelerate ASR as long as the OH$^-$ ion concentration remains over a certain limit. The results from Bérubé and Frenette [10] for concrete prisms immersed in 1 M NaCl and NaOH at 38 and 80 °C suggest that low-alkali concrete is sufficiently protected against ASR even when exposed to seawater or

deicing salt; however, high-alkali concrete prisms immersed in NaCl solution expanded in the medium term even more than high-alkali prisms immersed in NaOH; this was attributed to the combination of ASR and formation of expansive chloroaluminates.

Measurements on field concrete usually indicate that chloride does not penetrate more than ≈ 100 mm inside concrete, while all ASR-affected components expand their mass. Moreover, it must be recalled that the OH$^-$ ions, which control the pH, are the aggressive ions as regards ASR, and that NaCl does not contribute to the increase in OH$^-$ ion concentration as long as the Cl$^-$ ions do not transform to Ca-chloroaluminates and are not incorporated in the cement hydrates, then forcing portlandite to release OH$^-$ ions to reach equilibrium with alkali cations in the pore solution. Also, measurements on cores from concrete components subjected to seawater or deicing salts usually indicate that most chloride is still water-soluble, e.g., in the form of NaCl. Scanning electron microscopy (SEM) observations and microprobe analyses by Duchesne and Bérubé [11] on mortar bars stored in NaCl solution also suggest that binding of Cl$^-$ ions in chloroaluminates or cement hydrates

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is a slow process, but a little faster with a high initial alkali content. Their results also suggest that low-alkali concrete is sufficiently protected against ASR even when exposed to NaCl; however, the authors suggested that such an exposure should promote map-cracking at the surface of (high-alkali) concretes already affected by ASR, due to pH decrease in the near-surface layer of concrete.

Because of the limited size of the mortar and concrete specimens tested by Duchesne and Bérubé [11] and Bérubé and Frenette [10], their results could be rather representative of what takes place in the near-surface layer of concrete that is constantly exposed to seawater. The present study was undertaken to determine the actual effect of sodium chloride on ASR. To obtain more realistic results, experiments and measurements at different depths were performed on larger specimens, with measurements also made on cores from existing structures.

2. Materials and methods

2.1. Laboratory concretes

2.1.1. Concrete specimens

High- (H) and low-alkali (L) concrete cylinders, 255 in diameter by 310 mm in length, were made with a highly alkali–silica reactive limestone (Spratt), a nonreactive granitic sand, 365 kg/m^3 of a cement containing 0.50% (low-alkali concrete) or 0.93% Na_2O_e (high-alkali concrete), a water-to-cement ratio of 0.48, and an air-entraining admixture. The high-alkali cement was increased to 1.25% Na_2O_e by adding NaOH to the mixture water, for a total concrete alkali content of 4.6 kg/m^3 Na_2O_e, compared with 1.8 kg/m^3 Na_2O_e for the low-alkali concrete. A total of eight cylinders were made, four low-alkali and four high-alkali.

2.1.2. Storage conditions and periodic measurements

After 24 h in their molds in a moist room at 23 °C, all prisms were demolded and stored for 14 days at 23 °C and 100% RH (above water in sealed plastic pails; one cylinder per pail). One cylinder of each concrete was then subjected to one of the following storage conditions, all at 38 °C: (1) immersion in 3% or 0.5 M NaCl (L-3% NaCl and H-3% NaCl); (2) immersion in 6% or 1 M NaCl (L-6% NaCl and H-6% NaCl); (3) storage in moist air at 100% RH, i.e., above water in sealed plastic pails (L-100% RH and H-100% RH); and (4) 14-day cycling including 12 days in moist air at 100% RH (as above), 2 days of drying at ≈30% RH, and 3 h of immersion in 6% NaCl (L- and H-cycled). Conditions (1) and (4) were established to simulate constant immersion in seawater (≈3% NaCl) and periodic exposure to seawater (tides, splashing, etc.) or deicing salt, respectively. For cylinders in NaCl solutions [Conditions (1), (2), and (4)], the [immersion solution]/[concrete] was 2 by volume, which corresponds to an [immersion solution]/[concrete pore solution] over 35. For Conditions (1) and (2), the salt solution was never renewed

during the 1-year period of testing, while a fresh solution was used at each immersion period for Condition (4). Two diametrically opposed series of two demec gauges were installed on lateral sides of each cylinder, for periodic length change measurements. For the specimens subjected to cycling, the measurements were always performed after a period in humid air.

2.1.3. Chemical analysis and SEM observations

After 1 year, six of the eight cylinders were drilled to obtain dry powder samples from different depths (0–10, 10–20, 20–30, 30–50, 50–70, and 70–100 mm). Many drill-holes were made in each cylinder to obtain samples of about 50 g, which were analyzed at the Ministère des Transports du Québec (MTQ) for total chloride, water-soluble chloride, water-soluble potassium, and water-soluble sodium. For more confident results, all samples from a given cylinder were analyzed at the same time (same series of measurements). Concrete fragments were also examined using SEM.

2.2. Field concretes

Cores of 100 mm in diameter by 65–120 mm in length were taken by the MTQ in concrete components from bridges in the Trois-Rivières area (Québec, Canada), either exposed to deicing salts or not (Table 1). Each core was split into two parts. After the concrete was dried, petrographic examinations were performed on one-half, the other half being cut to obtain slices from different depths (Table 1). These concrete slices were crushed and ground to pass the 160-μm sieve. Representative samples were then chemically analyzed as above for total chloride, soluble chloride, soluble potassium, and soluble sodium. Results for the first slice (0–10 mm) are not presented here, the values obtained for the chloride contents being usually abnormally low in this slice compared with the next one (10–20 mm), which is likely attributed to leaching by rain or by water used during the coring process. Moreover, the first slice is more susceptible to carbonation and more severely cracked.

2.3. Procedures for chemical analysis

The method AASHTO T260 was used to determine the total chloride content. The water-soluble chloride content was obtained using the method described in CSA A23.2-4B or ASTM C 114, except that boiling water was used rather than nitric acid. The method used to determine the water-soluble alkali content of the concrete, expressed in kilograms per cubic meter of Na_2O_e, is discussed elsewhere by Bérubé et al. [12]. This method is considered to be able in extracting most alkalis originally dissolved in the pore solution before the concrete was dried, while not extracting alkalis incorporated in cement hydrates and most of those progressively incorporated in reaction products of ASR. For a better precision, all samples from a given laboratory

Table 1
Field concrete components investigated and sampling depths

No.	Type of component	Age (years)[a]	Core length (mm)	Subsamples[b] Depths (mm)	Number
1211	parapet	40	90	0–10, 10–20, 20–30, 30–50, 50–70, 70–90	6
1308	parapet	22	70	0–10, 10–20, 20–30, 30–50, 50–70	5
1315	parapet	23	80	0–10, 10–20, 20–30, 30–50, 50–80	5
1334	parapet	23	70	0–10, 10–20, 20–30, 30–50, 50–70	5
1377	abutment	45	110	0–10, 10–20, 20–30, 30–50, 50–75, 75–110	6
1380[c]	parapet	33	80	0–10, 10–20, 20–30, 30–50, 50–80	5
1390	parapet	39	65	0–10, 10–20, 20–30, 30–50, 50–65	5
1391	parapet	34	120	0–10, 10–20, 20–30, 30–50, 50–70, 70–90, 90–120	7
1408[c]	parapet	38	100	0–10, 10–20, 20–30, 30–50, 50–75, 75–100	5

[a] At the time of coring, during summer 1997.
[b] Results for all slices 0–10 mm are not presented (chloride leaching, carbonation, cracking).
[c] Not exposed or just slightly exposed to deicing salt.

cylinder or concrete core were analyzed at the same time, i.e., within the same series of measurements. For each series of measurements, the procedures used were the following:

(1) Two 10-g subsamples of each ground concrete sample and one subsample of a ground control concrete were immersed in 100 ml of boiling water for 10 min.

(2) The pulp was allowed to rest overnight at room temperature.

(3) The pulp was then filtered with the solids retained washed with distilled water and the volume of solution was adjusted to 100 ml by adding distilled water.

(4) Na and K concentrations of the solution were measured by atomic emission spectroscopy.

(5) The average results for the two subsamples of each concrete were expressed in kg/m^3 of Na_2O, K_2O, and Na_2O_e, using an estimate of 2400 kg/m^3 for the concrete density.

(6) All results were corrected with respect to the result obtained for the control concrete (corrected value = measured value × known value for the control/measured value for the control).

(7) All results were also corrected for the estimated alkali contribution by aggregates in the test method, according to Bérubé et al. [12]. For laboratory concretes, 0.011% Na_2O and 0.013% K_2O obtained for the combination of coarse and fine aggregates used (i.e., 1050 kg/m^3 of Spratt limestone + 700 kg/m^3 of granitic sand) were subtracted. The same corrections were applied to field concretes that also contained coarse limestone aggregates and granitic sand.

2.4. Expression of results and calculations

For an easier comparison, all results were expressed in moles per kilogram of concrete, using Eqs. (1)–(4):

$$\text{Soluble Na (mol/kg)} = (\% \text{ soluble } Na_2O/100) \times 32.269 \quad (1)$$

$$\text{Soluble K (mol/kg)} = (\% \text{ soluble } K_2O/100) \times 21.233 \quad (2)$$

$$\text{Soluble Cl (mol/kg)} = (\% \text{ soluble } Cl/100) \times 28.206 \quad (3)$$

$$\text{Total Cl (mol/kg)} = (\% \text{ total } Cl/100) \times 28.206 \quad (4)$$

where 32.069, 21.233, and 28.206 are the number of moles of Na, K, and Cl per kilogram of Na_2O, K_2O, and Cl, respectively. The amount of insoluble Cl was then obtained using Eq. (5):

$$\text{Insoluble Cl (mol/kg)}$$
$$= \text{total Cl (mol/kg)} - \text{soluble Cl (mol/kg)}. \quad (5)$$

Considering that the only significant ions in the pore solution are usually Na^+, K^+, Cl^-, and OH^-, and that cations are in equilibrium with anions, the OH^- ion content was obtained using Eq. (6):

$$OH^- \text{ ions (mol/kg)}$$
$$= \text{sol. Na (mol/kg)} + \text{sol. K (mol/kg)} - \text{sol. Cl (mol/kg)}. \quad (6)$$

However, exposure to NaCl may cause portlandite close to the surface to be completely leached out and the pH to decrease [13,14]. In such a situation, Ca ions can reach high concentrations in the near-surface layer of concrete, such that Eq. (6) does not hold anymore and may lead to negative OH^- ion concentrations, which is impossible. Eq. (6) was then used as a routine but a minimum value of zero was used.

3. Results and discussion

3.1. Laboratory concretes

3.1.1. Concrete expansion

All low-alkali (L) cylinders did not significantly expand under all exposure conditions (Fig. 1). This suggests that low-alkali concretes made with highly reactive aggregates are not affected by ASR even when constantly or periodically subjected to seawater or deicing salt. On the other hand, all high-alkali (H) cylinders largely exceeded the 1-year, 0.04% expansion limit criterion. However, the highest expansion was observed for the high-alkali cylinder never exposed to NaCl, followed by the cylinder exposed periodically to 6% NaCl. This suggests that exposure to seawater or deicing salt does not accelerate ASR, which rather appears to be slightly slowed down in the short term. However, one can observe in Fig. 1 that the cylinder constantly immersed in 6% NaCl started to expand in the long term at a higher rate than all other cylinders, which may suggest that NaCl could accelerate ASR in the long term or at least may produce additional expansion as a result of another expansive mechanism.

3.1.2. Chemical profiles in control cylinders stored in air at 100% RH

For cylinders always stored in air at 100% RH, the soluble Na and K contents are quite constant with depth (Fig. 2C,D). On average, the total soluble-alkali content is much lower in the low-alkali cylinder (0.025 mol/kg) than in the high-alkali one (0.051 mol/kg), which complies with the cements used that contained 0.50% and 1.25% Na_2O_e (with added NaOH), respectively. In the absence of sodium chloride, the OH^- ion content calculated using Eq. (6) is necessarily equal to the total soluble-alkali content (Fig. 2E).
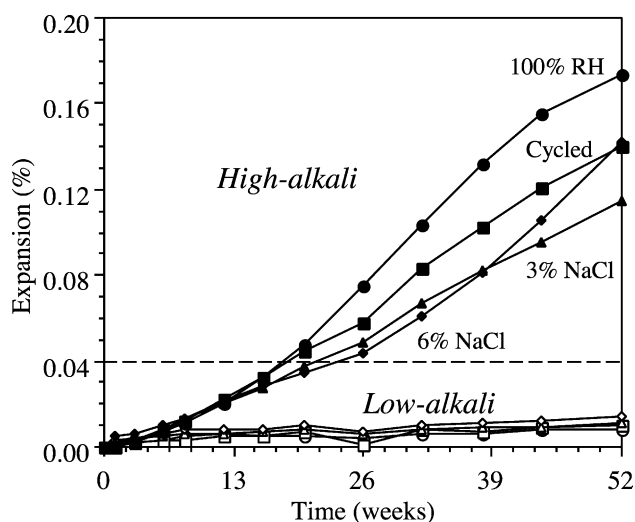
3.1.3. Chloride profiles in cylinders exposed to NaCl solution

All cylinders subjected to 6% NaCl solution present high total and soluble chloride contents near surface, which rapidly decrease with depth to reach very low values (Fig. 2A,B). The chloride contents in cylinders immersed for only 3 h every 2 weeks in NaCl solution are not lower than those in cylinders constantly immersed. This could be attributed to the fact that the cycled cylinders were always immersed in fresh NaCl solution at pH 7 after being dried for 2 days, which likely promoted a more rapid absorption and diffusion of sodium and chloride ions. The proportion of soluble Cl (in the form of NaCl) to total Cl (more likely in the form of Ca-chloroaluminate) is significantly higher in the near-surface layers of concrete ($\approx 80\%$) than at depth ($\approx 40\%$) (Fig. 2F). Cl is thus mostly in the form of NaCl even after 1 year of immersion in NaCl solution. A lower [soluble Cl]/[total Cl] at depth in concrete is attributed to the fact that, on average, chloride found at depth penetrated earlier in concrete, thus had more time to transform to chloroaluminate or to be incorporated in cement hydrates than chloride found near surface, which, on average, penetrated more lately.

3.1.4. Soluble sodium and potassium profiles in cylinders exposed to NaCl solution

All low- and high-alkali cylinders subjected to NaCl solution also present a high Na content near surface, which rapidly decreases at depth to reach about the same level as in corresponding cylinders always exposed to humid air (Fig. 2C). The soluble K content decreases from the surface to inside the concrete for all cylinders subjected to NaCl solution, as a result of diffusion out towards the salt solution (Fig. 2D). Such a decrease is more important for the high-alkali cylinders, most likely because the diffusion rate is directly proportional to the concentration difference between the immersion solution (initial K^+ ion concentration = 0) and the concrete pore solution (K^+ ion concentration higher in the high-alkali concrete). Again, at depth, the soluble K content approaches the one in the corresponding low- or high-alkali cylinder stored in air at 100% RH.

3.1.5. Hydroxyl ion profiles in cylinders exposed to NaCl solution

For all cylinders exposed to NaCl solution, the calculated OH^- ion content using Eq. (6) led to negative values (zero by default in Fig. 2E) in the first 20 mm of concrete. In other words, the Cl^- anions are always in excess with respect to the alkali cations (i.e., $Na^+ + K^+$) (see Fig. 2). This suggests that another cation is not taken into account in Eq. (6), most likely Ca^{2+}, as discussed before. According to Delagrave et al. [13,14], it is possible that portlandite is mostly consumed in the near-surface layer of concrete and that the OH^- ion content is close to zero. One point is clear from the above: The OH^- ion content clearly decreased from the surface to inside the specimens subjected to NaCl solution, the pore solution in the near-surface layers of

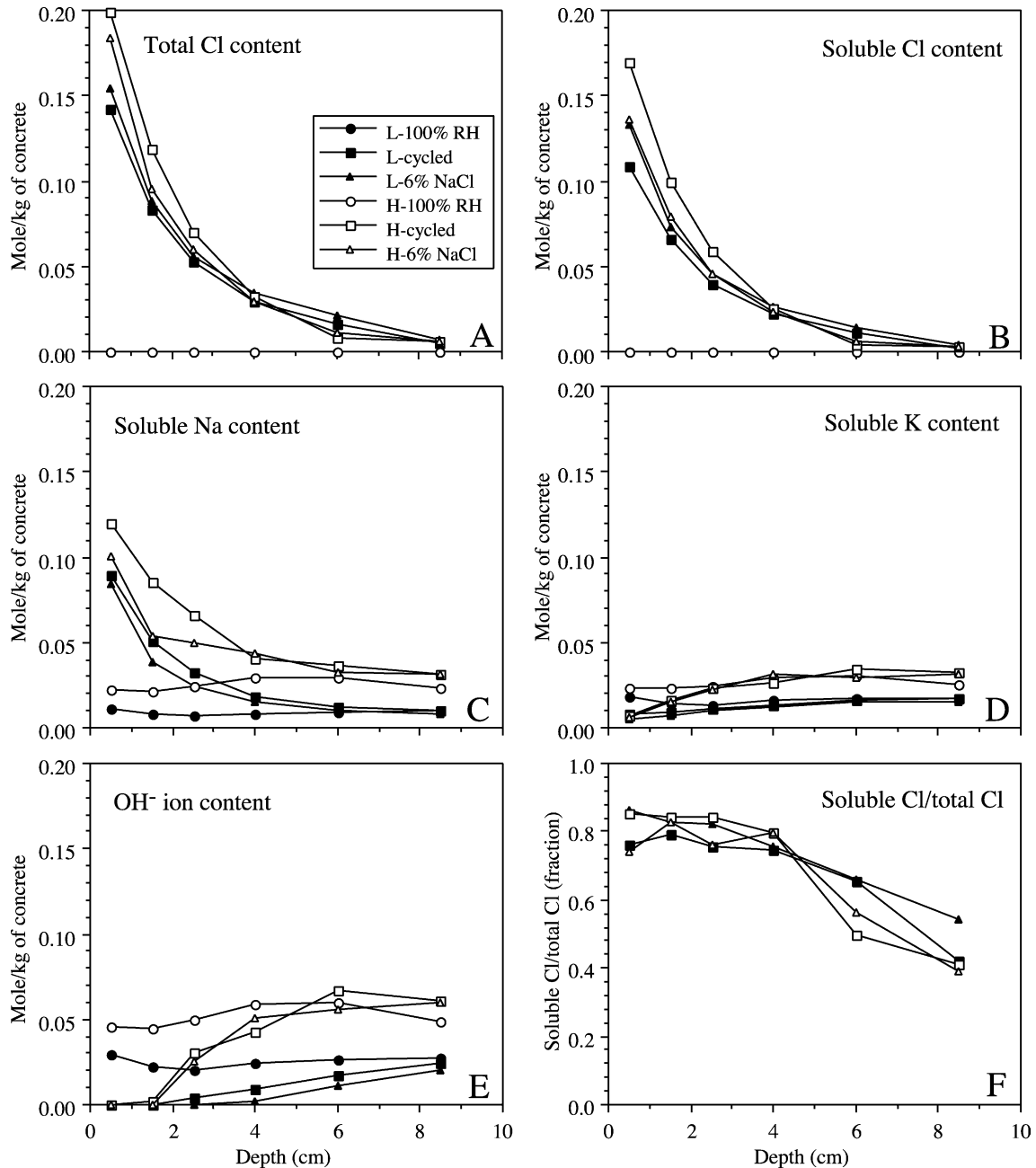Fig. 1. Expansion results for the laboratory cylinders.

Fig. 2. Chemical profiles with depth after 1 year in the concrete cylinders: (A) Total chloride content. (B) Soluble chloride content. (C) Soluble sodium content. (D) Soluble potassium content. (E) Calculated OH^- ion content. (F) Soluble chloride/total chloride.

concrete mostly containing NaCl, KCl, and likely $CaCl_2$, while NaOH and KOH are predominant at depth, but never increased with respect to the control concrete never exposed to NaCl solution.

3.1.6. Scanning electron microscopy

Fragments of cylinders L-6% NaCl, H-3% NaCl, and H-6% NaCl, were examined using the SEM, covering from the exposed surface to about 30 mm in depth. In all specimens observed, the cement paste is more porous in the near-surface layer of concrete than at depth as a result of port-

landite dissolution. Badly crystallized small crystals of chloroaluminate were observed in the cement paste (Fig. 3A,B), while well-formed crystals were present in air voids near surface (Fig. 3C). Fibrous ettringite was also observed in a number of pores, with or without chloroaluminate crystals. It was not possible to detect any difference among the three concretes examined except that chloroaluminate crystals were found in greater amount and at a greater depth in the high-alkali concrete stored in the 6% NaCl solution. Moreover, chloroaluminate was locally found along interfaces between cement paste and aggregate particles in the H-6%
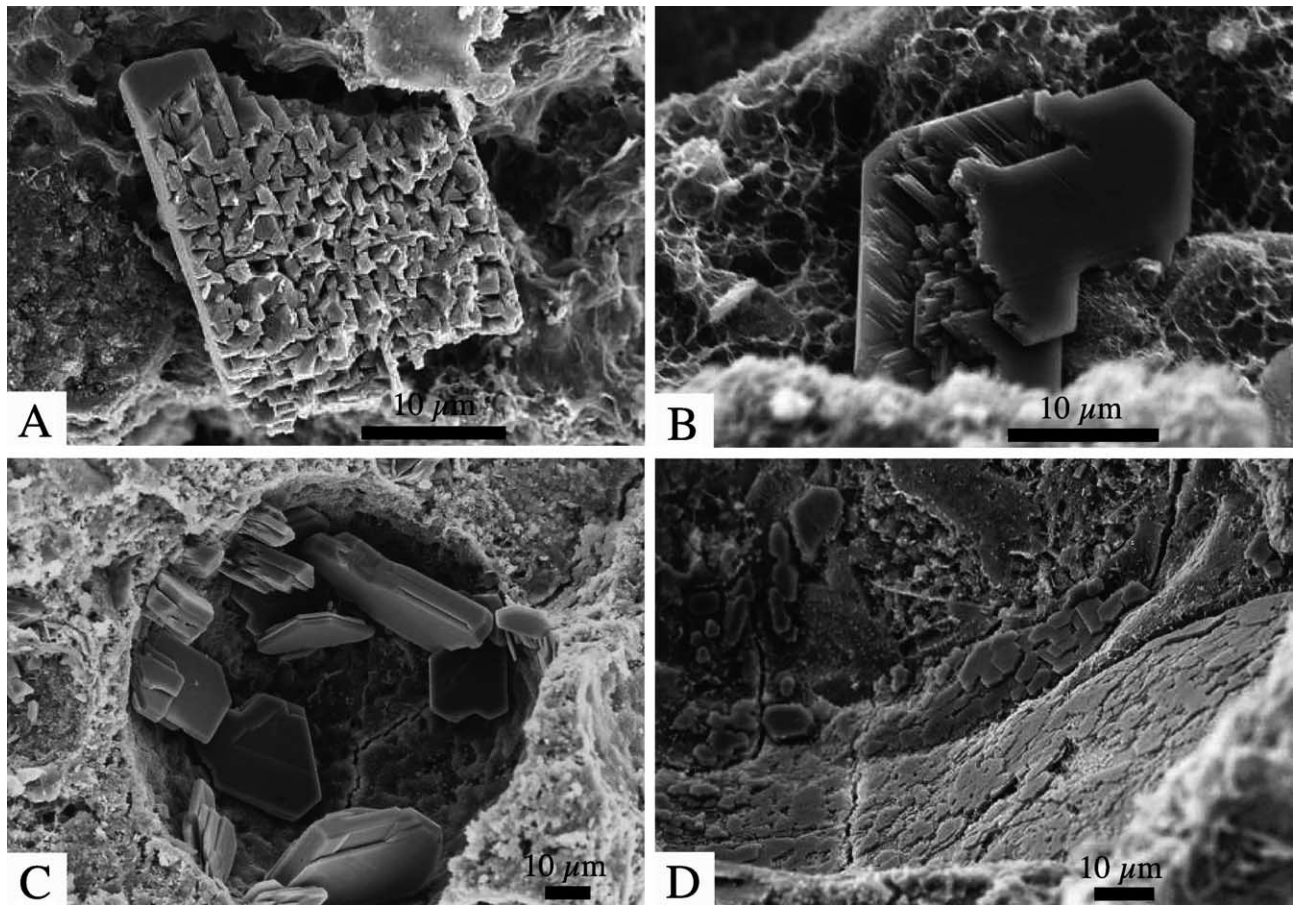
Fig. 3. SEM: (A) Badly crystallized chloroaluminate crystal in cement paste of concrete L-6% NaCl. (B) Same in concrete H-6% NaCl. (C) Well-formed chloroaluminate crystals in an air void of concrete H-6% NaCl. (D) Chloroaluminates crystals covering an aggregate–cement paste interface in concrete H-6% NaCl.

NaCl concrete (Fig. 3D). Chloride was also detected by X-ray in cement hydrates; however, one cannot say if it corresponds to precipitation of saline solution when the concrete was dried for the SEM observations or to previous chemical reaction with hydrates.

3.2. Field concretes

3.2.1. Chloride profiles

As for the laboratory cylinders, the total and soluble Cl contents progressively decrease with depth in the field components (Fig. 4A,B). However, (1) the chloride contents greatly vary from one component to another, from about zero to very low values in components not exposed (no. 1380) or just slightly exposed (no. 1408) to deicing salt, up to a maximum of 0.07–0.08 mol/kg in the most exposed components (nos. 1334, 1377, and 1308); this can be easily explained by differences in concrete permeability, age, application rates of deicing salt, and environmental conditions; (2) the decrease in chloride content with depth is more progressive than in laboratory cylinders (i.e., not exponential); (3) the chloride contents in the near-surface layer of concrete are significantly lower in the field compo-

nents than in the laboratory cylinders, but are much higher at depth for the last three components; (4) the [soluble Cl]/ [total Cl] still decreases with depth but is significantly lower than in the laboratory cylinders in the near-surface layer of concrete while decreasing at a lower rate with depth (Fig. 4F).

More progressive decreases with depth and higher contents at depth than in laboratory concretes can be attributed to the fact that the field components are much older (≥ 20 years) than the laboratory concretes (1 year old), the penetration of chloride being more advanced with a certain tendency to homogeneity. However, except in one case (no. 1211), the [soluble Cl]/[total Cl] is not significantly lower in the aged field concretes than in the young laboratory concretes; at least 50% of total Cl is still in the form of NaCl in the aged concretes, which suggests in turn that it takes time before soluble Cl transform to chloroaluminates or be incorporated in hydrates, as suggested by Duchesne and Bérubé [11].

3.2.2. Soluble sodium and potassium profiles

The soluble K content tends to slightly increase or to be quite constant with depth (Fig. 4D) as for laboratory
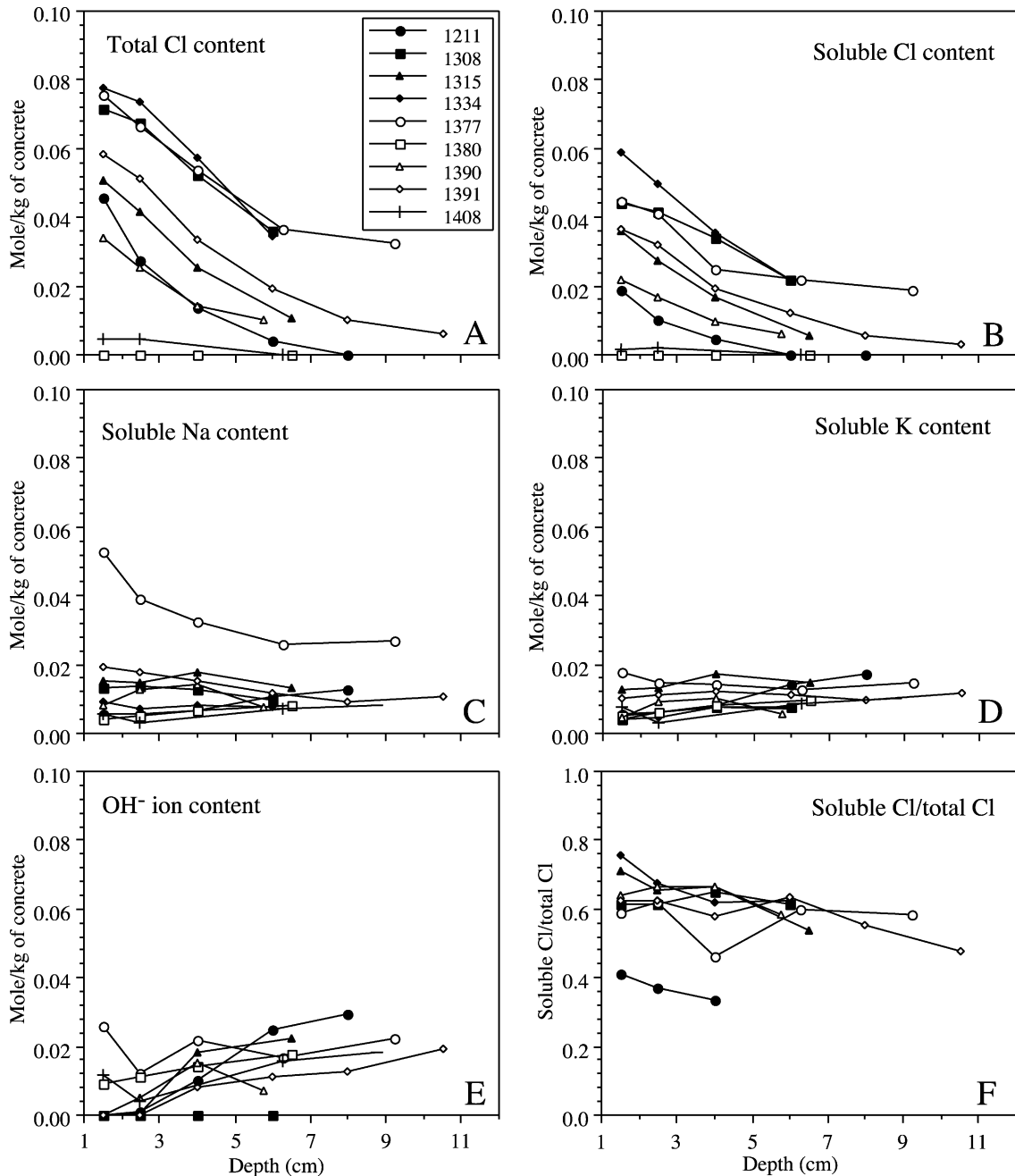
Fig. 4. Chemical profiles with depth in the field components: (A) Total chloride content. (B) Soluble chloride content. (C) Soluble sodium content. (D) Soluble potassium content. (E) Calculated OH^- ion content. (F) Soluble chloride/total chloride.

cylinders (Fig. 2D). Again, leaching by rain or salt solution can explain the observed decreases in the near-surface layer of concrete. The soluble Na content tends to be constant with depth for the two components that have not been exposed or that have just been slightly exposed to deicing salt (nos. 1380 and 1408), while slightly decreasing with depth in all components exposed to deicing salt (Fig. 4C). It is likely that the component no. 1377 with the highest soluble Na content be made with a higher water-to-cement ratio and/or a higher alkali (Na) content. Globally, the soluble Na content in the near-surface layers of concrete

is much lower than in laboratory cylinders, most likely because of less severe exposure to NaCl. Also, it must be mentioned that diffusion of Cl^- and Na^+ ions was always in the same direction in the case of the laboratory cylinders, i.e., from outside to inside, while the field concretes are exposed to rain during spring, summer, and fall, which tends to reverse the diffusion of these ions from inside to outside. This may partly explain why the chloride and sodium contents are more stable with depth and globally lower in the field concretes compared with the laboratory cylinders.

3.2.3. Hydroxyl ion profiles

As for the concrete cylinders, the OH^- ion content tends to progressively decrease towards the surface while never reaching values corresponding to the high-alkali laboratory concretes. This clearly suggests that exposure to NaCl does not increase the OH^- ion concentration and the pH in the concrete pore solution, which then would not promote ASR.

4. Conclusions and recommendations

- The Cl^- and Na^+ ion concentrations increased in the pore solution of the near-surface layers of field and laboratory concretes investigated and exposed to NaCl solution, while the K^+ and OH^- ion concentrations, and then the pH, decreased. However, the exposure to NaCl solution did not change the chemistry of the pore solution at depths greater than 60–80 mm in concrete.

- All specimens constantly or periodically immersed in NaCl solution never significantly expanded, for the following reasons: (1) the penetration depth of Na^+ and Cl^- ions was limited and, more important, (2) the OH^- ion concentration, and then the pH, tends to decrease in the near-surface layer of concrete. Making concrete with a low-alkali content then appears to be an effective way in preventing expansion due to ASR even for components exposed to seawater or deicing salt.

- ASR damage should be reduced in thin high-alkali concrete components incorporating alkali–silica reactive aggregates and exposed to NaCl, as a result of pH decrease.

- ASR should not be significantly modified in relatively thick high-alkali concrete components incorporating alkali–silica reactive aggregates, as a result of limited penetration of NaCl. However, the pH decrease in the near-surface layer of concrete could generate anisotropic expansion, thus promoting tension cracks (i.e., map-cracking) in the less-expanding concrete skin.

- Nevertheless, there is a possibility that the overall expansion of such concrete components be increased in the very long term due to the formation of chloroaluminates, as suggested before [10,11]. However, this mechanism seems not present in the case of low-alkali concretes exposed to NaCl solution. The exact role of chloroaluminates still needs to be investigated in more detail.

- The results obtained for laboratory and field concretes suggest that the incorporation of soluble chloride in chloroaluminates or cement hydrates is a slow process, as suggested before [11].

- The results obtained hold for 1-year concretes made with highly reactive aggregates and a relatively high water-to-cement ratio (0.48). The same conclusions should apply for concretes incorporating less reactive aggregates. However, it is likely that all differences observed in the near-surface layer of concrete compared with the concrete at greater depth should be reduced in the case of less permeable concretes made with a lower water-to-cement ratio, while increased in more permeable concretes. However, the exposure to seawater or deicing salt should never promote ASR in such concretes. Nevertheless, it should be of interest to acquire similar chemical profiles at different times in concretes made with different water-to-cement ratio and with cements with various compositions (e.g., SO_4, C_3A, and C_4AF contents). In addition, it could be more realistic to perform chemical analyses on pore solutions extracted under high pressure from the concrete specimens. This would allow the direct determination of Ca^{2+} and OH^- ion concentrations.

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