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Chloride resistance of high-performance concretes subjected to accelerated curing

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Abstract

The strengths and chloride penetration resistance of a series of high-performance concretes were measured after curing either at 23 °C or accelerated by heating to 65 °C. The results confirm that concretes containing silica fume (SF) or ternary blends of SF and ground granulated blast-furnace slag (GGBFS) exhibit improved chloride penetration resistance compared to those of plain Portland cement concretes. In addition, chloride penetration resistance of Portland cement concrete is adversely affected by accelerated curing. With the use of the ternary ordinary Portland cement (OPC)–SF–GGBFS binders, accelerated curing did not have detrimental effects on chloride penetration resistance and provided 18-h strengths in excess of 40 MPa.

Keywords: Curing; Chloride diffusion; Permeability; Ternary blends

1. Introduction

Accelerated curing is often used in the production of precast concrete. The elevated temperature of steam curing increases the initial rate of strength development and the high relative humidity provides the moisture required. In a production line where a quick turnover is required, the early strength gain and shorter active curing time offered by accelerated curing makes it an appealing choice [1]. Most precast plants operate on a 24-h cycle to maximize production and minimize formwork costs. Typically, forms are stripped 14 to 16 h after batching and concrete must achieve adequate strength within this time, especially if prestressing steel is to be released [2].

Increased temperature accelerates the chemical reactions of hydration. Although this initial high rate of hydration results in high early strength, long-term strength gain and durability can be adversely affected. This is primarily because accelerated temperature curing often results in formation of a coarser pore structure. The hydration products have insufficient time to diffuse evenly before they harden [3]. Instead, the hardened hydration products form a dense region around the cement particles and less dense regions farther away. This results in a more porous and nonuniform structure of the hydration products. As well, these dense zones impede further hydration of these encapsulated cement grains.

Measures can be taken to mitigate the potential negative effects of accelerated curing. One is the choice of the accelerated curing cycle. The delay or preset period before commencing the accelerated curing, the temperature rise period, the maximum temperature achieved and cooling period can all be adjusted to minimize the detrimental effects of accelerated curing for a given mix design.

Detwiler et al. [3] investigated the effectiveness of using supplementary cementing materials to increase the chloride resistance of accelerated cured concrete. They found that concretes containing supplementary cementing materials performed better than the Portland cement concretes. As well, use of supplementary cementing materials can also prevent deleterious expansions related to both delayed ettringite formation (DEF) [4] and alkali–silica reaction (ASR) [5]. The objective of this study was to further investigate the effectiveness of supplementary cementing

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Table 1
Chemical analysis of cementitious materials

	Portland cement	SF	Slag
SiO_2	21.17	95.65	37.41
Al_2O_3	4.07	0.23	9.01
Fe_2O_3	2.67	0.07	1.21
CaO	62.65	0.31	35.75
MgO	3.61	0.04	12.42
SO_3	2.65	0.17	2.87[a]
K_2O	0.46	0.56	0.37
Na_2O	0.19	0.15	0.31
TiO_2	0.22	0.02	0.37
P_2O_5	0.07	0.1	0.07
Mn_2O_3	0.04	0.07	1.05
SrO	0.06	0.03	0.05
LOI	2.18	2.27	0
Total	99.94	99.67	100.91

[a] Expressed as SO_3 but mainly in the form of sulfide sulfur.

materials in reducing the negative effects of accelerated curing on chloride penetration resistance.

2. Experimental

2.1. Mix design and materials

Six air-entrained concrete mixtures were cast with different cement replacement levels (by mass) of slag and silica fume (SF). Chemical compositions are given in Table 1. The volumetric mix designs are presented in Table 2 for a design air content of 7%. The water to cementitious materials ratio (w/cm) was kept constant, at 0.30, for all mixtures. The total cementing materials content was 460 kg/m^3 for all mixtures except for F3 where it was 500 kg/m^3. The ordinary Portland cement (OPC) used was a low-alkali CSA Type 10, similar to an ASTM Type I/II. Supplementary cementing materials used were ground granulated blast-furnace slag (GGBFS) from Hamilton, Ontario, and undensified SF from Becancour, Quebec. As well, a CSA Type 10SF blended SF cement (approximately 7.5% SF) was used for one mixture (G4A). Ten-millimetre crushed limestone coarse aggregate with a density of 2670 kg/m^3 and absorption of 1.67% was used. The fine aggregate used was a glacial sand with a density of 2700 kg/m^3, absorption of 1.4%, and fineness modulus of 2.56. All mixtures contained a lignosulphonate-based water reducer (ASTM Type A), a sodium naphthalene formaldehyde condensate superplasticizer (ASTM Type F) and a pure polymeric-based air-entraining agent.

2.2. Casting

The concretes were mixed in a 150-l countercurrent flat pan mixer in 90-l batches. For each mixture, five slabs were cast as well as 18 cylinders for compressive strength tests. Wooden moulds, which were 75 mm deep × 250 mm × 350 mm were used to cast the slabs and 200 mm by 100-mm-diameter plastic moulds were used to cast the cylinders. As well, two containers of mortar were sieved from the concrete and used for determination of the time of set by ASTM C 403. The slabs were consolidated using a vibrating table. The slabs were initially finished with a wood float followed by a final finish by using a magnesium trowel. It was noted that the ternary cementitious mixtures were easier to place and finish than the SF mixtures.

2.3. Curing

Two initial curing conditions were used: ambient curing at 23 °C (labelled A) and accelerated temperature curing (labelled S). The ambient curing consisted of wet burlap covered with plastic for the first 24 h followed by 6 days of moist curing in saturated limewater at 23 °C. The accelerated curing regime chosen is typical of that used in the precast concrete industry, with no subsequent moist curing (except for mixture D3S, which was subsequently moist cured until 7 days). Once the initial curing regime was completed, the slabs were stored at room temperature and 50% relative humidity until tests were initiated at 28 days of age.

Accelerated curing was done in a programmable environmental chamber. Temperature and relative humidity,

Table 2
Concrete mixture designs (kg/m^3 except as noted)

Concrete mixture code	E5	D3	D4	F3	F4	G4	M3	N3	N4
Portland cement	460	442	442	355	327	345[a]	423	308	308
Slag	0	0	0	125	115	115	0	115	115
SF	0	18	18	20	18	0[a]	37	37	37
Coarse aggregate—10 mm	1100	1100	1100	1100	1100	1100	1100	1100	1100
Fine aggregate	605	612	612	535	605	610	593	585	585
Water	137	137	137	149	137	137	137	137	137
Water reducer—25 XL (ml/100 kg)	325	325	325	325	325	325	325	325	325
Superplasticizer—SPN (ml/100 kg)	900	700	700	700	700	700	900	900	900
Air entrainer—micro air (ml/100 kg)	90	90	90	90	90	90	90	90	90
Design density (7% air)	2309	2316	2316	2291	2308	2313	2297	2289	2289

[a] Twenty-six kilograms of silica fume in blended Type 10SF cement.

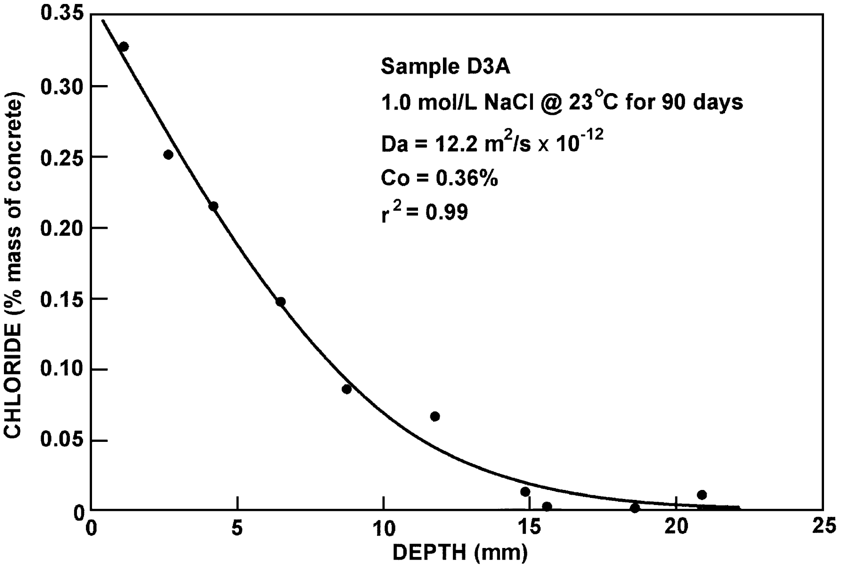

Fig. 1. Typical chloride penetration profile from bulk diffusion test.

including rates of increase and decrease, can be programmed into the machine. The cycle used met the recommendations of CSA A23.4 [6]. The temperature rise was commenced only after initial set had occurred. Initial set was determined using a penetrometer according to ASTM C 403. Typically, this took 6 to 8 h (this long set time likely resulted from the combination and dose of chemical admixtures used). The accelerated cure cycle used had a rise period of approximately 2 h (20 °C/h) to a maximum temperature of 65 °C, which was maintained for 9 h, followed by a cooling period of 2 h to again reach 23 °C. Relative humidity was held constant at 100% throughout, and distilled water was automatically atomized to maintain the relative humidity at 100%. As well, the slabs were covered with wet burlap and plastic while in the accelerated curing chamber.

3. Test procedures

3.1. Rapid chloride permeability test

The rapid chloride permeability test (RCPT) was performed at 28 days of age as per ASTM C 1202. The results for each mixture are the average of three replicate samples cored from the slabs.

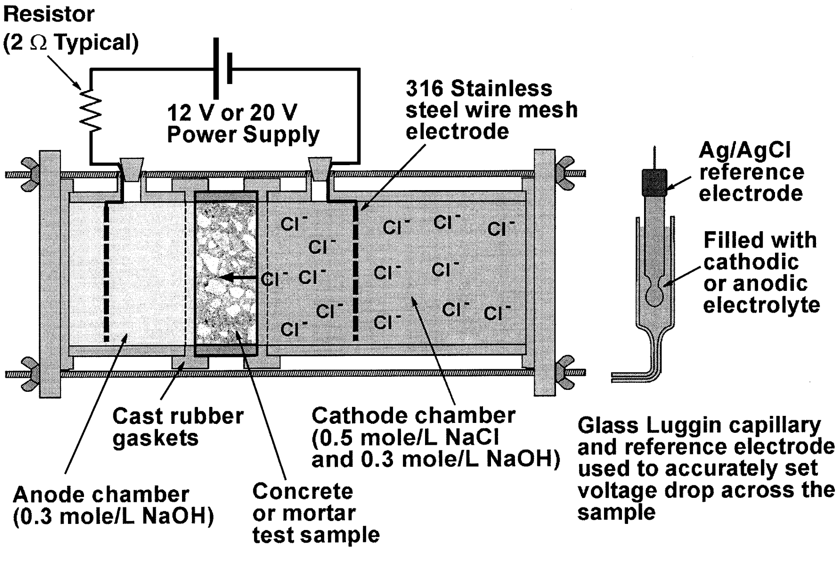

Fig. 2. Schematic of chloride migration cell and luggin capillary.

3.2. Bulk diffusion test

A chloride bulk diffusion test was performed to determine apparent diffusion coefficients. At 28 days of age, the samples were immersed in 5 mol/l NaCl solution at 40 °C for 120 days, similar to that developed by Wood et al. [7] and used by McGrath and Hooton [8]. This study was completed prior to the standardization of ASTM C1556 [9], which uses a 2.8 N NaCl solution at 23 °C for 35 days. The samples were cored from the 28-day old slabs. The cores were 100 mm in diameter and 50 mm long, with the bottom formed face being the test face.

All sides, except the formed face, of each cored specimen were sealed with epoxy. The samples were then vacuum saturated as per the procedure in ASTM C 1202.

After the samples were removed from the NaCl solution, a milling machine was used to grind off individual layers of the sample. Each layer was 0.5 mm thick and was ground with a 50-mm-diameter diamond-tipped core bit. Not all layers were collected, as only 6 to 10 points are needed to generate a diffusion curve. The ground powder samples were dried for 24 h in a 105 °C oven, then weighed and digested using a nitric acid digestion described below. Total chloride content at the different depths was determined using a potentiometric autotitrator. The digestion process consisted of adding 35 ml of distilled water and 7 ml of nitric acid (1 part distilled H_2O to 1 part HNO_3). This solution was then stirred for 10 s with a glass stirring rod and then covered with a watch glass and allowed to stand for 4 min. The beaker was then placed on a hot plate until the solution just came to a boil. It was then immediately removed and allowed to cool. The samples were then filtered to remove the solid residue.

The chloride content in parts per million (ppm) was determined using a Metrohm DMS 760 potentiometric autotitrator with 0.01 mol/l silver nitrate titrant. The apparent diffusion coefficient can be determined from the total chloride content versus depth distribution profile. Fick's second law of diffusion is as follows:

Table 3
Properties of fresh concrete

Mix	Slump (mm)	Air content (%)	Fresh density (kg/m^3)	Time of initial set (ASTM C 403) h
E5—100% OPC	230	8.5	2270	10.0
D3—4% SF	120	8.5	2280	7.3
D4—4% SF	165	7	2410	7.3
M3—8% SF	175	7	2350	8.3
F3—4% SF, 25% slag	210	6.5	2330	–
F4—4% SF, 25% slag	150	6.5	2400	–
G4—Type 10SF, 25% slag	140	7	2400	–
N3—8% SF, 25% slag	175	7.5	2300	–
N4—8% SF, 25% slag	150	7.5	2389	8.1

Table 4
Compressive strength results (MPa)

Mixture	Curing	18-h	7-day	28-day	56-day
E5A—100% OPC	Ambient	24.2	38.4	46.7	ND
E5S—100% OPC	Accelerated	31.3	38.2	45.4	ND
D3A—4% SF	Ambient	25.6	45.6	58.5	64.4
D3S + 6 day moist—4% SF	Accelerated + moist	40.7	46.1	52.8	56.2
D4S—4% SF	Accelerated	40.9	46.4	55.8	59.4
M3A—8% SF	Ambient	24.0	49.6	66.7	ND
M3S—8% SF	Accelerated	45.4	51.2	54.1	ND
F3A—4% SF, 25% slag	Ambient	17.5	42.6	63.4	66.4
F4A—4% SF, 25% slag	Ambient	14.9	47.9	57.5	68.1
G4A—Type 10SF, 25% slag	Ambient	25.6	44.6	60.2	61.2
N3A—8% SF, 25% slag	Ambient	12.7	44.6	62.8	63.1
N4S—8% SF, 25% slag	Accelerated	40.5	48.9	58.1	62.0

A = ambient cured; S = 65 °C accelerated cured; ND = not determined.

$$\frac{dC}{dt} = D_a \frac{d^2 c}{dx^2} \tag{1}$$

The chloride profile is fit to Crank's solution [10] to Eq. (1), as follows:

$$C = C_s \left[1 - \operatorname{erf}\left(\frac{x}{2\sqrt{D_a t}} \right) \right] \tag{2}$$

where C is the chloride concentration at a given depth (% of concrete mass), C_s is the concrete surface chloride concentration (% of concrete mass), x is the depth from concrete surface (mm), t is the exposure time (s), D_a is the apparent diffusion coefficient (m^2/s), and erf is the standard error function.

The value used for C, the concentration at a given depth, was taken as the background concentration subtracted from the concentration obtained. The background concentration, C_b was taken as 0.070% of concrete mass. Most of this

Table 5
Average RCPT results

Mix	Total charge 30 min × 12 (C)	Total charge 6 h (C)
E5A—100% OPC	1910	2280
E5S—100% OPC	2450	3120
D3A—4% SF	490	520
D3S—4% SF + 6 days moist	910	980
D4S—4% SF	990	1050
M3A—8% SF	260	270
M3S—8% SF	210	230
F4A—4% SF, 25% slag	310	310
G4A—Type 10SF, 25% slag	250	260
N3A—8% SF, 25% slag	160	175
N4S—8% SF, 25% slag	120	125

Table 6
Bulk diffusion test results (120 days)

Mix	Apparent diffusion coefficient, D_a (10^{-12} m^2/s)	Surface concentration, C_s (% of concrete mass)
E5A—100% OPC	32.7	0.735
E5S—100% OPC	61.4	0.802
D3A—4% SF	4.3	0.980
D3S—4% SF + 6 days moist	10.6	0.884
D4S—4% SF	13.4	0.762
M3A—8% SF	2.4	1.046
M3S—8% SF	3.8	0.995
F4A—4% SF, 25% slag	4.4	1.058
G4A—Type 10SF, 25% slag	4.5	0.965
N3A—8% SF, 25% slag	2.7	0.942
N4S—8% SF, 25% slag	3.3	1.039

background chloride is from the crushed limestone coarse aggregate and is typical of that used in Toronto. The unknowns in this equation are D_a and C_s. By using a curve fit software package, different values of these variables are input until convergence on the solution with the highest r^2 value occurred. A typical chloride profile is shown in Fig. 1. It should be noted that typically the first point in the chloride profile, which is at a depth of approximately 1 mm, is not included in the calculation of D_a and C_o. This is because this value is often affected by the higher paste volume near the formed surface.

3.3. Chloride migration test

Another method used to measure diffusion coefficients was the chloride migration cell test developed by McGrath and Hooton [11]. Two samples from each of the ambient and accelerated curing conditions were analysed for each concrete mixture. The specimens used for this test were slices taken from the interior of cores obtained at 28 days of age. The 100-mm-diameter cores were covered with a 5-mm-thick layer of sand-filled epoxy. The 30-mm disk samples were cut from the centre of the core.

Before being placed in the migration cell, the specimens were vacuum saturated as per ASTM C 1202.

The setup of the migration cell is as follows (see Fig. 2). The specimen is placed between two chambers. One chamber, the cathodic (upstream) chamber, contains 1.5 l of 0.5 mol/l NaCl and 0.3 mol/l NaOH solution. The other chamber, the anodic (downstream) chamber, contains 0.6 l of 0.3 mol/l NaOH solution. The electrodes consisted of 100-mm-diameter pieces of 316 stainless steel wire mesh that were connected to a DC power supply. The power supply was set to approximately 22.3 V in order to obtain an actual potential of approximately 20 V across the sample. This difference is due to polarization and IR losses as described in Ref. [11]. The actual voltage across the sample was measured by means of double junction Ag/AgCl electrodes placed inside Luggin capillary tubes (see Fig. 2). The outer chamber of the electrodes and the Luggin capillary tubes were both filled with the solution of the chamber into which they were inserted. This was measured after breakthrough had occurred.

Ten-millilitre samples of solution were taken from the anodic chamber every few days. The exact time at which the sample was taken was recorded. To maintain a constant volume in the downstream chamber, the sample, which was taken was replaced with 10 ml of the 0.3 mol/l NaOH anodic solution. There was a correction made in the analysis for the dilution effect this would have. The samples were stored in sealed plastic vials until the end of the test.

When the test was complete, all the samples were analysed at one time in order to reduce variability in the titration procedure. The samples were analysed for chloride content using the automatic potentiometric titrator described previously.

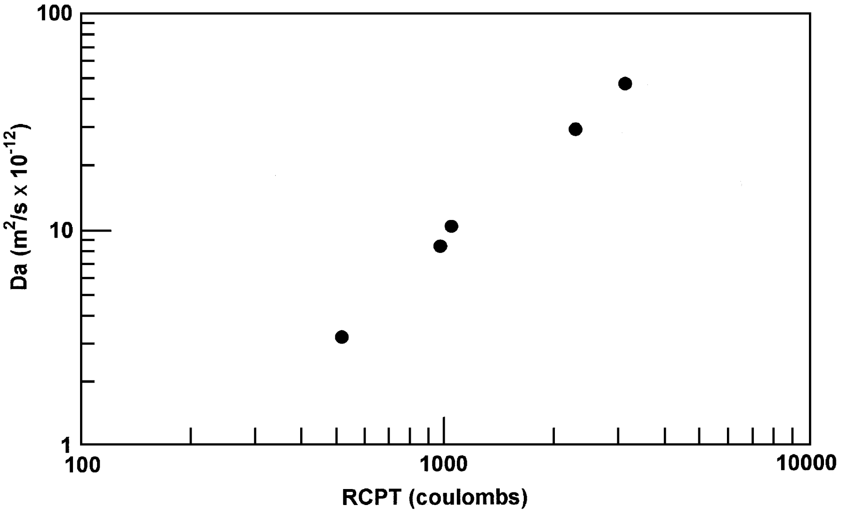

Fig. 3. Relationship between apparent bulk diffusion (D_a) and RCPT results.

Table 7
Chloride migration test results nonsteady state (D_{M25}) and steady state (D_{mf}) diffusion coefficients

Mixture	Non-steady-state migration, D_{M25} ($\times 10^{-12}$ m^2/s)	Steady-state migration, D_{Mf} ($\times 10^{-12}$ m^2/s)
E5A—100% OPC	4.4	4.0
E5S—100% OPC	20.1	18.0
D3A—4% SF	1.1	0.92
D3S—4% SF + 6 days moist	2.9	2.7
D4S—4% SF	7.2	4.5
M3A—8% SF	0.48	0.28
M3S—8% SF	1.0	0.95
F4A—4% SF, 25% slag	0.86	0.55
G4A—Type 10SF, 25% slag	0.91	0.52
N3A—8% SF, 25% slag	0.41	0.18
N4S—8% SF, 25% slag	0.57	0.54

Both non-steady-state (from breakthough time using Ficks second law) and steady-state diffusion coefficients (using the Nernst–Plank equation) were obtained as described by McGrath and Hooton [11].

4. Results and discussion

Properties of the plastic concrete are given in Table 3. The extended times of set are likely due to the combination and dose rates of chemical admixtures used. Compressive strength results for 18 h, 7, 28 and 56 days are given in Table 4. Compressive strengths in excess of 40 MPa at 18 h of age were obtained for accelerated cured concretes with 4% SF (D4S), 8% SF (M3S) and 8% SF + 25% slag (N4S). Strengths of this level are comparable to those required for release of prestressing.

The RCPT values are presented in Table 5. As an alternative method of analysis, in order to minimize heating effects on the results of higher coulomb concretes, the 30-min values were multiplied by 12 as described by McGrath and Hooton [12]. As can be observed, at low coulomb values the results using 12 times the 30-min value were similar to the 6-h standard value. According to ASTM C 1202, very low permeability concretes have RCPT values below 1000 coulombs (C), while moderate permeability concretes have an RCPT value between 2000 and 4000. Coulomb values above 4000 indicate high-permeability concrete. All the results in this study are below 4000 C, which would be expected since a low water/cement ratio, 0.30, was used in all the mixtures. The 100% OPC mixture, whether accelerated or ambient cured, was in the moderate permeability category (between 2000 and 4000 C). All mixtures containing supplementary cementing materials, for both accelerated and ambient curing conditions, were in the very low permeability category of under 1000 C. Of the mixtures containing supplementary cementing materials, mixtures D3 and D4 (4% SF) had RCPT values between 500 and 1000 C, while all other mixes were below 500 C. Values under 500 C indicate extremely low permeability concrete, and it is thought that small differences in this range are insignificant.

The 120-day bulk diffusion test results are presented in Table 6. Similar to the RCPT results, the general conclusions that can be drawn are that the supplementary cementing materials can be used to counteract the negative effects of accelerated curing on the chloride resistance of concrete. Accelerated cured concretes containing 4% SF perform significantly better than ambient-cured concretes containing no supplementary cementing materials. All of the concretes with higher replacements of supplementary materials such as 8% SF, 4% SF plus 25% slag, or 8% SF plus 25% slag

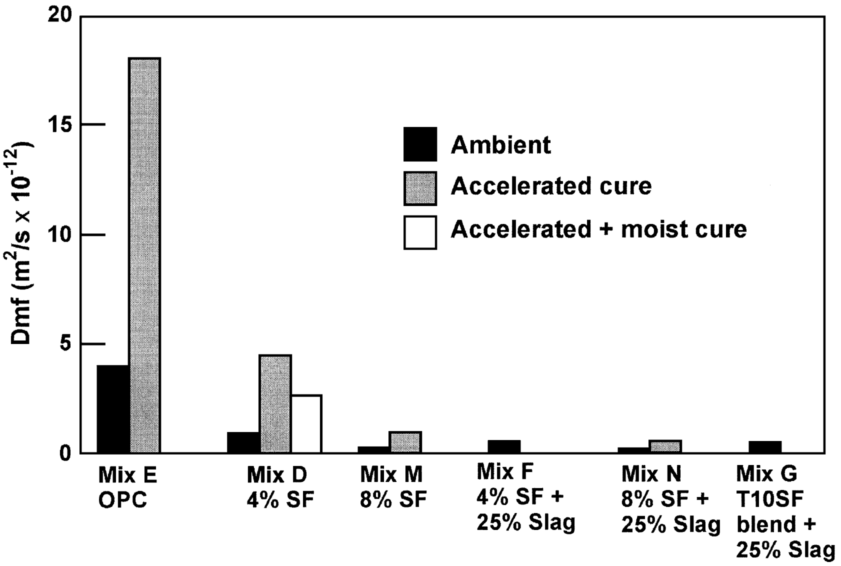

Fig. 4. Effect of cementing materials and curing procedures on steady-state migration results.

show significantly lower than apparent diffusion values, 4% SF only, but are not significantly different from each other.

The relationship between the 120-day bulk diffusion value and the RCPT data (30 min × 12 C values) are shown in log–log space in Fig. 3. Similar to previous findings [8,12,13], there is a good relationship (correlation coefficient, r^2=.973).

The results for the chloride migration test (Table 7, Fig. 4) are very similar to the RCPT results and the bulk diffusion test results. Mix E5 (100% OPC) had the highest D_a value for both accelerated and ambient-curing conditions. For each mixture, accelerated curing resulted in higher diffusion coefficients. The accelerated cured Mix E5 (100% OPC) concrete had the highest diffusion coefficient followed by the accelerated cured Mix D (4% SF) concrete, which was followed by the ambient cured Mix E5 (100% OPC) concrete. The differences between all other mixes for both ambient and accelerated curing conditions were very small. The addition of only 4% SF significantly reduced the negative impacts of accelerated steam curing. The accelerated cured 4% SF concrete had a diffusion coefficient approximately equal to that of the ambient cured 100% OPC mix. Higher replacement levels of SCMs such as 8% SF or 4% SF in combination with 25% slag or 8% SF in combination with 25% slag were significantly better than the accelerated cured Mix D4S (4% SF) concrete but were not significantly different from each other.

5. Conclusions

The 65 °C accelerated cured SF (4% and 8%) concretes as well as the ternary cement concrete (8% SF plus 25% slag) provided 18 h strengths exceeding 40 MPa, typical of what might be expected using high-early-strength cement concrete. The latter two mixtures exhibited the highest chloride penetration resistance of those exposed to 65 °C accelerated curing.

The results for all three chloride penetration resistance tests show a similar trend. Although it is clear that accelerated curing has negative impacts on the chloride resistance of concrete (even when a proper preset time is used), the use of supplementary cementing materials can be effective in terms of mitigating these negative effects. The replacements of Portland cement by only 4% SF significantly reduced the negative impacts of accelerated curing. Higher replacement levels of supplementary materials such as 8% SF, 4% SF plus 25% slag or 8% SF plus 25% slag are significantly better than using only 4% SF, but are not significantly different from each other. The ternary cementitious mixtures have the advantage of being easier to place and finish than the SF mixture.

Concrete mixtures containing 8% SF and 25% slag appear to have good potential for use in precast operations employing accelerated curing. They combine high early strength with superior chloride penetration resistance and are easier to place and finish than concretes that contain 8% SF alone.

It should also be noted that the ASTM C 1202 rapid test results relate well to both chloride bulk diffusion and chloride migration test results.

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