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Long-term testing of the chloride-penetration resistance of concrete containing high-reactivity metakaolin

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Abstract

This paper presents the long-term results of a study investigating the chloride penetration resistance of concrete containing high-reactivity metakaolin (HRM). The metakaolin evaluated in this study is a highly processed kaolinite clay that has been heat-treated under controlled conditions to produce high pozzolanic activity. Six concrete mixtures were cast with 0%, 8%, or 12% by mass replacement of portland cement with HRM at water-to-cementitious materials ratios (w/cm) of 0.30 or 0.40. The early age test results are summarized and include (1) strength, (2) 28-day and 90-day bulk diffusion, (3) rapid chloride permeability, and (4) resistivity. The long-term tests performed include bulk diffusion testing at 140 days, 1 year, and 3 years and chloride migration testing. It was found that strength increased at all ages with decreasing w/cm and increasing content of HRM. The results from all of the early-age experimental work showed that higher metakaolin content and lower w/cm decreased diffusion, permeability, and conductivity and increased resistivity. Resistance to chloride migration increased with increasing metakaolin content and decreasing w/cm. The long-term bulk diffusion testing showed a continued improvement in chloride resistance for all of the mixtures. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Chloride; Diffusion; High-reactivity metakaolin; Permeability; Concrete

1. Introduction

High-quality, low-permeability concretes improve durability by inhibiting the ingress of solutions containing soluble chloride ions towards reinforcing steel. These concretes often incorporate low water-to-cementitious materials ratios (w/cm) and use one or more supplementary cementing materials.

High-reactivity metakaolin (HRM) is one of the newest supplementary cementing materials to be used to produce high-performance concrete. High-purity kaolin clay is treated by controlled thermal activation to drive off the water "bound in the interstices of the kaolin," so "the structure collapses, resulting in an amorphous aluminosilicate," effectively converting the material to the metakaolin phase [1]. The end result is an almost 100% reactive pozzolan, which will chemically combine with calcium hydroxide to form additional cementitious products. Findings by other researchers have shown that with appropriate replacement levels, mixtures containing HRM yield comparable performance to those containing silica fume in terms of strength, permeability, chemical resistance, and drying shrinkage resistance [2–5]. Also, HRM, because of its white color, does not darken concrete, making it suitable for color matching and other architectural applications.

The objective of this study was to investigate the chloride penetration resistance of concrete containing HRM at various w/cm and replacement levels. This was accomplished by measuring the chloride ingress using a variety of test procedures. Both early- and late-age results are reported in this paper.

2. Experimental program

2.1. Materials, casting, and sample preparation

Six air-entrained concrete mixtures were cast at w/cm of 0.30 or 0.40 with 0%, 8%, or 12% by mass replacement of cement with HRM. The portland cement was of

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Table 1
Chemical analyses for cementitious materials

	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	TiO_2	K_2O	Na_2O	LOI	Na_2O_{eq}
HRM	0.05	52.06	45.29	0.60	0.00	0.00	1.64	0.16	0.21	0.51	0.32
OPC	62.65	21.17	4.07	2.67	3.51	2.65	0.22	0.46	0.19	2.18	0.49

Type I/II low-alkali classification. Chemical analyses for both the OPC and HRM are provided in Table 1. The fine aggregate had a density of 2700 kg/m^3, an absorption of 1.4%, and a fineness modulus of 2.56. Because of differences in specific gravity between the cement and the HRM, adjustments were made in the fine aggregate to maintain the yield. The HRM had a specific gravity of 2.50 and particles are less than 0.2 μm in size. Crushed 10-mm limestone with a density of 2670 kg/m^3 and an absorption of 1.67% was used as the coarse aggregate. All mixtures included a Type A water-reducer, a sodium naphthalene sulfonate-based superplasticizer and a polymer-based air-entraining admixture. The volumetric mixture proportions and fresh concrete properties are summarized in Table 2.

Three 75-mm deep × 250 × 350-mm slabs were cast from each mixture and were compacted on a vibrating table and finished with a wood float followed later by a magnesium trowel. Eight 100 × 200-mm cylinders were also cast for compressive strength testing. All of the concrete specimens were initially covered with wet burlap and plastic for 20 h. The cylinders for compressive strength testing were removed from their molds and stored in limewater at 23°C until their respective testing ages. The strength testing results have been reported elsewhere [6]. After demolding, the slabs were cured for 5 days in limewater, and then stored at 23°C and 50% RH. At 25 days of age, six 100-mm diameter cores were cut from the formed face of each slab. All cores were saw-cut to a thickness of 50 mm, with the exception of the samples used in the chloride-migration test, which were cut to a 30 mm thickness. After sealing their sides with a sand-filled epoxy, the samples were placed in a 50% RH, 23°C environment until the specific sample preparation for the specific test method was initiated at approximately 28 days of age.

3. Summary of early age results

Previous work on the same concrete mixtures involved testing in terms of strength, early-age bulk diffusion, AASHTO T259 90-day ponding, water sorptivity, the commonly called rapid chloride permeability test (ASTM C 1202, Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration), and resistivity. It was found that strength increased at all ages with increasing contents of metakaolin and decreasing water-to-cementitious ratio. The results from all of the other experimental work showed that higher metakaolin content and lower w/cm decrease diffusion, permeability, and conductivity, and increased resistivity. The early-age research performed led to the conclusion that HRM will substantially increase a concrete's resistance to chloride ingress. A detailed explanation of these test methods, early-age results, and analyses have been presented previously by Hooton et al. [6].

Table 3 shows test results from specimens of each of the six concrete mixtures. The resistivity and RCPT tests were carried out at 28 days of age. The rapid chloride permeability test followed the standard ASTM C1202 procedure. The resistivity test applied was based on the cyclic DC potential approach developed by Monfore [7].

4. Resistance to chloride migration

4.1. Test procedure

Fig. 1 shows the chloride migration apparatus used in this study. This apparatus was developed by McGrath and

Table 2
Summary of mixture proportions and properties of fresh concrete

Mixture	AB1	AB2	AB3	AB4	AB5	AB6
w/cm	0.40	0.40	0.40	0.30	0.30	0.30
OPC (%)	100	92	88	100	92	88
HRM (%)	0	8	12	0	8	12
OPC (kg/m^3)	380	350	334	460	423	405
HRM (kg/m^3)	–	30	46	–	37	55
Coarse aggregate (kg/m^3)	1100	1100	1100	1100	1100	1100
Fine aggregate (kg/m^3)	655	647	641	619	608	601
Water (kg/m^3)	152	152	152	138	138	138
25 XL water reducer (ml/100 kg)	325	325	325	325	325	325
SPN superplasticizer (ml/100 kg)	400	600	800	700	900	1100
MicroAir entrainer (ml/100 kg)	40	40	40	40	40	40
Slump (mm)	135	125	200	170	90	145
Air content (%)	9.5	7	6	7.5	5	5
Fresh concrete density (kg/m^3)	2275	2353	2424	2346	2459	2473

Table 3
Average 28-day resistivity and RCPT results

Mixture	Resistivity (Ω cm)	RCPT (6 h) (coulombs)
1 — 0.4 w/cm, 0% HRM	9960	2770
2 — 0.4 w/cm, 8% HRM	34910	560
3 — 0.4 w/cm, 12% HRM	72260	310
4 — 0.3 w/cm, 0% HRM	11370	2350
5 — 0.3 w/cm, 8% HRM	51960	400
6 — 0.3 w/cm, 12% HRM	99360	230

Hooton [8]. As can be seen in Fig. 1, a vacuum-saturated, 28-day-old, 30-mm thick, 100-mm diameter sample was placed in the plastic conduction cell casing and was sandwiched between two chambers on either side of the sample. The cathodic chamber contained a 0.5 mol/l NaCl and 0.3 mol/l NaOH solution, while the anodic chamber contained simply a 0.3 mol/l NaOH solution. A voltage of either 12 or 20 V was applied across the sample, depending upon the predicted quality of the concrete. The electrodes used to apply this potential consisted of circular pieces of 316 stainless steel wire mesh. Reference electrodes consisting of double junction Ag/AgCl electrodes placed inside glass Luggin capillary tubes were used to accurately set and monitor the voltage drop across the sample. Ten-milliliter samples were taken from the anodic chamber and analyzed for chloride content using potentiometric auto-titration with silver nitrate solution.

Reported results from chloride migration testing by other researchers have typically been analyzed in one of two ways, either steady state chloride flux [9–12] or initial nonsteady state chloride penetration [10,13].

4.1.1. Nonsteady state migration coefficient

The Nernst–Planck, or modified Fick's second law, can be used to describe chloride ion transport in conduction tests. This equation and its exact analytical solution shown as Eqs. (1) and (2), respectively [14]:

$$\frac{dC}{dt} = -\frac{dJ}{dx} = D\left(\frac{d^2C}{dx^2} - \frac{zF\Delta V}{RTl}\frac{dC}{dx}\right) \tag{1}$$

$$C = \frac{C_0}{2}\left[e^{ax}\mathrm{erfc}\left(\frac{x + aDt}{2\sqrt{Dt}}\right) + \mathrm{erfc}\left(\frac{x - aDt}{2\sqrt{Dt}}\right)\right] \tag{2}$$

where

$$a = \frac{zF\Delta V}{RTl}$$

and z = ion valence, F = Faraday's constant, ΔV = potential drop across the sample, R = gas constant, T = temperature in Kelvin, l = sample length, erfc() = standard error function, C = pore solution concentration at any depth, x, and any time, t, and C_0 = pore solution concentration at the surface.

A breakthrough time technique was used to solve Eq. (2) as described by Halamickova et al. [13] and later by McGrath and Hooton [8]. Breakthrough time was assumed to be at the first reliable inflection of the anode chamber chloride concentration, i.e., $C/C_0 = 0.003$.

4.1.2. Steady state migration coefficient

When steady state analysis is used and the diffusion component is taken to be small, Eq. (1) reduces to:

$$J = D\frac{zF\Delta VC}{RTl}. \tag{3}$$

The terms in Eq. (3) are defined as explained for Eqs. (1) and (2).

The D value obtained from Eq. (3) is similar to "an effective diffusion coefficient in a steady state diffusion test in that it relates the pore solution concentration gradient to the flux based on the total sample cross section area and it is

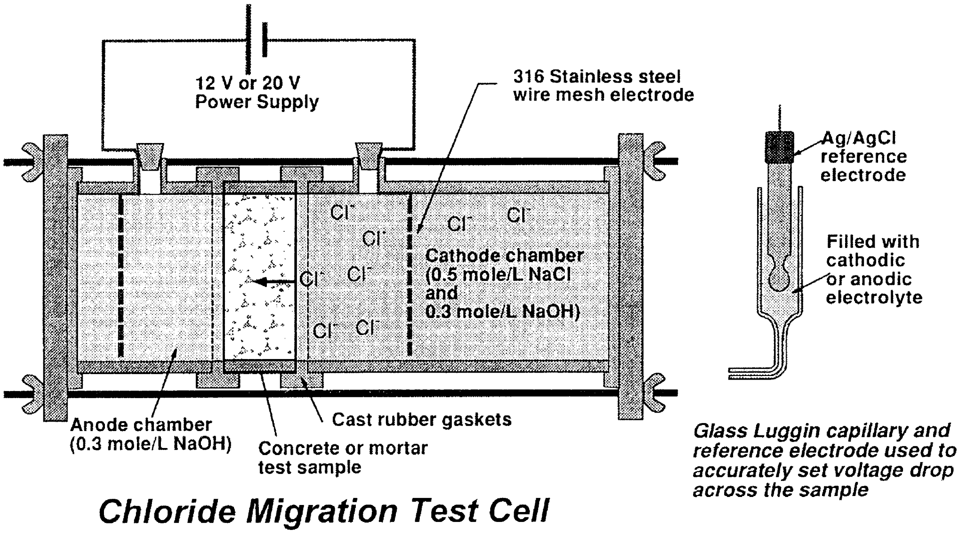

Fig. 1. Chloride conduction cell and luggin capillary with reference electrode.

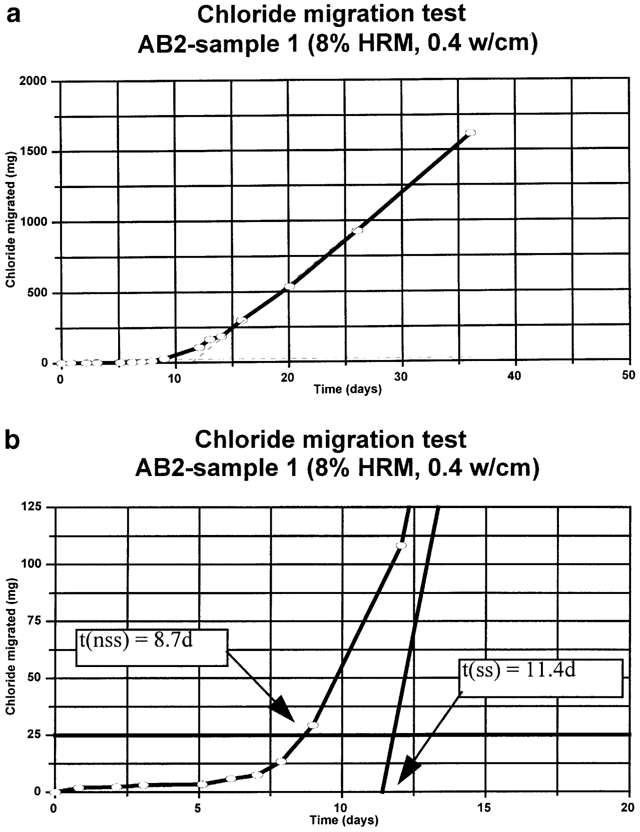

Fig. 2. (a) Typical plot of results from chloride migration test. (b) Schematic of determination of t(SS) and t(NSS).

assumed that chemical reaction sites have been filled, i.e. steady state conditions" [8]. This diffusion value is related to pore solution concentrations and pore area fractions, and, as such, a correction factor for pore area fraction must be made in order to make comparisons with nonsteady state migration coefficients [8]. The D values determined using Eq. (3) were divided by porosity volume fraction (n) as a simple approximation of pore area fraction.

A sample analysis of the data obtained from the chloride migration test is shown in Fig. 2a and b.

4.2. Results

Table 4 and Fig. 3 show the chloride migration coefficients for the concretes studied, using both steady state (D_{MSS}) and nonsteady state (D_{MNSS}) analysis. The results presented are based on the average obtained from two replicate samples tested for each mixture. Good reproducibility was obtained, as is shown in Fig. 4, which compares the results for each sample of the 0.40 w/cm mixtures.

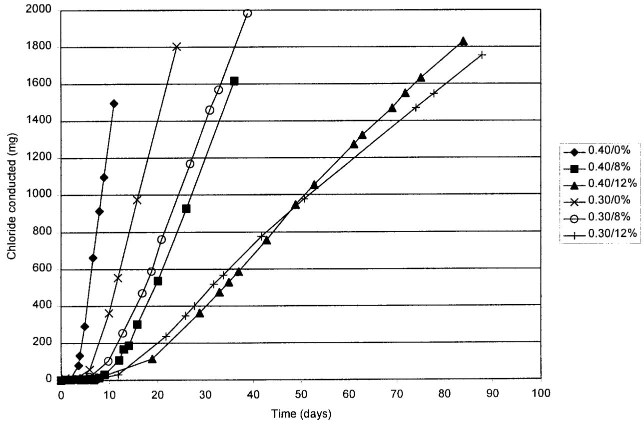

Fig. 3. Chloride migration test results.

The values presented in Table 4 show that the lowest value for D_{M}, using either steady or nonsteady state analysis, was obtained with the 0.30 w/cm, 12% HRM mixture. Overall, the migration coefficients also decreased with a decrease in w/cm and an increase in HRM content.

Fig. 3 shows that for the OPC control mixtures tested, the w/cm makes a significant difference in the resulting steady state and nonsteady state migration coefficient. When 8% or 12% HRM were included in the mixtures, a decrease in w/cm had almost no impact on the concrete's abilities to resist migration of chloride ions. However, at the same w/cm an increase in the HRM content of a mixture resulted in a greater decrease in both the D_{MNSS} and D_{MSS} coefficients.

5. Resistance to chloride diffusion

5.1. Test procedures

The apparent diffusion coefficient for each concrete mixture was determined using a bulk diffusion test [15]. Each 50-mm thick, 100-mm diameter sample was coated with sand-filled epoxy on all sides except the

Table 4
Chloride migration coefficient results

Mixture	Voltage (V)	Porosity (n)	D_{MNSS} ($\mathrm{m^2/s} \times 10^{-12}$)	t(NSS) (days)	D_{MSS} ($\mathrm{m^2/s} \times 10^{-12}$)	t(SS) (days)
0.4 w/cm, 0% HRM	12	0.157	9.91	2.0	7.06	3.3
0.4 w/cm, 8% HRM	12	0.130	2.18	8.6	2.03	10.8
0.4 w/cm, 12% HRM	12	0.112	2.08	9.0	1.19	12.6
0.3 w/cm, 0% HRM	12	0.128	5.77	3.6	4.63	6.2
0.3 w/cm, 8% HRM	20	0.104	1.78	6.2	2.05	10.0
0.3 w/cm, 12% HRM	20	0.100	1.12	11.0	0.89	12.8

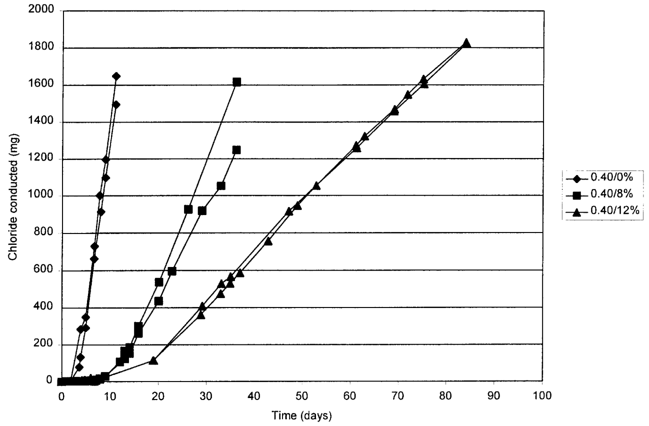

Fig. 4. Reproducibility of chloride migration test.

test face. The samples were then vacuum saturated in water as per ASTM C1202 (3 h in vacuum dry, water admitted under vacuum and maintained for 1 h, followed by immersion in water at atmospheric pressure for 18 h). At 28 days of age, they were sealed in plastic containers with the test face exposed to 1.0 mol/l NaCl solution at 23°C, for periods ranging from 28 to 1095 days (3 years). Because the samples are initially saturated and then submerged in chloride solution, this test measures the chloride transport solely as a result of pure diffusion. The test does not, however, correct for chloride binding effects as total (acid-soluble) chloride contents are measured. The beneficial effects of HRM in increasing chloride binding are presented elsewhere [16].

Two replicate samples were tested for each mixture. After removal from solution, the samples were profile ground on a milling machine using a 50-mm diameter diamond drill bit. Ten to twelve 0.5-mm layers were collected at different depths to a maximum of 25 mm. The powdered samples were dried in an oven at 105°C for 24 h. Subsequently, the samples were put through a nitric acid digestion process, and then filtered to remove excess solids, as explained in detail by McGrath [15]. Determination of the total chloride content at the different depths was made using automatic potentiometric silver-nitrate titration of the remaining liquid.

The resulting plot of chloride content versus depth was fit with Fick's Second Law of diffusion using Crank's solution to determine the apparent diffusion coefficient, D_a, and surface concentration, C_0. These relationships are listed as Eqs. (4) and (5), respectively:

$$\frac{dC}{dt} = D_a \frac{d^2C}{dx^2} \tag{4}$$

$$C(x,t) = C_0\left[1 - \mathrm{erf}\left(\frac{x}{2\sqrt{D_a t}}\right)\right] \tag{5}$$

where: C_0 = surface chloride concentration, C = the chloride concentration at depth, x, and time, t, and erfc() = standard error function.

It should be further noted that the background chloride concentration, that is the chloride in the original mix ingredients, was subtracted from the chloride concentrations determined at all depths. Largely due to the limestone coarse aggregate, background concentrations of acid-soluble chloride ranged from 650 to 750 ppm. Also, Jandell-Table Curve, a software program, was used to determine C_0 and D_a from the best curve fit to the data using a nonlinear, least-squares method. The best fit was determined by adjusting both of the unknowns to achieve the highest coefficient of determination, r^2.

6. Results

Table 5 and Fig. 5 show the bulk diffusion coefficients (D_a) and surface chloride concentrations at various exposure times up to 3 years.

After 3 years of ponding, the diffusion coefficients continue to decrease for all mixtures, as was expected due to further hydration with time and the resulting infilling of the porosity. As well, the HRM concretes would be expected to show decreased chloride penetration due to increased chloride-binding capacity [15]. The best performance at all ages was exhibited by the 0.30 w/cm, 12% HRM concrete. It should also be noted that, at all ages, replacing 8% cement with HRM in a 0.40 w/cm concrete improved diffusion characteristics as much or more than only reducing the w/cm to 0.30. For

Table 5
Bulk diffusion ponding test results

	Bulk diffusion coefficient ($\times 10^{-12}$ m^2/s) and surface concentration (%) for various exposure times									
	28 days		90 days		140 days		365 days		1095 days	
Mix design	C_0	D_a	C_0	D_a	C_0	D_a	C_0	D_a	C_0	D_a
1 — 0.40 w/cm, 0% HRM	0.33	19.1	0.34	11.8	0.64	6.1	0.63	6.3	–	–
2 — 0.40 w/cm, 8% HRM	0.69	8.2	0.58	5.7	0.61	3.8	0.68	2.3	0.73	1.7
3 — 0.40 w/cm, 12% HRM	0.58	5.8	0.53	3.5	0.60	3.2	0.58	1.7	0.68	1.0
4 — 0.30 w/cm, 0% HRM	0.42	9.6	0.44	5.9	0.56	5.4	–	–	0.52	3.4
5 — 0.30 w/cm, 8% HRM	0.52	3.7	0.49	2.9	0.72	2.2	0.83	1.4	0.76	0.8
6 — 0.30 w/cm, 12% HRM	0.50	2.9	0.48	2.7	0.73	1.5	0.69	0.9	0.69	0.5

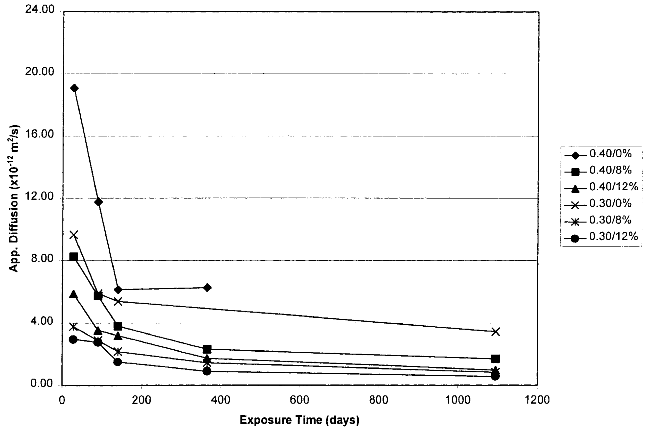

Fig. 5. Change in apparent diffusion coefficients of HRM concretes with time.

exposure times of 1 and 3 years, the 0.40 w/cm, 12% HRM mixture performed similar to the 0.3 w/cm, 8% HRM mixture.

The results in Fig. 5 show clearly that diffusion coefficient values decrease with time. This fact has important implications on service life predictions. The incorporation of the time dependence of diffusion coefficients in chloride diffusion models used to predict long-term chloride concentrations should lead to greater accuracy [17].

It is well known that transport by diffusion in saturated concrete occurs in the presence of a chloride concentration gradient, created when at least one face is continuously exposed to water and salt. The rate of ion diffusion through concrete can be represented by the following relationship [18]:

$$D(t) = D_{\mathrm{ref}}\left(\frac{t_{\mathrm{ref}}}{t}\right)^{m}. \tag{6}$$

The m coefficient in Eq. (6) is a constant that depends on mix proportions and has been developed to account for the rate of reduction of diffusivity. Further research to properly quantify this parameter would continue to improve the accuracy of service life predictions.

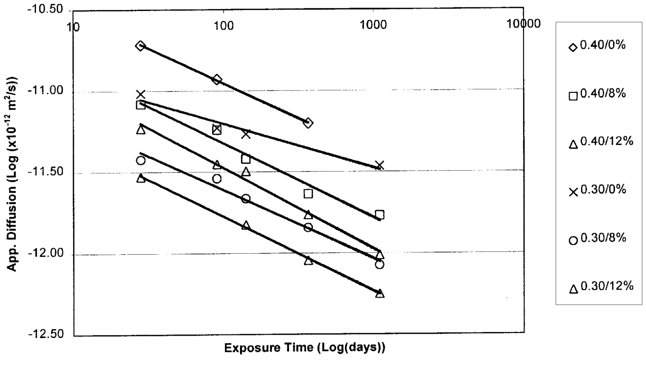

Fig. 6. Time dependent diffusion coefficients for HRM concretes.

Table 6
Summary of m values for HRM concretes

Mix design	m value	r^2
0.40 w/cm — 0% HRM	0.43	.99
0.40 w/cm — 8% HRM	0.44	.98
0.40 w/cm — 12% HRM	0.50	.99
0.30 w/cm — 0% HRM	0.27	.96
0.30 w/cm — 8% HRM	0.38	.96
0.30 w/cm — 12% HRM	0.46	.99

Fig. 6 and Table 6 show the results for the time-dependent diffusion coefficients, or m values, for the HRM concretes studied in this program. Diffusion values were first plotted with their respective exposure times on a log–log scale. The m values were determined by a linear regression analysis of the data. Good linear fits were found with r^2 values ranging from .96 to .99. It should be noted that alternative methods for determining m values using instantaneous diffusion values at different ages have been proposed by other workers [19].

The m coefficient determined for the 0.40/0% OPC mixture appears high compared to other reported values such as those from Bamforth (OPC: $m = 0.17$) [20], and Tang and Nilsson (OPC with w/cm of 0.7: $m = 0.25$; OPC with w/cm of 0.32: $m = 0.31$) [21]. The data for the 0.30 w/cm mixtures appear consistent and indicate that increasing the replacement level of HRM results in a corresponding increase in the reduction coefficient, m.

7. Conclusions

(1) Both resistivity and coulomb values of concretes were greatly improved with inclusion of HRM.

(2) Diffusion coefficients obtained from migration tests also showed the benefits of increasing HRM replacement levels of portland cement.

(3) Bulk diffusion values continue to reduce (improve) with increased periods of chloride exposure and support the beneficial effects of HRM relative to 100% OPC mixtures.

(4) The time-dependent reduction in apparent diffusion coefficients appears to be more pronounced with increased levels of HRM. This would have a significant beneficial effect on time-to-corrosion of reinforced concrete in chloride environments.

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