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Influence of high-temperature and low-humidity curing on chloride penetration in blended cement concrete

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Abstract

The influence of high-temperature and low-humidity curing on chloride penetration in concrete containing cement replacement materials was investigated. Three different mixes were studied: a control mix in which no cement replacement materials were added and two mixes where cement was partially replaced by 20% fly ash and 9% silica fume (by weight), respectively, at a constant water-to-binder ratio of 0.45. High-temperature curing was employed to simulate concrete temperature in hot climate. The results show that at early periods of exposure, initial curing has a substantial influence on chloride penetration in concrete. The effect of initial curing is much reduced after a long period of exposure. The chloride penetration at early ages of exposure is directly related to the porosity of the binder phase and the absorption of concrete. Higher chloride penetration resistance was observed when cement is partially replaced with either fly ash or silica fume.

Keywords: Blended cement; Chloride; Curing; Diffusion; Temperature

1. Introduction

Initial curing to which concrete is subjected, prior to exposure to chloride environment, plays an important role in determining the chloride penetration rate in it, especially at early ages of exposure [1]. The durability of concrete depends largely on the extent to which the hydrates produced in cement paste fill the space originally occupied by water [2]. Drying of concrete particularly at the concrete surface, caused by a poor curing regime, leads to a restricted hydration in the surface layers and thus higher porosity and permeability [3].

A considerable volume of research has been conducted on chloride penetration of cement paste and concrete, with and without cement replacement materials [1,4–14]. Some researchers [4,5,11] have adopted curing regimes that allow maximum hydration to take place—i.e. specimens were immersed in water or sealed before the exposure to chloride environment. Some others [7,8] have adopted curing that simulates more realistic curing conditions. They found an increase in chloride uptake when 26% by weight of cement was replaced with fly ash [8]. Initial curing was conducted in air at ambient conditions for 14 days prior to exposure to chloride environment. On the other hand, it was found that replacing 15%, 30% and 40% of cement with fly ash yielded a much lower chloride intake when compared with pure cement paste [4]. Paste specimens were either stored in saturated calcium hydroxide solution for about 14 months or stored for 60 days in saturated air prior to the chloride diffusion test. The chloride diffusion rate was also found to be lower when 10% and 20% of cement is replaced with silica fume [4,15].

This paper investigates the effect of high-temperature curing at different relative humidities on chloride ingress characteristics into concrete. High-temperature curing was chosen to simulate temperatures in hot regions. The partial substitution of cement with either fly ash or silica fume on chloride penetration is considered. Correlation between the intruded pore volume of the paste and the chloride level in the concrete is investigated. The absorption values (by shallow immersion and capillary action), which were obtained in a previous investigation, are also correlated with chloride levels at the surface.

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2. Experimental

2.1. Mix proportions

Three concrete mixes listed in Table 1 were used to study the influence of initial curing regime on chloride penetration into concrete. The control mix A, in which ordinary Portland cement (OPC) was used, the proportion by weight was 1:2.0:3.4 with a water/cement ratio of 0.45 and a cement content of 350 kg/m^3. In Mixes B and C, 22% and 9% of cement was replaced with fly ash and silica fume, respectively. The proportion of fine aggregate (sand) was altered to compensate for the density variation of fly ash and silica fume. Superplasticiser and air entraining agent were added to all three mixes. The dosage of superplasticiser was 1.2% by weight of cementitious material (binder) for Mixes A and B and 1.5% for Mix C, which contained silica fume. The air entraining agent addition was 95 ml/50 kg of binder for Mixes A and C, whereas 118 ml was added to Mix B. The slump and air content for all mixes were 95 ± 10 mm and 3%, respectively.

2.2. Materials

Composition of the cement and cement replacement materials is given elsewhere [16,17]. The fine aggregate was unwashed glacial pit sand, which conformed to Category M of BS882. Unwashed crushed gravel coarse aggregate of 20 mm nominal size was used.

The superplasticiser is based on the sodium salt of copolymer of an unsaturated carboxylic acid and the alkyl of such an acid. It conforms to Types F and G materials of ASTM-C494. The air entraining agent is based on neutralised salts of wood resins and conforms to ASTM designation C260 for air entraining admixture.

2.3. Casting and curing

Prism specimens measuring 100 × 100 × 300 mm were cast in steel moulds. After 24 h, the specimens were demoulded. Three initial curing methods were employed, which are as follows.

(a) After casting, specimens were covered with wet burlap and plastic sheeting for 14 days at 37 °C temperature and 25% relative humidity. After that period, the covers were removed, and specimens were left to cure in air under the same conditions of temperature and humidity (i.e. 37 °C and 25% RH) for another 14 days. Specimens were designated as MA and are called moist-cured/air-cured at high temperature.

(b) After casting, specimens were left to cure in air at 37 °C and 25% RH for 28 days. Specimens were designated as AH and are called air-cured at high temperature.

(c) Specimens were left to cure in the laboratory under ambient conditions after casting for 28 days. The temperature was fairly controlled at 20 °C, but humidity was uncontrolled and generally ranged between 60% and 80%. Specimens were designated as AA and are called air-cured at ambient temperature.

2.4. Specimen preparation and testing

After an initial curing period of 26 days, specimens were sealed on all faces except one face in order to ensure that chloride penetrated from one face only. Specimens were left to dry for 2 more days in their environmental conditions (i.e. MA, AH and AA). After 28 days from casting, specimens were then immersed in a 3-M (175 g/l) sodium chloride solution. The temperature of the chloride solution was 37 °C for initially AH- and MA-cured specimens and ambient (approximately 20 °C) for AA-cured specimens. The chloride concentration test was conducted after 28 days, 6 months and 12 months of immersing the specimens in chloride solutions.

After the eighth month of immersing the specimens in the chloride solution at 37 °C, the temperature was increased to 45 °C due to transfer of the research project to a new laboratory accommodation.

After the required period of immersing the specimens in the chloride solution (i.e. 28 days, 6 months and 12 months), they were taken out of the solution and were cut into two halves. Powder samples were then taken at different depths from the concrete surface.

The chloride content at various depths was determined by chemical analysis on powder samples obtained from the

Table 1
Details of concrete mixes

Mix	OPC (kg/m^3)	Fly ash (kg/m^3)	SF[a] (kg/m^3)	Water (kg/m^3)	FA[b] (kg/m^3)	CA[c] (kg/m^3)	SP[d]	AEA[e]
A	350	–	–	157.7	708.6	1201.4	1.2	95
B	273	77	–	157.7	687.1	1201.4	1.2	118
C	318.5	–	31.5	157.5	698.6	1201.4	1.5	95

[a] Silica fume.

[b] Fine aggregate.

[c] Coarse aggregate.

[d] Superplasticiser (% by weight of binder).

[e] Air-entraining agent (ml/50 kg binder).

concrete specimens. The concentration of chloride was determined by Mohr's titration [18]. The chloride content, expressed as percent by weight of cementitious materials (binder), was calculated from the following formula [19,20]:

$$\%\mathrm{Cl}^{-} = \frac{35.453 V_s N_s (1 + A_g)}{10 W_p}$$

where $C1^{-}$ is the chloride content expressed in percent by weight of binder, V_s is the volume of $AgNO_3$ used in milliliters to neutralise $C1^{-}$, N_s is the normality of $AgNO_3$ (N), W_p is the weight of powder sample (g) and A_g is the proportion of aggregate in the concrete mix.

Further details on the initial curing methods, sample preparation and testing are reported elsewhere [21].

3. Results and discussion

3.1. Chloride diffusion profile

The rate of chloride penetration into concrete as a function of depth from the concrete surface and time can reasonably be represented by Fick's Law of diffusion [22], which is given by the following expression:

$$\frac{\delta C}{\delta t} = D_c \frac{\delta^2 C}{\delta x^2}$$

Solving the above differential equation gives:

$$C_{(x,t)} = C_0 \left[1 - \mathrm{erf}\left(\frac{x}{2\sqrt{D_c t}} \right) \right]$$

where $C_{(x,t)}$ is the chloride ion concentration at a distance x (cm) from the concrete surface after time t (s), C_0 is the equilibrium chloride concentration on the concrete surface, D_c is the chloride diffusion coefficient in cm^2/s and erf is the error function.

In this investigation, the experimental data, which represent the chloride concentration with depth, were analysed by a computer using a BMDP statistical package. The best-fit diffusion curve was, therefore, obtained, and the values of C_0 and D_c were determined.

Although concrete properties (e.g. capillary suction) can vary depending on the depth from the exposed surface, however, the above equation assumes that D_c is constant at a particular period of exposure to chlorides and does not vary with depth.

3.2. Chloride ingress after 28 days

Chloride penetration profiles after 28 days of exposure to chloride solutions are shown in Figs. 1–3 for the control mix (Mix A), 22% fly ash mix (Mix B) and 9% silica fume mix (Mix C) for specimens initially subjected to AH, MA and AA curing regimes, respectively. After 28 days of exposure to the chloride solution, initially air-cured specimens at high temperature (AH) display higher chloride uptake in comparison with initially moist-cured/air-cured specimens at high temperature (MA). This is due to the fact that dry curing at high temperature leads to relatively higher loss of water from the surface of specimens and restricted hydration. Therefore, higher porosity and permeability are expected in specimens subjected to AH curing as compared with initially moist-cured specimens. The lowest chloride uptake is found in initially moist-cured specimens at high temperature (MA). Initially air-cured specimens at ambient temperature (AM) exhibit intermediate values that range between the values obtained for initially AH- and CH-cured

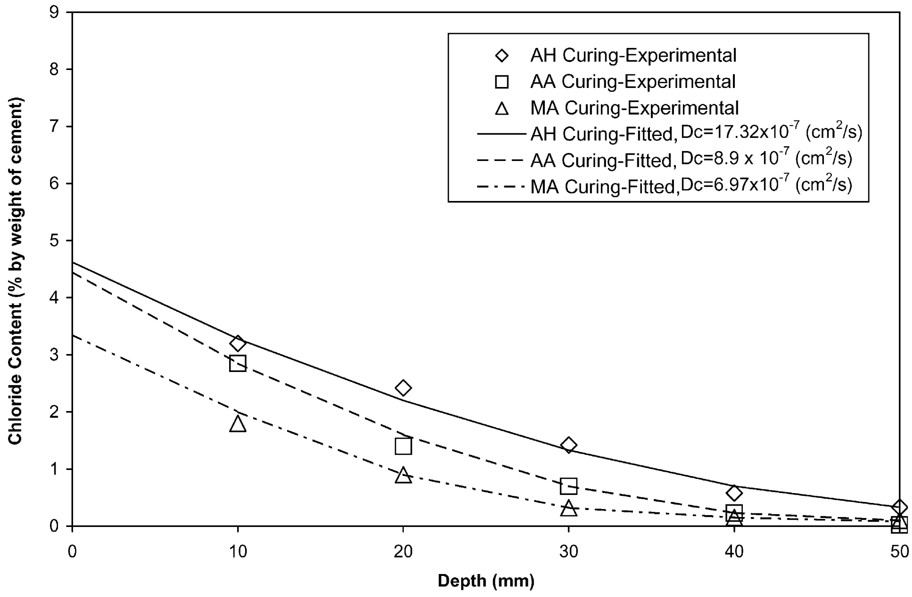

Fig. 1. Chloride diffusion profiles for Mix A exposed to different initial curing regimes after 28 days of exposure to chloride.

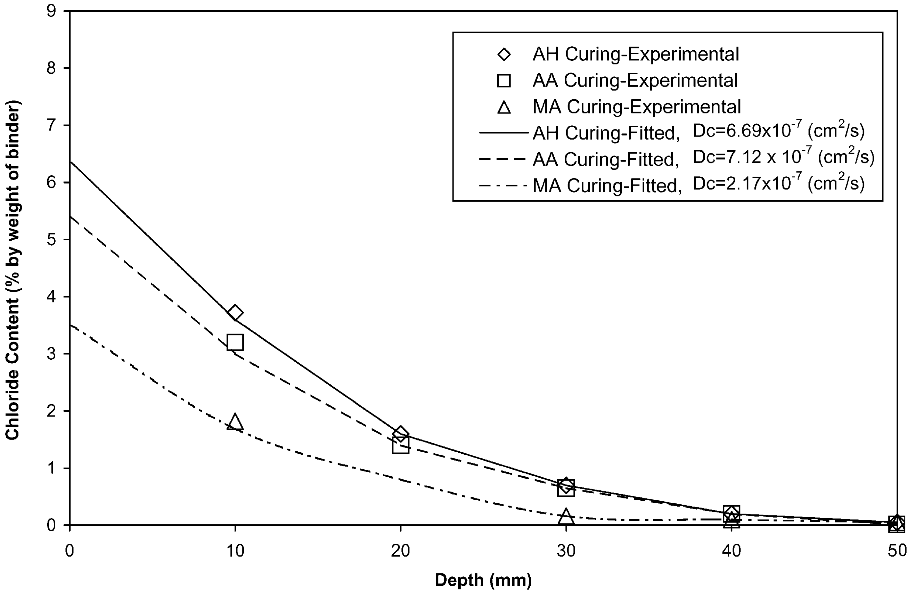

Fig. 2. Chloride diffusion profiles for Mix B exposed to different initial curing regimes after 28 days of exposure to chloride.

specimens. Chloride levels at depths remote from the concrete surface (i.e. depths more than 40 mm) are quite low, and the curing methods employed do not show a marked difference in terms of chloride uptake.

The chloride diffusion coefficient, which was determined by applying Fick's Law of diffusion to the experimental data, is affected by the initial curing conditions in a similar way to chloride penetration profiles. Specimens subjected to initially high-temperature air curing (AH) showed the highest values. The lowest values of D_c are for specimens subjected to MA curing. Initially ambient-cured specimens yield values of D_c between those obtained for AH- and MA-cured specimens.

The partial replacement of cement with cement replacement materials is quite beneficial, in that lower values of chloride levels are obtained as compared with the control mix especially at depths beyond 10 mm from the surface. In concrete containing 22% fly ash (Fig. 2), the chloride contents are generally lower than those of the control mix, except for chloride levels at 10 mm from the concrete surface for AH- and AA-cured specimens where slightly higher values are obtained. The fly ash particles near the concrete surface underwent little hydration due to the lack of availability of water especially for specimens subjected to dry curing and high temperature. The partial substitution of cement with silica fume is even more beneficial in reducing

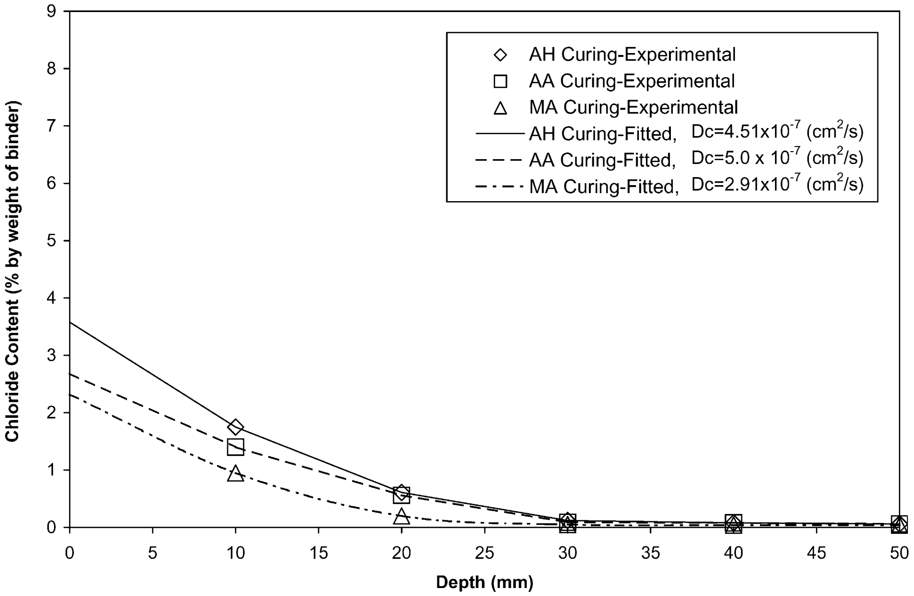

Fig. 3. Chloride diffusion profiles for Mix C exposed to different initial curing regimes after 28 days of exposure to chloride.

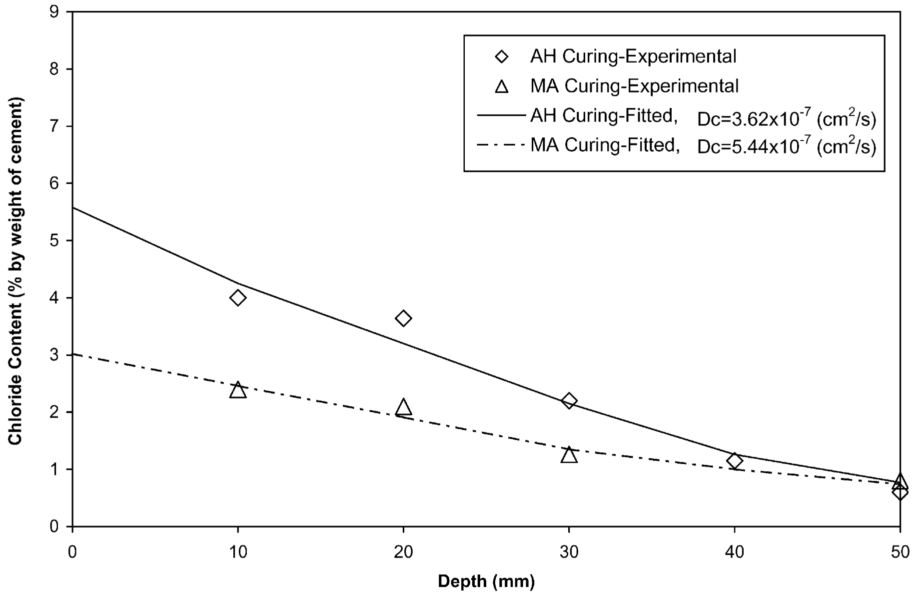

Fig. 4. Chloride diffusion profiles for Mix A exposed to different initial curing regimes after 6 months of exposure to chloride.

chloride penetration as shown in Fig 3. The D_c values for concrete containing silica fume or fly ash are considerably lower than those obtained for the control mix. The improved resistance to chloride penetration due to replacing cement with either fly ash or silica fume supports results reported elsewhere [11–13].

3.3. Chloride ingress after 6 months

After 6 months of exposure of specimens to the chloride solution, initially AH-cured specimens still exhibit higher chloride uptake in comparison with CH specimens. This is illustrated in Figs. 4–6, where the chloride penetration profiles after 6 months of immersion in the chloride solution, are plotted for Mixes A, B and C, respectively, for initially AH- and MA-cured specimens. As can be expected, the values of chloride contents are higher after 6 months of immersion than those after 28 days of immersion.

Generally, concretes containing cement replacement materials (i.e. fly ash or silica fume) showed lower values of chloride ingress when compared with control concrete, especially at depths beyond 20 mm from the concrete surface. The values of the diffusion coefficient (D_c), which are shown in the figures are systematically lower after

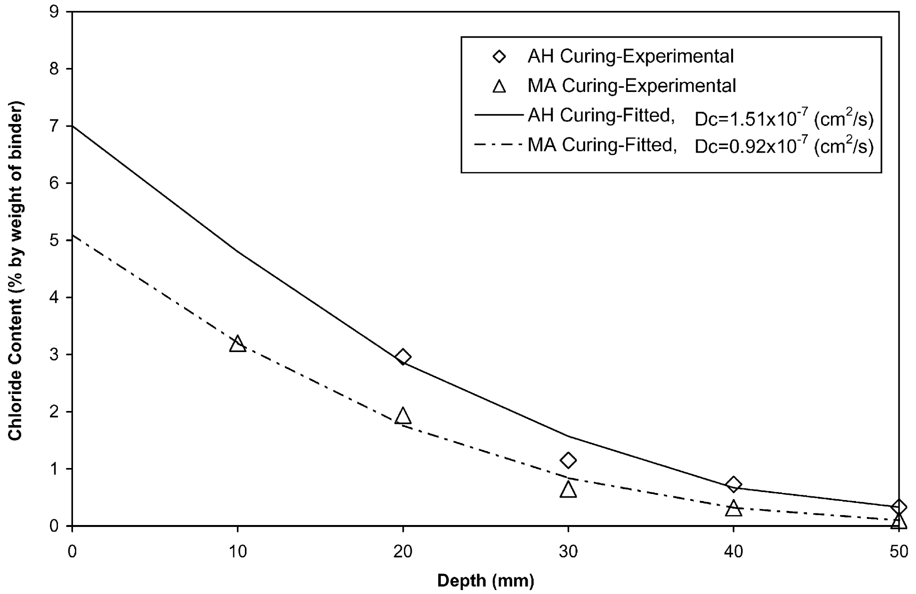

Fig. 5. Chloride diffusion profiles for Mix B exposed to different initial curing regimes after 6 months of exposure to chloride.

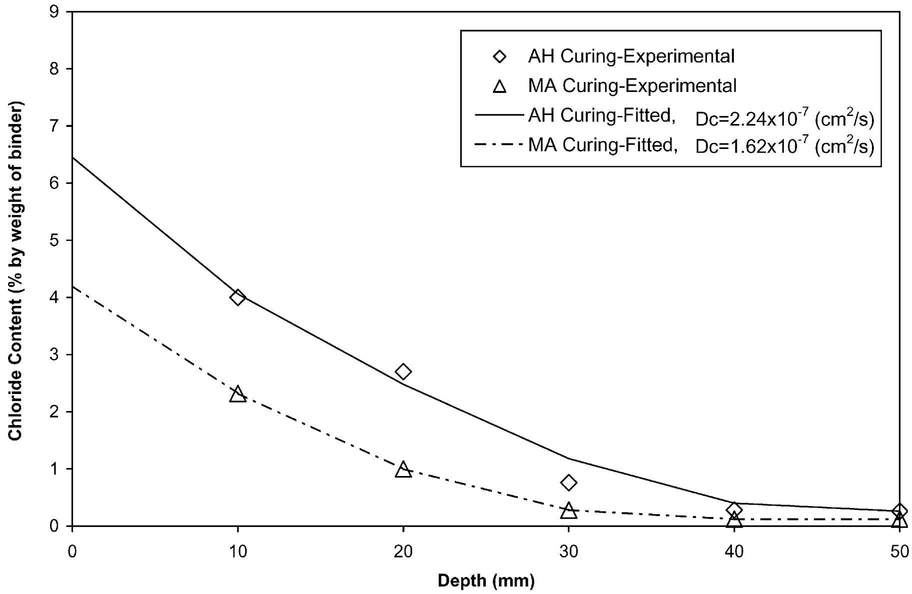

Fig. 6. Chloride diffusion profiles for Mix C exposed to different initial curing regimes after 6 months of exposure to chloride.

6 months of exposure as compared with the values after 28 days of exposure for all mixes considered in this investigation. This seems to be independent of the initial curing regime and is also independent of the presence or absence of cement replacement materials.

3.4. Chloride ingress after 12 months

Results of chloride ingress after 12 months of immersion in the chloride solution are shown in Figs. 7–9 for Mixes A, B and C, respectively, for specimens subjected to AH, MA and AA curing. The results show that the initial curing regime does not have a clear influence on the chloride level after the long immersion period of 12 months. In some cases (e.g. Fig. 8), the chloride contents are even higher for MA-cured specimens than AH-cured specimens. The chloride contents after 12 months of exposure are higher than those obtained after 28 days of exposure for all mixes considered. The trend is not clear for specimens immersed in the chloride solution for 6 and 12 months. In some cases, the chloride levels after 12 months of exposure are slightly lower than those after 6 months of exposure.

The partial replacement of cement with cement replacement materials is shown to have a beneficial influence. The

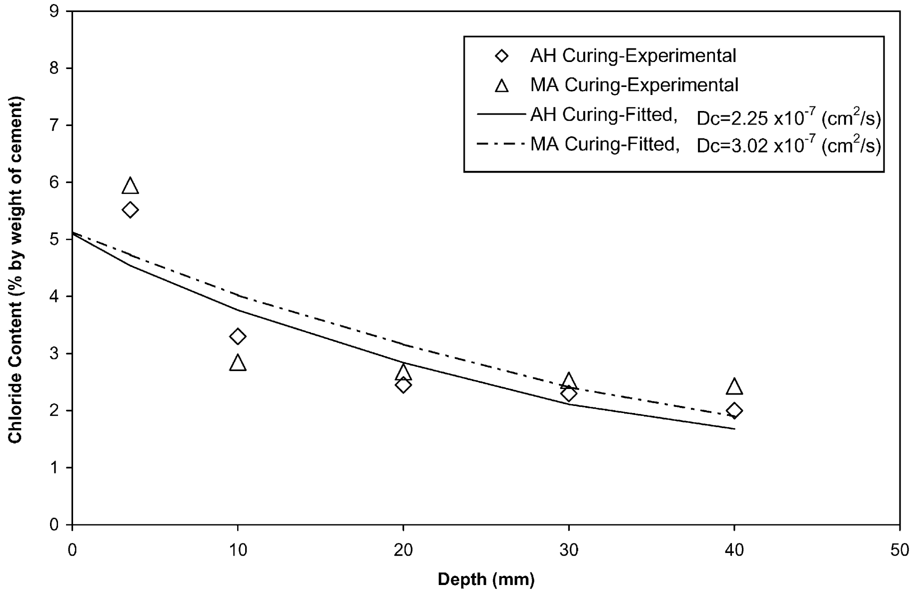

Fig. 7. Chloride diffusion profiles for Mix A exposed to different initial curing regimes after 12 months of exposure to chloride.

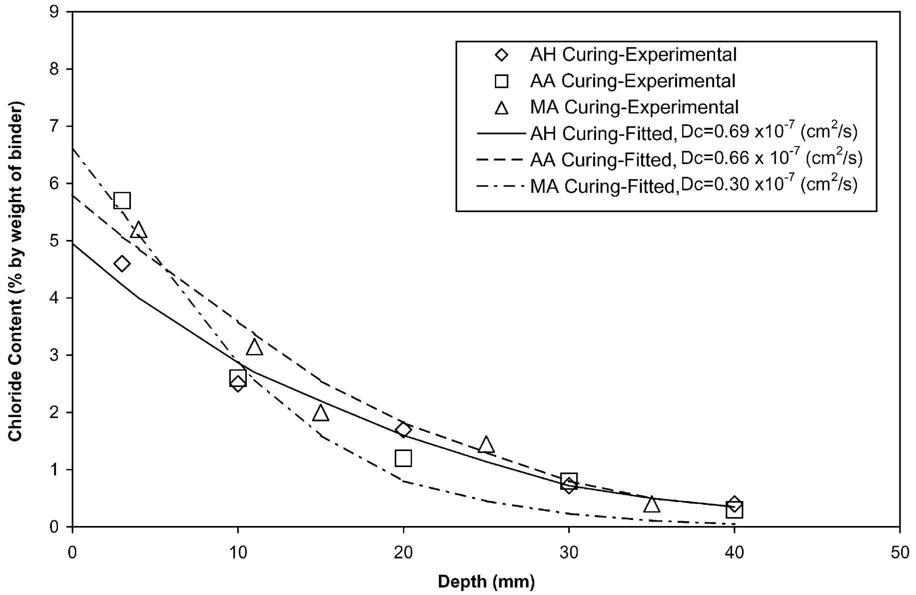

Fig. 8. Chloride diffusion profiles for Mix B exposed to different initial curing regimes after 12 months of exposure to chloride.

chloride uptake of the control mix increases with depth of penetration after 12 months of exposure as compared with 28 days and 6 months of exposure. The control mix still shows a considerable chloride concentration even at large depth (e.g. 40 mm from the concrete surface). Mixes containing fly ash or silica fume, however, show much smaller chloride uptakes than the control mix, at depths beyond 20 mm from the surface. This supports the previous argument that it is just a matter of time before a detrimental amount of chloride reaches the embedded steel even through high-quality concrete when ordinary Portland cement is used in the mix without cement replacement materials [9].

3.5. Variation of D_c with duration of exposure

The variation of D_c with duration of exposure to the chloride solution is presented in Fig. 10 for all mixes and all curing methods. The diffusion coefficient values (D_c) are obtained by applying Fick's law of diffusion to the experimental data of this investigation. It is clear that D_c decreases systematically with the period of exposure. This seems to be the case regardless of the curing regime adopted or the presence of cement replacement materials. The decrease in D_c with the increase in the exposure period to chloride environment is in agreement with results repor-

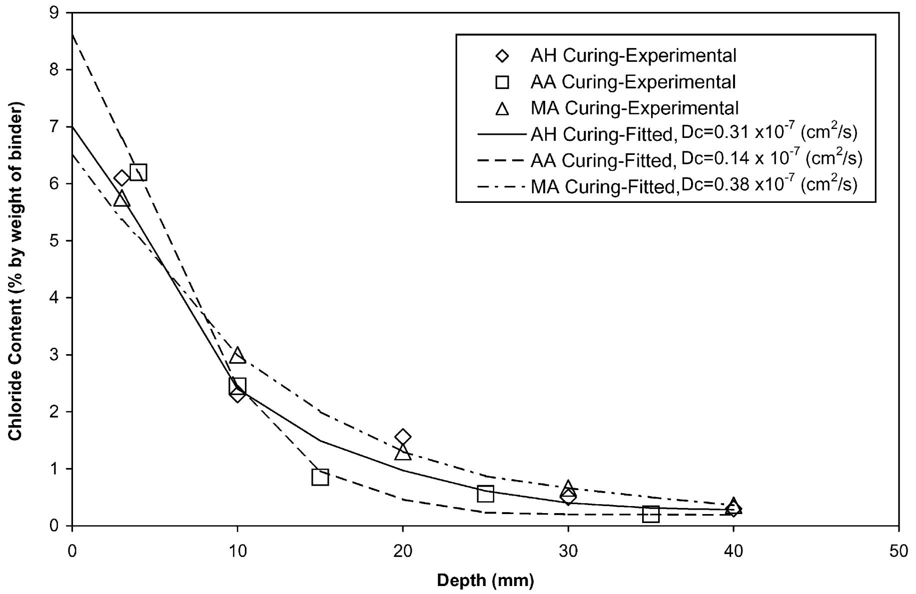

Fig. 9. Chloride diffusion profiles for Mix C exposed to different initial curing regimes after 12 months of exposure to chloride.

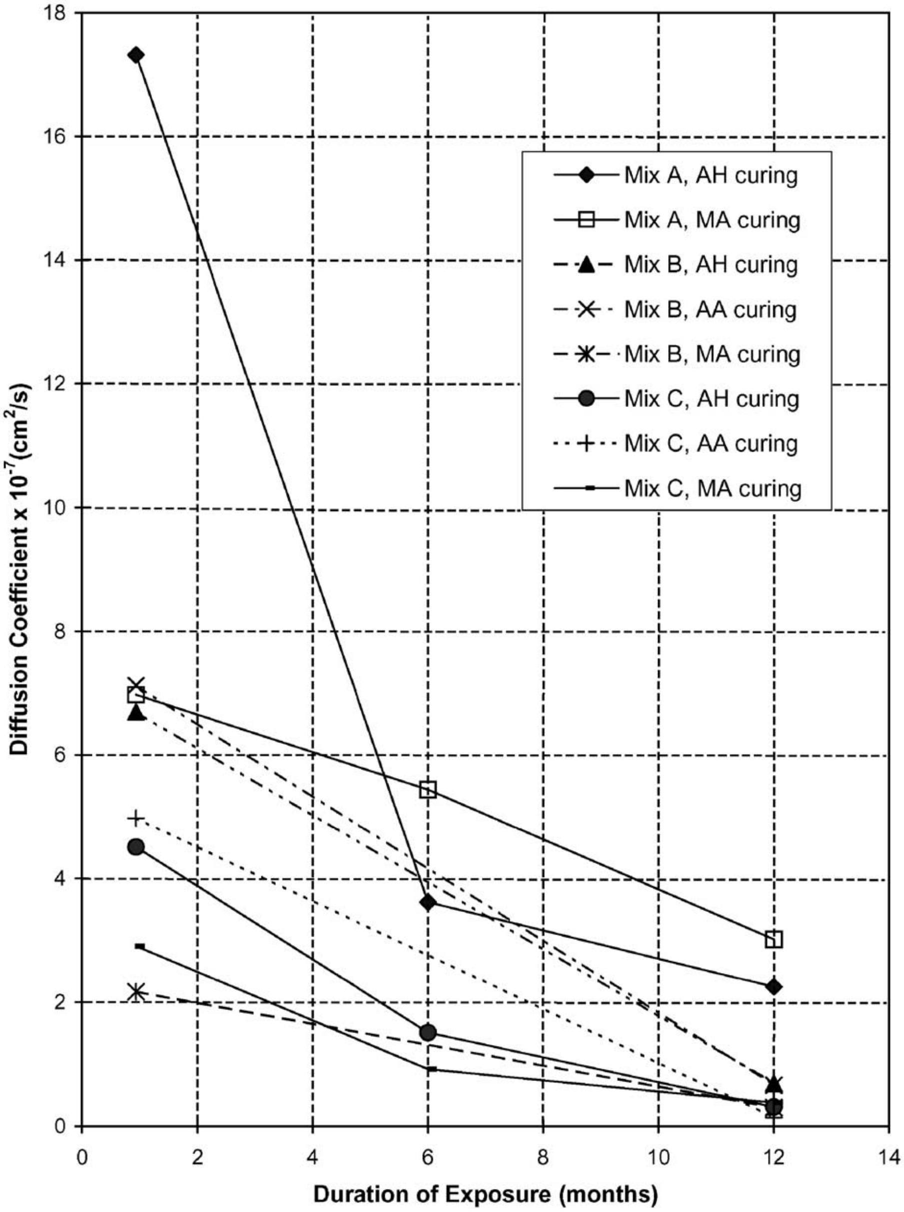

Fig. 10. Variation of chloride diffusion coefficient with duration of exposure.

ted elsewhere [10,14]. This decrease in the values of D_c is partly attributed to the ongoing production of hydrated gel, which leads to a reduction in permeability and hence less chloride penetration.

Based on the results of Fig. 10, the variation of D_c with time can be described using the following equation:

$$D_{(c,t)} = at^{-b}$$

where $D_{(c,t)}$ is the diffusion coefficient after time t, a is related to the diffusion coefficient at early ages of exposure (i.e. 28 days in the case of the present investigation) and b is an empirical constant. Mangat and Molloy [10] used a similar equation for the prediction of the long-term diffusion coefficient as a function of the diffusion coefficient after 1 s of exposure. Different values of a and b for each of the concrete mixes used were suggested.

3.6. Variation of C_0 with duration of exposure

The variation of the equilibrium chloride concentration on the concrete surface (C_0) is presented in Fig. 11. The values of C_0 were obtained from the chloride diffusion profiles (Figs. 1–9)—i.e. the intercept of the best-fit curve with the vertical axis. Generally, the results suggest that C_0 tends to increase with the duration of exposure. This increase with time is in agreement with results reported elsewhere [9,23]. However, some others [7,10] found that

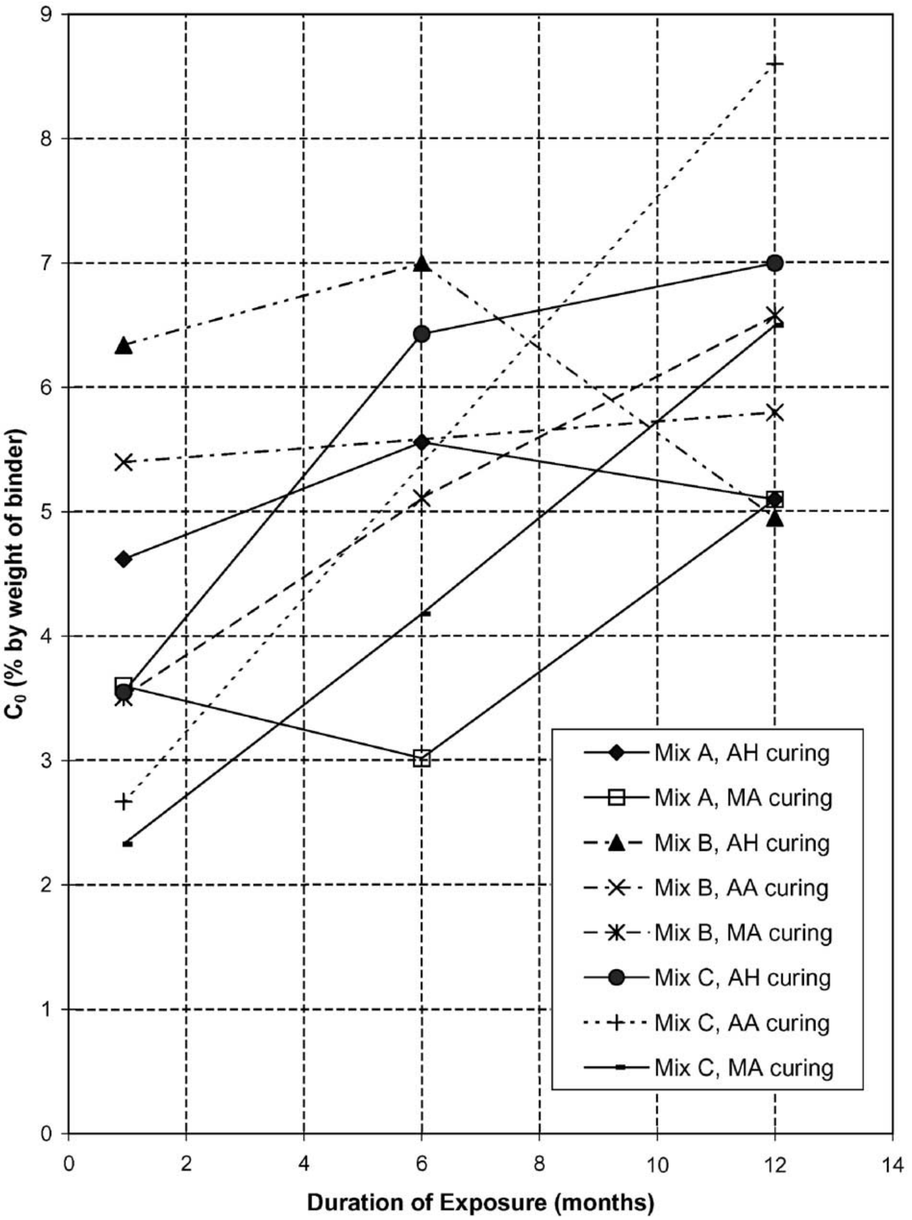

Fig. 11. Variation of chloride concentration on the concrete surface with duration of exposure.

C_0 remains constant with the duration of exposure. This can be due to the method of exposure, where specimens were subjected to alternate wetting and drying to simulate concrete in the tidal zone [10]. In the case of this investigation, specimens were continuously immersed in the chloride solution.

3.7. Curing, porosity and chloride ingress

Table 2 presents the results on pore volume and the proportions of large pore volumes (i.e. pores whose diameters > 0.1μm) and small pore volumes (i.e. pores whose diameters < 0.1μm) for pastes representing the binder phase of the concrete mixes for specimens subjected to AH and MA curing. Details on paste preparation, curing and testing are given in Ref. [17]. The absorption values of the concrete mixes subjected to similar curing are also presented in the table. Two parameters of water absorption were reported, viz. water absorption (WA) obtained by shallow immersion and water absorption coefficient (WAC) obtained by capillary suction. Further details about testing and curing are given in Ref. [24]. In addition, the values of the chloride diffusion coefficient (D_c) are included for these mixes (i.e. Mixes A, B and C) after 28 days, 6 months and 12 months of exposure to the chloride solution for specimens subjected to AH and MA curing.

The data obtained show that higher intruded pore volume and higher proportions of large pores result in higher chloride penetration at early age of exposure. For example, the intruded pore volumes of the control paste are 0.178 and

Table 2
Relation among porosity, absorption and chloride characteristics

		Paste			Concrete							
		P_v^a	Pores (% of P_v)				$D_c \times 10^{-7}$ (cm^2/s)			Chloride concentration (%)[d]		
Mix	Curing	(cm^3/g)	Large	Small	WA[b]	WAC[c]	After 28 days	After 6 months	After 12 months	After 28 days	After 6 months	After 12 months
A	AH	0.178	65.4	34.6	6.5	33	17.32	3.62	2.25	3.3	4.0	3.3
A	MA	0.135	38.7	61.3	5.8	22	6.97	5.44	3.02	2.0	2.4	2.85
B	AH	0.215	79.8	20.2	6.2	27	6.69	1.51	0.69	3.7	–	2.5
B	MA	0.156	17.2	82.8	5.4	23	2.17	0.92	0.30	1.84	3.2	3.15
C	AH	0.236	69.5	30.5	6.5	31	4.51	2.24	0.31	1.76	4.02	2.3
C	MA	0.187	18.4	81.6	6.0	20.0	2.91	1.62	0.38	0.95	2.32	3.0

[a] Pore volume of paste.

[b] Water absorption (% by weight of concrete).

[c] Water absorption coefficient $\times 10^{-6}$ (g/mm^2 s$^{0.5}$).

[d] Chloride concentration (% by weight of binder) at 10 mm from the concrete surface.

0.135 cm^3/g for initially AH and MA specimens, respectively. This corresponds to chloride levels at 10 mm depth of 3.3 and 2.0% by weight of cement, respectively, after 28 days of exposure to chloride, which indicates that higher intruded pore volume of the paste leads to higher chloride ingress. Similar cases are found after 6 months of exposure to the chloride solution. For example, the 9% silica fume mix shows that a higher intruded pore volume of silica fume paste for specimens subjected to AH when compared with MA-cured specimens results in a higher chloride ingress after 28 days and 6 months of exposure. However, the intruded pore volume of the silica fume paste is larger than that of the control paste, yet, the chloride contents are less in the silica fume mix than the control mix. This is due to the fact that the presence of silica fume produces a denser, less permeable concrete at the aggregate–paste interface and therefore less levels of chloride.

The values of WA and WAC also indicate that they are directly related to the chloride penetration after 28 days and 6 months of exposure to chloride. Higher values of WA and WAC result in higher chloride uptake. In AH-cured specimens, more chloride uptake is expected at the concrete surface than that of MA-cured specimens, since the former have higher values of WA and WAC. After a long period of exposure (i.e. 12 months), the dependency of chloride ingress on initial absorption values is reduced.

The intruded pore volume of the paste is heavily dependent on the initial curing regime adopted, and so are the values of WA and WAC [17,24]. Since chloride ingress into concrete at early stages of exposure is dependent on intruded pore volume and absorption, therefore, the initial curing plays a role in determining the chloride penetration into concrete at these early stages. Thomas et al. [25] indicated that the penetration of chloride ions into initially dry-cured specimens during early exposure to chloride environment is controlled by a capillary suction or sorption mechanism rather than diffusion. This is in agreement with the results of the present investigation, in that chloride penetration during early exposure (28 days and 6 months) to the chloride solution is directly affected by capillary suction (WAC). In addition, others [1,26] reported that chloride penetration is clearly dependent on the initial curing regime at an early age of exposure. However, the curing regimes did not have any significant effect on chloride concentration at a later stage of exposure [1]. During the early period of exposure, chloride ions penetrate mainly into the surface layers. The porosity and capillary absorption of the skin layers are heavily dependent on the curing regime, especially under hot and dry conditions [1,26]. The deeper the chlorides diffuse into the concrete, the less they will be affected by the properties of the outer skin [1]. This explains the nonsignificant effect of curing regimes after long periods of exposure (e.g. the 12 months in this investigation). A better description of chloride penetration with depth may be achieved if the variation in concrete properties such as capillary sorption is taken into consideration.

4. Conclusions

(1) Initial curing conditions of concrete have a substantial influence on the rate of chloride penetration during the early periods of exposure to chloride environment. After a long period of exposure, the effect of initial curing on chloride penetration becomes insignificant.

(2) Concrete containing ordinary Portland cement only as the cementitious material is prone to an ongoing penetration of chloride. Chlorides penetrate deeper as these concretes are continuously exposed to chloride environment.

(3) The replacement of cement with 22% fly ash is quite beneficial in resisting chloride penetration into concrete especially at depths beyond 20 mm from the concrete surface. The diffusion coefficient of concrete containing fly ash is lower than concrete without fly ash.

(4) Replacing 9% of cement with silica fume leads to increased resistance to chloride penetration during the early ages of exposure to chloride. In addition, at later ages of exposure, concrete containing silica fume has lower chloride uptake, especially at depths beyond 10 mm from the surface

and negligible chloride levels at depths beyond 30 mm from the surface. The replacement of cement with silica fume reduces the diffusion coefficient.

(5) The intruded pore volume of paste is directly related to chloride penetration at early age of exposure. The larger the pore volume of the paste, the higher the chloride content, except in the case of silica fume mix where chloride levels were low, although the pore volume of the paste was larger than that of the control paste. This is due to the denser matrix at the paste–aggregate interface due to high surface area of silica fume particles.

(6) An increase in WA and WAC results in an increase in chloride penetration in the surface zone of concrete at early ages.

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