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Scientific paper

Proposal of Analysis Methods Based on Stagnation of Chloride Ion in Concrete

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Abstract

The service life deterioration of concrete is mainly a function of the quality of cover concrete. Whereas most past designers considered the cover size and diffusion property as the key quality parameters for durability design, whereas recent research has come to the conclusion that the water penetration front is another key issue regarding cover quality that dramatically affects the service life as well as LCC of RC structures.

In this study, Cl^- ion profiles, the absorption of water, and the liquid water front position along with its variation for two types of concrete, concrete from a bank protection structure located in Okinawa and slag concrete with 70% w/c ratio in the laboratory were examined. The methods of analysis were divided into three groups. Each type of concrete was analyzed with each of the methods and compared with the actual chloride profile data. It was found that optimum method of analysis depends on the type of concrete to be analyzed. A classification of concrete types along with their respective analysis methods that modifies Fick's law of diffusion based on the behavior of stagnation of the liquid water front in concrete is proposed.

1. Introduction

It is very difficult to predict the actual performance scenario of actual in-situ structures without carrying out the inspections. In this context, a structure situated in Okinawa was inspected and cores were taken to the laboratory to obtain the actual profile of chloride ingress. Immersion tests were conducted for 3 months. This work was done by Takahashi Yuya (Takahashi 2010) and led to the conclusion that water penetration depth is the governing position beyond which chloride does not penetrate. This statement was proved for two types of fly ash concrete but OPC did not show similar behavior. For this aspect immersion tests in salt and normal water were continued and a new parameter i.e. the variation of the front position along with the mean front position is found to be important.

One case of slag concrete having 70% w/c ratio was cast and tested in the laboratory to determine the Cl^- ion profile, absorption and liquid water front. Surprisingly, this group of concrete having high w/c ratio follows Fick's law with regard to Cl^- ion penetration. The Cl^- profiles obtained from the inspected cores and experiments suggest modifying Fick's law by the inclusion of liquid water front. Verification of Cl^- ion ingress was done with the mean value and standard deviation of the liquid water front- for the case of the inspected cores

from Okinawa.

A classification is proposed using three types of concrete (two types from Okinawa, and one cast in laboratory) based on Cl^- ion profiles with differing water penetration behaviors. It is also shown here that the adoption of suitable methods of analysis for these types of concrete can minimize the root mean square error between the actual data and the predicted data.

2. Background and purpose

The chloride profile of the cores taken from Okinawa and the immersion test results of the same clearly show that the liquid water front stops chloride ion penetration, as shown in **Figs. 1a** and **1b** (Takahashi 2010).

The chloride stopping effect by the liquid water front (w_f) will change the service life of a structure, as shown in **Fig. 2**. In this figure, the liquid water front varies from 2 to 10 cm and the concentration of chloride is computed for a depth of 6 cm. We can see a large difference in service life that will also affect the life cycle cost for concretes having different surface quality with respect to the liquid water front.

The influence of the water front on chloride movement and on service life motivates this study to establish a concept that allows the inclusion of the liquid water front in the prediction of chloride profile.

3. Concept

The conceptual framework to use the liquid water front in chloride profiling is described briefly in sections 3.1, 3.2 and 3.3.

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Fig. 1 Comparison between immersion test and real profile.

3.1 Model modification

Chloride diffusion into concrete, like any diffusion process, is controlled by Fick's Second Law for non-steady state diffusion.

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \quad (1)$$

This includes the effect of changing concentration with time (t). This has been solved using the boundary condition $C_{(x=0, t>0)} = C_0$ (the surface concentration is constant at C_0), the initial condition $C_{(x>0, t=0)} = 0$ (the initial concentration in the concrete is 0) and the infinite point condition $C_{(x=\infty, t>0)} = 0$ (far enough away from the surface, the concentration will always be 0). The solution is,

$$\frac{C(x,t)}{C_0} = 1 - \operatorname{erf}\left(\frac{x}{2\sqrt{Dt}}\right) \quad (2)$$

where $\operatorname{erf}(y)$ is the error function, a mathematical constant found in math tables or as a function in common computer spreadsheets, x is the cover depth, and $C(x,t)$ is the chloride concentration at time t and at depth x from the surface.

However, in actual practice, the flow is governed by the diffusion and convection manner (Bear *et al.* 1988; Freeze *et al.* 1979), and thus we have to use the following equation.

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} - V \frac{\partial C}{\partial x} \quad (3)$$

Fig. 2 Effect of liquid water front on service life.

Ogata and Banks (1961) derived the solution for the above equation considering a semi-infinite column of porous media as follows.

$$\frac{C(x,t)}{C_0} = 0.5 \left[\operatorname{erfc} \frac{x-Vt}{2\sqrt{Dt}} + \exp\left(\frac{Vx}{D}\right) \operatorname{erfc} \left(\frac{x+Vt}{2\sqrt{Dt}}\right) \right] \quad (4)$$

where V is the average linear rate of flow (cm/s).

$$V = \frac{k}{\phi} \frac{\partial h}{\partial x} \quad (5)$$

where $\partial h / \partial x$ is the hydraulic gradient, which is considered constant as 0.1 MPa/m in this study, k is the hydraulic permeability (m/s), and ϕ is the porosity of the material. Porosity is dependent on other properties as the following equation, Eq. 6. The key parameters in this study are *diffusion coefficient* and *liquid water front*. In Eq. 5, the rate of flow is the function of hydraulic permeability (k) and porosity (ϕ), and these are dependent on the *diffusion coefficient* and *liquid water front*. Thus to determine the effects of the parameters only, the hydraulic gradient is kept constant.

$$\phi = \frac{1000 \times M}{\omega_f} \quad (6)$$

where M is the absorption capacity (gm/mm^2) and ω_f is the liquid water front (cm).

Hydraulic permeability and the critical pore radius are determined by the experimental results stated in the references (Halamicova *et al.* 1995).

Fig. 3 Permeability vs. critical pore radius.

The critical pore radius, which is designated as r in this study, can be determined by capillary theory according to the following equation.

$$r(m) = \sqrt{\frac{4\mu}{P_o}} \times \frac{\omega_f}{\sqrt{t}} \quad (7)$$

where t is the time for short term absorption in seconds, ω_f is the water front in cm, μ is the viscosity in pa-s, and P_o is atmospheric pressure in Pa.

In Eq. 3, the diffusion coefficient is found from experiments done in the laboratory and the solution is based on the Finite Difference Method that will be discussed in the next section.

The surface chloride concentration C_o is considered to increase with time and is calculated as follows (Takeda *et al.* 1999).

$$C_o(t) = C_{ou} (1 - \exp^{-\alpha t}) \quad (8)$$

where α is an environmental factor adopted as 0.50, t is time in years, and C_{ou} is the ultimate surface chloride.

Diffusion coefficient D is assumed to obey the following rule to make compatible with the stopping criterion of chloride ion up to the liquid water front. In **Fig. 4**, MFD, MFP and F stand for Modified Fick Deterministic, Modified Fick Probabilistic and Fick respectively.

Figure 4c shows the conventional way of thinking, which does not take into account the liquid water front, whereas the Modified Fick Deterministic and Modified Fick Probabilistic ways of thinking include the influence of the liquid water front on the chloride stopping criterion, diffusion coefficient D , which is set to the value shown in **Figs. 4a** and **4b**. Under the conventional Fick's law, the diffusion coefficient is set as a constant throughout the specimen depth, whereas in the case of the modified Fick models, the diffusion coefficient is set differently in step with the liquid water front. In this paper, concrete having 70% w/c ratio is treated as bad quality concrete. In the case of bad quality concrete, saturation drops in the deeper zone from the surface where the diffusion coefficient drops slightly, but for ease of calculation, it is assumed to be constant throughout the specimen depth, based on the premise that the specimen is saturated almost in its entirety. In the case of large pores, Cl⁻ can move through the unsaturated zone inside. In the case of good quality concrete, satura-

tion drops with the sharpness of the water front, and thus two values are needed for the diffusion coefficient, as shown in **Fig. 4a**. The medium quality type of concrete has both large and small pores, which makes the water front penetrate in a distribution pattern where the diffusion coefficient should be considered to decline along a slope until it reaches zero.

Matric pressure or capillary pressure is thought of as the pressure of water in a pore of the medium relative to the pressure of the air. If the matric pressure is close to zero, air-water interfaces are broadly curved, nearly all pores are filled and water content is high. If matric pressure is much less than zero the interfaces are more tightly curved, they can no longer go across the largest pore, and the pores have less water inside. Actually the curvature of the air-water interface is inversely related to the pressure. Tighter curvature is associated with smaller pores with more negative pressure. That is why in bad quality concrete having many larger pores, the water front variation is large and Cl⁻ can move through unsaturated parts with less wall friction. However, in good quality concrete, it is difficult to achieve the fully saturated condition and the pores inside remain disconnected. Moreover, good cover concrete having fine pores, a sharp front of water is expected, and if water can move through the even finer pores, Cl⁻ flow will be restricted by the wall friction.

3.2 FDM scheme

Each of the proposed equations was calculated using Microsoft Excel to be able to efficiently calculate the concentration distribution and input the necessary data at suitable stages in order to find the output concentration.

Equation 3 is solved for the diffusion-advection of flow using Finite Difference scheme considering three points on the x-axis separated by dx . By using Taylor expansion and rearrangement, the final equation to solve is as follows.

$$C_{i+dt} = C_i + D \frac{dt}{dx^2} [C_{i-1} - 2C_i + C_{i+1}] - V \frac{dt}{2dx} [C_{i+1} - C_{i-1}] \quad (9)$$

According to the assumption of the effect of the liquid water front on chloride penetration and experimental models (which will be discussed in another paper), and with the help of the above finite differentiation scheme, the concentration profile in concrete can be calculated.

Hindmarsh and Sousa (Hindmarsh *et al.* 1984; Sousa 2003) showed under which the conditions that the finite difference scheme in Eq. 9 is stable are,

$$2D \frac{dt}{dx^2} \leq 1 \quad (10)$$

$$\left(\frac{Vdt}{dx} \right)^2 \leq 2D \frac{dt}{dx^2} \quad (11)$$

Rearrangement and simplification of both of these conditions gives the restriction on the size of the time

Fig. 4 Coupling between liquid water front and diffusion coefficient.

step in terms of the parameters and the grid size:

$$dt \leq \min \left\{ \frac{dx^2}{2D}, \frac{2D}{V^2} \right\} \quad (12)$$

3.3 Parameter quantification

The important parameters governing the flow through concrete can be determined as shown in the following figure.

Parameter D can be set based on the w/c ratio and material used in the concrete. Usually, the health of the structure is judged during the inspection by torrent permeability kT , which can be correlated with the absorption of water as well as the liquid water front. Finally these parameters can be used as input in the broader sense of life cycle cost computation.

4. Application of variation of liquid water front to verification (case study of Okinawa structure)

Three types of concrete, namely B0, F1, and F2, which stand for Ordinary Portland Cement concrete, fly ash 1,

Fig. 5 Conceptual flowchart.

Fig. 6 Division of core specimens.

and fly ash 2 concrete, were taken as cores having the compositions listed in Table 1.

4.1 Parameters from core data

4.1.1 Profile from inspection data

The cores were collected from Okinawa at the inspection age of 8.75 years. Each of the cores was divided into several parts to utilize in various experiments as shown in Fig. 6. One part of each core was treated as horizontal part and another part was treated as a vertical open part to simulate the thickness of the wall where the effect of relative humidity inside the concrete wall may affect the penetration of water. Horizontal indicates a thicker wall with high humidity inside, while vertical open indicates a thin wall with low humidity inside.

Previous inspection was done by Prof. Yamada (University of the Ryukyus) at 1.5 and 3.5 years of the structure's age and the profiles of past data including data at 8.75 years are plotted in Figs. 7, 8 and 9.

The chloride stopping position changes according to the composition of materials but does not become deeper as the concrete ages.

4.1.2 Experimental study

To implement the concept of using the liquid water front

Fig. 7 Chloride profile for core (B0).

Fig. 8 Chloride profile for core (F1).

Table 1- Mix proportion (Okinawa structure).

Name	W/C (%)	W/(C+F) (%)	Unit Volume (kg/m³)				
			W	C	F	S	G
B0	56	56	169	302	0	860	979
F1	70	56.3	172	245	60	852	955
F2	59	46.7	175	295	80	688	1052

Fig. 9 Chloride profile for core (F2).

Fig. 10 Water absorption of Okinawa cores.

for chloride profiling, it is necessary to know the diffusion coefficient, water absorption and liquid water front. To extract those data, the end part (10 to 20 cm) of the core specimens was coated with primer and epoxy to make it non penetrable. Only the required surfaces were kept open. The specimens were placed in a 10% NaCl solution. The chloride concentration profile was measured after 1 year by titration (JCI SC5 1987) to determine the diffusion coefficient. The profile from 1 year of exposure and the real profile clearly show the stopping position of chloride movement at the same distance from the surface as later described in the verification section.

The remaining two parts were placed in pure water with two different exposure conditions as horizontal and vertical open. The weights were measured continuously to determine absorption until 1 year and are shown in **Fig. 10**. Bo, F1 and F2 indicate the respective concrete types, namely OPC and two different compositions of fly ash. M indicates the middle height, i.e. the core was taken at a height of 1.5 m above the surface of water. 'H' and 'VO' indicate horizontal and vertical open, respectively, as in the laboratory test. Finally the specimens were broken and the water penetration fronts were measured according to **Fig. 11**. Dry cutting and coating of the surfaces took as many as 7 days. The specimens became almost dry during that time. To get concrete without previous penetration of chloride, samples were taken from the end part (10 to 20 cm) of the core. To avoid the effect of saturation, the specimens were dried thoroughly and the liquid water front was measured after absorption became constant.

Fig. 11 Measurement of liquid water front.

Fig. 12 Correlation between absorption and liquid water front (Okinawa Cores).

Table 2 Parameters (Okinawa structure).

Name	C_o (kg/m ³)	D (cm ² /yr)	w_f (mm)
B0	21.74	2.308	47.48
F1	27.30	0.125	40.05
F2	30.22	0.053	24.76

The relation between the absorption amount of water and the liquid water front is shown in **Fig. 12**.

Surface chloride C_o , diffusion coefficient D and liquid water front w_f of the different types of concrete are listed in **Table 2**.

4.2 Verification

Fick's law is modified taking liquid water front into consideration. Verification is done by matching the 1-year chloride profile for two types of fly ash concrete with the profile derived from the modified Fick's law along with the liquid water front.

Figures 13 and 14 above agree well in terms of justifying taking into account the liquid water front for chloride profiling.

The exposure conditions explored the risk or variation of the liquid water front for different types of concrete as shown in **Fig. 15**.

Fig. 13 Verification with MFP (Okinawa F1).

Fig. 14 Verification with MFP (Okinawa F2).

Table 3 Variation of water penetration front (Okinawa structure).

Name	Water Penetration Front, w_f (mm)	
	Mean	Standard Deviation
B0	47.48	21.67
F1	40.05	7.99
F2	24.76	6.96

The mean value and standard deviation of the liquid water front for the three different types of concrete are given in **Table 3**.

A comparison graph between standard deviations, assumed as 4.748, and real standard deviation of 21.67 as measured for B0 concrete is shown in **Fig. 16**. The sharpness of the liquid water front affects the penetration position of chloride inside concrete. This sharpness depends on the type of concrete. If the standard deviation of the water front along the mean position is larger, the penetration exceeds the mean value of depth. It is therefore, necessary to evaluate the water penetration behavior to determine the stopping position of chloride ingress. In **Fig. 16**, the mean water penetration front in the concrete is 4.748 cm and the standard deviation along the mean is 2.17 cm. If the standard deviation is assumed as 10% of the mean value, chloride penetrates up to 6 cm, but the actual value of standard deviation shows that the chloride penetration depth is greater than 8 cm. Thus the above assumption will definitely cause overestimation of the predicted lifetime of the structure depending on the position of the reinforcement from the exposed surface.

That suggests the importance of including variations of the liquid water front when judging RC structural

Fig. 15 Variation of liquid water front (real case).

Fig. 16 Effect of standard deviation on chloride profile (B0 concrete).

Fig. 17 Verification with MFP (Okinawa B0).

health. **Figure 17** verifies the chloride ion profile of B0 concrete taking into consideration the liquid water front along with the variation of the front position.

4.3 Experimental study (laboratory-cast slag concrete)

Slag concrete with 50% replacement of cement by slag was cast in the laboratory with w/c ratios of 40%, 55%, 70% respectively. The specimens were 100 mm in diameter and 200 mm in height. All the specimens were cut in 4 parts, coated with primer and allowed to dry for 24 hours. Two layers of epoxy coating were added over the primer finished surface and allowed to dry for 24 hours prior to exposure. This coating was applied to make the surfaces other than the required surface that was kept open non penetrable to liquid. The specimens were placed in a 10% NaCl solution in three different exposure

Fig. 18 Specimen exposure.

Fig. 19 Variation of liquid water front (experiment case).

Fig. 20 Mean water front of horizontal series (slag concrete).

situations, horizontal, vertical open and vertical coated, as shown in Fig. 18, where 'C' is a coated surface and 'O' is an open surface. Wet-dry cycles were maintained to simulate a splash zone. The wet condition was set as submergence in NaCl solution with temperature of 20°C and 100% RH for 1 day, and the dry condition was set as storage of the specimens inside a 40°C chamber with 60% RH for 6 days. The weights for all the specimens were measured twice a week after each condition. The mix proportions for the experimental cases of slag concrete are given in Table 4. Sealed curing was applied to all the specimens for 28 days.

After 6 months, the specimens were broken and their respective liquid water front was measured. The position of the front was measured at a number of points and the risk of variation is shown in Fig. 19.

Table 4 Mix proportions.

Type	W/C [%]	Quantity [kg/m ³]				
		W	C	BFS	S	G
B40	40	174.9	218.6	218.6	695.9	1001.2
B55	55	174.9	159.0	159.0	792.3	1008.7
B70	70	174.9	124.9	124.9	873.7	985.9

Table 5 Difference among methods.

Method	Liquid Water Front (cm)	
	Mean	Standard Deviation
MFD	Actual	No
MFP	Actual	Actual
F	Independent of water front	

Table 6 Water front of analyzed concrete.

Type	Liquid Water Front (cm)	
	Mean	Standard Deviation
F2	2.48	0.7
B0	4.75	2.17
B70	6.47	2.42

The mean front position of the horizontal series is shown in Fig. 20, whose value for the 70% w/c ratio will be used in the next section.

5. Classification of concrete and its application to determine suitable methods of analysis

Two types of concrete, concrete from Okinawa and slag concrete cast in laboratory were analyzed. Three analysis methods were considered namely, Modified Fick Deterministic (MFD), Modified Fick Probabilistic (MFP) and Fick (F). Table 5 shows the differences among these methods. The mean and standard deviation of the water front for the analyzed concretes are given in Table 6.

Each type of concrete was analyzed for Cl⁻ profile using three different methods and the results were compared with the actual profile data as shown in Fig. 21. The root mean square error (RMSE) was determined for all cases, as shown in Fig. 22.

The suitable method of analysis for each type of concrete was selected based on minimum RMSE. In the case of good concrete, although RMSE of MFP is slightly lower than that of MFD, MFD was selected as the most suitable method due to its ease of application.

Thus chloride distribution analysis using suitable methods according to the above figure for different grades of concrete can yield precise results, which is important for service life design and LCC computation.

6. Comparison of proposed method with diffusion coefficient recommended by JSCE

The experimental models are based on laboratory experiments where the liquid water front is used to modify

Fig. 21 Cl^- profile by different methods.

Fig. 22 RMSE for different methods.

Table 7 Variation of water penetration front (experimental case).

Name	Water Penetration Front, w_f (mm)	
	Mean	Standard Deviation
N40	26.37	4.32
N55	48.71	7.21
N70	59.46	11.60

the concept of Fick's law. The liquid water front and variation values used as input are given in **Table 7**. The diffusion coefficient used in the analysis is also based on experimental models. The Japan Society of Civil Engineers (JSCE) proposed equations to calculate the diffusion coefficient for OPC and slag concrete. It is better to compare the experimental data with analysis results. Analysis was done using different diffusion coefficients, one was from experimental model and the other was recommended by JSCE.

Figure 24 is the comparison between the proposed method and the JSCE recommendation for normal concrete. The dots in **Fig. 24** present the profile obtained from the experiments. N40, N55 and N70 represent OPC concrete with 40%, 55% and 70% w/c ratio, respectively. The diffusion coefficients used here are of two types. One is from fitting the experimental chloride concentrations. The other is from the JSCE recommended equation for normal concrete, which is dependent on the w/c ratio. Both diffusion coefficients are applied in the conceptual model coupled with liquid water front. For the case of 40% w/c ratio, Fick's law is modified with deterministic

Fig. 23 Classification of analysis methods.

way (**Fig. 4(a)**), for the case of 55% w/c ratio, Fick's law is modified with probabilistic way with the mean front position along with variation of the water front (**Fig. 4(b)**), and in the case of 70% w/c ratio, Fick's law is applied without modification (**Fig. 4(c)**). The importance of doing so is to reveal that the diffusion coefficient suggested by JSCE, if applied in this concept, can work well with the experimental profile. The profiles using both the JSCE 'D' and the experimental 'D' are the same, reflecting the low importance of taking into consideration the diffusion coefficient. The time average experimental 'D' and the 'D' from JSCE combined with the proposed method give the same profile, suggesting the importance of taking into consideration the liquid water front as the key parameter for analysis.

Fig. 24 Comparison with JSCE-D (normal concrete).

7. Conclusions

A concept was developed to take into account the liquid water front for service life and LCC prediction. The application of this concept to a real structure has yielded the following findings.

- (1) Fick's law overestimates the concentration of chloride for concrete with good surface quality.
- (2) The liquid water front and its variation play an important role in the prediction of the Cl^- ion profile. In the case of very good concrete having smaller pores,

the seepage front of water is stopped sharply and Cl^- cannot go beyond the water front position. In the case of the medium grade of cover quality concrete, the water front is not stopped sharply but it creates some variation along its mean stopping position due to having small and medium sized pores. Thus Cl^- moves beyond the mean position of the stopping position of the water front. In the case of bad quality concrete, although saturation drops inside, water flows deeper with larger pores and creates an unsaturated zone through which Cl^- can flow.

- (3) Methods of analysis for chloride distribution in concrete influenced by the liquid water front are proposed.

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