

Effect of measurement method and cracking on chloride transport in concrete

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Abstract. This paper aims to study the effect of measurement methods and cracking on chloride transport of concrete materials. Three kinds of measurement methods were carried out, including immersion test, rapid migration test and steady-state migration test. All of these measurements of chloride transport show that chloride ion diffusion coefficient decreased with the reduction of water to cement ratio. Results of the immersion test were less than that of rapid migration test and steady-state migration test. For the specimen of lower water to cement ratio, the external electrical field has little effect on chloride binding relatively. Compared with the results obtained by these different measurement methods, the lower water to cement ratio may cause smaller differences among these different methods. The external voltage can reduce chloride binding of concrete, and the higher electrical field made a strong impact on the chloride binding. Considering the effect of high voltage on the specimen, results indicate that results based on the steady-state migration test should be more reasonable. For cracked concrete, cracking can accelerate the chloride ion diffusion.

Keywords: chloride transport; immersion test; rapid migration test; steady-state migration test; cracking

1. Introduction

Concrete is the most widely used building material in the world, owing to its versatility and relatively low cost. Concrete has also become the material of choice for the construction of structures exposed to extreme conditions (Shi *et al.* 2010). Many environmental phenomena are known to significantly influence the durability of reinforced concrete structures (Ihekweba *et al.* 1996, Castro *et al.* 2000). Especially, chloride-induced reinforcing steel corrosion is one of the most important material deterioration problems in reinforced concrete structures (Wang *et al.* 2005). It affects a large number of infrastructures, particularly those exposed to environments where de-icing salts or coastal/marine conditions are encountered (Li *et al.* 2012). In normal circumstances when chloride reaches the reinforced steel, the depassivation of the steel occurs and the steel reinforcement starts to corrode (Chindaprasirt *et al.* 2008, Conciatori *et al.* 2010, Hussain and Ishida 2011). Chloride ingress into concrete causes reinforcement corrosion, and the diffusion coefficient of chloride ions in concrete affects the durability of coastal and offshore reinforced

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concrete structures (Tatsuhiko *et al.* 2006, Ann *et al.* 2009).

In order to protect the reinforcing steel from corrosion one has to understand how chloride penetration taking place in concrete. The chloride penetrates the concrete by means of diffusion of the chloride ions through surface cracks or the interconnected pores in the concrete. The interaction between chloride diffusion and microcracks in the structures affects the durability of these structures (Shi *et al.* 2010). The chloride ingress into concrete and other cementitious materials is a complex phenomenon involving multiple mechanisms. In reality, the determination of chloride diffusion coefficient is often affected by the method of chloride analysis.

Owing to its important role with regard to durability of concrete structures, many methods have been proposed for testing chloride ingress in concrete. However, chloride ingress into concrete involves complex physical and chemical processes, which have up to now hindered to reach a general agreement on a single test method. Actually, the complexity comes at least from three sources. Firstly, the external environment is not constant. For example, in marine environments, the amount of chlorides in contact with concrete depends on whether the structure is placed fully submerged. Secondly, the concrete is constituted by different types of cement and binder, with different mix proportions. Lastly, the mechanisms of chloride penetration are not single diffusion, but combined with convection, chemical and physical binding, etc. Although, many studies have been done on the chloride attack in concrete structures (Wang and Ueda 2011), and most of these work was to investigate the effect factors on chloride diffusion in concrete, such as water-to-cement ratio, degree of hydration, aggregate volume fraction, coarse and fine aggregate particle size distributions, interfacial transition zone thickness and air content, and so on (Bentz *et al.* 1998, Halamickova *et al.* 1995, Zibara *et al.* 2008). There is opposite less study on the measurement method of chloride transport. A collection of more than ten different testing methods is available through an international committee, RILEM TC-178 TMC. These methods can be categorized into three categories: diffusion tests, migration tests and indirect tests based on resistivity or conductivity. Since conventional chloride diffusion test for the cement-based materials is time-consuming, the accelerated chloride ions diffusion test methods were developed. An effective method is to apply an external electrical field for accelerating chloride diffusion or penetration (Yang and Cho 2003). In order to recommend reliable methods for testing chloride transport, among those many methods, three of them were evaluated in this paper. That is immersion test, rapid migration test and steady-state migration test. Moreover, Cracking is an inevitable characteristic of reinforced concrete structures, which is usually a result of various physical and chemical interactions between concrete and environment, and it may develop at different stages throughout the life of the structure. In practice, the transport of chlorides in reinforced structures is the main cause of corrosion, and it is very common to have cracks in concrete structures in chloride ion environment (Win *et al.* 2004, Peter *et al.* 2009). Therefore, the influence of cracking on chloride diffusivity in concrete was also examined.

2. Materials and test methods

2.1 Materials

Samples were prepared with ordinary Portland cement (OPC) produced by Onada Corp. in Jiangsu Province, China. The chemical composition of the cement is presented in Table 1. The crushed stone used was basalt (5 mm to 20 mm), and middle sand with 2.3 fineness modulus was

Table 1 Chemical compositions of cement

Sample	Chemical composition /wt%						
	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	Na ₂ O _{eq}
Cement	66.6	21.74	5.06	3.56	1.60	0.81	0.50

Table 2 Mix proportion of concrete

W/C	Cement/kg/m ³	Water/kg/m ³	Sand /kg/m ³	Crushed stone /kg/m ³	Admixture /kg/m ³	Steel fiber /kg/m ³
0.4	440	176	665	1235	1.368	0
0.45	440	198	665	1235	1.368	0
0.5	440	220	665	1235	1.368	0
0.45	400	180	710	1110	0.25	0.28

used. Steel fiber with 13 mm length and 0.2 mm diameter was also used. The concrete admixture used was JM-PCA produced by Jiangsu Bote New Materials Co., Ltd.

2.2 Test methods

The concrete specimens with size of $\Phi 100$ mm \times 160 mm and 100 mm \times 100 mm \times 100 mm were prepared. The mix proportions of the concrete are listed in Table 2. The experimental specimens were demolded after 24 h and were cured in a room with a temperature of 20°C \pm 3°C and a relative humidity of 90% \pm 5% for 28d. Then they were cut into 50 mm thick slices. Considering the boundary effect, the slices were cut from the middle part of the specimen.

Immersion test method is based on natural diffusion under a very high concentration gradient, and gives chloride contents at different depths after different exposure duration. The test also provides values of D (non-steady state diffusion coefficient) and C_s (surface total chloride content) by curve-fitting the measured chloride profile to an error-function solution of Fick's 2nd law. This method is also time-consuming and takes relative long time. The specimen with size of 100 mm \times 100 mm \times 50 mm by sawing a portion of the prism specimen was used, and five faces of each specimen were coated with epoxy, leaving only one face uncoated after drying. The specimens were immersed in 5% NaCl solution for 60 days in a controlled room at 20°C \pm 3°C. A drill with an adjustable bit size was used to obtain samples. The bit was moved at regular intervals. After each milling step, the resulting powder was collected and was then used to extract acid-soluble chloride contents (Berman 1972). Mohr titration was used to determine the chloride concentration in the solution.

Rapid migration test is a non-steady state migration using an external electrical field for accelerating chloride penetration. The test is relative simple and rapid with the test duration in most cases 24 hours. 3 specimens of diameter 100 mm and thickness 50 mm were tested. A 30 V external potential was imposed across the specimen with the test surface exposing in the 10% NaCl solution and the opposite surface in the 0.3 M NaOH solution for a certain duration, then the specimen was splitted, and the penetration depth of chlorides can be measured by using a colourimetric method.

For steady state migration testing method, the driving force is an electrical potential that forces ions to migrate between the anode and the cathode. The schematic diagram of steady state migration testing is shown in Fig. 1. This migration test consists in accelerating a chloride ion diffusion test by a constant electrical field. The effective chloride ion diffusion coefficient is assessed from the chloride ion flux in the cathodic cell in steady state. The 50 mm slices were prepared including vacuuming, saturation and setting up the test, and the lateral surfaces of specimens were coated with epoxy, and then placed between two cells. Each specimen was placed between the two compartments of a cell. Flat silicone circular seals ensured that the system was leakproof. The solutions comprised NaCl (3%) in the upstream compartment, and NaOH (0.2 mol/l) in the downstream compartment. An 8-v potential was applied over the specimen, and the test was conducted at $20\pm 5^{\circ}\text{C}$. Electrical current and chloride-ion concentration were simultaneously monitored in the migration test. This migration testing consists in accelerating a chloride diffusion testing by a constant electric field. The effective chloride diffusion coefficient is assessed from the chloride ion flux in the cathodic cell in steady state.

Steel fiber was used to improve embrittlement of concrete. At 28 days of age, the prisms were pre-cracked by a splitting tensile test. It was to generate a fine crack at the surface of the prism. Crack width was measured along this line using an image analysis technique. Firstly, collect the crack image by a certain image collecting system such as an optical microscope. Then, the edge of the crack is orientated by the improved grads threshold way. In the end, the crack feature of width is calculated based on the edge obtained above.

3. Results and discussion

3.1 Immersion test method

This method is based on natural diffusion under a very high concentration gradient, and it is also relatively laborious and takes relatively long time. The positions of sampling for chloride profiling on the specimen are shown in Fig. 1. All the concentration data presented are in mass percentage of concrete, derived from the chemical analysis test. The concentration value at a particular depth indicates an average concentration with a 2 mm depth. That chloride ion concentration profiles of concrete after 60d immersion was shown in Fig. 2. From Fig. 2, as expected, from the surface to interior, a clear trend was observed in which the chloride ion concentration was high near the surface. The chloride ion concentration decreased from surface to interior.

The immersion test provides coupled values of D_{imm} (chloride diffusion coefficient) and C_s (surface chloride content) by curve-fitting the measured chloride profile to an error-function solution of Fick's 2nd law, which is under the assumption of constant chloride binding capacity. In the reality, chloride binding capacity is non-linearly dependent on free chloride concentration and also dependent on type of cementitious binder (Tritthart 1989, Tang and Nilsson 1993). The total chloride content is a sum of the free chlorides in the pore solution and the bound chlorides on the surfaces of hydrates. C_s value is also dependent on the porosity and the type of binder. Even under the same exposure condition, different types of binder or different water to cement ratio will give different values of C_s .

Fick's 2nd Law was widely adopted, in which the concentration gradient was assumed as the only driving force of the transport of chloride ions in concrete (Mangat and Molloy 1994, Berke

and Hicks 1994, Tang 1996, Onyejekwe and Reddy 2000). In this paper, the chloride ion transport into concrete is assumed to be one-dimensional in a semi-infinite medium complying with Fick's 2nd Law of Pure Diffusion given as (Crank 1975, Gowripalana *et al.* 2000)

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

An analytical solution to Eq. (1) is given by

$$C_x = C_s \left[1 - \operatorname{erf} \left(\frac{x}{2\sqrt{Dt}} \right) \right] \quad (2)$$

where D is non-steady state diffusion coefficient, m^2/s ; x is average depth where the chloride sample was ground, m ; t is immersion duration, s ; C_x is chloride content at depth x , mass % of sample; C_s is surface chloride content, mass % of sample; erf is error function.

According to Eq. (2), the chloride diffusion coefficient was calculated, the results were given in Fig. 3. From Fig. 3, it was found that the chloride diffusion coefficient of the specimen of $w/c=0.4$ was the lowest, and it increased with the water to cement ratio. That is to say, higher water to cement ratio resulted in greater chloride ion diffusion.

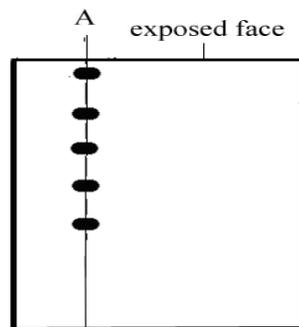


Fig. 1 Instruction of testing sites for chloride profiling

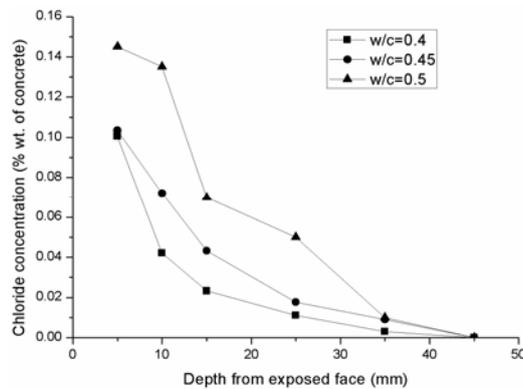


Fig. 2 Chloride ion concentration of concrete samples

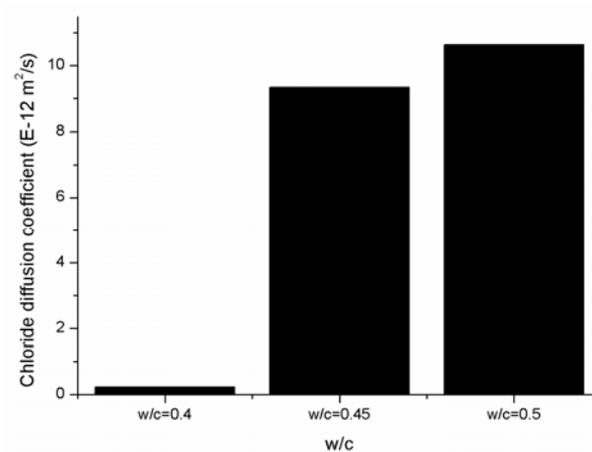


Fig. 3 Chloride diffusion coefficient of immersion test

3.2 Rapid migration test

This method is a non-steady state migration test using an external electrical field for accelerating chloride penetration. For rapid migration test, after a certain test duration, the specimen is split, and a silver nitrate solution is sprayed on to one of the fresh section. The chloride penetration depth can be measured from the visible white silver chloride precipitation, then the chloride migration coefficient can be calculated from the chloride penetration depth. The Rapid migration test provides value of D_{RCM} , which is also under the assumption of constant chloride binding capacity during the test. D_{RCM} was deduced according to Eq. (3)

$$D_{RCM} = 2.872 \times 10^{-6} \frac{Th(x_d - \alpha \sqrt{x_d})}{t} \quad (3)$$

$$\alpha = 3.338 \times 10^{-3} \sqrt{Th} \quad (4)$$

where D_{RCM} is chloride ion diffusion coefficient, m²/s; T is average value of early and final temperature of anolyte, K; h is the height of the sample, m; x_d is the diffusion depth of chloride ion, m; t is the testing time, s; α is auxiliary variable.

Fig. 4 presents the results of chloride ion diffusion depth, and we can see that chloride ion diffusion depth also increased with the water to cement ratio. Based on the data of chloride ion diffusion depth, the chloride ion diffusion coefficient D_{RCM} was calculated by Eq. (3). Fig. 5 gives the chloride ion diffusion coefficient D_{RCM} . From Fig. 5, the chloride ion diffusion coefficient of the specimen with $w/c=0.4$ was still the lowest, and it increased with the water to cement ratio.

3.3 Steady state migration test

Chloride binding has an influence upon time lag but it does not alter the value of the diffusion coefficient (David *et al.* 2010). Therefore, we are able to determine the diffusion coefficient of

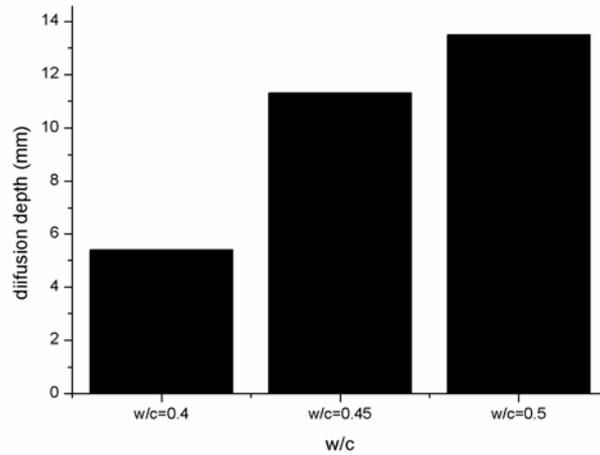


Fig. 4 The chloride ion diffusion depth

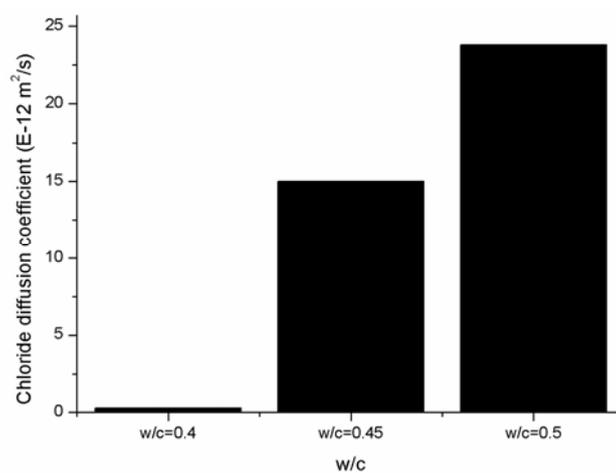


Fig. 5 Chloride diffusion coefficient of rapid migration test

concrete within a short period using the migration test. In chloride migration testing, chloride ions are transported in concrete under an applied voltage. As the chloride flux becomes constant, the Nernst–Planck equation describes transport processes in a solution (Misra *et al.* 1994)

$$J = -D \frac{\partial C}{\partial x} - \frac{zF}{RT} DC \frac{\partial V}{\partial x} + Cu \quad (5)$$

where D is the diffusion coefficient of ion (m²/s), which is later defined as a migration coefficient of mortar; T is the concentration of ion (mol/m³); R is the universal gas constant (8.3 J/mol/K); T is absolute temperature (K); V/x is the strength of the electric field between the anode and the cathode (V/m); and u is the velocity of solute (m/s). The first and the third terms of Eq. (5) are the contributions of diffusion and convection, respectively. Since the concrete is saturated, the velocity of the solute can be neglected. Under the influence of an electrical field across the sample, the

contribution of diffusion in concrete is small and can be neglected. Then, only taking into account the migration due to the electrical field, the migration coefficient D can be calculated by rearranging the middle term of Eq. (5) as follows

$$J = -\frac{zF}{RT} DC \frac{\partial V}{\partial x} \quad (6)$$

Then the migration coefficient of chloride ion for concrete, D_{cl} , is calculated as

$$D_{cl} = \frac{RT}{zFC(E/l)} J_{cl} \quad (7)$$

where J_{cl} is the constant flux of chloride in the downstream cell ($\text{mol m}^{-2} \text{s}^{-1}$), z is the electrical charge of chloride, F is the Faraday constant ($96,500 \text{ C mol}^{-1}$), and C is chloride concentration in the upstream cell at the cathode

$$J_{cl} = \frac{V}{A} \frac{\partial C}{\partial t} \quad (8)$$

Based on Eqs. (4) and (7), the migration coefficient of chloride ion of concrete is calculated from a stable steady-state current as follows (Yang *et al.* 2003)

$$D_{state} = \frac{RTt_{cl}}{zCF^2 A(E/l)} I_s \quad (9)$$

The chloride migration coefficients of concrete samples calculated from Eq. (9) are also shown in Fig. 6. It is apparent that the chloride migration coefficient of the sample with $w/c=0.5$ is greater than that of the sample with $w/c=0.4$ and $w/c=0.45$. Furthermore, the higher water to cement ratio also caused greater chloride migration coefficient.

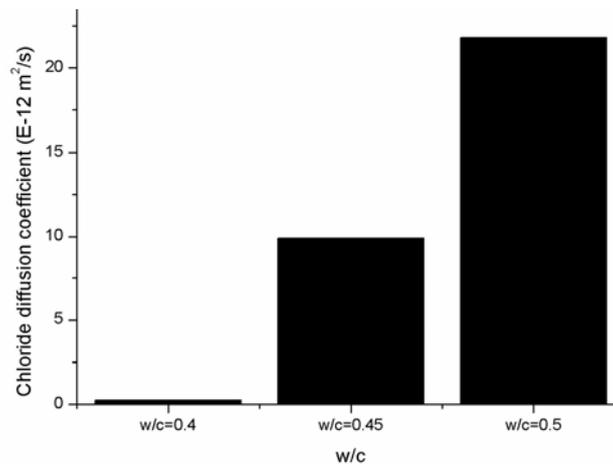


Fig. 6 Chloride diffusion coefficient of steady state migration test

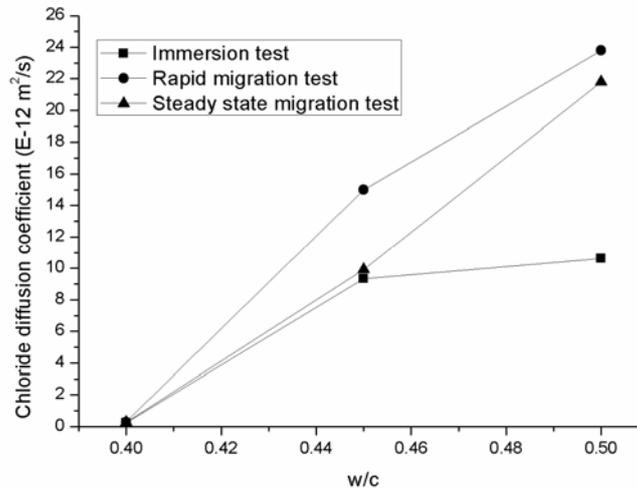


Fig. 7 Comparison of the results according to three kinds of test methods

Fig. 7 gives the relationship among chloride ion diffusion coefficient D_{imm} , D_{rapid} and D_{state} obtained by immersion test, rapid migration test and steady state migration test respectively. It can be observed that the trend of results obtained by immersion test appears to be consistent with the results obtained by rapid migration test and steady state migration test. The resistance of concrete to chloride penetration was increased with the reduction of w/c . For the specimen with water to cement ratio 0.4, difference among the results based on three kinds of measurement of chloride transport was very small, while for the specimen with water to cement ratio 0.5, the difference was great relatively. All of these different measurements are operated under the assumption of constant chloride binding capacity during the test. Different from the immersion test, this assumption may better hold in the rapid migration test and steady state migration test, due to the strong external electrical field and short testing duration. These objective factors tend to reduce the amount of bound, especially physically bound, chlorides. Chloride binding removes chloride ions from the pore solution, and slows down the rate of penetration. Therefore, the results based on rapid migration test and steady state migration test describes the property of chloride transport under a condition of reduced chloride binding (Tang 1996). According to Fig. 7, the higher the water to cement ratio was, the greater the difference between the results from immersion test and rapid migration test or steady state migration test was, and it may be because the effect of external electrical field on chloride binding increases with the water to cement ratio. So, when water to cement ratio was 0.5, the chloride diffusion coefficient based on immersion test was lower than that based on rapid migration test and steady state migration test, obviously. Since the external electrical field was 30 V in rapid migration test, while a 8 volt potential was applied in steady state migration test. The gap between the results based on immersion test and rapid migration test was greater than that between the results based on immersion test and steady state migration test. It indicates that the higher the electrical field, the greater the influence of it on chloride binding. However, since the high potential will cause heated concrete, which may bring other problems, such as ionization of the solution. So, the imposed voltage should not be too high. The voltage used in rapid migration test is too high, which may affect the accuracy of results. Steady state migration test not only reduces the effect of chloride binding, but also improves the work

efficiency, due to the application of 8 volt potential. So, steady state migration test should be more reasonable among these three kinds of measurement methods.

3.4 Chloride transport of cracked concrete

An image analysis technique was implemented to rapidly extract pertinent crack width information along a crack path. Fig. 8 provides an overview of the image processing and analysis procedure that was used in this study. From this figure it can be seen that the process involves: image acquisition, image processing, crack feature determination and measurement. The image is processed by serials of ways such as thresholding, filtration, sharpening, grid-mask subtraction, etc. According to conclusions about measurement of chloride transport, steady state migration test was used to evaluate the chloride transport of cracked concrete. Fig. 9 gives the results of chloride ion diffusion for the concrete with different width cracking. Fig. 9 clearly shows that the chloride diffusivity in the cracked concrete is greater than that in the sound concrete. Furthermore, the wider cracking leads to greater chloride diffusion coefficient when the cracking width increases from 0 to 135 μm . Therefore, it indicated that cracking can accelerate the chloride ion diffusion. Results from this study can be employed in the future by others to evaluate the effect of crack parameters on the amount of chloride penetration.

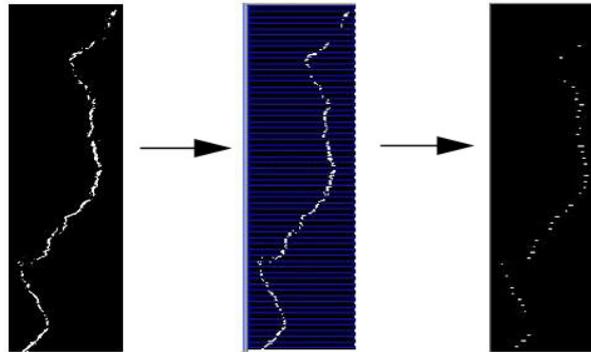


Fig. 8 Image analysis procedure

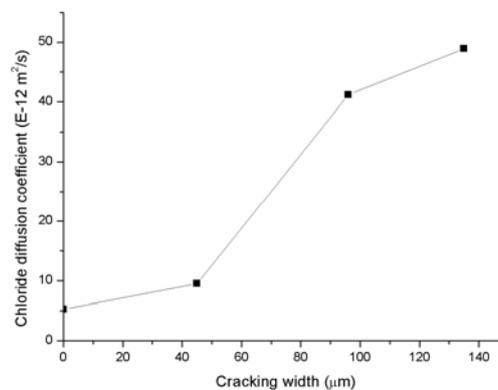


Fig. 9 Chloride ion diffusion coefficient of cracked concrete

4. Conclusions

Three kinds of measurement methods have been carried out in order to study chloride transport of concrete, including immersion test, rapid migration test and steady-state migration test. It was shown that that chloride ion diffusion coefficient decreased with reduction of water to cement ratio based on immersion test, rapid migration test and steady-state migration test. Furthermore, results of chloride ion diffusion coefficient based on the immersion test were less than that based on rapid migration test and steady-state migration test. The external voltage can reduce chloride binding of concrete, and the higher electrical field had great influence on chloride binding. For the specimen of lower water to cement ratio, the effect of the external electrical field on chloride binding was also small relatively. Compared with the results obtained by these different measurements, the difference among these methods was minor for the samples with lower water to cement ratio. Since the rapid migration test needs so high voltage which may cause heated concrete, steady state migration test should be more reasonable among these three kinds of measurement methods. Furthermore, cracking can accelerate the chloride ion diffusion. The wider cracking leads to greater chloride diffusion coefficient when the cracking width increases from 0 to 135 μm .

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