

# Transport and Interactions of Chlorides in Cement-Based Materials



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# Transport and Interactions of Chlorides in Cement-Based Materials

Caijun Shi, Qiang Yuan,  
Fuqiang He, and Xiang Hu



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# Contents

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<i>Preface</i>	xiii
<i>Authors</i>	xv
<b>1 Introduction</b>	<b>1</b>
1.1 Chloride-Related Corrosion	1
1.2 Chloride Transport in Cement-Based Materials	4
1.3 Interaction of Chloride with Hydration Products of Cement	7
1.4 Organization of This Book	10
References	12
<b>2 Mechanisms of Chloride Transport in Cement-Based Materials</b>	<b>19</b>
2.1 Introduction	19
2.2 Transport of Chloride in Cement-Based Materials	20
2.2.1 Electrical Migration	23
2.2.2 Diffusion	26
2.2.3 Hydrostatic Advection	29
2.2.4 Thermal Migration	29
2.2.5 Capillary Effect	30
2.2.6 Coupled Effects	31
2.3 Summary	32
References	33
<b>3 Chemical and Physical Interactions between Chlorides and Cement Hydrates</b>	<b>35</b>
3.1 Introduction	35
3.2 The Formation of Friedel's Salt	36
3.2.1 Reaction between Chloride Ions and Aluminum Phase	37

3.2.2	<i>Reaction between Chloride Ions and AFm Phase</i>	38
3.3	<i>The Stability of Friedel's Salt</i>	42
3.4	<i>Physical Adsorption of Chloride</i>	45
3.4.1	<i>Chloride Adsorption by C-S-H Gel</i>	45
3.4.2	<i>Chloride Adsorption by AFt Phase</i>	47
3.5	<i>Chloride Concentrate Phenomenon</i>	47
3.5.1	<i>Formation of EDL within Cement-Based Materials</i>	51
3.5.2	<i>Zeta Potential</i>	54
3.5.3	<i>Chloride Distribution within EDL</i>	59
3.5.4	<i>Variations of EDL during Pore Solution Expression</i>	62
3.6	<i>Chloride Binding with Other Compounds</i>	67
3.7	<i>Summary</i>	69
	<i>References</i>	70

## **4 Chloride Binding and Its Effects on Characteristics of Cement-Based Materials**

77

4.1	<i>Introduction</i>	77
4.2	<i>Chloride Binding of Cement-Based Materials</i>	78
4.2.1	<i>Chloride Concentration</i>	78
4.2.2	<i>Cement Composition</i>	80
4.2.2.1	<i>C<sub>3</sub>A and C<sub>4</sub>AF</i>	80
4.2.2.2	<i>C<sub>3</sub>S and C<sub>2</sub>S</i>	82
4.2.2.3	<i>SO<sub>3</sub> Content</i>	82
4.2.3	<i>Supplementary Cementitious Materials</i>	83
4.2.3.1	<i>Fly Ash</i>	83
4.2.3.2	<i>Ground Granulated Blast Furnace Slag (GGBFS)</i>	83
4.2.3.3	<i>Silica Fume</i>	85
4.2.4	<i>Hydroxyl Ion Concentration</i>	87
4.2.5	<i>Cation of Chloride Salt</i>	87
4.2.6	<i>Temperature</i>	88
4.2.7	<i>Carbonation</i>	89
4.2.8	<i>Sulfate Ion</i>	89
4.2.9	<i>Electrical Field</i>	90
4.3	<i>Chloride Binding Isotherm</i>	92
4.3.1	<i>Linear Binding Isotherm</i>	92
4.3.2	<i>Langmuir Isotherm</i>	94
4.3.3	<i>Freundlich Binding Isotherm</i>	94
4.3.4	<i>Brunauer, Emmett, Teller (BET) Isotherm</i>	95

4.4	<i>Experimental Determination of Binding Isotherm</i>	95
4.4.1	<i>Equilibrium Method</i>	96
4.4.2	<i>Pore Solution Expression</i>	96
4.4.3	<i>Diffusion Cell Method</i>	97
4.4.4	<i>Migration Test Method</i>	98
4.5	<i>Determination of Physically Absorbed Chloride Distribution in EDL</i>	98
4.6	<i>Effect of Chloride Binding on Microstructure</i>	104
4.6.1	<i>Effects of Chloride Binding on Hydration Products</i>	104
4.6.2	<i>Effects of Chloride Binding on Pore Structure</i>	105
4.7	<i>Summary</i>	108
	<i>References</i>	111
<b>5</b>	<b>Testing Methods for Chlorides Transport in Cement-Based Materials</b>	<b>119</b>
5.1	<i>Introduction</i>	119
5.2	<i>Some Chloride-Related Tests</i>	120
5.2.1	<i>Chloride Profile</i>	120
5.2.2	<i>Chloride Analysis</i>	121
5.2.2.1	<i>Determination of Total Chloride</i>	124
5.2.2.2	<i>Determination of Water-Soluble Chloride</i>	126
5.2.2.3	<i>Relationship between Water-Soluble and Total Chlorides</i>	127
5.3	<i>Testing Methods for Chloride Transport in Concrete</i>	129
5.3.1	<i>Brief Overview of Testing Methods</i>	129
5.3.2	<i>Fick's First Law</i>	132
5.3.3	<i>Fick's Second Law</i>	134
5.3.3.1	<i>NT Build 443</i>	134
5.3.3.2	<i>Short-Term Immersion Test</i>	135
5.3.4	<i>Nernst–Planck Equation</i>	136
5.3.4.1	<i>NT Build 355</i>	137
5.3.4.2	<i>Upstream Method (Truc's Method)</i>	139
5.3.4.3	<i>NT Build 492</i>	140
5.3.4.4	<i>Breakthrough Time Method</i>	143
5.3.4.5	<i>Andrade and Castellote's Method</i>	145
5.3.4.6	<i>Samson's Method</i>	147
5.3.4.7	<i>Friedmann's Method</i>	148
5.3.5	<i>Nernst–Einstein Equation</i>	150

5.3.6	Formation Factor	151
5.3.7	Other Methods	152
5.3.7.1	ASTM C 1202/AASHTO T227 Test Method	152
5.3.7.2	Salt Ponding Test (AASHTO T 259)	154
5.3.7.3	Water Pressure Method	155
5.3.7.4	AC Impendence Method	156
5.4	Standards on Testing Methods for Chloride Transport	157
5.5	Relationship between Test Results Obtained from Different Test Methods	159
5.5.1	Non-Steady-State Migration and Diffusion Coefficients	159
5.5.2	Steady-State and Non-Steady-State Migration Coefficients	164
5.5.3	ASTM C1201 (or Initial Current) and Migration Coefficients	168
5.5.4	NT Build 443 Results Obtained from Free and Total Chlorides	169
5.6	Summary	171
	References	172

## **6 Determination of Chloride Penetration in Cement-Based Materials Using $\text{AgNO}_3$ -Based Colorimetric Methods** 177

6.1	Introduction	177
6.2	Determination of Chloride Ingress Depths	178
6.2.1	$\text{AgNO}_3$ + Fluorescein Method	178
6.2.2	$\text{AgNO}_3$ + $\text{K}_2\text{CrO}_4$ Method	179
6.2.3	$\text{AgNO}_3$ Method	179
6.2.4	Comparison of the Three Methods	181
6.2.5	Measurement of Chloride Penetration Depth	182
6.3	Chloride Concentration at Color Change Boundary	185
6.3.1	Parameters of Colorimetric Reactions	185
6.3.2	Sampling Methods	187
6.3.3	Methods for Free Chloride Measurement	187
6.3.3.1	Pore Solution Expression Method	188
6.3.3.2	Water Extraction Method	189
6.3.4	Spraying of $\text{AgNO}_3$ Solution and Representative Value of $C_d$	190
6.3.4.1	Spraying Method and Amount of $\text{AgNO}_3$ Solution	190
6.3.4.2	Representative Value of $C_d$	191

6.4	<i>Application of Colorimetric Method to Determine Chloride Diffusion/Migration Coefficient</i>	193
6.4.1	<i>Measurement of Non-Steady-State Chloride Diffusion</i>	193
6.4.1.1	<i>Measurement of Chloride Penetration Kinetics</i>	193
6.4.1.2	<i>Measurement of Apparent Chloride Diffusion Coefficient</i>	194
6.4.2	<i>Measurement of Non-Steady-State Electrical Migration</i>	195
6.4.2.1	<i>Effect of <math>C_d</math> on <math>D_{nssm}</math> Error</i>	197
6.4.2.2	<i>Error of <math>D_{nssm}</math> Based on Controlled Amount of Sprayed <math>AgNO_3</math> Solution</i>	198
6.4.3	<i>Evaluation for Corrosion Risk of Steel in Concrete</i>	199
6.5	<i>Chloride Ion Types in <math>C_d</math> and Absence of Color Change Boundary</i>	199
6.6	<i>Depth Dependence of Chloride Diffusion Coefficient Based on <math>AgNO_3</math> Colorimetric Method</i>	200
6.7	<i>Summary</i>	200
	<i>References</i>	202
<b>7</b>	<b>Factors Affecting Chlorides Transport in Cement-Based Materials</b>	<b>207</b>
7.1	<i>Introduction</i>	207
7.2	<i>Effect of Interaction between Ions on Chloride Transport</i>	208
7.2.1	<i>Model Describing Multi-Ion Transport</i>	208
7.2.2	<i>Theory on Interaction between Ions</i>	210
7.2.2.1	<i>The Chemical Potential between Ions</i>	210
7.2.2.2	<i>Lagging Motion of the Cations</i>	212
7.2.2.3	<i>Interaction between the EDL and Ionic Clouds</i>	213
7.2.3	<i>Concentration Dependence of Chloride Transport</i>	213
7.2.4	<i>Effect of Species on Chloride Transport</i>	215
7.3	<i>Effect of Micro-Structure on Chloride Transport</i>	216
7.3.1	<i>Effect of Pore Structure on Chloride Transport</i>	216
7.3.1.1	<i>Relation between Porosity and Chloride Transport</i>	216
7.3.1.2	<i>Relation between Pore Diameter and Chloride Transport</i>	218
7.3.2	<i>Effect of ITZ on Chloride Transport</i>	219

7.3.2.1	<i>Transport Properties of Chloride in ITZ</i>	220
7.3.2.2	<i>Effect of Aggregate Volume on Chloride Transport</i>	220
7.3.2.3	<i>Effect of Aggregate Shape on Chloride Transport</i>	223
7.3.3	<i>Coupling Effect of Pore Structure and ITZ on Chloride Transport</i>	223
7.4	<i>Effect of Chloride Binding on Chloride Transport</i>	225
7.4.1	<i>Describing Effect of Chloride Binding on Chloride Transport by Binding Isotherm</i>	226
7.4.2	<i>Transportable Chloride in Cement-Based Materials</i>	229
7.5	<i>Effect of Cracking on Chloride Transport</i>	230
7.5.1	<i>Cracking Formation Method in Laboratory Studies</i>	230
7.5.2	<i>Characterization of Cracking</i>	231
7.5.3	<i>Effect of Cracking on Chloride Transport</i>	232
7.5.3.1	<i>Effect of Cracking Width</i>	232
7.5.3.2	<i>Effect of Cracking Depth</i>	234
7.5.3.3	<i>Effect of Cracking Tortuosity, Orientation, and Density</i>	234
7.5.4	<i>Cracking Effect under Different Loading Level</i>	235
7.6	<i>Summary</i>	236
	<i>Bibliography</i>	237
<b>8</b>	<b>Simulation and Modeling of Chloride Transport in Cement-Based Materials</b>	<b>245</b>
8.1	<i>Introduction</i>	245
8.2	<i>Modeling Chloride Transport in Saturated Concrete</i>	247
8.2.1	<i>Empirical Model (Fickian Model)</i>	249
8.2.2	<i>Physical Models</i>	255
8.2.3	<i>Probabilistic Model</i>	274
8.3	<i>Modeling Chloride Transport in Unsaturated Concrete</i>	277
8.3.1	<i>Deterministic Model</i>	277
8.3.2	<i>Probabilistic Model</i>	279
8.4	<i>Chloride-Related Durability Codes</i>	282
8.4.1	<i>ACI Standards</i>	282
8.4.2	<i>Eurocode</i>	282



8.4.3	<i>Chinese Code</i>	284	
8.4.4	<i>Japanese Code</i>	284	
8.5	<i>Summary</i>	285	
	<i>References</i>	288	
	<i>Index</i>		293



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# Preface

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Chloride-induced corrosion of steel bar in concrete ranks number one in durability issues of reinforced concrete structures. Understanding the transport and interactions of chlorides in concrete is the first step in controlling the chloride-induced corrosion of reinforced concrete. Due to extensive research funded by governments or industries around the world, a great deal of knowledge has been generated, great advances have been made, and many engineering experiences have been obtained over the past few decades. Durability design has become dominant in the design of important costal infrastructures which may subject to chloride attack. However, this doesn't mean that we have completely understood the transport and interactions of chloride in concrete and can accurately predict and control it. Still, many scientific issues are yet to be clarified.

This book focuses on the chloride transport and interactions in cement and concrete. It reviews the general knowledge, technologies, and experiences involved in these areas, and presents the state-of-art progress made recently. The authors of this book have long been involved in research related to chloride transport and interactions in concrete at Hunan University, Ghent University, Central South University, and Xiamen University of Technology. Over the past few decades, many graduate students have obtained their M.Sc. or Ph.D. degrees in this field of study, and the theses are important sources for this book. Obviously, without the knowledge and experiences obtained by them, without their extraordinary works, this book would not be possible. The authors would like to express their gratitude to each and every one of them who have made contributions to the content of this book. Special thanks go to Dr. Runxiao Zhang, a post-doctoral fellow in the Department of Civil Engineering at the University of Toronto, for his participation in the preparation of Chapter 7, and the students who helped with organizing the format of entire book, as well as drawing the new graphs and tables.

The intended audience of this book includes students, researchers, and practicing engineers in the concrete community. This book can also be used as a reference book or extensive reading book for graduate courses on durability of reinforced concrete structures. Researchers may be inspired by

the comprehensive overview on the chloride transport and interaction in cement and concrete. Practicing engineers can also benefit from this book through learning the basic knowledge and practical techniques.

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# Introduction

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The deterioration of reinforced concrete structures caused by reinforcement corrosion is a worldwide concrete durability problem, particularly when the concrete structures are located in marine environments. Mostly, concrete constructions can be submitted to attacks from aggressive substances, such as chloride and carbon dioxide, chemically. Among these, chloride-related reinforcing steel corrosion accounts for the most of them. When chloride concentration in the pore solution around steel bars reaches a threshold value and breaks the passivation film, corrosion can be initiated at some connection points of the steel bar until the local damage of concrete. For concrete structures exposed to extreme conditions, such as marine exposition or highway structures where de-icing salt is used, the damage caused by chloride ingress does great harm to the durability of the reinforcement concrete structure and gives rise to high repairing and reconstruction costs.

### **I.1 CHLORIDE-RELATED CORROSION**

Normally, the concrete embedded in concrete is highly resistant to corrosion under the protection of cover layer. In well-designed and properly cured concrete with relatively low water to binder (w/b) ratio, the penetration of aggressive substances such as chloride ions and carbon dioxide can be effectively restricted from concrete surface to steel surface. The condition of high alkalinity ( $\text{pH} > 13.5$ ) in pore solution of cement-based materials provides an excellent condition for the passivation of steel (Singh and Singh 2012). In one view, steel can be protected from corrosion by the surface layer film (approximately 10000 Å thick), which generally consists of ferric oxide ( $\text{Fe}_2\text{O}_3$ ) and is thought to passivate the steel from corrosion (Shan et al. 2008; Waseda et al. 2006; Tittarelli and Bellezze 2010). It can be concluded that in a properly designed, constructed, and maintained reinforced concrete structure, the problem of steel corrosion should be neglected during the service life. However, these requirements on the design, construction, and maintenance of concrete structure are always not achieved in practice, and the corrosion of steel bar in reinforced structures

has become a common cause of the decreased durability and service life of concrete structures (Alexander and Nganga 2014).

Generally, it is considered that two stages are included in process of corrosion, especially for pitting corrosion: initial and propagation stages. The initial stage of corrosion relates to the breakdown of the passivation layer, while the propagation stage of corrosion describes the reaction between steel and electrolytes and the formation of corrosion products. There is general agreement about the mechanism of the propagation stage which is the transformation of electrons from anode to cathode surface of steel and the formation of electrical current in steel. In the propagation stage, corrosion products such as  $\text{Fe}(\text{OH})_3$ ,  $\text{FeSO}_4$ ,  $\text{Fe}_3\text{O}_4$ ,  $\text{FeO}(\text{OH})$ ,  $\text{HFeOOH}$ , and  $\text{HFeO}_2$  will be generated based on different composition and ionic concentration of pore solution (Bazant 1979). In this period, re-passivation of steel surface can occur when chloride concentration in pore solution around the steel surface is reduced. It was reported that sufficient chloride concentration was necessary for the propagation of steel corrosion and to keep the steel surface away from re-passivation (Eichler et al. 2009).

Generally, a passive oxide layer can be formed on the steel surface in alkaline conditions, which is followed by electrochemical deposition of polyaniline (PAni) (Jafarzadeh et al. 2011). The doped PAni layer is in contact with the oxidized surface and can stabilize the oxide ions from dissolution or take part in the corrosion process. It was reported (DeBerry 1985) that a PAni layer could be electrochemically deposited above the passive oxide layer formed on steel surfaces, which provide anodic protection from sulfuric acid corrosion. Even though some papers (Kraljić et al. 2003; Sathiyarayanan et al. 2008) have shown that no corrosion resistance could be provided, the corrosion protection of PAni and the oxide layer in different types of steel have been revealed by many researches (Chaudhari and Patil 2011; Johansen et al. 2012; Karpakam et al. 2011).

In order to describe the initial corrosion stage, several theories have been proposed (Kuang and Cheng 2014), such as local acidification theory (Galvele et al. 1978; Galvele 1976), depassivation–repassivation theory (Dawson and Ferreira 1986; Richardson and Wood 1970), chemical dissolution theory (Hoar and Jacob 1967), point defect models (PDM) (Chao et al. 1981; Urquidi and Macdonald 1985), chemical–mechanical models (Hoar 1967; Sato 1971), and anion penetration/migration models (Okamoto 1973; Rosenfeld and Marshakov 1964). Among all of these theories, a consensus was obtained that the adsorption of aggressive ions, such as chloride ions, plays an important role in the initial stage of steel corrosion (Angst et al. 2011; Cheng et al. 1999). The depassivation of protection layer on the surface of steel is directly induced by reaching the threshold value of chloride concentration in pore solution in contact with steel surface (Ghods et al. 2012). Bertocci and Ye (1984) reported that the most important role of chloride ions on steel corrosion rested on the increasing possibilities of local breakdown of the passive oxide layer. In some studies (Angst et al. 2011;



Liao et al. 2011), it was found that when chloride ions were introduced into the steel surface, no protective oxide layer could be formed in the anodic side and a chloride-ion film was formed to initiate the corrosion. The roles chloride ions played in steel corrosion can be: increase of iron solubility and conductivity of electrolytes, and dissemination of corrosion products (Lou and Singh 2010). Electronically, the existence of chloride ions accelerates the corrosion initiation by increasing the susceptible sites (Burstein et al. 1993) and decreasing the value of pitting potential (Tang et al. 2014; Xu et al. 2010).

Extensive studies (Otieno et al. 2016a; Tennakoon et al. 2017; Hou et al. 2016; Borg et al. 2018; Borade and Kondraivendhan 2019) have been conducted to investigate the effects of chloride-induced corrosion on durability and performance of concrete structures. It was considered that the corrosion of steel bars due to chloride penetration dominated the durability of concrete structures exposed to extreme condition, such as marine exposition or highway structures where de-icing salt was used. The damage caused by chloride ingress does great harm to the durability of reinforcement concrete structures and gives rise to high repairing and reconstruction costs. The deterioration of reinforced concrete due to chloride-related corrosion can be normally divided into four stages, as shown in Figure 1.1 (Berke et al. 2014; Budelmann et al. 2014): the initial cracking of concrete due to external applied pressure or shrinkage of concrete; the penetration of chloride ions in concrete cover and accumulates around the steel surface; the corrosion of steel which results in the formation and accumulation of corrosion products; the generation of cracks in concrete cover and fracture deterioration. Generally, the permeability of concrete cover and the diffusion rate of chloride ions are dominant factors of the initial stage, while oxide dissolution, moisture condition, and electrical resistance of concrete control the last three stages of steel corrosion.

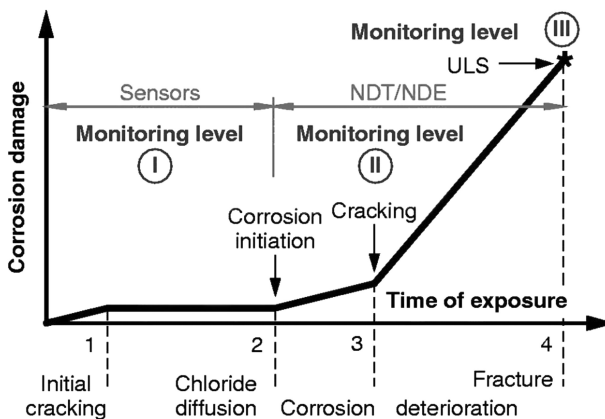


Figure 1.1 Steel corrosion stages in concrete. (From Budelmann et al. 2014.)

As it has become a major problem threatening concrete structures, studies on damage prevention in chloride-induced steel corrosion have been extensively conducted (Ann and Song 2007; Pour-Ali et al. 2015; Van Belleghem et al. 2018). According to the chloride-related corrosion mechanism described previously, efforts that have been made to prevent chloride corrosion involve keeping chloride ions from penetrating into steel surface (Pack et al. 2010; Song et al. 2008) and increasing the chloride binding capacity of cement matrix from the concrete point of view (Glass and Buenfeld 2000; Yuan et al. 2009).

### **1.2 CHLORIDE TRANSPORT IN CEMENT-BASED MATERIALS**

Generally, chloride ion can be introduced into concrete in two ways: (1) as an admixture (internal chloride); (2) penetration from external environment (external chloride), mainly from seawater and de-icing salt. The internal chloride can be well controlled by using chloride-free ingredients and choosing raw materials with low chloride content. But the chloride ions penetrated from the environment are mostly inevitable and difficult to predict and control. The service life of reinforced concrete depends more on chloride ions penetrating from the external environment. Thus, the transport of chloride in concrete has attracted a lot of attention. For the external chloride, no matter from where the chloride externally originated, by de-icing salt or seawater, chloride generally penetrates into concrete with the transportation of water within materials. When chloride ions continue to penetrate into concrete or chloride ingress is repeated, the chloride concentration around the steel surface can be relatively high.

As a homogenous porous material, cement concrete is mostly permeable by chloride ions from the surrounding environment. Based on different driven forces, the transport of chloride ions in cement concrete can be divided into five classification groups (Yuan et al. 2009): hydrostatic advection, capillary suction, diffusion, electrical migration, and thermal migration. The saturation degree of a concrete structure is always the foremost factor controlling the chloride transport process. For some marine constructions, concrete structures may always experience exposure to sea water and be exposed to dry condition periodically. When concrete is exposed to salt water or a high relative humidity environment, the salt water can enter and increase the chloride concentration in pore solution. If the external environment becomes dry with the removal of exposure water or decrease of relative humidity, the internal water of the concrete can evaporate and shift in different directions with the movement under wet conditions. During this process, only water can be evaporated, and salts are left. Based on the duration of the dry process, the water in the area of the concrete surface can be totally removed. Thus, the chloride concentration in this zone can

be increased and diffuses toward parts with lower concentration under the concentration gradient forces. Therefore, in dry conditions, the internal water moves outward from the concrete while salts move inward. Thus, in the next cycle of the wet process with salt water, more salts will be brought into samples with water. The chloride concentration profile along the penetration side may first be decreased, then increased. Generally, the wetting process occurs rapidly while longer a period of time for drying is needed. Therefore, the chloride ingress process and penetration rate depend on the length of the wetting and drying processes.

Studies have been done on ion and fluid transport in cement concrete structures with different saturation degrees, and several models have been proposed to describe the chloride ingress process within concrete structures (Jin et al. 2008; Yang et al. 2006; Otieno et al. 2016). By taking into account the diffusion and sorption of chloride ions in concrete, a durability simulator model was designed to simulate the marine environment with wetting–drying cycle (Iqbal and Ishida 2009). Chloride profiles were simulated by the proposed model with the moisture conductivity. In 1931, Richards (1931) firstly studied the mechanism of chloride transport in unsaturated porous solids, and an equation was proposed to describe the water flow under the capillary suction. Based on this work, Samson et al. (2005) developed a model to describe the ion transport in unsaturated cement systems by coupling the ionic and water transport models within materials. Generally, a parameter or model of dynamical moisture within systems is applied to take the water saturation degree into account during the chloride transport studies.

Besides the wetting–drying circles, an external applied electrical field can also accelerate the penetration of chloride salts into concrete, and it has been widely applied in rapid chloride migration tests. Compared to concentration gradient force, the external applied voltage can accelerate the ingress of chloride salts into concrete samples more significantly. According to the rapid chloride migration (RCM) test, where external electrical voltage was applied, it was reported that the obtained chloride migration coefficient was larger than chloride diffusion coefficient obtained from the natural diffusion test. Even though the RCM test has been widely applied to evaluate the chloride penetration resistance of concrete samples, many controversial issues still remain unsolved. Regarding the movement of chloride ions within cement-based materials, parts of them can be fixed by chemical or physical works between chloride ions and hydration products or other solid phases. These parts of chloride ions are defined as bound chloride, and this phenomenon is known as chloride binding. Free chloride ions in pore solution will be reduced due to chloride binding, and the flow of chloride ions with pore solution can be slowed down. However, the external applied voltage can bring some unexpected changes on chloride binding of cement matrix. On the other hand, the shortening of the ingress period due to electrical voltage can also change the content of chloride ions reacted with solid phase in samples.

Some researchers (Krishnakumark 2014; Spiesz and Brouwers 2013; Voinitchi et al. 2008) reported that the adsorption of chloride ions after reaching the steady state was independent of the applied voltage, and the applied voltage mainly affects the free chloride concentration in pore solution under steady state. Numerical simulation on non-steady-state diffusion and RCM tests showed that the free chloride ions in pore solution could be instantaneously combined, and no variation of free and bound chloride content was found between samples after diffusion and migration tests (Spiesz and Brouwers 2012). However, Spiesz and Brouwers (2013) showed that it took seven days or even longer for chloride binding in cement matrix to reach equilibrium in diffusion tests, which was much longer than the testing duration of RCM test. Castellote et al. (1999, 2001) analyzed the chemically bound and free chloride ions of the samples after RCM test using X-ray fluorescence technique and leaching method, respectively. In their studies, the obtained chloride adsorption isotherm was compared with that from diffusion test by Sergi et al. (1992). The applied electrical field suppressed chloride binding at lower free chloride concentration ( $<97$  g/L) while enhanced it at higher concentration. The decreased contact time and altered electrical double layer (EDL) property were considered as reasons to cause these differences.

It is apparent that under concentration gradient, wetting and drying circles, or external applied voltage, the chloride ions may progressively penetrate through the concrete cover layer toward the surface of steel. Then, along with the different depth from the concrete surface, a chloride profile can be established. As shown in Figure 1.2 (Toumi et al. 2007), the total chloride content gradually decreased with the increase of distance

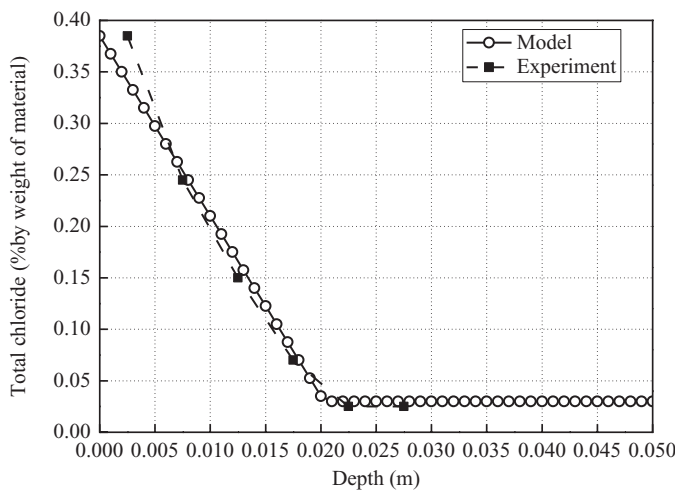


Figure 1.2 Concrete chloride content profile in immersion test. (From Toumi et al. 2007.)

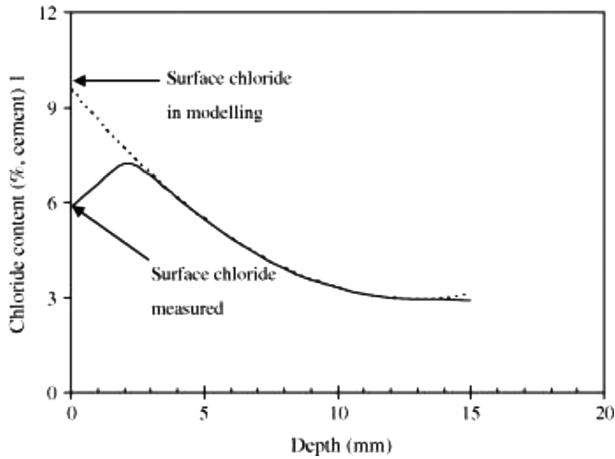


Figure 1.3 A decrease of the surface chloride content at the right surface of concrete in a conventional chloride profile. (From Ann et al. 2009.)

to concrete surface. Sometimes, the chloride concentration in the outmost layer will be reduced, as shown in Figure 1.3 (Ann et al. 2009). There is a sink of the chloride profile in the outermost layer of concrete. The rapid water movement during wetting and drying cycle and the decrease of bound chloride content due to the portlandite precipitation were considered as the reasons for this phenomenon (Ann et al. 2009). Generally, the chloride content results of the surface layer are ignored and mathematical fitting is applied to calculate the surface chloride content, as shown in Figure 1.3. Besides the total chloride content, the chloride profile for free chloride and chemically or physically bound chloride content have been also studied in the literature (Glass and Buenfeld 2000; Ishida et al. 2009).

### 1.3 INTERACTION OF CHLORIDE WITH HYDRATION PRODUCTS OF CEMENT

For concrete structures, chloride ions may derive from aggregates/mixing water (internal chloride) or penetrate into steel surface during exposure to chloride-bearing environments (intruded chloride). During the ingress process into cement concrete, the chloride ions in pore solution can be captured by the solid phases, including unhydrated cement component and hydration products of cementitious materials. The interaction between chloride ions and cement hydrates can be chemical or physical, and are defined as chemical binding and physical adsorption respectively. It was generally considered that the free-state chloride ions in pore solutions were mainly responsible for the corrosion of reinforcement steel (Yuan et al. 2009). However, Glass and Buenfeld (1997, 2000) stated that the bound

chloride ions could also induce the corrosion of reinforcement steel when they were released into pore solutions under certain conditions. The effects of chloride binding on chloride penetration and chloride-related corrosion can be considered as three aspects: (1) reduction of the free chloride concentration in the vicinity of the reinforcing steel, which will reduce the chance of corrosion; (2) removal of chloride from the diffusion flux, thus retarding the penetration of chloride to the level of the steel (Li et al. 2015); (3) formation of Friedel's salt, which results in a less porous structure and slows down the transport of chloride ion. Therefore, the effect of chloride binding must be taken into account when studying chloride ion transport in concrete. It can be seen from Figure 1.4 that the consideration of chloride binding can change the chloride profile (Martín-Pérez et al. 2000). However, it was also reported that chloride binding could have no remarkable effects on penetration depth (Ye et al. 2016).

Due to the retardation effect of bound chloride, the free and bound chlorides must be distinguished from each other in service life prediction models. Chemical binding is generally considered as the result of reaction between chlorides and  $C_3A$  or AFm phases to form Friedel's salt or the reaction with  $C_4AF$  to form a Friedel's salt analog (Florea and Brouwers 2012; Ipavec et al. 2013; Yuan et al. 2009). Physical binding is due to the adsorption of chloride ion to the C-S-H surfaces. Studies on mechanism of chloride binding of cement-based materials mostly focus on C-S-H (Shi et al. 2017) and AFm phase (Chen et al. 2015), as the former controls the physical adsorption while the latter dominates the chemical binding. However, the portlandite and ettringite (Ekolu et al. 2006; Hirao et al. 2005), also the Friedel's salt (Elakneswaran et al. 2009), formed through the interaction of other AFm phases and intruding chlorides can bind chloride ions (Florea and Brouwers 2012). Besides, the alumina phase in mineral component of cementitious materials such as  $C_3A$  and  $C_4AF$  can also bind the chloride ions and transfer into Friedel's salt. Usually, binding occurs instantaneously or at a much greater rate than transport velocities. The pore system is always considered to be at equilibrium. This assumption may be valid, when the chloride ion travels slowly, just in the case of diffusion alone, but this may not be valid when the ions are moving quickly and the test duration is short, as in the case of the rapid migration test. In this case, transport would be occurring too quickly for equilibrium to be maintained (Barbarulo et al. 2000; Samson et al. 2003). Tang and Nilsson (1993) reported that when crushed particles (with the size of 0.25–0.2 mm) were immersed in chloride solution, chemical binding almost completed after less than 14 days. However, Arya et al. (1990) found that bound chlorides were still increasing after 84 days of immersion in 2% chloride solution. Olivier (2000) believed that the rate of chloride binding on crushed mortar particles was very high. Indeed, more than 80% of the bound chlorides are bound in less than 5 h.

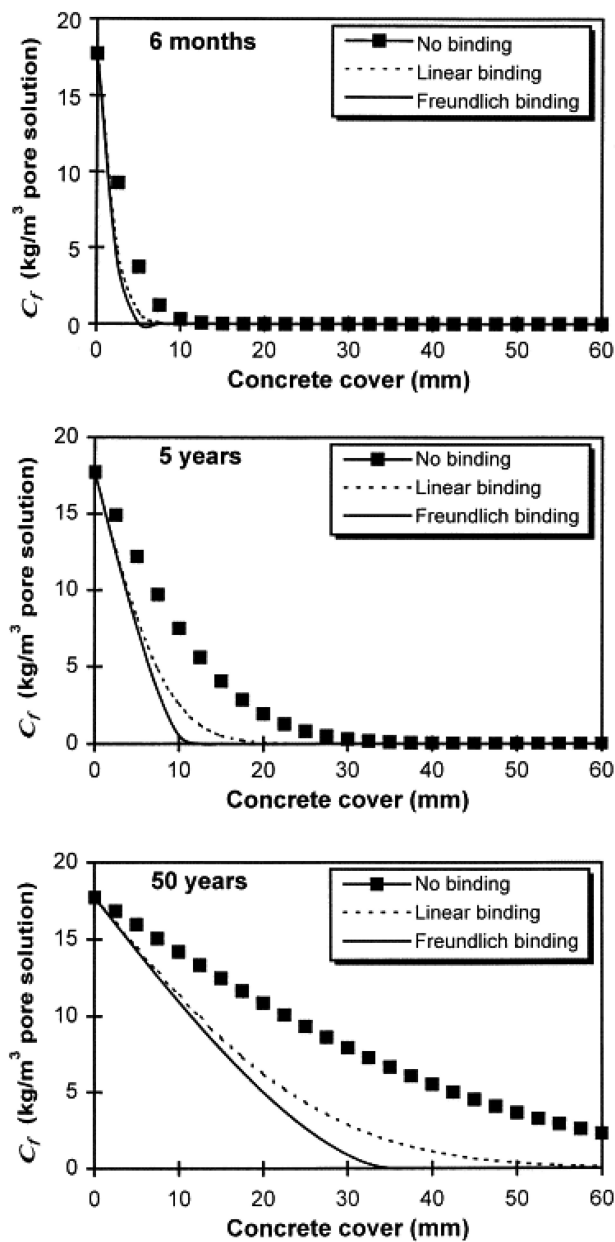


Figure 1.4 Free chloride concentration profiles at (a) 6 months, (b) 5 years, and (c) 50 years for 0.5 M exposure conditions. (From Martín-Pérez et al. 2000.)

The research on chloride binding has been carried out for a long time and in different cementitious systems, including cement-based materials (Gbozee et al. 2018; Shi et al. 2016) or alkali-activated materials (Ke et al. 2017a, b). In 1998, Justnes (1998) did an excellent work on reviewing the chloride binding in cementitious materials. The effects of cement type, mineral additives or replacement, cement content, water to binder ratio, curing and exposure condition, and chloride source on chloride binding of cementitious materials were discussed in detail. Besides, many experimental and review papers have also studied on different factors (De Weerd et al. 2014, 2015; Florea and Brouwers 2012) which can affect the chloride binding capacity of cementitious materials. According to these studies, it has been confirmed that the content of  $C_3A$  and  $C_4AF$  dominate the chemical binding of chloride ion, while  $C_3S$  and  $C_2S$  dominate physical adsorption. Hydroxyl and sulfate ions may decrease the chloride binding capacity of cementitious materials.

## **1.4 ORGANIZATION OF THIS BOOK**

This book summarizes recent progress in chloride-related issues in cement-based materials and structures. Focused on chloride-related corrosion occurring in cement concrete structures, the chloride ingress process, interaction between chloride ions and cement hydrates, influential factors, and testing methods for chloride transport in cement concrete are presented in detail. A brief introduction on the background of the topic of every chapter is firstly presented and is followed by a detailed summary of all related aspects of the topic.

### **1.4.1 Chapter 2—Mechanisms of Chloride Transport in Cement-Based Materials**

In this chapter, the chloride transport process in cement-based materials is studied, and different mechanisms based on the driven forces are presented. The mechanism and influential factors of different chloride transport methods, including hydrostatic advection, capillary suction, diffusion, thermal and electrical migration, are discussed. The contents of this chapter can help to understand the transport process of chloride ions in cement-based materials and also lay the foundation for discussions in the chapters that follow it.

### **1.4.2 Chapter 3—Chemical and Physical Interactions between Chlorides and Cement Hydrates**

In this chapter, mechanisms of chloride binding, including chemical binding and physical adsorption, are presented. Formation and stability of Friedel's salt are introduced as the main factors controlling the chemical-binding



capacity of cement-based materials. The properties of electrical double layer formed at solid–liquid interface are discussed to explain the phenomenon of “chloride concentrate,” which can be considered as an instable physical adsorption between hydration products and chloride ions.

### **1.4.3 Chapter 4—Chloride Binding and Its Effects on Characteristics of Cement-Based Materials**

In this chapter, different forms of binding isotherm used to describe the chloride binding capacity of cement-based materials are presented. The application of binding isotherms in chloride binding capacity evaluation is discussed. The effects of chloride binding on microstructure and properties of cement concrete structure are summarized.

### **1.4.4 Chapter 5—Testing Methods for Chloride Transport in Cement-Based Materials**

In this chapter, classifications of testing method for chloride transport in cement-based materials are reported. Testing procedure, parameters, and influential factors of these measurements are overviewed. Relationship and comparison of testing results of these different methods are also provided.

### **1.4.5 Chapter 6—Determination of Chloride Penetration in Cement-Based Materials Using $\text{AgNO}_3$ -Based Colorimetric Methods**

As an important testing method on chloride penetration depth determination,  $\text{AgNO}_3$  colorimetric method has been widely applied. The factors affecting the testing results for penetration depth and chloride concentration at the color change boundary are discussed. The applications of  $\text{AgNO}_3$  colorimetric method in chloride diffusion and migration coefficients determination are also reviewed.

### **1.4.6 Chapter 7—Factors Affecting Chlorides Transport in Cement-Based Materials**

Chloride transport is a complex process in which chemical and physical reactions simultaneously occur. During the chloride transport process, any changes of properties, microstructure of materials, and environmental conditions can cause effects on the chloride transport process. In this chapter, factors that may influence the chloride transport of cement-based materials, including ion interaction, microstructure, chloride binding, and cracking are introduced and discussed in detail.

### 1.4.7 Chapter 8—Simulation and Modeling of Chloride Transport in Cement-Based Materials

Chloride transport is one of the main aspects in the establishment of service life prediction of concrete structures, and different models simulating the chloride transport process in cement-based materials have been proposed. Based on the knowledge and discussion in the previous chapters about chloride transport, chloride binding, and their relationship with the microstructure of the structure, this chapter summarizes different chloride transport models proposed for cement-based materials and their application in different materials and environmental conditions.

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