



# Influence of carbonation on “maximum phenomenon” in surface layer of specimens subjected to cyclic drying-wetting condition

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## ABSTRACT

Numerous researches have reported that there is a tendency chloride content first climbs to the maximum then declines with depth increasing in the surface layer of concrete under cyclic drying-wetting environments, which is temporally called ‘maximum phenomenon’ in this paper. This research focuses on the impact of different carbonation conditions on this phenomenon for both pastes and mortars. The distribution of chloride suggests that coupled effect of carbonation and capillary suction/moisture evaporation is more likely to lead to the formation of maximum phenomenon than the merely effect of capillary suction/moisture evaporation. Furthermore, analysis of pore structure and phase composition reveals that this particular phenomenon is directly related to the release of bound chloride fixed in Friedel's salt triggered by carbonation. In addition, the forming process of maximum phenomenon is proposed in the end based on Friedel's salt decomposition under cyclic drying and wetting condition.

## 1. Introduction

One major cause of the durability problems of reinforced concrete structure is steel corrosion. Steel corrosion not only reduces the cross-sectional area of steel bars but also leads to the dilating and cracking of concrete protective layer. In consequence, the service life and safety of reinforced concrete structures are in peril. Carbonation and chloride ingress are accountable for the destruction of the passivity layer protecting steel from corroding. Carbonation decreases the pH of concrete in the proximity of steel bars, which invalidates the protective layer of concrete. Meanwhile, when chloride ions intrude from chloride attack environment (like marine environment and deicing salt environment) or are introduced from inward incidentally, the passivity layer will also be destroyed, and the steel starts to corrode even if at high pH values [1,2].

According to previous researches, cyclic drying-wetting chloride attack environment is the most aggressive exposure condition for reinforced concrete structures [3–5]. Under such condition, the rate of chloride ingress increases significantly due to the combined action of capillary suction and diffusion. Furthermore, the effect of carbonation is more serious compared with that in total submersion condition since CO<sub>2</sub> can penetrate into matrix directly during drying process while the rate of carbonation is very low in total submersion condition [6,7].

Researches on chloride transport in cyclic wet-dry environments have been widely conducted, many of which [8–12] discovered a phenomenon that chloride content first climbs to the maximum and then declines with depth increasing in the surface layer of concrete. It is temporarily called “maximum phenomenon” in this paper for convenience. And this phenomenon is believed to generally exist in specimens subjected to cyclic dry-wet environments though it was not observed in chloride profiles of some studies [13–15]. The “not appearing” of it in some studies is supposed to be caused by either short exposure time [16], or ignorance of chloride content in the surface layer [13,14], or large intervals between points where chloride concentration is detected [8]. Maximum phenomenon is getting increasing attention recently because its appearance influences the accuracy of using the basic diffusion equation of Fick's second law to predict the service life of concrete [3,8,9,13,14,18]. Four possible reasons responsible for its appearance have been recognized so far: skin effect, rain washing, capillary suction/moisture evaporation and carbonation. However, the mechanisms about how these aspects work to cause the phenomenon are remained unclear.

The surface layer of concrete usually has a different composition from the internal concrete due to phenomena such as contact with molds or segregation of aggregates [70], which results in different pore structure in the two parts. In consequence, the chloride resistance in

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surface layer and inner part differs. And chloride ions could accumulate at the interface between the two parts, inducing the formation of chloride content peak gradually. This is called “skin effect” [19], and the maximum phenomenon caused by it usually appears at superficial position, at about 2.0 mm. Some experiment results [20,71] could be explained by this effect, but the position of maximum phenomenon detected in many other researches [8,21,26,73,77] are clearly beyond the influence of it. In terms of rain washing effect, in the natural environments, rain water dissolves and washes away chloride ions in the surface layer of concrete, which decreases surface chloride concentration and may probably lead to the maximum phenomenon [21–24]. Yet this phenomenon also occurs in indoor cyclic dry-wet conditions where rain washing cannot reach.

Capillary suction/moisture evaporation refers to a process that first salt solution quickly enters into some depth of the surface layer driven by the capillary suction force when wetting starts, and then in the following drying process water evaporates from the exposed surface and the dissolved salts are deposited there in the form of crystal [25]. Combined action of capillary suction and moisture evaporation will not only leads to the mass accumulation of chloride in the surface layer [21,23,26–28] but also makes it accumulate at a certain depth due to hysteretic moisture behavior [5,29,30]. Although the occurrence of maximum phenomenon can be successfully predicted by models merely based on capillary suction/moisture evaporation theory [30–32], it is difficult to verify the reliability of these models. It is mainly because the maximum phenomenon does not necessarily happen when capillary suction/moisture evaporation seems to be exclusively taking place, in disagreement with models' prediction [30,32]. Another challenge is the complete precluding of carbonation during the course of experiment, since both carbonation and capillary suction/moisture evaporation can influence the chloride distribution [26], especially pozzolanic materials are contained within cement composites.

Models taking carbonation into consideration can also predict the occurrence of maximum phenomenon [33,34,69], among which it is found that this phenomenon would take place in a more profound way with the accelerated carbonation effect. More interestingly, the peak of chloride concentration appears to be close to carbonation front [34–37]. Moreover, the electron probe macro analyzer results of Liu [38] present that chloride ions are redistributed under the mere action of carbonation and there appears the maximum phenomenon. Therefore, it is believed that carbonation also plays a very important role in leading to the formation of maximum phenomenon under cyclic drying-wetting conditions.

As a matter of fact, the influence of carbonation on chloride transport is manifested both physically and chemically. The former refers to that carbonation can lead to the reduction of total porosity and redistribution of pore size [33,39–42], which may retard inward chloride penetration [43,44]. And the latter means that some chloride ions fixed by chloro-aluminate compound, especially by Friedel's salt, are released into pore solution under the action of carbonation [34,35,45,46,78]. More investigations are needed to clarify the significance of these two influences under cyclic drying-wetting environments.

Therefore, to reveal the impact of carbonation on maximum phenomenon in the surface layer of specimens under cyclic drying-wetting conditions, pastes and mortars with different water to binder ratios ( $W/B$ ) and mineral admixtures were exposed to five carbonation conditions. Free chloride profiles and pH profiles of the same specimen under different conditions were determined to reflect the relation between chloride distribution and carbonation. Besides, mercury intrusion porosimetry (MIP) and quantitative X-ray diffraction (XRD) were also conducted as a supplementary analysis.

**Table 1**  
Chemical composition (%) of cement P.II.52.5, SL and FA.

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	Na <sub>2</sub> O	TiO <sub>2</sub>
P.II. 52.5	19.5	4.32	63.6	2.92	0.658	1.35	0.129	0.262
SL	32.1	16.7	39.3	0.746	0.348	6.41	0.151	0.895
FA	45.4	36.3	6.97	5.30	0.864	0.599	0.314	1.65

## 2. Experiments

### 2.1. Materials and mixing proportion

In this research, Portland Cement P.II. 52.5, which is produced in Jiangnan-xiaoyetian Cement Co., Ltd., Nanjing, China, was used. And the chemical composition of this cement is given in Table 1. The granulated blast-furnace slag (SL) with the fineness of 423 m<sup>2</sup>/kg and fly ash (FA) with the fineness of 625 m<sup>2</sup>/kg were used as mineral admixtures, and their mineral composition are also listed in Table 1. The fine aggregate is river sand with a fineness modulus of 2.20.

Eight mixes of paste and three mixes of mortar specimens were molded to explore the influence of different  $W/B$  and mineral admixtures on chloride transport. The mix proportions are shown in Table 2.

### 2.2. Sample preparation

The size of paste specimens is 40 mm × 40 mm × 40 mm. To avoid segregation of high  $W/B$  paste specimens, the special molds were sealed with plastic sheet and adhesive tape after casting, and then these molds were rotated slowly until pastes were totally hardened. The size of mortar specimens is 70 mm × 70 mm × 70 mm. After casting, the molds were sealed with cling film on the surface to reduce moisture evaporation. Then, both paste and mortar specimens were stored at 20 ± 1 °C and 90% relative humidity for 24 h. Afterwards, they were demolded and cured for 28 days at 20 ± 1 °C 20 mm thickness of each mortar specimen was cut off from the mold free surface to guarantee the homogeneity of specimens after curing. Before subjecting to prescribed exposure conditions, all the faces of the paste and mortar specimens were sealed with epoxy resin except for the cutting surface.

### 2.3. Exposure condition

The specimens were subjected to five different exposure conditions, as shown in Table 3. The dosage of NaCl for all solutions is 3.5% by mass. The ratio of drying time to wetting time is 6:1, one cycle a week. Paste specimens are exposed for 4 weeks(t1), 8 weeks(t2), and 12 week (t3), mortar specimens are exposed for 12 weeks(t3). The reasons for the design of each exposure condition are as follows:

Under condition A, specimens were immersed into chloride solution, and chloride transport is dominated only by diffusion, without the influence of capillary suction/moisture evaporation. And carbonation can be neglected due to the fairly low CO<sub>2</sub> concentration in the solution. Samples exposed to condition A can serve as reference since diffusion

**Table 2**  
Mix proportions of pastes and mortars.

	W40	W50	W60	W70	SL20	SL40	FA20	FA40
Cement (%)	100				80	60	80	60
SL substitution (%)	0				20	40	0	0
FA substitution (%)	0				0	0	20	40
Paste $W/B$	0.40	0.50	0.60	0.70	0.50			
Mortar $W/B$		0.50			0.50		0.50	
Sand-cement ratio	3							

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