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EFFECT OF CYCLIC NaCl SOLUTION IMMERSION CURING ON THE MECHANICAL PROPERTIES OF PLAIN CONCRETE

MEHANSKE LASTNOSTI NEOJAČANEGA GRADBENEGA BETONA PO OBDELAVI Z IZMENIČNIM POTAPLJANJEM V SLANICI

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The effects of cyclic curing (CC) alternating between NaCl solution immersion and drying for 28 d on the microstructure, phase composition and mechanical properties of ordinary concrete are studied in comparison with those cured by standard curing using municipal water. Compared to standard curing, CC enhances the compressive strength, flexural strength and static compressive elastic modulus of concrete by (5.71, 11.14 and 8.06) %, respectively. $Ca(OH)_2$ is significantly reduced, while C-S-H is increased, and a minor amount of $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ is formed. Hydration products become finer, more uniform and denser. The use of CC is proposed as it improves the strength.

Keywords: concrete, microstructure, phase compositions, mechanical property, curing, NaCl solution

Avtorji v članku opisujejo analizo učinkov cikličnega potapljanja običajnega betona v slanico (vodno raztopino s 3,5 % NaCl) in njegovo alternativno obdelavo s staranjem oziroma sušenjem pri sobni temperaturi za 28 dni (angl.: CCA curing). Analizirali so mikrostrukturo, fazno sestavo in mehanske lastnosti tako obdelanih vzorcev betonov in jih primerjali s standardno obdelanim betonom z uporabo mestne (pitne) vode. Primerjava lastnosti CCA betona s standardno obdelanim betonom je pokazala, da ima CCA beton za 5,71 % višjo tlačno trdnost, 11,4 % višjo upogibno trdnost in izboljšan statični tlačni elastični modul za 8,06 %. Vsebnost $Ca(OH)_2$ se je pomembno zmanjšala in povečal se je delež C-S-H vezi, tvorila se je zanemarljiva vsebnost $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$. Produkti hidracije so postali finejši (drobnejši), bolj enakomerne velikosti in z večjo gostoto. V članku so avtorji razložili mehanizem utrjevanja CCA betona in v zaključku članka priporočili uporabo CCA obdelave betona, če se zahteva njegova večja trdnost.

Ključne besede: beton, mikrostruktura, fazna sestava, mehanske lastnosti, obdelava s potapljanjem v solno raztopino

1 INTRODUCTION

Concrete is the most widely used building materials at present.[1] Mixing and curing are essential steps in the process of concrete preparation and they have a direct impact on the performance of concrete.[2–7] In most cases, the mixing and curing of concrete require the use of municipal water, usually fresh water, as recommended in EN 1008-2002,[8] C1602/C1602M–12,[9] ACI 308.1M-11,[10] C31/C31M-21a,[11] JGJ 63-2006 (Chinese standard),[12] etc.

There is a serious shortage of water resources in the arid saline alkali areas of Northwest China, such as the Tarim Basin, Turpan Basin, Jungar Basin, Qaidam Basin, etc. In these areas, it is usually difficult to ensure sufficient fresh water supply, but it is relatively easy to obtain chlorinated salt (mainly composed of NaCl) water at a construction site.[13–15] Therefore, it is a promising option to use chlorinated salt water around a construction site for concrete curing.

It is generally recognized that chlorine usually results in a strength degradation of concrete due to the reaction between chloride ions and the cement aluminate phase (C3A) as well as the leaching of calcium hydroxide and C-S-H.[16–20] On the other hand, it is revealed that chloride ions can react with the aluminate phase to form Friedel's salt, which can block the pores in cement and is therefore beneficial for the strength improvement. In addition, the research of the effect of sea water as the mixing water on hydration and mechanical properties of ordinary Portland cement (OPC) revealed that the chloride in sea water can obviously accelerate the tricalcium silicate hydration rate at the early age. And thus, the early compressive strength (7 and 14) d of the OPC mortar using sea water as the mixing water increased rapidly compared to that using distilled water as the mixing water.[21,22] However, it is found that at a later age of 28 d, C-S-H remains non-hydrated in the OPC paste using sea water as the mixing water and its compressive strength is lower than that of the OPC paste using distilled water as the mixing water. Experimental results demonstrated that, compared with the curing of OPC with fresh water, the curing with continuous immersion in a sodium chloride (NaCl) solution deteriorates the strength of concrete.[20,23,24] The research shows that the strength of concrete may be different even if the curing medium is the

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Table 1: Physical properties of the coarse and fine aggregates

Aggregate	Water absorption (%)	Apparent density ($kg \cdot m^{-3}$)	Bulk density ($kg \cdot m^{-3}$)	Stacking density ($kg \cdot m^{-3}$)	Clay content (%)	Crushing index (%)
Fine	5.3	2640	1570	1940	2.3	-
Coarse	0.22	2720	1522	1650	0.9	11.7

same and only the curing process is different.[3,25–27] For example, the work by Raza et al. demonstrated that for plain concrete, the ponding method of concrete curing is more effective than sprinkling or wet cover curing method.[3] Compared to continuous immersion in water, the curing by alternating between the wetting and drying methods, during which concrete samples are subjected to 12-h curing in water, followed by 12-h curing in ambient air, has an adverse effect on the strength.[27] At a construction site, the curing of concrete by alternating between wetting and drying is more economical and feasible. Therefore, it can be concluded that the effect of chlorinated salt water on the concrete cured by alternating between wetting and drying might be different from that of curing with continuous immersion in chlorinated salt water. Unfortunately, reports on curing concrete with chlorinated salt water by alternating between wetting and drying is lacking.

In the present work, ordinary-concrete samples are cured for 28 d with standard curing using municipal water and with cyclic curing, alternating between NaCl solution immersion and drying. The effects of the cyclic curing with NaCl solution immersion and drying on the phase composition, compressive and flexural strength of OPC are investigated.

2 MATERIALS AND METHODS

The used cement is P.O. 42.5R ordinary Portland cement (Jidong Cement Co., Ltd., China). The used-water reducing agent is SBTJM–II (Sobute New Materials Co., Ltd., China). Gravel of (5 and 20) mm is used as the coarse aggregate. Sand from the banks of the Weihe River with a fineness modulus of 2.2 and apparent density of 2.64 g cm^{-3} is adopted as the fine aggregate. **Table 1** lists the physical properties of the coarse and fine aggregates. **Figure 1** shows the grading curve of the aggregates obtained with a screening test.

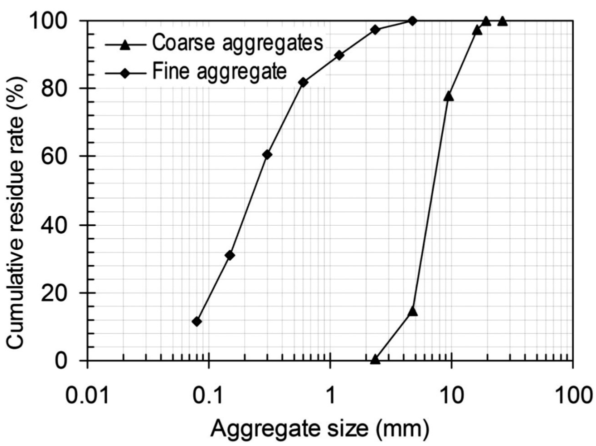

Figure 1: Aggregate grading curve

Municipal water from the Yangling City is used as the mixing water. The mix proportions of concrete are shown in **Table 2**. The tested air content of the mixture and the slump are 4.4 % and 94 mm, respectively. The measured concrete density is 2350 $kg \cdot m^{-3}$.

Table 2: Mix proportions of concrete ($kg \cdot m^{-3}$)

W/C ratio	Cement	Water	Coarse aggregate	Sand	Water reducing agent
0.50	314	157	1069	840	3.14

For the tests, three types of samples are molded. Cubic samples with nominal dimensions of (100 × 100 × 100) mm are molded for the compressive strength test. Cylindrical samples with a diameter of Ø150 mm and height of 300 mm are molded for the static compressive modulus test. Prism samples with nominal dimensions of (100 × 100 × 400) mm are molded for the bending strength test.

After 24 h, samples are demolded and then cured with two methods. The first one is standard curing in a chamber at 20 °C with a relative humidity (R.H.) of 95 % for 28 d. The second one is cyclic curing alternating between NaCl solution immersion and drying, designated as CC. During the latter, samples are immersed in an NaCl solution with a concentration of 3.5 % for 4 d, then dried for 3 d.

The uniaxial compressive strength and the flexural strength of the samples are carried out on an Instron 1195 machine at room temperature (25 °C and 65 % R.H.); every test involves the testing of three samples in each configuration. The loading rate for the compressive strength test and flexural strength test is 0.3 $MPa \cdot s^{-1}$ and 0.2 $mm \cdot min^{-1}$, respectively. Compressive strength is calculated with the average value of the test values that are selected with an error range within 10 %. The flexural strength of the samples is tested using the three-point bending method with a span of 300 mm. A WE–1000 hydraulic universal testing machine and MT–II elastic modulus tester are used for static compressive elastic modulus testing as shown **Figure 2**. An MT–II elastic modulus tester is placed in the middle part of a sample with a distance to the upper end surface and lower end surface of 75 mm. The samples are continuously loaded at a rate of 0.3–0.50 $MPa \cdot s^{-1}$ until they are damaged. The secant elastic modulus with the stress ranging from

Figure 2: Photo of static compressive elastic modulus testing

0.5 MPa to 40 % of the failure stress is taken as the static compressive elastic modulus. The static compressive elastic modulus testing is conducted on four samples of each group.

The flexural strength of the samples, σ_f, is calculated with the following equation[28]

$$\sigma_f = \frac{3P_{\max} L}{2bh^2} \quad (1)$$

where L denotes the span during the bending test and is equal to 300 mm; b and h are the width and height of the cross-section of a bending sample, respectively.

X-ray diffraction (XRD; BRUKER, D8 ADVANCE A25 X) is used for phase composition characterization of the samples. The samples are washed with water and dried before conducting the XRD examination. XRD is operated at 40 KV and 40 mA, in a range of 2θ = 10–70° and with a 0.02 step, respectively. The microstructures of the samples are checked with a scanning electron microscope (SEM; ZEISS Gemini 300) equipped with EDS (OXFORD Xplore).

3 RESULTS AND DISCUSSION

Table 3 lists the failure load, and the corresponding compressive and flexural strengths of the concrete samples with the two curing methods. The compressive strength (converted to the equivalent strength of a standard sample with dimensions of (150 × 150 × 150) mm and flexural strength of the concrete cured with standard curing are 44.25 MPa and 5.02 MPa, respectively. The compressive strength (converted to the equivalent strength of a standard sample with dimensions of (150 × 150 × 150) mm and flexural strength of the concrete cured with cyclic curing including NaCl solution immersion and drying are 46.77 MPa and 5.58 MPa, respectively. Therefore, compared with those samples cured with standard curing, the compressive strength and flexural strength of the concrete cured with cyclic curing including NaCl solution immersion and drying are improved by 5.71 % and 11.14 %.

Figure 3 shows the typical load-displacement curves of the concrete during bending. The load-displacement curves of the concrete cured with the two methods during bending are similar, suggesting typical brittle damage. Therefore, it seems that cyclic curing alternating between NaCl solution immersion and drying does not change the damage model of concrete. However, the breaking load and corresponding deformation of the concrete after cyclic curing including NaCl solution immersion and drying for 28 d are higher than those of the concrete after 28 d of standard curing.

Figure 4 shows the representative stress-strain data and their fitting results for the concrete samples during

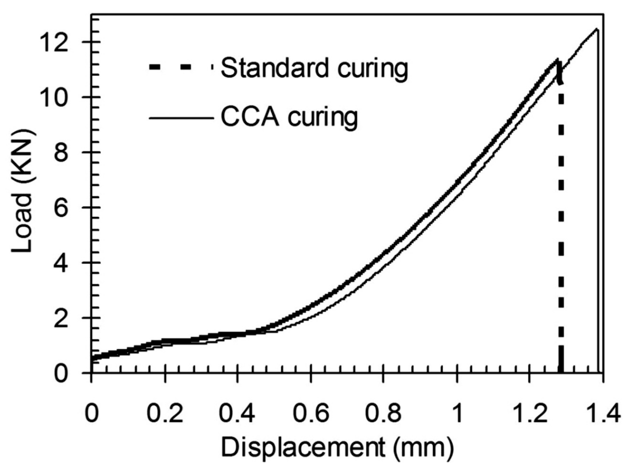

Figure 3: Typical load-displacement curves of the concrete during bending

Table 3: Compressive and flexural strength of the concrete

Curing method	Compressive strength				Bending		
	Maximum load (kN)	Strength (MPa)	Average strength (MPa)	Effective strength (MPa)	Maximum load (kN)	Strength (MPa)	Average strength (MPa)
CC	516.91	51.69	49.24	46.77	12.49	5.62	5.58
	492.41	49.24			12.18	5.48	
	467.77	46.78			12.54	5.64	
Standard	480.91	48.09	46.58	44.25	11.32	5.09	5.02
	415.34	41.53			11.29	5.08	
	501.00	50.10			10.87	4.89	

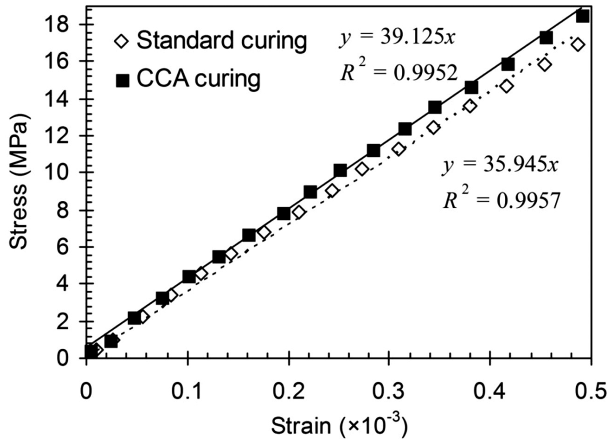

Figure 4: Linear fitting of the representative stress-strain data for the concrete samples during static compressive elastic modulus testing

the static compressive elastic modulus testing with the stress ranging from 0.5 MPa to 40 % of the failure stress. It seems that the stress and strain during the static compressive elastic modulus testing of the concrete samples cured with the two methods can be well fitted by linear fitting.

The static compressive elastic modulus of the concrete cured with standard curing is 35.51 GPa, while that of the concrete cured with cyclic curing alternating between NaCl solution immersion and drying is 38.37 GPa, enhanced by 8.05 %. The above mechanical-property results indicate that cyclic curing including NaCl solution immersion and drying has a positive effect on the mechanical properties of concrete. And these results are different from those for the samples cured with NaCl solution immersion.[20,23,24]

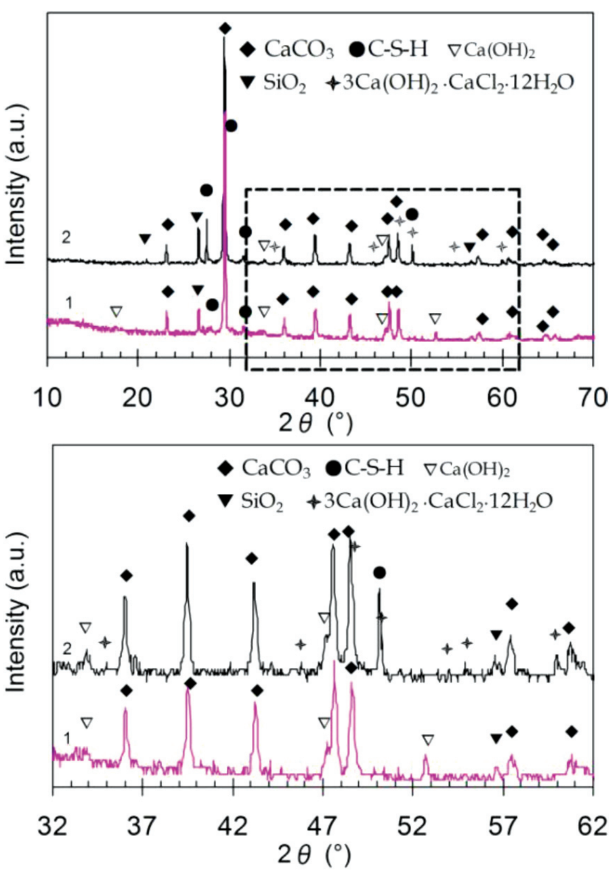

Figure 5: XRD patterns of the concrete after 28 d curing: 1 – standard curing; 2 – CCA curing

Figure 5 shows XRD patterns of the concrete after standard curing and cyclic curing alternating between NaCl solution immersion and drying for 28 d under the same diffraction conditions. It can be found that the main products of both cured samples are $CaCO_3$, $Ca(OH)_2$ and C-S-H. In addition, from the magnified view at a 2θ range of 32–62°, a weak signal of $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ can be detected in the concrete cured with cyclic curing including NaCl solution immersion and drying. However, it should be noted that the content of $Ca(OH)_2$ is significantly reduced, while the content of C–S–H is significantly increased in the sample cured with cyclic curing including NaCl solution immersion and drying, compared with the sample cured with standard curing.

Figure 6 shows the microstructures of the concrete after standard curing and cyclic curing alternating between NaCl solution immersion and drying for 28 d. It seems that the sample cured with cyclic curing including NaCl solution immersion and drying is denser than that cured with standard curing. Moreover, from a magnified view, it can be seen that the sample cured with cyclic curing including NaCl solution immersion and drying has fine and uniform spherical crystals as shown in **Figure 6c**, while the sample cured with standard curing has relatively large, short, columnar or platelet-shaped crystals, shown in **Figure 6d**. Finer and more uniform crystals as well as a denser morphology suggest that a hydration process enhances nucleus formation and is thus beneficial for the strength.

Due to the porous structure of silicate concrete, chloride ion can diffuse inward easily. During immersion in the NaCl solution, free Cl^- inside the concrete might react with hydration-formed Ca^{2+} as shown with Equation (2)[29] and thus forms $CaCl_2$ as shown with Equation (3).[25] Furthermore, $CaCl_2$ reacts with $Ca(OH)_2$ to form insoluble solid phase $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ as illustrated with Equation (4).[21,30] The formation of $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ leads to an accelerated hydration of concrete,[21] accompanied with an increased hydration temperature. This in return results in an accelerated formation of C–S–H[29–33] as illustrated with Equation (5). As a result, the cement shows finer and more uniform crystals as well as a denser morphology. Moreover, ionic exchange between calcium silicate hydrates and NaCl, shown with Equation (6), also leads to $Ca(OH)_2$ consumption.[34,35] Therefore, a minor amount of $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ can be detected; the content of $Ca(OH)_2$ is significantly reduced, while that of C-S-H is significantly increased in the sample cured with cyclic curing including NaCl solution immersion and drying, com-

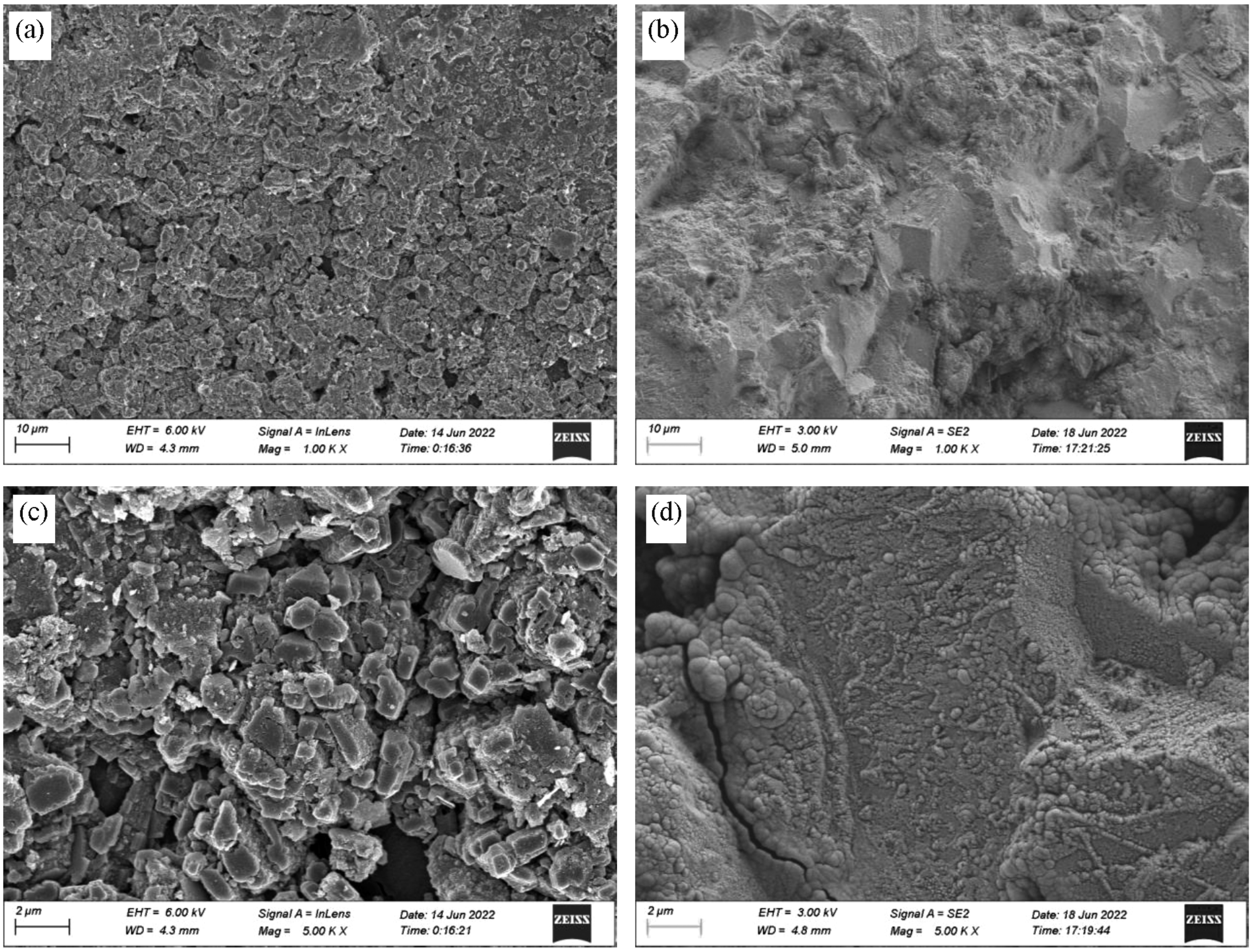

Figure 6: Micromorphologies of the concrete after 28-d curing: a), c) standard curing; b), d) CC

pared with the sample cured with standard curing. According to possible reactions (3) and (4), the NaOH and $Na_2SiO_3{\cdot}H_2O$ products are easily dissolved in water and their contents are very small. The concerned samples are washed with water and dried before the XRD examination. Therefore, the signal of Na^+ cannot be detected by XRD for the CC-cured sample.

$$Ca_3SiO_5 + 3H_2O \rightarrow 3Ca^{2+} + 4OH^- + H_2SiO_4^{2-} \quad (2)$$

$$3Ca^{2+} + 4OH^- + H_2SiO_4^{2-} + 2NaCl \rightarrow 2Ca(OH)_2 + CaCl_2 + Na_2SiO_3 \cdot H_2O \quad (3)$$

$$3Ca(OH)_2 + CaCl_2 + 12H_2O \rightarrow 3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O \quad (4)$$

$$2Ca^{2+} + 2OH^- + H_2SiO_4^{2-} \rightarrow C\text{–}S\text{–}H \quad (5)$$

$$C\text{–}S\text{–}H\text{–}OH^- + Na^+ + Cl^- \rightarrow C\text{–}S\text{–}H\text{–}Cl^- + Na^+ + OH^- \quad (6)$$

During curing with continuous immersion in the NaCl solution, the rapid formation of cementitious $Ca(OH)_2$ and C–S–H due to accelerated hydration is beneficial to the strength. However, the hydrated cementitious layer rapidly forms on the periphery of the sample, filling in the pores by forming expansive $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ that prevents the diffusion of chloride ions into the interior,[19,36] thus reducing internal hydration. Moreover, the expansion of $3Ca(OH)_2{\cdot}CaCl_2{\cdot}$ $12H_2O$ may deteriorate the rapidly formed cementitious layer on the periphery. As a result, curing with continuous immersion in the NaCl solution deteriorates the strength of concrete as reported by others.[20,23,24] In the case of cyclic curing alternating between NaCl solution immersion and drying, the accelerated formation of cementitious $Ca(OH)_2$ and C–S–H, filling in the pores by forming expansive $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ during the first cycle of immersion in the NaCl solution is limited. Therefore, this curing cycle is favorable for the strength enhancement. The drying cycle makes the concrete more evenly hydrated inside and outside so that the periphery of the sample can maintain a certain degree of pore opening. During the subsequent NaCl solution immersion, chloride ions can still diffuse inside along the pores and thus accelerate the inner hydration. The previously formed $3Ca(OH)_2{\cdot}CaCl_2{\cdot}12H_2O$ and C–S–H on the pore walls prevent chloride ions to diffuse through the pore walls and thus impede the plugging of the pores. As a result, the internal strength can be gradually improved during the subsequent curing cycles. Consequently, unlike the curing with continuous immersion in the NaCl solution, the cyclic curing including NaCl solution immersion and drying is beneficial for the mechanical properties of OPC.

It should be noted that Friedel's salt is not detected by XRD in the present work. It is supposed that the formation of $3Ca(OH)_2·CaCl_2·12H_2O$ and accelerated formation of hydrated calcium silicate due to the acceleration of hydration lead to a reduction of $Ca(OH)_2$. On the other hand, the ionic exchange between calcium silicate hydrates and NaCl also leads to the $Ca(OH)_2$ consumption. Thus, the reduction of $Ca(OH)_2$, which can participate in the reaction producing Friedel's salt, limits the formation of Friedel's salt.

4 CONCLUSIONS

Compared to standard curing in water, cyclic curing alternating between 3.5 % NaCl solution immersion and drying (CC) has a positive effect on the mechanical properties of concrete. The compressive strength, flexural strength and static compressive elastic modulus of the concrete cured by CC for 28 d are enhanced by (5.71, 11.14 and 8.06) %, respectively, compared to those obtained with standard curing using municipal water.

The main products for both cured samples are $CaCO_3$, $Ca(OH)_2$ and C–S–H. Compared to standard curing using municipal water, CC results in significantly reduced $Ca(OH)_2$, while C–S–H is significantly increased; in addition, a minor amount of $3Ca(OH)_2·CaCl_2·12H_2O$ is formed. The hydration accelerated due to CC makes hydration products finer, more uniform and denser than those formed during standard curing.

The strength improvement due to CC can be explained with the following mechanisms. During the alternating curing process, immersion in NaCl accelerates hydration, while the subsequent drying helps to limit possible damage caused by expansive $3Ca(OH)_2·CaCl_2·12H_2O$. Multiple curing cycles gradually accelerate hydration from the outside to the inside, thus improving the overall strength of the samples.

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