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Improvements of the Carbonation Resistance of Concrete after Steam Autoclave Curing

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ABSTRACT

Incorporation of mineral admixtures, secondary water curing, and surface treatments were performed to improve the carbonation resistance of concrete after steam autoclave curing. The mineral admixtures used were fly ash, slag, and a mixture of both, and the surface treatments applied were those of organic silicone (OS) water repellent and epoxy resin (ER) coating. Accelerated carbonation experiments were performed on specimens at 28, 90, 180, and 360 d. Experimental results indicated that steam autoclave curing significantly reduces concrete carbonation resistance compared with standard curing. Secondary water curing for one week can replenish water in concrete and further hydrate the residual unhydrated cement particles in concrete, effectively improving the concrete carbonation resistance. The reduction of concrete carbonation depth can reach 81% at 360 d. The incorporation of mineral admixtures cannot improve the early-age carbonation resistance of concrete. However, it can provide several benefits to long-term carbonation resistance. Comparatively, slag behaves better than fly ash, and the maximum improvement ratio by 20 wt.% replacement of slag can reach 36.9% at 360 d. ER coating can substantially improve the carbonation resistance of concrete due to the formation of a compact membrane on the concrete surface, with an average improvement ratio of 77.2%. By contrast, the OS repellent can only improve the hydrophobic properties of the concrete surface, which is not effective enough.

Keywords: concrete; carbonation; steam autoclave curing; mineral admixture; secondary water curing;

1.0 INTRODUCTION

As an accelerated curing method for precast concrete, steam autoclave curing is extensively used in the production of pre-stressed, high-strength concrete pipe piles (Yan, 2015; Tan and Zhu, 2017). In the manufacturing industry, the compressive strength of concrete is given considerable attention, whereas durability, especially long-term durability, is not sufficiently considered. Generally, elevated temperature curing will shorten the curing period and improve the early-age strength of concrete. However, several studies confirmed that accelerated curing also causes long-term strength reduction and durability degradation because of the loose microstructure in concrete and the uneven distribution of cement hydration products (Mehta and Gerwick, 1982; Tan and Liu, 2006; García *et al.*, 2016).

Several studies have aimed to reduce the negative effects of accelerated curing (Detwiler *et al.*, 1994; Aldea *et al.*, 2000; He *et al.*, 2012; Li *et al.*, 2016). Detwiler *et al.* (1994) found that the partial replacement of cement by 30% blast furnace slag or 5% silica fume can reduce the chloride ion penetration of steam-cured concrete. Aldea *et al.* (2000) studied the effects of slag replacement on the transport properties of concrete after autoclaving (175 °C, 0.5 MPa) or steam curing (80 °C) compared with normal curing (20 °C, 100% RH). They observed that increasing slag replacement can significantly reduce the chloride permeability and penetrability of steamed concrete; however, this effect was not observed for autoclaved concrete. He *et al.* (2012) investigated the effects of subsequent curing on the properties of steam-cured concrete and found that exposure to air conditioning had deleterious effects on concrete properties. They also found that adopting 20 °C subsequent water curing can reduce the capillary water absorption coefficient and total porosity of steam-cured concrete. Li *et al.* (2016) studied the carbonation resistance of concrete under different curing temperatures from 20°C to 80°C. They found that the carbonation resistance of concrete incorporated with mineral admixtures gradually improves with increasing curing temperature up to an upper limit of 60 °C and then declines sharply, and that the replacement of pozzolanic materials, such as fly ash, slag, and silica fume, reduces concrete carbonation resistance compared with that of plain concrete.

In summary, numerous studies have been conducted on the improvements of the strength or durability of concrete under elevated temperature curing, and many valuable results have been obtained. However, studies on concrete under steam autoclave curing remain limited. Carbonation resistance is a critical durability property of reinforced concrete elements (Li *et al.*, 2016). The present study adopted several measures, such as the addition of mineral admixtures, subsequent water curing, and application of surface treatments, to reduce the negative effects on carbonation resistance of concrete after steam autoclave curing.

2.0 EXPERIMENT

2.1 Raw Materials

P·O 52.5 ordinary Portland cement (OPC), S95 granulated blast furnace slag, and class II low-calcium fly ash were used as binding materials. Their chemical composition and properties are shown in Table 1. Natural river sand with a fineness modulus Mx of 2.24, crushed limestone with particle sizes ranging from 5 mm to 25 mm, and tap water were used as fine aggregate, coarse aggregate, and mixing water, respectively. A polycarboxylate superplasticizer was also used as a water-reducing agent.

Table 1. Chemical composition and density of cement, fly ash, and slag (%)

Item	Al_2O_3	MgO	CaO	Fe_2O_3	SO_3	Density (g/cm^3)
Cement	5.4	0.9	64.5	2.6	-	3.1
Fly ash	31.53	1.14	5.74	6.21	-	2.7
Slag	13.8	7.9	36.4	-	0.7	2.9

2.2 Fabrication, Curing, and Testing of Specimens

Compared with OPC concrete, three mineral admixture concrete, namely, fly ash concrete (FAC), slag concrete (SLC), and fly ash + slag concrete (FSC), were designed. The replacement ratio of cement by fly ash in FAC, by slag in SLC, or by a mixture of both in FSC was 20 wt.% in total. The detailed concrete mixture proportions are listed in Table 2.

Concrete was mixed by using a forced mixer and compacted by using a table vibrator. Cubic specimens 100 mm × 100 mm × 100 mm in size were demolded 24 h after casting. Subsequently, three curing methods were adopted, such as steam autoclave curing, subsequent water curing and standard curing. Steam autoclave curing was performed by means of a two-stage curing regime (Fig. 1). The first stage was steam curing , which was carried out in a steaming pool (Fig. 2) by elevating the temperature to 90 ± 5°C for 1.5h, maintaining the temperature for 3.5 h, and then cooling down to ambient air temperature for 1.5h. The second stage was autoclave curing, which was carried out in an autoclave (Fig. 3) by heating up to 180 ± 5°C and 1.0 ± 0.05 MPa for 1.5h, maintaining that condition for 3h, and then cooling down for 1.5h. Subsequent water curing consisted of the initial steam autoclave curing and the secondary curing in 20 °C water tank for one week. Standard curing was performed in an automatic standard curing chamber (T=20 ± 2°C, RH= 70 ± 5%) for 28d. After the curing, all specimens were placed in an indoor natural environment prior to accelerated carbonation experiments.

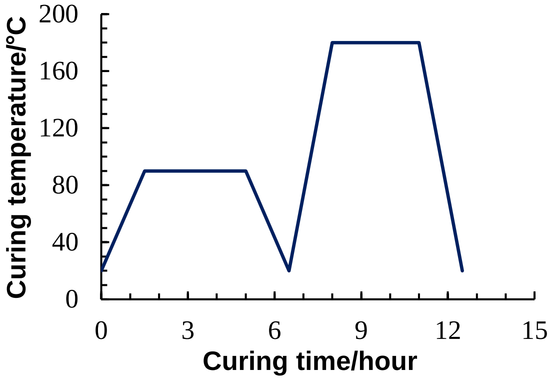

Fig. 1. Steam autoclave curing regime

To study the influences of surface treatments on the improvements of carbonation resistance for steam-autoclaved concrete, two surface treatments, such as epoxy resin (ER) painting and organic silicon (OS) waterproofing repellent were applied on partial steam-autoclaved OPC specimens at the age of 21d. ER coating consists of an epoxy zinc primer and an epoxy finishing coat. Seven days after the application of surface treatments, specimens were placed in an indoor environment prior to accelerated carbonation experiments.

Table 2. Concrete mixture proportions (kg/m^3)

Item	Cement	Fly ash	Slag	Sand	Stone	Water	Superplasticizer
OPC	470	0	0	660	1280	150	10.5
FAC	376	94	0	656	1272	150	10.5
SLC	376	0	94	658	1276	150	10.5
FSC	376	47	47	657	1274	150	10.5

Fig. 2. A steaming pool

Fig. 3. An autoclave

At the age of 28, 90, 180, and 360 d, specimens were placed in a chamber with high CO_2 concentration (T = 20 °C ± 2°C, RH = 70% ± 5% and CO_2 concentration of 20% ± 3%) for 10 d according to the Chinese test method (GB/T50082-2009). Phenolphthalein solution was used as an indicator to measure the concrete carbonation depth.

3.0 RESULTS AND DISCUSSION

3.1 Carbonation Resistance of Concrete under Different Curing Methods

Figure 4 shows the carbonation depths of OPC specimens under different curing methods based on the obtained data. Clearly, different curing regimes produce different effects on the carbonation depth of concrete at all testing ages. Generally, at the same testing age, the carbonation depth of concrete under steam autoclave curing was higher than that under subsequent water curing or standard curing. For example, the carbonation depth of concrete under steam autoclave curing was 1.61 and 3.84 times that of the depth under subsequent water curing and standard curing at 28d, respectively, while it was 5.25 and 21.96 times at 360d. The findings confirmed that steam autoclave curing produces considerable damages on concrete carbonation resistance, an outcome that is consistent with the findings in previous studies (Ho *et al.*, 2003; Tian *et al.*, 2009).

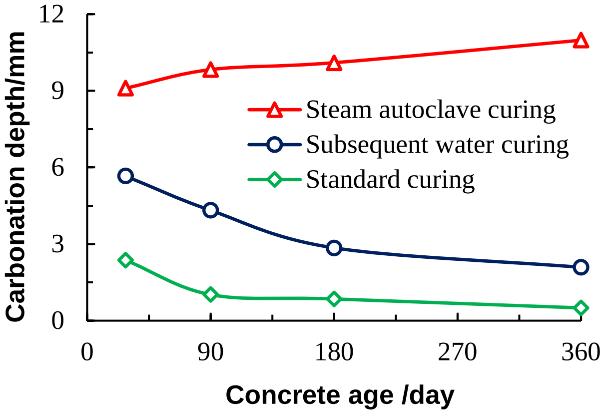

Fig. 4. Carbonation depths of concrete under different curing methods

The carbonation depths of concrete under different curing methods also varied. The carbonation depths under subsequent water curing and standard curing decreased gradually, whereas the values under steam autoclave curing exhibited an increasing trend. Thus, the differences between steam autoclave curing and subsequent water curing or with standard curing increased. Therefore, steam autoclave curing will not only damage early-age concrete carbonation performance but also long-term carbonation resistance.

The hydration of cementitious materials in concrete is a long-term chemical reaction process in which water participation is critical (Wang *et al.*, 2011). A high level of humidity was observed under conventional standard curing. A significant amount of water remains in concrete even after curing, which can continue the hydration of residual unhydrated cementitious particles in concrete. This condition makes concrete denser with increasing age and is beneficial for improving long-term concrete carbonation resistance. In the case of steam autoclave curing, the water in concrete will evaporate during cooling and make concrete dry. Thus, even if a considerable amount of unhydrated cementitious particles remain in concrete, they cannot be hydrated further due to the lack of water. Subsequent water curing provides an opportunity to replenish water into concrete, which is beneficial to the continuation of hydration for residual unhydrated binding materials in concrete and will increase the density of concrete (He *et al.*, 2012). After subsequent water curing for one week, although the carbonation depth of concrete was still higher than that under standard curing, the carbonation depth of concrete under subsequent water curing decreased significantly compared with that under steam autoclave curing at each testing age. Compared with steam autoclave curing, the reductions in carbonation depth of concrete under subsequent water curing were 37.8%, 56.1%, 71.8%, and 81.0%, corresponding to the testing ages of 28, 90, 180, and 360d, respectively. These results indicate that one week of subsequent water curing can effectively improve the carbonation performance of steam-autoclaved concrete, and that improvement efficiency increases with testing age.

3.2 Influences of Mineral Admixtures on the Carbonation Resistance of Steam-autoclaved Concrete

Concrete carbonation resistance depends mainly on the $Ca(OH)_2$ content in concrete, the proportion of harmful pores, and other factors (Papadakis *et al.* 1991; Tian *et al.* 2009). For the mineral admixture concrete, the amount of $Ca(OH)_2$ in concrete can be reduced by the partial replacement of cement with an equivalent amount of mineral admixtures and further pozzolanic reactions. By contrast, the active ingredients of SiO_2 and Al_2O_3 in mineral admixtures will react with $Ca(OH)_2$ and produce calcium silicate hydrate gels and hydrated calcium aluminate, which will reduce the porosity of concrete and thus make concrete more compact (Gonen and Yazicioglu, 2007; Shi, Xu, and Zhou, 2009; Wang *et al.*, 2011). Figure 5 illustrates the carbonation depth of steam-autoclaved concrete incorporated with mineral admixtures.

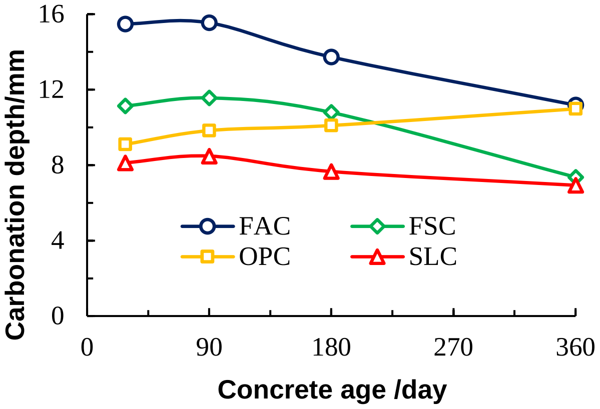

Fig. 5. Carbonation depth of steam-autoclaved concrete with mineral admixtures

The carbonation resistance of steam-autoclaved concrete was altered after the addition of mineral admixtures into concrete. Mineral admixtures typically increase the risk of carbonation for concrete incorporated with mineral admixtures because of pozzolanic reactions (Li *et al.*, 2016). Compared with OPC specimens, most of the carbonation depths of mineral admixture concrete increased at 28d, except that of SLC specimens. The carbonation depths of FAC and FSC specimens were 1.7 and 1.22 times that of the OPC specimen, respectively. Therefore, the incorporation of mineral admixtures.

However, the carbonation depth developments of concrete with or without mineral admixtures were different. As previously mentioned, the development of OPC specimen showed an increasing trend. The development of mineral admixture concrete exhibited a descending trend, which indicates an increase in the carbonation resistance of concrete with testing age. Therefore, at 360d, the carbonation depth of FAC specimen decreased to a value approaching that of OPC specimen, and the carbonation depths of FSC and SLC decreased to values below that of the OPC specimen. The specific improvement ratios of FAC, FSC, and SLC on concrete carbonation resistance were −1.8%, 33%, and 36.9% at 360d, respectively. SLC behaves better than FSC and FAC, which can be ascribed to the higher CaO content in slag than that in fly ash. Although the addition of mineral admixtures cannot improve the early-age carbonation resistance for steam-autoclaved concrete, it can contribute several benefits on the long-term carbonation resistance.

3.3 Influences of Coatings on the Carbonation Resistance of Steam-autoclaved Concrete

Surface treatments are often used to improve the durability of concrete (Almusallam *et al.*, 2003; Li *et al.*, 2016; Pan *et al.*, 2017). Carbonation depths of steam-autoclaved concrete with surface treatments are shown in Fig. 6. The application of surface treatments reduced the carbonation depths of steam-autoclaved concrete at all testing ages.

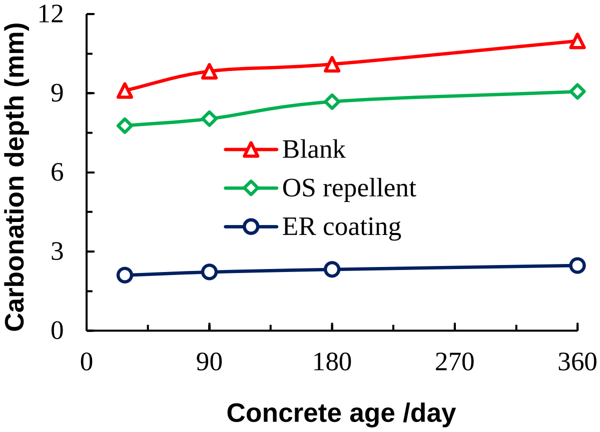

Fig. 6. Carbonation depths of steam-autoclaved concretes with coatings

OS repellent is a kind of hydrophobic agent that can prevent the penetration of water into concrete. However, this agent cannot effectively prevent gas permeation into concrete (Almusallam *et al.*, 2003; Pan *et al.*, 2017); thus, the average improvement ratio of OS repellent on concrete carbonation resistance was only about 16.1%. By contrast, ER coating can form an airtight film on the concrete surface, which is effective for blocking the penetration of water and gas. Thus, the average improvement ratio of ER coating was approximately 77.2%. ER coating is evidently more effective in the improvement of carbonation resistance for steam-autoclaved concrete than OS repellent

Further observation shows that the carbonation depth developments of concrete with surface treatments were nearly the same as those of blank concrete. The surface treatments simply provide an exterior protection on concrete and do not change the material properties in concrete.

4.0 CONCLUSIONS

Through the experiment in this study, the following conclusions can be obtained:

1) Compared with standard curing, steam autoclave curing can significantly reduce concrete carbonation resistance. Owing to the replenishment of water into concrete, subsequent water curing can effectively improve the carbonation resistance of steam-autoclaved concrete, and the improvement efficiency increases with the testing age. The reduction of concrete carbonation depth can reach 81% at 360d.

2) The addition of mineral admixtures cannot improve the early-age carbonation resistance for steam-autoclaved concrete. However, it can provide several benefits for long-term carbonation resistance. Comparatively, slag behaves better than fly ash. The maximum improvement ratio of SLC can reach 36.9% at 360d.

3) ER coating can effectively improve the carbonation resistance of steam-autoclaved concrete, while OS repellent is not effective enough. The average improvement ratios of ER coating and OS repellent are 77.2% and 16.1%, respectively.

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