Long-age Wet Curing Effect on Performance of Carbonation

Resistance of Fly Ash Concrete

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Abstract: To investigate the carbonation regularity in concrete of 100 mm×100 mm×400 mm, in the environment, (20±3)% (volume fraction) concentration of CO₂, (70±5)% relative humidity and (20±2)°C temperature. The concrete specimens were divide into two parts: curing 28 days with temperature of (20±2)°C and the relative humidity ≥95%(named wet curing), and then curing for 3 years(named A condition)with the relative humidity of (60±5)% and the temperature of (20±2)°C(named dried curing)and long-age wet curing for 90 days(named B condition). The water-binder ratio was 0.37, 0.45 and 0.53, the content of fly ash substituting for the equal quality of cement by mass was 0%, 30% and 60%, respectively. Based on the scanning electron microscope (SEM) morphology of the interfacial transition zone of concrete's coarse aggregate and X-ray diffraction (XRD) analysis, the influence mechanism of wet curing for 90 days on the fly ash concrete carbonation resistance ability was analyzed. The results show that the wet curing for 90 days had the positive effect of dense interfacial zone and the negative effect of calcium hydroxide consumption on the concrete carbonation resistance ability. In the case of high fly ash content with low water-binder ratio or low fly ash content with high water-binder ratio, the dense effect of the interfacial transition zone of concrete's coarse aggregate occupied a dominant position, and the carbonation resistance ability was greatly improved after wet curing for 90 days. The average increment value of carbonation depth of fly ash concrete in each group decreased by 53.5% compared with the specimens cured under A condition. When the water-binder ratio and the fly ash content were either higher or lower, the positive effect of dense interfacial zone and the negative effect of calcium hydroxide consumption had little significant effect on the concrete carbonation resistance ability, and the wet curing for 90 days had no obvious influence on improving the carbonation resistance ability. At present, the average increment value of carbonation depth of fly ash concrete in each group decreased only by 11.5% of that under A condition.

Key words: fly ash concrete; wet curing; long- age; carbonation; mechanism

Introduction

The durability of concrete structures was a major concern of building owners as it was related to safety, economy and sustain-ability. The most relevant issue affecting the durability of concrete structures was anti-carbonation ability ^[1-2]. In resent years, with the development of the concrete technology, fly ash had become one of the main components of modern concrete ^[3]. The secondary hydration of fly ash would consume the Ca(OH)₂, which reduces anti-carbonation of concrete, and leads to steel corrosion in concrete, and then affects the durability of concrete^[4]. Therefore, anti-carbonation was the key factor which restricts the development of fly ash concrete.

Fly ash content and water-binder ratio were two important parameters that influence the carbonation resistance of fly ash concrete ^[5-6]. The carbonation resistance of fly ash concrete was decreased with the increased of water-binder ratio and fly ash content ^[7-8]. Nevertheless, the carbonation resistance of concrete

1 with low fly ash content would increase. For the concrete that have the low volume fly ash mixtures (less
2 than 30%) the carbonation coefficients of concrete was approximately equal to those of the concrete of
3 cement-only mixtures, and the carbonation depth of the specimens containing 20% of fly ash was only
4 77.6% of specimens with cement-only mixtures^[9-10]. Based on the above analysis, it could be obtained that
5 these studies did not consider the carbonation resistance of fly ash concrete under different curing
6 conditions.

7 Early curing period had important effect for the anti-carbonation of fly ash concrete^[11]. The
8 incorporation of fly ash postpones the development of both wave velocity and attenuation coefficient, and
9 achieves lower one-day wave velocity values^[12]. This would slow down the rate of hydration of cement.
10 Thus, extending the early curing time would promote the hydration of gelled material, and improve the
11 anti-carbonation of fly ash concrete, especially for the concrete with large content of admixture which
12 should be more appropriate to extend the early curing period^[13-14]. The anti-carbonation ability of concrete
13 could be improved by extending the wet curing age. Under wet curing for 28 days, there was no obvious
14 difference in the anti-carbonation of concrete with different content of fly ash, and the carbonation depth of
15 concrete standard curing for 3 days was about 2.35 times for standard curing 28 days^[15-18]. Curing
16 temperature and humidity changes would affect the anti-carbonation ability of fly ash concrete^[19-22]. Li^[19]
17 had studied the carbonation resistance of fly ash concrete which were curried for 3 days, 7 days, 14 days,
18 28 days in the water with the temperature of 20°C, 30°C and 40°C respectively. The results showed that
19 extend the initial curing time and increasing the curing temperature was beneficial to improve its resistance
20 to carbonation. And the concrete cured at 60°C hot water for 24 hours could promotes changes in the pore
21 size distributions and generates certain modifications in the C-S-H gels formed, and increase the
22 carbonation resistance of concrete^[20-21]. The anti-carbonation of concrete was greatly improved when the
23 curing relative humidity increased from 60% to 100% according to Li Xuebin's study^[22]. For the concrete
24 with different water-binder ratio and different curing conditions, the studies found that the early standard
25 curing time less than 3 days compared with the standard curing for 28 days of concrete carbonation depth
26 increased by 50%~100% and the longer standard curing the stronger anti-carbonation of concrete would
27 be^[23-24]. Above these studies, they did not consider the influence of curing age for the concrete carbonation
28 resistance with different content of fly ash and water-binder ratio at the same time. In addition, concrete
29 carbonation test was used with a standard carbonation test in the study of the past^[25-26], this method was the
30 standard curing 28 days after the specimens shape, then carried out the carbonation experiment of 28 days

1 in the carbonation chamber. Through the carbonation depth of concrete showed the anti-carbonation of
2 concrete. This method could intuitively and conveniently reflect the influence factors in the
3 anti-carbonation of concrete and their influence degree. But for the fly ash concrete, studies [27-28] showed
4 that the secondary hydration of fly ash mainly happened between the 28 days and 90 days, and the
5 hydration reaction of fly ash was accompanied by the rapid carbonation of concrete. And the carbonation of
6 hydrated cement paste caused a change in porosity and pore size distribution, the pore structure and
7 morphology of hydrated products would have an effect on the carbonation resistance of concrete [29-30].
8 These would affect the carbonation of the uncertainty.

9 Based on the above researches, the carbonation regularity in concrete of 100 mm×100 mm×400 mm
10 was studied, in the environment, $(20\pm3)\%$ (volume fraction) concentration of CO_2 , $(70\pm5)\%$ relative
11 humidity and $(20\pm2)^\circ\text{C}$ temperature. The concrete specimens were divide into two parts: curing 28 days
12 with temperature of $(20\pm2)^\circ\text{C}$ and the relative humidity $\geq 95\%$ (named wet curing), and then curing for 3
13 years(named A condition)with the relative humidity of $(60\pm5)\%$ and the temperature of $(20\pm2)^\circ\text{C}$ (named
14 dried curing)and long-age wet curing for 90 days(named B condition). The water–binder ratio was 0.37、
15 0.45、 0.53, the content of fly ash substituting for the equal quality of cement was 0%, 30% and 60%. The
16 purpose of setting A condition was to close to the engineering practice and the purpose of setting B
17 condition is to make the secondary hydration of fly ash concrete more fully and reduce the secondary
18 hydration of fly ash of uncertain influence on carbonation during accelerated carbonation. According to the
19 scanning electron microscope (SEM) morphology of the interfacial transition zone of concrete's coarse
20 aggregate and X-ray diffraction (XRD) analysis, the influence mechanism of wet curing for 90 days on the
21 fly ash concrete carbonation resistance ability was analyzed.

22 1 Experimental

23 1.1 Raw Materials

24 The cementitious materials used in the experiment were Portland cement and fly ash, whose chemical
25 components and specific surface area were shown in the Table 1. Fine aggregate was river sand with the
26 fineness modulus of 2.8 and the apparent density of 2650 kg/m^3 . Coarse aggregate was crushed lime stone
27 with the size of 5–20 mm and the apparent density of 2660 kg/m^3 . The polycarboxylic water reducer with a
28 water-reducing rate of 21% was used to achieve target workability.

Table 1 Chemical composition, loss on ignition and specific surface area of cement and fly ash

Materials	Chemical compositions (%)												Specific surface area (m ² ·kg ⁻¹)
	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	MnO	TiO ₂	P ₂ O ₅	SO ₃	LOI*	
Cement	63.70	20.30	4.83	3.25	3.28	0.03	1.28	0.12	0.33	0.12	2.67	3.26	350
Fly ash	7.50	49.10	32.50	4.78	0.80	0.51	1.88	0.08	1.49	0.23	0.58	1.60	390

*LOI = loss on ignition

1.2 Experimental methods

1.2.1 Preparations of specimens and accelerated carbonation experiment

In the concrete mix design, the equivalent mass substitution method of fly ash was taken in this article according to the GBJ146-90 《Technical specification for fly ash concrete application》. Reference the literature[31-35], this article directly determined the uniform water-binder ratio was 0.37, 0.45 and 0.53. The concrete specimens with 100 mm × 100 mm × 400 mm, by using the ratio of Table 2. The slump of each mixture was controlled within (100±10) mm by adjusting the dosage of superplasticizer. In the specimens, one part of the 3 years concrete specimens were the wet curing for 28 days and dry curing for 3 years (condition of A), the rest of specimens were wet curing for 90 days(condition of B). Stop curing after reaching a predetermined age. The initial carbonation depth of concrete under two curing conditions were measured before the experiment in the environment of the relative humidity of (60±5)% and the temperature of (20±2)°C, and then the specimens were sealed with a heated paraffin wax. One part of the specimens which content of fly ash was 60% left one side without paraffin wax, the other sides were sealed. One part of the specimens which content of fly ash was 0% and 30% left two sides without paraffin wax, the other sides were sealed. Accelerated carbonation experiment of concrete specimens under two curing conditions referring to the Chinese standard GB/T 50082-50082 《Ordinary concrete long-term performance and durability test method standard》. When the carbonation time (3 days, 7 days, 14 days and 28 days) reached, the specimens were cut from the set position. Drop the phenolphthalein with the concentration of 1% (mass fraction) on the split surface. When the color of concrete was not changed, the carbonation depth from the edge to the test surface without carbonation zone boundary along the border was measured once per 10 mm. And the average was the carbonation depth of the concrete specimens. After the measurement was completed, the split face was sealed and continued to be put into the carbide chamber until the next test time.

Table 2 The mix proportion of concrete

Mix No.	m_w/m_B	$\omega_{FA}/\%$	Mix proportion/(kg·m ⁻³)				
			Fly ash	Cement	Water	River sand	Coarse aggregate
A1	0.37	0	0	460	170	684	1114
B1	0.37	30	138	322	170	684	1114
C1	0.37	60	276	184	170	684	1114
A2	0.45	0	0	390	175	695	1135
B2	0.45	30	117	273	175	695	1135
C2	0.45	60	234	156	175	695	1135
A3	0.53	0	0	340	180	715	1165
B3	0.53	30	102	238	180	715	1165
C3	0.53	60	204	136	180	715	1165

* In the absence of special expository writing all percentage for quality percentage in the article.

1.2.2 The SEM test of aggregate interface transition zone

According to the test method described in the literature [36], observing the morphology characteristic of the interfacial transition zone of concrete's coarse aggregate after 28 days rapid carbonation under A and B curing conditions by SEM respectively.

1.2.3 The XRD test of hydration product component

The component of hydration product was determined by XRD. The paste specimens were taken from the non-carbonated zone of concrete specimens. At the testing age, the specimens were processed as following steps. First, the non-carbonated specimens were split into small pieces and put in ethanol and were ground into fine powder particles till they all passed the sieve with the diameter of 0.12 mm. Second, the powder was filtered in a vacuum by the suction filter bottle and diluted 5 times with ethanol and twice with ether. Then the powder particles were dried in a vacuum drying oven with desiccant at 105 °C for 24 h. Finally, the powder particles were placed in the small glass bottles to be used for XRD. The 2θ values ranged from 10 ° to 90 ° and the scanning rate applied was 4 °/min for all specimens.

2 Results and discussion

2.1 Analysis method

The carbonation depth of different content of fly ash concrete cured in conditions A and B with the water-binder ratio of 0.37, 0.45, and 0.53 for each age was shown in figure 1.

It can be seen from the three dash lines in figure 1 that in the case of 60% content of fly ash and different water-binder ratio, the carbonation depth of concrete cured in A condition was higher than or close to that of concrete cured in B condition after the rapid carbonation of 28 days. Analyzing the effect of

90 days wet curing on the properties of fly ash concrete carbonation resistance by the absolute value of carbonation depth, the early natural carbonation layer had a great influence on the carbonation depth of concrete, this method would make the inaccurate analysis result. When analyzing the effect of 90 days wet curing on the properties of fly ash concrete carbonation resistance by the increment value of carbonation depth, the early natural carbonation layer had inhibition for the later carbonation, and this method may underestimate the effect of 90 days wet curing^[37-38]. Above these, this article analyzed the effect of 90 days wet curing on the properties of fly ash concrete carbonation resistance by the increment value of carbonation depth.

Fig. 1 Different water-binder ratio of concrete carbonation depth under two curing conditions

(Three dash lines were the natural carbonation depth of the fly ash concrete under A curing condition at the content of fly ash was 60%, respectively)

2.2 The influence in anti-carbonation of the fly ash concrete with long-age wet curing

Under different content of fly ash, the concrete carbonation depth with water-binder ratio of 0.37 and 0.45 was smaller than that with the water-binder ratio of 0.53. In order to clearly reflect the effect of wet curing 90 days on the anti-carbonation of concrete, figure 2 lists the concrete with different content of fly ash which water-binder ratio was 0.53 carbonizing for 28 days in two curing conditions. The pictures in figure 2 were the squares with the side length of 100 mm.

Fig. 2 The pictures of concrete carbonation 28 days with different content of fly ash under two curing conditions

The figure 2 showed that the concrete non-carbonation zone decreases with the increase of content of fly ash under the same curing condition and carbonation age; And the non-carbonation zone of concrete under B condition was significantly greater than that of A condition under different curing conditions and at the same carbonation age, namely the anti-carbonation concrete increases under B curing condition.

The increment value of concrete carbonation depth in two kinds of curing conditions for 28 days was shown in figure 3.

It can be seen from figure 3 that when the content of fly ash and water-binder ratio were same, the increment value of concrete carbonation depth under the curing condition A was greater than that of condition B and the concrete carbonation depth developed slowly under the curing condition B, which suggested that the 90 days wet curing generally improved the anti-carbonation of fly ash concrete.

Fig. 3 The increment value of concrete carbonation depth under the two curing conditions for 28 days

2.3 Quantitative analysis about the influence in anti-carbonation of the fly ash concrete with long-age wet curing

To quantitatively analyze the improvement degree of anti-carbonation of fly ash concrete with 90 days wet curing. The coefficient H (hereafter called H) was defined to represent it. The calculation of H value was given in formula (1). It showed that under the 90 days wet curing, the smaller of the H , the more obvious function of fly-ash concrete's anti-carbonation would be.

$$H = \frac{M_1}{M_2} \quad (1)$$

In the formula, M_1 was the increment value of concrete carbonation depth under the B curing condition for 28 days, mm; M_2 was the increment value of concrete carbonation depth under the A curing condition for 28 days, mm.

H value under the condition of different content of fly ash and water-binder ratio was shown in table 3 through the analysis of concrete carbonation data processing.

Table 3 H value under the condition of different content of fly ash and water-binder ratio

W/B	FA/%		
	0%	30%	60%
0.37	0.88	0.84	0.33
0.45	0.87	0.58	0.46
0.53	0.54	0.49	0.93

It could be seen from table 3 that when the content of fly ash was 0% and 30%, H value reduced with the increase of water-binder ratio. However, in the case of 60% content of fly ash, H value increased gradually with the increase of water-binder ratio. This suggested that the anti-carbonation of concrete with low content of fly ash and high water-binder ratio increased more obvious under the 90 days wet curing. So did that with high content of fly ash and low water-binder ratio. Under the curing condition B, the average carbonation depth of fly ash concrete in each group decreases by 53.5% compared with the specimens

cured under A condition; H value was closer to 1 except the shaded part in the table 3, and this showed that when the water-binder ratio and the content of fly ash were either high or low, the effect of improvement in fly-ash concrete's anti-carbonation was not obvious under the condition of 90 days wet curing. At present, under the curing condition B, the average carbonation depth of fly ash concrete in each group decreases only by 11.5% compared with the specimens cured under A condition.

3 The mechanism of long-age wet curing's influence on anti- carbonation of the fly ash concrete

In order to clarify the mechanism of wet curing for 90 days' influence on anti- carbonation of the fly ash concrete, observe the morphology characteristic of the interfacial transition zone of concrete's coarse aggregate and the hydration product component by SEM and XRD respectively after 28 days' rapid carbonation under A and B curing conditions.

3.1 The effect of long-age wet curing on the interfacial transition zone of concrete's coarse aggregate

The morphology characteristic of the interfacial transition zone of concrete's coarse aggregate after 28 days rapid carbonation under A and B curing conditions was shown in figure 4.

W/B=0.37

W/B=0.45

W/B=0.53

B condition

(a) FA0%

W/B=0.37

W/B=0.45

W/B=0.53

A condition

W/B=0.37

W/B=0.45

W/B=0.53

B condition

(b) FA30%

W/B=0.37

W/B=0.45

W/B=0.53

A condition

Fig.4 SEM photographs of concrete coarse aggregate interface transition zone under two curing conditions at the different content of fly ash
(A named aggregate, M named substrate in figures)

By comparing the SEM morphology of the interfacial transition zone of concrete's coarse aggregate under A and B curing conditions in Figure 4, it could be obtained that with the same curing condition and content of fly ash, the density of coarse aggregate interfacial transition zone decreased with the increase of water-binder ratio; In the same water-binder ratio of different curing conditions, the density of the transition zone of the coarse aggregate interface decreased with the increase of content of fly ash; In the curing of A condition, the structure of hydration product, in the concrete coarse aggregate interfacial transition zone, was obviously loose and highly porous. The bonding between paste and aggregate was poor, and there were obvious interface cracks. In the curing of B condition, the dense structure was formed by the cross connection of the hydration products in the concrete coarse aggregate interfacial transition zone. The hydration products of the cement cover the unhydrated fly ash particles into a whole structure, which made the interface transition zone denser. The smaller water-binder ratio, the better effect of the interface transition zone, and the stronger the anti-carbonation performance. The dense degree in the interfacial transition zone of concrete's coarse aggregate under B condition was better than that of A condition.

3.2 XRD analysis of the fly ash concrete under different curing conditions

Fig. 5 shows the XRD patterns of fly concrete specimens cured under different curing conditions.

Fig. 5 XRD patterns of fly ash concrete specimens cured under two curing conditions

It was apparent that the peaks of paste specimens (CaCO_3 , SiO_2 , Ca(OH)_2 , C_3S , and C_2S) diminished under different curing conditions. It could be seen that the hydration product of paste specimens under different curing conditions included mainly a large amount of C-S-H in various patterns, Ca(OH)_2 , CaCO_3 , C_3S , C_2S and SiO_2 . The C-S-H gel and Ca(OH)_2 were the main hydration products of the cementitious phase, in which C-S-H gel could not be identified by XRD because it was amorphous. The CaCO_3 contained in the hydration product was caused by the error of the test specimens. Comparing the peaks of

Ca(OH)₂ at the $2\theta=18^\circ$ ($d=0.490$ nm) of the paste specimens cured under two curing conditions, the peaks of Ca(OH)₂ of the paste specimens (3 a-A2, 3 a-B2, 3 a-C1, 3 a-C2, 3 a-C3) and (90 d-A2, 90 d-B2, 90 d-C1, 90 d-C2, 90 d-C3) gradually diminished under A and B curing conditions respectively. The results showed, under the same curing condition, the peaks of Ca(OH)₂ decreased with the increase of water-binder ratio in the same content of fly ash; The peaks of Ca(OH)₂ decreased with the increase of content of fly ash in the same water-binder ratio. The peaks of Ca(OH)₂ of the paste specimens cured under A condition are higher than the paste specimens cured under B condition. The result showed that wet curing 90 days could motivate the activity of fly ash and promote the fly ash to react with Ca(OH)₂.

3.3 Mechanism analyses

Wet curing 90 days had two aspects effect on the anti-carbonation ability of fly ash concrete. On the one hand, wet curing 90 days would promote the hydration of fly ash, and could generate more C-S-H gel, which was helpful to reduce the porosity of concrete and improve the dense degree of concrete. This could improve the carbonation resistance of concrete. On the other hand, the hydration of fly ash would consume some calcium hydroxide produced by cement hydration in concrete. This would have a negative impact on the anti-carbonation ability of concrete.

For the concrete without fly ash, it was the main factor affecting anti-carbonation ability that the dense degree in the interfacial transition zone of concrete's coarse aggregate. When water-binder ratio was 0.37 and 0.45, the concrete was enough density, and wet curing for 90 days had little effect on the dense degree in the interfacial transition zone of concrete's coarse aggregate, and it had no obvious influence on improving the anti-carbonation ability. When the water-binder ratio was 0.53, the internal structure of concrete was looser than the concrete with low water-binder ratio, and wet curing of 90 days could improve the dense degree in the interfacial zone and anti-carbonation ability. For fly ash concrete, wet curing 90 days could motivate the activity of fly ash and promote the fly ash to react with Ca(OH)₂, the peaks of Ca(OH)₂ of the paste specimens under A condition are higher than the paste specimens under B condition. When it was high content of fly ash and low water binder ratio or low content of fly ash and high water-binder ratio, the dense degree effect in the interfacial transition zone of concrete's coarse aggregate occupied a dominant position compared with the negative effect of calcium hydroxide consumption, and the anti-carbonation ability was greatly improved in wet curing for 90 days. When the water-binder ratio and the content of fly ash were either high or low, the positive effect of dense interfacial zone and the

negative effect of calcium hydroxide consumption had little obvious effect on the concrete anti-carbonation ability, and the wet curing for 90 days did not have obvious influence on improving the anti-carbonation ability.

4 Conclusions

From the test and the analytical results, the following conclusions can be drawn.

1. Wet curing for 90 days had the positive effect of dense interfacial zone which would improve the concrete's anti-carbonation ability. Besides, it had the negative effect of calcium hydroxide consumption which would have an uncertain influence on the concrete's anti-carbonation ability.

2. When it was high content of fly ash and low water binder ratio or low content of fly ash and high water-binder ratio, the dense effect in the interfacial transition zone of concrete's coarse aggregate occupied a dominant position, and the carbonation resistance ability was greatly improved under wet curing for 90 days. At present, the average carbonation depth of fly ash concrete in each group decreases by 53.5% compared with the specimens cured under A condition

3. When the water-binder ratio and the content of fly ash were either high or low, the positive effect in dense interfacial zone and the negative effect of calcium hydroxide consumption had little obvious effect on the concrete's anti-carbonation ability, and the wet curing for 90 days did not have obvious influence on improving the anti-carbonation ability. Under the curing condition B, the average carbonation depth of fly ash concrete in each group decreases only by 11.5% of that under A condition.

Acknowledgements

The authors would like to acknowledge the financial supports provided by the National 973 Plan Project No. 2015CB655100, the National Science Foundation (NSF) of China under Grant No. 51578477 and the Graduate Innovative Funding Program of Hebei Province under Grant No. 00302-6370018.

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