



Time dependence of carbonation resistance of concrete with organic film coatings



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HIGHLIGHTS

- Organic film coatings can remarkably improve concrete carbonation resistance.
- Organic film coatings' carbonation resistance will decrease with aging.
- Coating's carbonation resistance exhibits an S-shaped curve with aging time.
- S-shaped curve models can be used to predict organic coatings' service lives.

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ABSTRACT

Concrete specimens with three types of organic film coatings, namely, polyurethane (PO), epoxy resin (EP), and chlorinated rubber (CR), were fabricated to study the time-dependent relationship between the aging of organic film coating and the corresponding degradation of concrete carbonation resistance. Then, three coating aging methods were adopted: artificial ultraviolet radiation, coupled ultraviolet radiation and moisture, and natural outdoor climate exposure. After different aging stages, the specimens were subjected to accelerated carbonation experiments, and partial specimens were examined by scanning electron microscopy. Results indicated that organic film coatings can significantly improve concrete carbonation resistance, and a thicker film coating can obtain better improvement. According to the parameter α for the improvement of concrete carbonation resistance per unit thickness, the ranking order of the three types of coatings is as follows: PO > CR > EP. Defects in organic coatings caused by aging, such as granulation, porosity, and cracking, are the causes of degradation of coating carbonation resistance. As an organic film coating ages, its carbonation resistance gradually decreases and exhibits an S-shaped curve. On this basis, regression models for the carbonation depth with aging time of the coated specimens were established. These models were used to predict the service lives of the coatings. The ranking order of the service lives of the coatings is PO > EP > CR. Increasing the thickness of a coating can enhance its service life.

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1. Introduction

Surface coatings on concrete are among the important measures for the durability protection of concrete structures [1–2]. Organic film coatings, owing to their good adornment, convenient construction, and high protective efficiency, among other desirable characteristics, have become an important option to improve the

carbonation resistance of structures, avoid the corrosion of steel bars in concrete, and prolong the service lives of concrete structures [3–10].

Ahmed et al. [5] conducted accelerated carbonation experiments on concrete coated with acrylic and epoxy resins (EPs) and found that the application of coatings can improve concrete carbonation resistance, with the improvement of EP coating better than that of acrylic coating. Li et al. [6] conducted an accelerated carbonation experiment on concrete specimens with acrylic resin, cementitious waterproof coating, and organic silicone emulsion, and found that the improvements of acrylic resin on concrete carbonation resistance are significantly higher than those of the other

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two coatings. Sun et al. [7] indicated that, aside from organic silicone emulsion, other organic coatings, such as acrylic emulsion, styrene–acrylic emulsion, tertiary carbonate emulsion, all improve concrete carbonation resistance. Park [8] investigated the CO₂ diffusion coefficients of four types of organic coatings and concluded that significant differences in CO₂ diffusion coefficients exist across different coatings; the order of carbonation resistance from high to low is as follows: polyvinyl chloride, polyurethane (PO), epoxy, and acrylic. Ho and Harrison [9] suggested that coating effectiveness depends not only on the coating material, but also on its mode of application and the conditions of its concrete substrate.

Generally, the aforementioned studies show that organic coatings can effectively improve concrete carbonation resistance. However, organic materials are prone to aging because of weathering as a consequence of the environmental climate, which will gradually deteriorate the concrete protection [3,4,10–12]. Khanzadeh et al. [10] performed long-term natural exposure experiments on concrete specimens with six types of organic coatings in the Persian gulf tidal zone for five years and found that the protection by coating on concrete is decreasing and time dependent. Li et al. [11] conducted artificial accelerated aging and carbonation experiments on concrete specimens with four types of coatings. They found that carbonation resistance decreases with aging time for the specimens with PO and EP coatings, whereas the carbonation resistance only slightly changes for specimens with cementitious waterproof coating or organic silicone emulsion. Kozak [12] indicated that the barrier properties of an acrylic coating against CO₂ permeability decreases quite rapidly in the aging process of UV radiation. However, other researchers presented different findings. Beushausen and Burmeister [13] conducted accelerated ultraviolet aging and carbonation experiments for concrete specimens coated with acrylic emulsion, acrylic resin, polymer modified cementitious mortar, and cementitious fiber reinforced mortar and observed that UV radiation on coatings do not decrease the specimens' carbonation resistance but instead further enhances their carbonation resistance.

The aging of organic coatings and degradation of protection effects on concrete are considerably complex problems because they involve many factors, and only very few studies on them have been published [14–18]. The study on the time-dependent relationship between coating aging and carbonation resistance degradation is of importance to the evaluation of the time-varying effectiveness of organic coatings. The purpose of the current study is to obtain the quantitative development of carbonation resistance with respect to coating aging through aging and carbonation experiments on concrete specimens with three typical organic film coatings. Moreover, it aims to predict the service life of a coating based on its carbonation resistance.

2. Materials and methods

2.1. Raw materials

Chinese standard P·O42.5R ordinary Portland cement produced by Zhonglian Cement Plant was used. Table 1 shows the chemical composition of the cement. Natural river sand with a fineness modulus of 2.5, crushed stone with a particle size of 5–20 mm, and ordinary tap water were chosen as fine aggregate, coarse aggregate, and mixing water, respectively. The concrete water/cement ratio was set to 0.6, and the detailed mixture ratio of concrete was cement: sand: stones: water = 360:730:1094:216 (in kg/m³).

Table 1
Chemical composition of ordinary Portland cement (wt%).

Item	SiO ₂	SO ₃	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	Loss
Cement	22.30	3.58	3.16	5.05	64.78	0.92	1.32

Three typical concrete organic film coatings available in the market, namely, PO, EP, and chlorinated rubber (CR) coatings, were adopted. The first two coatings are composed of two components, whereas the CR coating is composed of only one component.

2.2. Specimens

The size of each specimen is 100 × 100 × 300 mm³. The specimens were fabricated at normal indoor temperature and demolded 24 h after casting. They were then transferred to a water tank for curing at a temperature of 20 ± 2 °C until the age of 28 d. Before the application of coatings, the specimens were stored in an oven for 48 h at 60 °C for drying.

According to the manufacturer's instructions, coatings were brushed on the surface of the specimens. One-layer and two-layer for each type of coating were applied, and the brushing interval was 24 h. After the completion of coating, specimens were placed in an indoor environment for at least 7 d before the experiments were initiated. The thickness of each dry coating film was measured using a USB digital microscope, and the measurements are listed in Table 2.

2.3. Experimental methods

The speed of coating aging is associated with the prevailing environmental climate conditions. Generally, more severe environmental climate conditions always correspond to a faster coating aging speed. Under natural climate conditions, the aging speed of organic coating is relatively slow [12,15]. Two kinds of artificial accelerated aging methods, namely, ultraviolet light radiation aging (ultraviolet aging) and coupled ultraviolet light radiation and moisture aging (coupled aging), were selected to shorten the experimental time. At the same time, natural outdoor exposure aging (natural aging) was used for comparison.

The ultraviolet aging and coupled aging experiments were all conducted in an artificial climate chamber, which was equipped with two 400 W ultraviolet lamps. The vertical distance from the ultraviolet lamps to the surface of the specimens was 20 cm. For the ultraviolet aging experiment, the internal temperature was set to 60 ± 0.5 °C, but the relative humidity (RH) was not controlled. For the coupled aging experiment, the same internal temperature was adopted, but the RH was set to ≥90%. The natural aging experiment was conducted on the roof of a three-story laboratory building, where the annual average temperature was 14 °C and the annual average RH was 68.8%.

Coated specimens under these three aging methods were retrieved periodically and subjected to accelerated carbonation experiments. Partial specimens were also examined by scanning electron microscopy (SEM). Accelerated carbonation experiments were performed according to the Chinese national standard (GB/T50082-2009). Specimens were put into an oven at 60 °C and dried for 48 h before carbonation, and then put into an accelerated carbonation chamber, in which CO₂ concentration was 20 ± 3%, RH was 70 ± 5%, temperature was 20 ± 2 °C, and carbonation time was 28 d. After carbonation, the specimens were split and then measured for carbonation depth with the use of 1% phenolphthalein ethanol solution. The measurement accuracy was 0.5 mm, and the average of the three specimens with the same coating was taken as the representative measurement of the carbonation depth of each coating type.

According to the experimental plan, a total of 216 pieces coated specimens were fabricated, and three pieces of specimens without coating were also fabricated and subjected to an accelerated carbonation experiment for comparison. The detailed coated specimens' making plan was shown in Table 3.

3. Results

3.1. Carbonation depth of coated concrete after ultraviolet aging

The CO₂ diffusion coefficients of the organic coatings before aging range between 10⁻¹² and 10⁻¹⁰ m²/s, whereas that of ordinary concrete is approximately 10⁻⁸ m²/s [8]. Organic film coatings have higher densities than normal concrete; thus, the former can restrain the diffusion of CO₂ into concrete and enhance concrete carbonation resistance. Fig. 1 presents the accelerated carbonation

Table 2
Film thickness of the three types of coatings (μm).

Item	Coatings		
	EP	PO	CR
One-layer	45	29	34
Two-layer	88	63	67

Table 3
Coated specimen's making plan.

Aging method	Aging time	EP coating/PO coating/CR coating	
		One-layer	Two-layer
Ultraviolet aging Coupled aging	120 h	$3 \times 2 \times 3 = 18$	$3 \times 2 \times 3 = 18$
	240 h	$3 \times 2 \times 3 = 18$	$3 \times 2 \times 3 = 18$
	360 h	$3 \times 2 \times 3 = 18$	$3 \times 2 \times 3 = 18$
	480 h	$3 \times 2 \times 3 = 18$	$3 \times 2 \times 3 = 18$
Natural aging	6 month	$3 \times 3 = 9$	$3 \times 3 = 9$
	8 month	$3 \times 3 = 9$	$3 \times 3 = 9$
	10 month	$3 \times 3 = 9$	$3 \times 3 = 9$
	12 month	$3 \times 3 = 9$	$3 \times 3 = 9$

depths of the coated specimens obtained from experimental results after different ultraviolet aging periods. In addition, the accelerated carbonation depth of uncoated specimen was also plotted.

As shown in Fig. 1, the carbonation depth of the uncoated specimen was 20.3 mm. Before the start of the aging, the carbonation depths of the coated specimens were significantly lower than that of the uncoated specimen, but the improvements of their carbonation resistance to concrete vary. After one-layer applications of PO, EP, and CR coatings on concrete, the carbonation depths of the specimens were 7.2, 8.0, and 9.1 mm, which were only 35.5%, 39.4%, and 44.8% of the carbonation depth of the uncoated specimen, respectively. After the two-layer application, the carbonation depths of the specimens were 4.0, 4.4, and 5.4 mm, which were only 19.7%, 21.7%, and 26.6% of the carbonation depth of the specimen, respectively. The application of coatings significantly improved the carbonation resistance of concrete, and the increasing number of layers (thickness) further improved concrete carbonation resistance. This result is consistent with the results obtained by Ahmed et al. [5] and Sun et al. [7].

In the process of coating aging, organic coatings will inevitably deteriorate, and the protective capability of the coatings on concrete will decrease [14–16]. The results in Fig. 1 confirmed this idea. With the growth of ultraviolet aging time, the carbonation depths of coated specimens gradually increased, indicating the gradual degradation of organic coatings. At the same time, the development of the carbonation depths of the specimens along the coating aging time presented a three-stage S-shaped curve. In the initial stage of coating aging, the carbonation depths of the coated specimens increased slowly; in the middle stage of coating aging, the rate of increase in the carbonation depths of the coated specimens increased; and in the later stage of coating aging, the rate of increase in the carbonation depths of the coated specimens slowed down again.

In the initial stage of coating aging, defects in the coating surfaces gradually appeared because of the degradation of polymers with coating aging. The coating defects increased slowly in the initial stage; as a result, the carbonation depth of coated concrete

gradually increased with an increase in aging time. When the aging of coating entered the middle stage, coating defects significantly increased, leading to the rapid increase in carbonation depth of coated concrete. By the time coating aging entered the later stage, a large number of defects were generated on the coating surface, but the new defects generated were getting fewer; thus, the increase in concrete carbonation depth slowed down again. By the end of coating aging, a coating loses its carbonation resistance completely, and the carbonation depth of an aged specimen comes close to that of the uncoated specimen.

As shown in Fig. 1(b), at the same aging time, the carbonation depths of the two-layer specimens were significantly less than those of the one-layer specimens. This result indicates that the increase in thickness of the coating can reduce its aging speed.

3.2. Carbonation depth of coated concrete after coupled aging

Fig. 2 presents the development in accelerated carbonation depth of specimens after different coupled aging time.

As shown in Fig. 2, the development in carbonation depth of the coated specimens under the coupled aging condition was similar to that under the ultraviolet aging condition, as both demonstrated that the longer the coating aging time, the more the degradation in carbonation resistance of the coated specimens. The carbonation resistance degradation of two-layer specimens was also lower than that of the one-layer specimens at the same aging time.

Furthermore, given that the deterioration effect of coupled aging on the coatings was more severe than that of ultraviolet aging, more degradation occurred on the coatings subjected to coupled aging at the same time. As a result, the decrease in carbonation resistance of the coated specimens under coupled aging condition was greater. Under the coupled aging condition, the carbonation depth development of one-layer specimens is nearly an S-shaped curve, further confirming the S-shaped development law of coating degradation with aging time.

By the end of the aging experiments, the carbonation depths of the one-layer specimens were significantly close to the carbonation depth of the uncoated specimen, indicating that all the coatings were on the edge of failure. However, for the two-layer specimens, the carbonation depths were still lower than that of the uncoated specimen, suggesting that increasing the thickness of a coating can enhance its aging resistance.

3.3. Carbonation depth of coated concrete after natural aging

Fig. 3 provides the development in accelerated carbonation depths of the specimens after different natural aging time.

The development speeds of the carbonation depths under natural aging condition were apparently slower than those under artificial ultraviolet aging and coupled aging conditions. For the specimens with two-layer coats, the carbonation depths of the coated specimens were still very low even after 12 months of aging

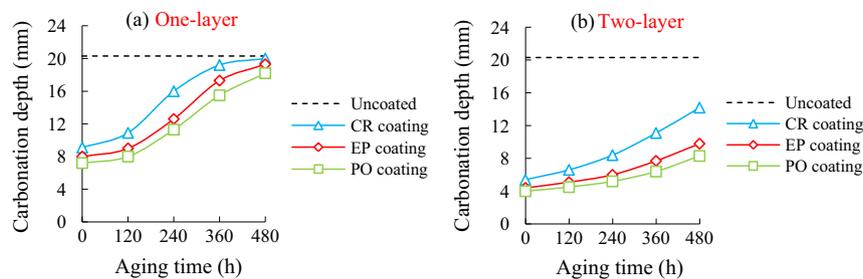


Fig. 1. Accelerated carbonation depths of coated specimens after ultraviolet aging.

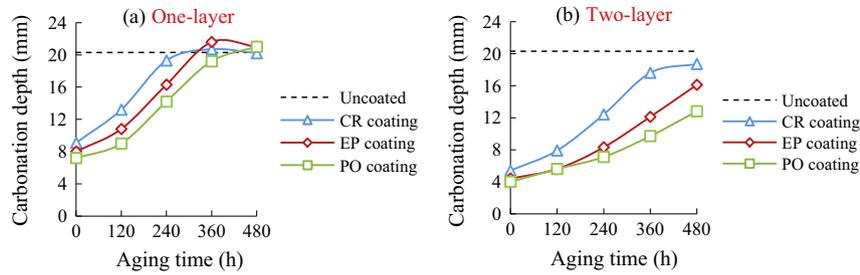


Fig. 2. Accelerated carbonation depths of coated specimens after coupled aging.

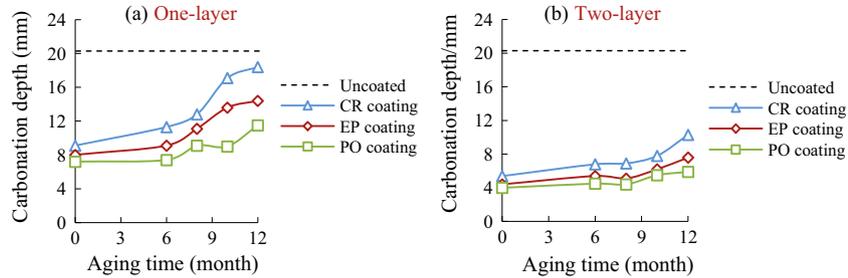


Fig. 3. Accelerated carbonation depths of coated specimens after natural aging.

time. Therefore, different climatic conditions obviously have different aging effects on coatings.

Although the degradation speeds of the coatings under different aging conditions were different, the ranking order of the aging resistances of the three coatings under every aging condition was the same: PO > EP > CR. Thus, PO coating has the best aging resistance, successively followed by EP coating and CR coating.

3.4. Microstructural changes in coatings before and after aging

Figs. 4–6 show some SEM photos of the coatings taken before and after different periods of aging.

As shown in Figs. 4(a), 5(a), and 6(a), the surfaces of the three types of coatings were all very dense, smooth, and with few defects, coinciding with the findings that coatings can provide good carbonation resistance before aging. Some differences could also be noted among the coatings. For the EP coating, some white strip flake fillers, which could strengthen its anti-aging capability and reduce coating contraction, were evident. For the CR coating, numerous uniformly distributed micro-holes were present because the CR coating was formed into a film by paint drying, and a significant number of micro-holes still existed after solvent evaporation.

The surface of the PO coating was relatively smoother and denser than those of the EP and CR coatings. Therefore, the PO coating had the best carbonation resistance among the three coatings.

Significant changes in the microstructures of the coatings occurred after a period of aging (Figs. 4(b–c), 5(b–c), and 6(b–c)). After 240 h of ultraviolet aging or coupled aging, the surfaces of the CR and EP coatings became rough, porous, and granular, and numerous tiny cracks formed in the CR coating. After 480 h of aging, the granular aging phenomenon became more severe, and the number and widths of the cracks continually increased.

The optical wavelength absorbed by PO is generally within the range of 290–400 nm. When the PO membrane absorbs a certain wavelength of light, the covalent bonds of the molecular chain in the coating fracture or crosslink as well as release CO₂, thereby making the coating porous [15]. The surface of the PO coating became rough after 6 months of natural aging and then became obviously granular after 12 months of natural aging. In addition, the micro-hole defects increased.

The given analysis shows that the formation of defects on the coating surface and the increase in the number of defects are the causes of degradation of the carbonation resistance of organic film coatings.

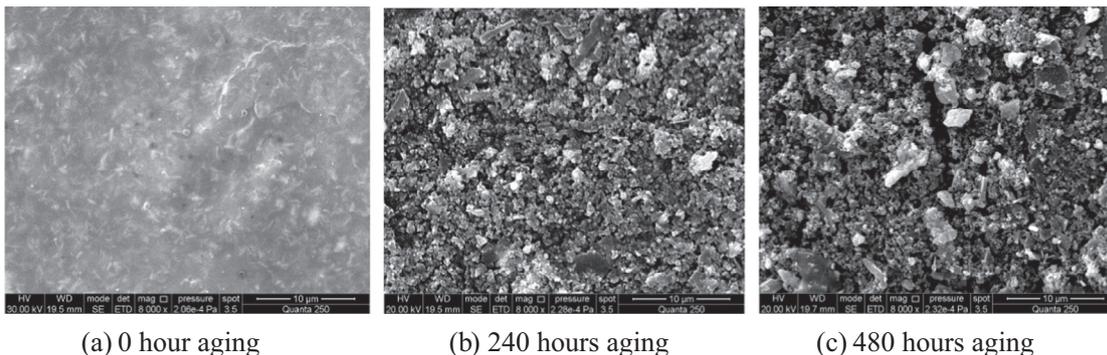


Fig. 4. SEM photos of EP coating before and after different coupled aging times.

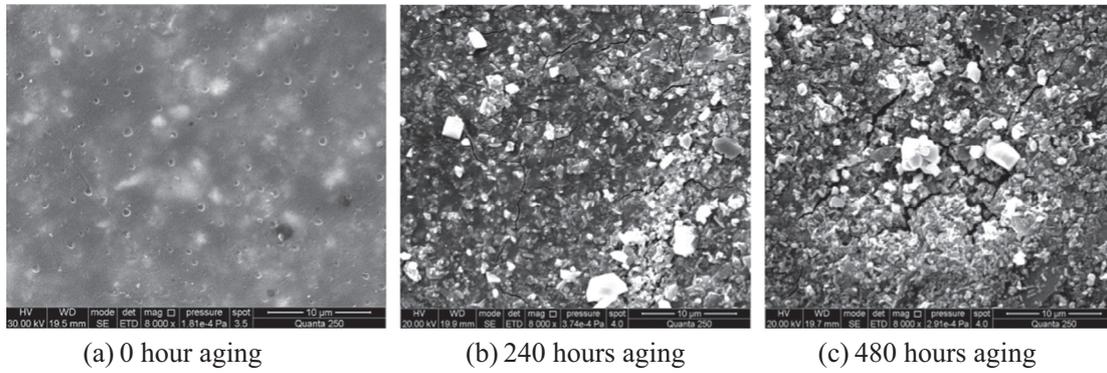


Fig. 5. SEM photos of CR coating before and after different ultraviolet aging times.

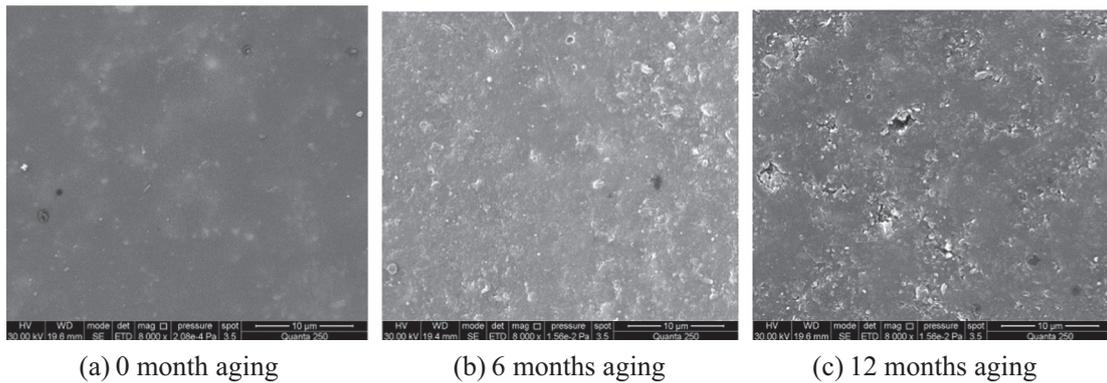


Fig. 6. SEM photos of PO coating before and after different natural aging times.

4. Discussion

4.1. Carbonation resistances of concrete coatings before aging

To quantitatively measure the carbonation resistance of a coating, an index of α is defined in Formula (1) to depict the improvement in carbonation resistance of a coating.

$$\alpha = \frac{x_u - x_{c0}}{d_c} \quad (1)$$

where x_{c0} and x_u are the respective accelerated carbonation depths of the specimens with and without a coating before aging (mm), and d_c is the thickness of a coating (μm).

Thus, α represents the contribution per unit thickness of a coating to concrete carbonation resistance. A higher value of α means that the unit thickness of a coating can provide a higher carbonation resistance improvement, that is, the efficiency of carbonation resistance of a coating is higher. On the basis of the data in Fig. 1, the values of α are 452, 273, and 329 for the one-layer specimens of PO, EP, and CR coating, respectively, and 259, 181, and 222 for the two-layer PO-, EP-, and CR-coated specimens, respectively. Of these specimens, PO coating is the highest, CR coating follows, and EP coating is the lowest. The ranking order of the unit thickness coatings to improve concrete carbonation resistance before aging is PO coating > CR coating > EP coating.

4.2. Aging resistances and service life prediction of concrete coatings

According to the previous experimental results, the carbonation depth developments of the coated specimens exhibit an S-shaped curve with aging time. Generally, a typical S-shaped curve equation is shown as Formula (2).

$$y = \frac{a}{1 + b \cdot e^{-mt}} \quad (2)$$

where a , b , and m are all constants, with $a > 0$, $m > 0$, and t is time.

According to the characteristics of an S-shaped curve, the limit of y in Formula (2) is a when the variable t tends to infinity. In this paper, a is the carbonation depth of a coated specimen after the complete failure of a coating. Theoretically, the carbonation depth of a coated specimen after the complete failure of its coating should be the same as that of an uncoated specimen. A prediction model for the carbonation depths of the coated specimens with aging time is established based on an S-shaped curve, as shown in Formula (3).

$$x = \frac{x_u}{1 + b \cdot e^{-mt}} \quad (3)$$

where x is the accelerated carbonation depth of a coated specimen after t time aging (mm), x_u is the standard accelerated carbonation depth of a specimen without a coating and is set to 20.3 mm in this paper (mm), and t is the coating aging time (d).

On the basis of Formula (3) and a nonlinear regression analysis, the carbonation depth prediction models for each kind of specimen with one-layer coating under different aging conditions are obtained and are shown in Table 4.

As shown in Table 4, the relevant coefficients (R^2) of the regression models for the specimens under ultraviolet aging and coupled aging conditions are all higher than 0.9. Given that a natural climate condition is more complex than artificial climate conditions, the carbonation depth data of the coated specimens obtained under natural aging condition are more discrete than those under artificial aging conditions. As a result, the relevant coefficients (R^2) of the regression models for the specimens under natural aging condition are relatively lower than those under artificial aging

Table 4
Carbonation depth prediction models for coated specimens.

Item	Coating	Regression equation	R ²	Predicted service life
Ultraviolet aging	PO	$x = 20.3 / (1 + 2.450e^{-0.132t})$	0.950	29.2 d
	EP	$x = 20.3 / (1 + 2.069e^{-0.146t})$	0.937	25.3 d
	CR	$x = 20.3 / (1 + 1.517e^{-0.174t})$	0.952	19.4 d
Coupled aging	PO	$x = 20.3 / (1 + 2.581e^{-0.199t})$	0.938	19.6 d
	EP	$x = 20.3 / (1 + 2.016e^{-0.229t})$	0.918	16.0 d
	CR	$x = 20.3 / (1 + 1.398e^{-0.253t})$	0.958	13.0 d
Natural aging	PO	$x = 20.3 / (1 + 2.116e^{-0.002t})$	0.733	3.0 y
	EP	$x = 20.3 / (1 + 1.843e^{-0.004t})$	0.866	2.6 y
	CR	$x = 20.3 / (1 + 1.544e^{-0.006t})$	0.819	1.5 y

conditions. However, even the lowest relevant coefficient (R²) is 0.733, suggesting that the confidence level of each regression model established in Table 4 is very high, and using the S-shaped curve to predict the carbonation depth of coated specimens with coating aging is feasible.

The time of coating failure should be determined to obtain the predicted service lives of organic film coatings in terms of their carbonation resistances. Theoretically, when a coating completely loses its protective capability, the carbonation depth of a coated specimen should be equal to that of an uncoated specimen. However, according to the characteristics of an S-shaped curve, the development of carbonation depth with aging time of a coated specimen is a gradual line of the carbonation depth of an uncoated specimen. No matter how long a coating ages, the carbonation depth of the coated specimen infinitely approaches that of the uncoated specimen. Therefore, the failure time of a coating cannot be determined, which is inconsistent with the actual situation. In this paper, the failure criterion for a coating is defined as the moment when the carbonation depth of a coated specimen reaches 95% of the carbonation depth of the uncoated specimen. On this basis, the predicted service lives of the coatings can be obtained through their respective regression models and are listed in Table 4.

As shown in Table 4, the service lives of the coatings under ultraviolet aging, coupled aging, and natural aging are within the ranges of 19.4–29.2 d, 13.0–19.6 d, and 1.5–3.0 y, respectively. Among the three types of coatings, the predicted service life of the PO coating is the longest, that of the EP coating is the second, and that of the CR coating is the shortest. Thus, the ranking order of the weathering resistances of the coatings is as follows: PO > EP > CR. Furthermore, different aging conditions induce different aging effects, and the aging speeds of the coatings under coupled aging and ultraviolet aging are significantly higher than that under natural aging. The respective accelerated aging factors of coupled aging and ultraviolet aging are nearly 52 and 34 times that of natural aging.

Given that the film thickness of one-layer application of a coating is relatively thin, the predicted service lives of a one-layer coating in the natural climate environment of Xuzhou are all short. For the best coating of PO, the predicted service life is only 3 y, whereas that of CR is only 1.5 y. Through the same method, the predicted service lives of two-layer of PO, EP, and CR coatings are 4.8, 3.9, and 2.5 y, respectively. Thus, increasing the thickness of the coating film is beneficial to enhancing the service life of a coating.

In this paper, the established concrete carbonation prediction models for coated specimens are only suitable for specific climate conditions, because of the variety of different environment climate and the variety of concrete and coating materials, further study should be done and more suitable models should be established in the future.

5. Conclusions

Through the experimental research and regression analysis in this study, some conclusions can be drawn as follows:

- (1) Organic film coating can provide effective carbonation resistance to concrete before aging, and a thicker film can provide better improvement. Based on the carbonation resistance improvement index α , the ranking order of the three types of coatings is PO > CR > EP.
- (2) Regardless of the aging condition, the aging of organic film coatings can decrease the carbonation resistance of coated specimens, and the aging efficiencies of the coatings under ultraviolet aging and coupled aging are significantly higher than that under natural aging.
- (3) Defects, such as granulation, porosity, and cracking of coatings caused by aging, are the actual causes of the degradation of coating carbonation resistance. Carbonation resistance gradually decreases as the organic film coating ages and exhibits an S-shaped curve with aging time.
- (4) Based on an S-shaped curve development of the degradation of coating carbonation resistance, models for the carbonation depth with aging time of coated specimens were established, and the service lives of the coatings were predicted. The ranking order of the coatings based on their service lives is as follows: PO > EP > CR. An increase in thickness of a coating can enhance its service life.

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