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EFFECT OF PRE-IMPREGNATED CARBONIZED AGGREGATE ON CARBONIZATION RESISTANCE OF FULLY RECYCLED CONCRETE

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In order to investigate the effect of treated recycled aggregates by pre-soaking calcium hydroxide (CH) combined with CO_2 curing on the carbonation resistance of fully recycled concrete. The carbonation depth of fully recycled concrete was tested at 3, 7, 14 and 28 days using the rapid carbonation method. The experimental results show that with the recycled aggregate replacement rate rising, the carbonation resistance of fully recycled concrete gradually decreases. CH pretreatment combined with CO_2 curing effectively improves the quality of the recycled aggregate, thereby improving the carbonation resistance of fully recycled concrete. The optimum effect of CO_2 curing on fully recycled concrete is achieved with the recycled fine aggregate replacement rate of 70 % and the recycled coarse aggregate replacement rate of 100 %. In addition, microscopic tests were used to reveal the enhancement mechanism of CH pretreatment combined with CO_2 curing increases the carbonation resistance of fully recycled concrete. Experimental data were used to fit and model the carbonation depth of fully recycled concrete formulated before and after CO_2 curing of recycled aggregates.

INTRODUCTION

The cumulative concrete production in China in 2020 is estimated to reach 2.84 billion m^3 , and the construction sector emitted 1.48 billion tons of carbon during the production process. Sand and gravel aggregate accounts for about 60-75% of the composition of concrete, and the over-exploitation of aggregate is bound to consume a large amount of natural resources, which will destroy the ecological environment, cause damage to the ecological environment of the origin of the aggregate and hinder the sustainable development of human society [1].

Under the background of double carbon, Xiao et al. [2] proposed the concept of fully recycled concrete (FRC). Three aspects make up the FRC concept, including concrete with 100 % recycled coarse aggregate (RCA) or recycled fine aggregate (RFA), concrete with both RCA and RFA, and concrete with RCA, RFA, and recycled micro powder. Fully recycled concrete is a new direction for the future development of concrete because of its ability to consume construction waste on a large scale, effectively solving the huge demand for sand and gravel and the problem of stacking and utilization of construction solid waste.

Due to the high water absorption, large porosity and low mechanical properties of the old mortar adhered to the surface of the recycled aggregate, recycled concrete has significantly lower physical properties and durability than natural concrete [3]. It is essential to reinforce recycled aggregates in order to improve its performance and ensure its durability. Shi et al. [4] compared several methods of aggregates reinforcement, carbonation reinforcement of recycled aggregates being an effective method to improve the performance of recycled concrete. The diffusion coefficient of chloride ions decreased by 36.4 % at 100 % RCA Under various environmental replacement [5]. conditions, Liang et al. [6] investigated the carbonation resistance of carbonated recycled aggregate concrete and found that carbonated recycled aggregates improved the carbonation resistance of recycled concrete. However, it is challenging to improve the performance of recycled aggregates derived from demolished concrete through regular CO₂ curing due to their long storage time and extremely low carbonizable alkaline substances. In order to solve this problem, Pan et al. [7] performed a calcium hydroxide (CH) pretreatment prior to the carbonation process. Through the CH pretreatment combined with CO₂ curing, the compressive strength of recycled concrete increased by 3.9 % to 7.6 % [7-9], and the water absorption of recycled aggregates decreased by almost 50 % [8]. According to Fang et al. [10], soaking recycled aggregates with calcium ionrich wastewater for cyclic CO₂ curing the resistance of recycled concrete to chloride ion infiltration. However, the improvement of mechanical properties was mainly studied regarding the effect of calcium hydroxide soaked carbonation treated recycled aggregates on recycled concrete, the effect of resistance to carbonation properties is still lacking.

The purpose of this paper is systematically studying the carbonation resistance of FRC in which the recycled aggregates are enhanced by CH pretreatment combined with CO₂ curing. Using the first type of FRC, the effects of different RFA replacement rates (0 %, 30 %, 50 %, 70 %, 100 %) on the FRC prepared with 100 % RCA, and different RCA replacement rates (0 %, 50 %, 100 %) on the carbonation resistance of the FRC prepared with 100 % RFA were analyzed. Various microscopic tests were used to analyze the effect of composite carbonation reinforcement on the phase composition and microstructure of FRC. For the effects of RCA, RFA replacement rates, and CO₂ curing aggregates, a prediction model of FRC carbonation depth was developed.

EXPERIMENTAL

Test material and matching ratio

In this experiment, P.O42.5 ordinary silicate cement, crushed stone with a particle size of 5-20 mm, and natural river sand with a particle size of less than 4.75 mm were used. RCA with particle sizes of 5-20 mm and RFA with particle sizes below 4.75 mm were obtained by crushing the C40 beams in the lab using a jaw crusher. With a water cement ratio of 0.49 and a strength class of C40, the FRC was prepared. In order to ensure the correct water cement ratio, the recycled aggregates were treated to a saturated dry surface before casting. The mixing ratios are shown in Table 1.

Table 1. Details of all the mix proportions $(kg \cdot m^{-3})$.

Testing Method CO₂ curing enhancement of recycled aggregates

First, the recycled aggregates were soaked in CH solution for 24 hours. Then, the pretreated recycled aggregates was dried in an oven at 105 °C and moved to a constant temperature and humidity chamber at 22 ± 2 °C and 60 - 70 % relative humidity until its moisture content met the experimental requirements. Finally, it was put into a carbonation reactor with a volume of 50 L, a CO₂ concentration of 99.9 %, a temperature of 20 °C, a humidity of 70 %, and a pressure of 0.3 MPa. After spraying the recovered aggregate with 1 % phenolphthalein reagent, the aggregate did not turn red and carbonation was completed.

Accelerated carbonation of FRC

According to the Chinese standard GB/T50082-2009, $100 \times 100 \times 100$ mm cubic specimens were used for accelerated carbonization test. The specimens were cured for 26 days, baked in an oven at 60 °C for 48 hours, waxed and then placed in a carbonation chamber with a temperature of 20 ± 2 °C, humidity of 70 ± 5 % and CO_2 concentration of 20 ± 3 %. After reaching the corresponding carbonation age, the specimens were split from the middle and the residual powder on the section was swept away. Phenolphthalein alcohol solution with a concentration of 1 % was sprayed, and the carbonation depth was measured by electronic calipers along the pre-marked points after 30 s of color development.

				RFA		RCA		
Group	Cement	NFA	NCA	Before	After	Before	After	water
				CO ₂ curing	CO ₂ curing	CO ₂ curing	CO ₂ curing	
NAC	378	680	1157	0	0	0	0	185
FRC (100, 0)	378	680	0	0	0	1157	0	185
FRC (100, 30)	378	476	0	204	0	1157	0	185
FRC (100, 50)	378	340	0	340	0	1157	0	185
FRC (100, 70)	378	204	0	476	0	1157	0	185
FRC (100, 100)	378	0	0	680	0	1157	0	185
FRC (50, 100)	378	0	578.5	680	0	578.5	0	185
FRC (0, 100)	378	0	1157	680	0	0	0	185
CFRC (100, 0)	378	680	0	0	0	0	1157	185
CFRC (100, 30)	378	476	0	0	204	0	1157	185
CFRC (100, 50)	378	340	0	0	340	0	1157	185
CFRC (100, 70)	378	204	0	0	476	0	1157	185
CFRC (100, 100)	378	0	0	0	680	0	1157	185
CFRC (50, 100)	378	0	578.5	0	680	0	578.5	185
CFRC (0, 100)	378	0	1157	0	680	0	0	185

Note: NAC-natural aggregate concrete; FRC is fully recycled aggregate concrete; CFRC is CO_2 cured aggregate fully recycled aggregate concrete; (x, y) where x is the recycled coarse aggregate replacement rate and y is the fine aggregate replacement rate.

Microscopic Testing

- (1) X-ray powder diffractometer (XRD). With 200 mesh square sieve, FRC bonded mortar samples were sieved and dried in a vacuum oven at 65 °C until their mass remained unchanged. The scanning rate was 5°·min⁻¹ and the scanning range was 5 70° using XRD.
- (2) Thermogravimetric analysis (TG). With 200 mesh square sieve, FRC bonded mortar samples were sieved and dried in a vacuum oven at 65 °C until their mass remained unchanged. Samples were examined using a Japanese TG-DTA7300 thermogravimetric analyzer with a temperature range of 0 − 1000 °C, a heating rate of 10 °C·min⁻¹, and a nitrogen flow rate of 30 ml·min⁻¹.
- (3) Scanning electron microscopy (SEM) and energy spectroscopy (EDS). After polishing and finishing, the FRC samples were dried to constant weight in an oven at 65 °C. The images obtained from scanning electron microscopy were used to examined the microscopic morphology and the energy spectrometry were used to analyze the chemical elements.

RESULTS AND DISCUSSION

Physical properties of CO₂ curing-treated aggregates

Table 2 shows the tests of water absorption, crushing value, and apparent density of RCA and RFA before and after CO₂ curing. CO₂ curing resulted in a 0.38 % and 1.49 % increase in apparent density, a 10.4 % and 16.49 % decrease of the crushing value, and a 19.24 % and 15.5 % decrease of the water absorption of RCA and RFA, respectively. The data shows that the CO₂ curing has a small improvement of the apparent density of recycled aggregates. There are two reasons for this results: (1) CH pretreatment combined with CO₂ curing generates a large amount of calcium carbonate, which can reduce the macropores in the mortar attached to the surface of the recycled aggregates, but have limited effect on the mesopores and micropores [11]; (2) the denser surface of the recycled aggregates caused by carbonation reduced the rate of CO₂ diffusion into the recycled aggregates [12]. When compound carbonation is enhanced, the recycled aggregates structure becomes denser, the solid phase volume increases by about 20 %, and the crushing value and water absorption rate are significantly reduced [4]. The enhancing effect of compound carbonation on RFA is more significant because the water absorption and crushing value of recycled aggregates are related to the mortar content on the surface of recycled aggregates. More mortar is attached to the surface of RFA and these mortars' relative specific surface area is larger, allowing for a more complete reaction between CO₂ gas and mortar [13].

The carbonation resistance of CO₂ curing-treated FRC Effect of recycled aggregates replacement rate on the carbonation resistance of FRC

Figure 1 illustrates the effect of recycled aggregates replacement rate on the carbonation depth of FRC, where the normal FRC is represented by Figure 1a, c and FRC formulated with recycled CO2-cured aggregate is shown in Figure 1b, d. The carbonation resistance of both FRCs decreases with the increase of replacement rate. At the carbonation age of 3 days, compared with natural aggregates concrete (NAC), in Figure 1a the carbonation depths of FRC (100, 0) and FRC (100, 30) decreased by 4.96 % and 8.51 %, and the carbonation depths of FRC (100, 50), FRC (100, 70) and FRC (100, 100) increased by 5.32 %, 10.64 % and 19.86 %, respectively. This is due to the fact that, at a lower replacement rate of RFA, the internal CH of the mortar reacted with CO₂ to generate CaCO₃, which partially filled the mortar pores, temporarily prevented CO2 intrusion, and enhanced the carbonation resistance of FRC. The carbonation depths of FRC in Figure1b are all higher than those of NAC, indicating that excessive old mortar lead to pore connections and infiltration within FRC when the replacement rate of RFA is high. The CaCO₃ produced in a short period of time was not sufficient to prevent CO₂ intrusion, thus reducing the carbonation resistance of the FRC. FRC carbonation depths in Figure 1c, d are also deeper than NAC, which shows that the effect of recycled CO₂-cured aggregates on FRC is not evident at the early stage of the carbonation test.

At the carbonization age of 28 days, the carbonation depths of FRC (100, 0) and FRC (0, 100) were 4.58 mm and 6.27 mm, and the carbonation depths of CFRC (100, 0) and CFRC (0, 100) were 4.33 mm and 5.57 mm, respectively. It can be seen that using or without CO₂ curing treatment, 100 % RFA replacement rate significantly reduces the carbonation resistance of FRC far more than FRC with 100 % RCA replacement. This is due to the fact that the content of bonded mortar on the surface of recycled aggregates is negatively correlated with the aggregates particle size.

Table 2. Physical properties of recycled aggregates.

	Apparent	Apparent density (kg·m ⁻³)		Crushing value (%)		Water absorption (%)	
	Before	After	Before	After	Before	After	
	CO ₂ curing	CO ₂ curing	CO ₂ curing	CO ₂ curing	CO ₂ curing	CO ₂ curing	
RCA	2640	2650	19.33	17.32	5.74	4.85	
RFA	2619	2658	32.32	26.99	9.72	7.85	

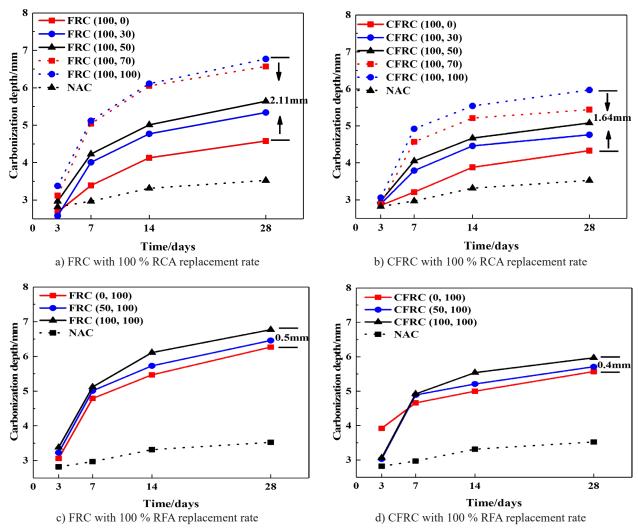


Figure 1. The relationship between carbonation depth and carbonation time.

The smaller the particle size of the recycled aggregates, the more old mortar will be adhered to the surface, which is consistent with the study of Yang et al. [14]. Although CO₂ curing improves the quality of old mortar adhered to the surface of recycled aggregates, the carbonation depth of CO₂ curing enhanced FRC is still higher than that of NAC. This is due to the fact that old mortar not only adheres to the surface of recycled aggregates, but also constitutes the recycled aggregates itself. The resistance of mortar to CO₂ erosion is much lower than that of natural aggregates. As the increase in the replacement rate of CO2 curing recycled aggregates, some of the natural aggregates are replaced by hardened mortar in FRC compared with NAC. Therefore, the carbonation resistance of FRC is still lower than that of NAC after CO₂ curing.

Effect of carbonization time on the carbonation resistance of FRC

The carbonation depth of FRC increases with increasing carbonation time. Combining with Figure 1, it can be seen that the carbonization depth of FRC

developed faster in the early stages of the experiment, and the carbonation depth of FRC had reached 50 % of the final carbonation depth when the carbonation age was 3 days. After that, the change rate of carbonation depth gradually slowed down with the growth of time. The increase of carbonation depth of FRC from 14 to 28 days was only about 10 % of the final carbonation depth when comparing the carbonization depth of FRC in 28 days with that in 14 days. This is due to the fact that the CO₂ gas inside the carbonation chamber is attached to the surface of FRC, and the CO₂ slowly penetrates from the surface to the interior. When the specimen is carbonated at a limited depth on the surface, the carbonate with larger particle size and insoluble in water is gradually deposited inside the material, which reduces the porosity inside the material and refines the pore structure, which slows down the diffusion of CO₂ to the concrete interior to a certain extent. Thus, the carbonation process makes FRC denser, and the carbonation resistance of FRC gradually increases with the growth of carbonation time.

Effect of CO₂ curing recycled aggregates on the carbonation resistance of FRC

Figure 2 shows the trend of the effect of CO₂ curing on the depth of FRC carbonation. In Figure 2a, at 3-day carbonation age, the carbonation depths of CFRC (100, 0), CFRC (100, 30) and CFRC (100, 50) are increased by 6.34 %, 15.12 % and 0 % compared with FRC (100, 0), FRC (100, 30) and FRC (100, 50), respectively. This is due to the fact that the carbonation resistance of concrete is related to its internal alkaline content and pore distribution, and the neutralization reaction between CO₂ and CH in the mortar during CO₂ reduces the alkalinity inside the recycled aggregates and increases the carbonation risk of FRC [15-16]. As the recycled aggregates replacement rate increases, CO₂ curing enhanced recycled aggregates reduce the carbonation depth of FRC. This indicates that although the CO₂ curing reduces the alkalinity within the recycled aggregates, the admixture ratio of the recycled mortar increases successively with the increase of the recycled aggregates replacement rate. The enhancement effect of CO₂ curing on the carbonation resistance of FRC is highlighted, and the carbonation depth of FRC is reduced.

In Figure 2b, c and d, the carbonation resistance of CO₂ curing FRC is better than that of plain FRC at accelerated carbonization test ages of 7, 14 and 28 days. The most significant improvement was achieved in FRC (100, 70), whose carbonation depth was reduced by 9.33 %, 13.88 % and 17.2 %, respectively. This is due to the generation of hydrophilic silica gel in the CO₂ curing recycled aggregates, which absorbs some water during concrete mixing to play the role of internal maintenance, so that the unhydrated cement clinker particles of the bonded mortar continue to hydrate. This part of water is gradually released for additional cement hydration, increasing the density of fully recycled concrete. However, FRC is a complex nonhomogeneous multiphase material. With the increase

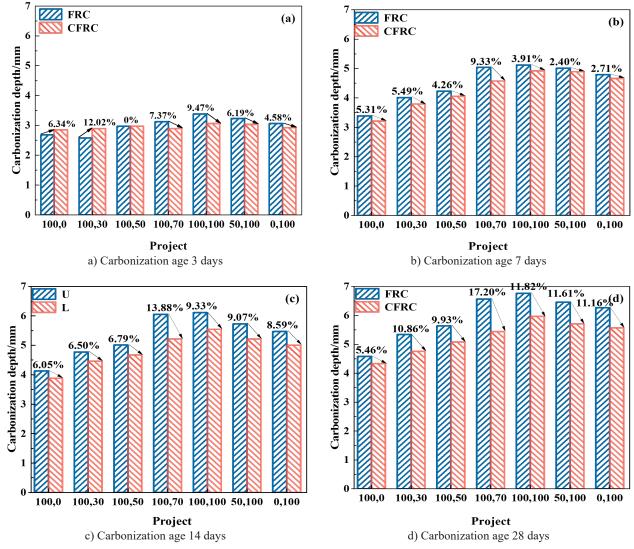


Figure 2. Relationship between carbonation depth and replacement rate of recycled aggregates.

in the replacement rate, the deterioration of properties is becoming more significant. Under the coupling of the two factors, FRC (100, 70) shows the most significant effect of CO₂ curing on the improvement of the carbonation resistance.

The CO₂ curing recycled aggregates significantly improve the carbonation resistance of RFC over time. This is due to the fact that the recycled aggregates include a lot of stone powder that adheres to its surface after crushing, thus hindering the connection between recycled aggregates and cement. Additionally, many angles of the recycled aggregates and the old mortar with the feature of loose and porous on its surface reduce the durability of the FRC [17]. However, the CO₂ curing densifies the microstructure of the mortar attached to the surface of the recycled aggregates, the calcite produced on the surface of the recycled aggregates emits CO₃², which reacts with the aluminate ions in the fresh cement matrix to form single carbon aluminate (Mc). This offers additional nucleation sites for C-S-H growth on the surface of the recycled coarse aggregates, resulting in an improvement of not only the performance of the recycled aggregates but also the strength of the interfacial transition zone in the FRC [18-19]. Meanwhile, the carbonation products seal the pores of the recycled aggregates, raise the pore curvature of FRC, and inhibit CO₂ penetration to the interior [20], with the enhancing effect becoming more substantial as the accelerated carbonation test progress.

Microscopic properties of FRC before and after CO₂ curing *Qualitative analysis of micro-components*

A comparison between the XRD patterns of the FRC (100, 70) and the CFRC (100, 70) is shown in Figure 3. Compared with FRC (100, 70), the diffraction peak of Ca(OH)₂ of CFRC (100, 70) greatly decrease at the 2θ peaks of 18° and 34°, while the diffraction peaks of calcite remarkably increases at the 2θ peaks of 29.5°, 36.1°, 39.4° and °43.1°. The crystalline phases of CaCO₃ are calcite, aragonite, and vaterite [21], while no aragonite or vaterite are found in the current XRD pattern. There are two reasons for these results: (1) the vaterite has been transformed into relatively stable aragonite and calcite during the storage of the recycled aggregates; (2) After CH pretreatment combined with CO curing, the main crystalline phase is calcite.

Quantitative analysis of micro-components

Figure 4a, b illustrates the thermo gravimetric changes in FRC (100, 70) and CFRC (100, 70). According to the corresponding exothermic peaks on the thermogravimetric and thermal difference analysis curves, the quantities of CaCO₃ and CH can be compared.Based on the data, CH mass decreases and CaCO₃ mass notably increases after CH

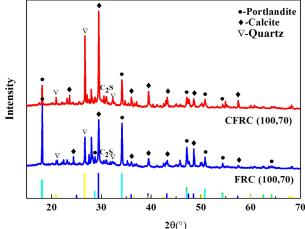
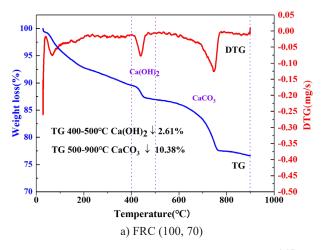


Figure 3. XRD pattern.



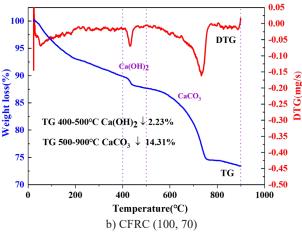


Figure 4. TG and DTG curves.

pretreatment combined with CO₂ curing. This is because the CaCO₃ and silica gel produced by carbonation enhancement react with cement hydration to produce calcium carboalumination and C-S-H during FRC preparation, which consumes the amount of CH in FRC. Furthermore, the carbonation product CaCO₃ promotes the hydration reaction of FRC due to its nucleation action.

Microscopic morphology

To further understand how CO₂ curing aggregates strengthen the microstructure of FRC, SEM tests were carried out on FRC (100, 100), CFRC (100, 100), and the results are shown in Figure 5. Compared with Figure 5a FRC (100, 100), Figure 5b CFRC (100, 100) has a denser overall structure, fewer pores, and no obvious cracks. Several needle-like calcium alumina and hexagonal platelet calcium hydroxide stone are found in specimens FRC (100, 100) in Figure 5c.

CFRC (100, 100) specimens show an increase in calcium carbonate and the presence of a large number of hexagonal flakes of calcium aluminate monocarbonate (Mc) produced by the reaction of calcite and aluminate in Figure 5d. Combined with the energy spectrum analysis in Figure 5e, f, there is also a small amount of Si in the CaCO₃ crystals, which may come from the silica gel wrapped around the outside of CaCO₃. These CaCO₃ crystals are stacked with silica gel to fill the pores and cracks in the recycled aggregates, making the FRC sample structure denser.

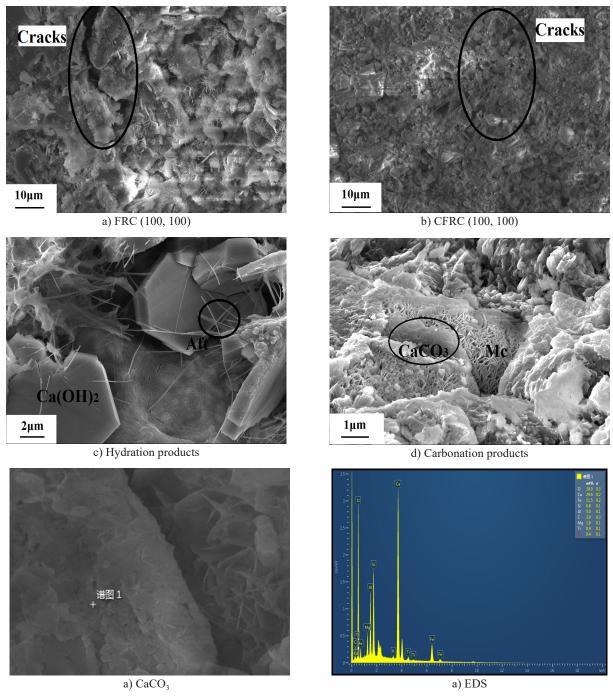


Figure 5. SEM and EDS images.

PREDICTIVE MODEL FOR CARBONATION DEPTH OF FRC

Similar to NAC, as the square root of time increases, the carbonation depth of FRC increases as a power function [6]. The experimental datas are brought into the NAC basic carbonation model, as the following Equation 1. Based on the theoretical model obtained from Fick's first law, the carbonation depth and carbonation coefficient are used to measure the carbonation resistance of FRC. A prediction model for the carbonation depth of FRC is establish as follows.

$$d = k\sqrt{t} \tag{1}$$

$$k = \sqrt{2DC_0} \tag{2}$$

Where C_0 is the concentration of CO_2 in the environment (20 % in the accelerated carbonation test), d is the carbonation depth (mm), k is the carbonation rate coefficient, t is the carbonation time (d), D is the diffusion coefficient of CO_2 gas in concrete. The carbonation depths of FRC before and after CO_2 curing at 3, 7, 14 and 28 days

are fitted according to Equation 1, and the fitting results are shown in Figure 6. The carbonation rate coefficient k is obtained, and the diffusion coefficient D is calculated according to Equation 2, and the results are shown in Table 3.

Table 3. Fitting parameters of carbonation depth of fully recycled concrete.

Project	k	R2	D
FRC (100, 0)	1.025	0.84	2.627
FRC (100, 30)	1.177	0.889	3.463
FRC (100, 50)	1.249	0.871	3.900
FRC (100, 70)	1.464	0.88	5.358
FRC (100, 100)	1.502	0.879	5.640
FRC (50, 100)	1.432	0.871	5.127
FRC (0, 100)	1.377	0.883	4.740
CFRC (100, 0)	0.978	0.784	2.391
CFRC (100, 30)	1.094	0.797	2.992
CFRC (100, 50)	1.158	0.812	3.352
CFRC (100, 70)	1.257	0.814	3.950
CFRC (100, 100)	1.358	0.845	4.610
CFRC (50, 100)	1.306	0.808	4.264
CFRC (0, 100)	1.294	0.696	4.186

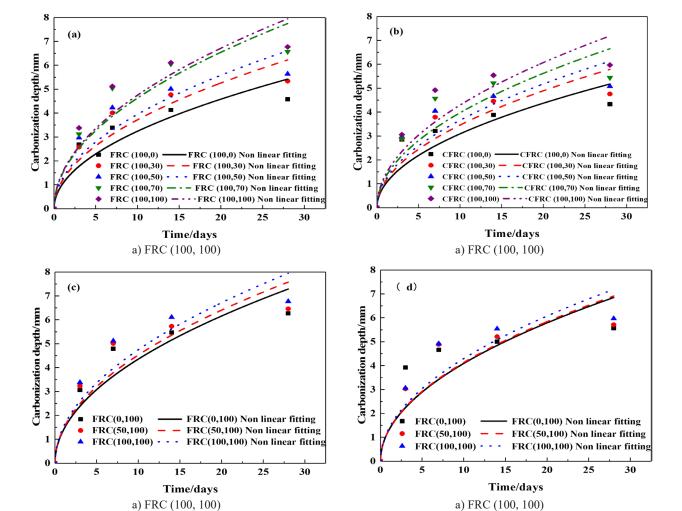


Figure 6. Fitting curve of carbonation depth of FRC.

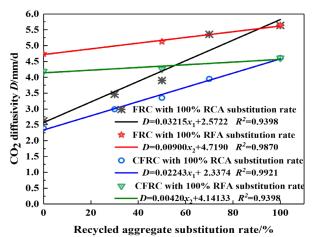


Figure 7. Fitted image of D value of diffusion coefficient of fully recycled concrete.

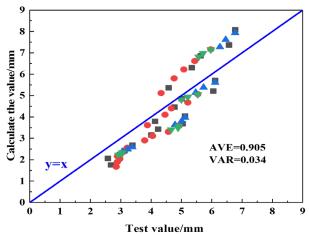


Figure 8. Comparison of calculated and tested carbonation depth values.

Table 4. Prediction model of carbonization depth.

Project	Carbonization depth model
FRC with 100 % RCA replacement rate	$d = \sqrt{2(0.03251x_1 + 2.5722)C_0} \times \sqrt{t}$
CFRC with 100 % RCA replacement rate	$d = \sqrt{2(0.02243x_1 + 2.3374)C_0} \times \sqrt{t}$
FRC with 100 % RFA replacement rate	$d = \sqrt{2(0.009x_2 + 4.719)C_0} \times \sqrt{t}$
CFRC with 100 % RFA replacement rate	$d = \sqrt{2(0.00420x_2 + 4.14133)C_0} \times \sqrt{t}$

According to Table 3, there is a strong linear relationship between the recycled aggregates replacement rate x (x_1 is the RFA replacement rate, and x_2 is the RCA replacement rate) and the diffusion coefficient D of CO_2 gas in FRC. The fitting results are displayed in Figure 7. Based on this, the carbonation depth prediction model for FRC is established and shown in Table 4.

The carbonation depth prediction model developed in this paper can accurately predict the carbonation depth of FRC before and after CO_2 curing, as shown in Figure 8. The mean value (AVE) of the ratio of calculated values to testing values of the FRC carbonation depth prediction model is 0.905 and the variance (VAR) is 0.034.

CONCLUSIONS

- (1) The carbonation depth of fully recycled concrete (FRC) steadily increases with an increasing replacement ratio, and RFA results in a more profound degradation of the carbonation resistance of FRC.
- (2) Compared to FRC made with raw recycled aggregates, FRC made with $\rm CO_2$ curing recycled aggregates exhibit better carbonation resistance. The optimum improvement effect is achieved with the replacement rate of RFA of 70 % and the replacement rate of RCA of 100 %.
- (3) XRD patterns and TG reveal that the CO₂ curing process produced CaCO₃. Because CaCO₃ generated

fills the pores of the recycled aggregates, the structure of the FRC is denser, and the performance of the FRC is refined, as indicated by SEM images.

(4) Based on the effect of recycled aggregates replacement rate on CO₂ diffusion efficiency, a prediction model for FRC carbonation depth before and after CH pretreatment combined with CO₂ curing is established. The mean values of the prediction results to the actual results are 0.905 with a variance of 0.034, which can be used to predict the FRC carbonation depth before and after CH pretreatment combined with CO₂ curing recycled aggregates.

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