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Carbonization Durability of Two Generations of Recycled Coarse Aggregate Concrete with Effect of Chloride Ion Corrosion

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Abstract: Carbonation durability is an important subject for recycled coarse aggregate concrete (RAC) applied to structural concrete. Extensive studies were carried out on the carbonation resistance of RAC under general environmental conditions, but limited researches investigated carbonation resistance when exposed to chloride ion corrosion, which is an essential aspect for reinforced concrete materials to be adopted in real-world applications. This paper presents a study on the carbonation durability of two generations of 100% RAC with the effect of chloride ion corrosion. The quality evolution of recycled concrete coarse aggregate (RCA) with the increasing recycling cycles was analyzed, and carbonation depth, compressive strength and the porosity of RAC were measured before and after chloride ion corrosion. The results show that the effect of chloride ion corrosion negatively affected the carbonation resistance of RAC, and the negative effect was more severe with the increasing recycling cycles of RCA. Chloride ion corrosion led to a decrease in compressive strength, while an increase in carbonation depth and the porosity of RAC. The equation of concrete total porosity and carbonation depth was established, which could effectively judge the deterioration of carbonation resistance of RAC.

Keywords: recycled coarse aggregate; repeated recycled concrete; carbonation durability; chloride ion corrosion

1. Introduction

The application of RAC in non-structural and structural concrete can effectively solve the shortage of natural coarse aggregate (NCA) and the environmental crisis caused by waste concrete, which is considered as an inevitable way for the sustainable development of concrete engineering. With the increasing popularity of applications, the recycled concrete structure faces the problem of recycling after its service life. Huda [1] studied the mechanical properties of three generations of recycled concrete with 100% replacement of recycled coarse aggregate (RCA) and found that although the increase in the recycling cycles led to a gradual decline in the compressive strength of RAC, RAC exhibited better deformation capacity and its peak strain was higher than that of ordinary concrete. Zhu et al. [2] evaluated the durability evaluation of three generations of 100% repeated RAC. The results showed that durability metrics of the RAC decreased gradually with the increasing number of recycling cycles, but the RCA in each cycle could meet the requirements for structural concrete. Ángel Salesa [3] studied three generations of waste precast concrete and found that the new concrete with 100% recycled and multi-recycled coarse aggregates from rejected precast concrete performed equally well, even slightly higher in compressive strength compared with control concrete. Pavlů et al. [4] used a lifecycle

assessment to compare two use cycles of natural aggregate concrete and recycled aggregate concrete. The obtained results confirmed the potential use of high-quality recycled aggregate originating from local sources in some applications in building structures and indicated the benefits of using recycled materials. These studies confirm the suitability of multi-recycled coarse aggregate concrete as materials for reinforced concrete.

To date, research shows that the durability of RAC restricts the application and development of multi-recycled coarse aggregate concrete [5–7]. The carbonization durability of concrete is an essential subject as it allows the understanding of the performance of concrete throughout the service life of a structure. The neutralization of concrete caused by carbonization leads to the failure of steel passivation film protection, which results in corrosion of steel bars, and seriously affects the normal use and safe operation of concrete structures. Extensive studies were carried out on the carbonization durability of RAC under general environmental conditions [8–11], but a limited number of researches investigate its carbonization performance when exposed to the environmental condition of chloride ion corrosion [12,13], which is an essential aspect for reinforced concrete materials to be adopted in real-world applications. Carbonation and chloride attack are the two main phenomena that initiate reinforcement corrosion, by destroying the passive coating on reinforcing bars. It is worth highlighting that research on the carbonation resistance of RAC, especially the influence of chloride ion corrosion on it, will help us to have a deeper understanding of the durability failure of recycled concrete, and can more accurately predict the service life of the structure.

1.1. Carbonation Durability of Recycled Concrete: Effects of Recycled Coarse Aggregate

The carbonation durability of concrete may be prompted by external environmental agents or internal agents of concrete. In terms of internal agents, many studies have analyzed the influence of RCA on the carbonation resistance of RAC, highlighting that the performance of RCA is an important factor affecting the carbonation resistance.

Existing studies [14,15] have shown that the effects of RCA on the carbonization of recycled concrete mainly include two aspects. On the one hand, the porosity of recycled aggregate is larger than that of natural aggregate, which leads to the greater porosity and water absorption of recycled concrete than that of ordinary concrete with the same water-cement (w/c) ratio. Carbon dioxide (CO₂) is more prone to penetrate into the recycled concrete, thus accelerating the carbonization speed and increasing the carbonization depth, which reduces the carbonization resistance. On the other hand, the old cement mortar attached to the surface of recycled aggregate increases the total cement content of recycled concrete, in other words, increases carbonizable substances, which is conducive to carbonization resistance. Therefore, the carbonation resistance of recycled concrete should be influenced by both the positive and negative aspects mentioned above, and the carbonation result is a combination of the two aspects.

Generally, according to the literature [16–18], as the replacement level of RCA increases, the carbonation depth of RAC also increases. It was found that when 100% RCAs are introduced, RAC mixes may show carbonation depths up to 2.15 and 6.03 times greater than those of the NAC. Silva [16] found when other factors were the same, the carbonation depth of concrete increased with the increase of the replacement rate of recycled aggregate, and the use of 100% RCA in concrete caused up to two times the carbonation depth than that of the corresponding NAC. Xiao et al. [19] found that the carbonation depth of RAC increased first and then decreased with the increase of the replacement rate of RCA. When the replacement rate was 70%, the negative impact of recycled coarse aggregate on recycled concrete reached the maximum, and its carbonation depth reached the peak; when the replacement rate exceeded 70%, the negative impact started to be smaller than its positive impact, and the carbonation depth decreased. Zhu et al. [20] investigated carbonation resistance of concrete with two generations of recycled fine aggregate (RFA) under a coupling of bending load and carbonation and suggested that the carbonation depth of recycled concrete increased with the bending load and replacement rate of RFA increasing.

However, it was perceived that some RAC mixes showed similar or higher resistance to carbonation than NAC. Xiao et al. [20] also suggested that the carbonation depth of RAC decreased with the increase of the strength of recycled coarse aggregate parent concrete. When the strength of recycled coarse aggregate parent concrete is large, the carbonation resistance of recycled concrete is better, even equivalent to that of ordinary concrete. Poon et al. [21] found that the carbonation depth of recycled concrete decreased with the increase of the attached mortar strength of recycled coarse aggregate. Shi et al. [22] summarized methods for improving RCA performance by removing or strengthening mortar adhesion. These methods have resulted in recycled concrete aggregate with less porosity and water absorption, thus reducing the w/c ratio of recycled concrete and equivalent carbonation depth to that of corresponding NAC.

1.2. Carbonation Durability of Recycled Concrete: Effects of Water Reducing Admixtures and Mineral Additions

Some scholars have improved the carbonation durability of RAC by using efficient water-reducing admixtures and mineral additions. Matias et al. [23] performed an experimental investigation to evaluate the effect of different superplasticizers on the carbonation resistance of concrete with RCA. It has been suggested that the use of superplasticizers allowed the carbonation depth of the RAC to be lower than that of the RAC at early ages. Over time, the relative efficiency of superplasticizers decreased in the RAC, even though the RAC with the high-performance superplasticizer always had lower carbonation depth than the one with the standard superplasticizer. This may be due to higher internal humidity associated with lower porosity, resulting in slower water evaporation, similar to prolonged curing, and possibly a partial reduction in the depth of carbonation. The introduction of superplasticizer delayed the curing time of cement hydration, which was equivalent to a prolonged cure, and thus improved the carbonization depth of the mix using superplasticizers. Silva [15] also discovered that the reduction in the w/c ratio by using a water-reducing agent could effectively improve the mechanical strength of recycled concrete and reduce the carbonation depth, which is consistent with Matias's research conclusions.

There have been many studies regarding the effect of mineral additions on the carbonation of RAC, but the results are inconsistent. Kurda [24] analyzed the carbonation resistance of low- and high-strength concrete mixes produced with various incorporation ratios of RCA and RFA. The results show that the carbonation depth of concrete increased up to three and six times when incorporating 30% and 60% of fly ash (FA), respectively, and 100% of RCA led to an up to 2.7-time increment. This trend was also observed by Sim and Park [25], who found that the carbonation resistance of concrete with 100% RCA and incremental ratios of RFA increased with increasing incorporation of RFA and FA. This could be explained in that the alkalinity of cement cementing material decreased and the calcium silicate hydrate (C-S-H) formed from the pozzolanic reaction absorbs more alkali ions, hence lowering the pH level in concrete.

On the contrary, Bhashya et al. [26] evaluated the effect of mineral additions on the carbonation durability of RAC and discovered that the use of heat-treated recycled aggregates and incorporating FA/silica fume (SF) in addition to cement reduced the carbonation depth in concrete. Reduction in carbonation depth of RAC was due to the minimization of pores in recycled aggregate after heating and abrasion treatment and incorporation of FA or SF in addition to cement in recycled aggregate concrete. Singh et al. [27] assessed the relative efficacy of silica fume and Metakaolin (MK) blended with Portland cement and FA in compensating the loss of carbonation resistance due to the inclusion of RCA in self-compacting concrete. It has been observed that the inclusion of FA/MK compensated the loss of carbonation resistance of self-compacting concrete up to 50% replacement of NCA with RCA. Based on the results, the carbonation performance of FA blended mixes was better than that of MK blended self-compacting concrete mixes.

1.3. Carbonization Durability of Recycled Aggregate Concrete Assessed by Permeability-Related Properties

Studies have shown that the permeability of recycled concrete affects its carbonation resistance, and recycled concrete with high permeability is more prone to carbonation [28]. The permeability of recycled concrete is affected by many factors, but it is generally believed that the porosity of concrete has a great influence on it, especially the connected porosity. Since the w/c ratio and the effectiveness of curing have a great influence on the compactness of concrete, the porosity of concrete with a high w/c ratio and inadequately cured is large, which makes the concrete more prone to carbonization. The porosity of concrete is governed by the porosity of the cement paste, while the surface of recycled aggregate adheres to a lot of old mortar, so the porosity of the cement paste of recycled concrete is generally higher than that of the corresponding natural concrete, leading to the high permeability of recycled concrete. Hence, the use of more porous aggregates increases the permeability of concrete and thus the carbonation rate [15,28].

Evangelista's research [29] confirmed that the durability of the fine recycled aggregate concrete mixture was directly affected by its permeability performance, which in turn was affected by the amount and quality of RFA. The higher porosity of these aggregates allowed CO₂ to migrate more easily through the matrix, leading to a potential increment of 60% of the carbonation coefficients compared to the corresponding concrete.

Kazmi [30] studied the effect of different treatment techniques of RCA on the durability properties of RAC. According to the results, regression models were established between the physical properties of coarse aggregate and the durability of concrete, and the carbonization durability of RAC could be predicted effectively according to the porosity of coarse aggregate.

Amorim [16] conducted a test to assess whether exposure of RAC to a drier environment had any effect on the carbonation depth of concrete with increasing RCA content. He found that the carbonization depth of specimens cured in the dry environment was greater than that in other environments. As the replacement level increased, the carbonization depth of these specimens increased significantly (30% increase when 100% RCA was used at 91 days).

1.4. The Effect of Chloride Ion Corrosion on the Carbonization Durability of RAC

The ability of chloride ions to penetrate the concrete cover is a key factor in the service life of structural concrete. It is now generally accepted that chloride ions can ingress concrete through a combination of transport mechanisms, namely absorption by capillary suction, diffusion, and permeation [31]. In general, the resistance to chloride attack of recycled concrete is worse than that of the corresponding natural concrete, which is usually attributed to the increase of water absorption characteristic of RCA. The majority of the published research on the effect of RCA on concrete durability has focused on rapid chloride migration and water absorption test methods to determine acceptable levels of replacement of natural aggregates.

The chloride attack involves a coupled physical-chemical interaction between environments and dynamic responses of concrete. It is known that the crystallization of chloride can potentially modify the properties of concrete (e.g., pore structure, hydrated phase assemblage, the binding capability of hydrates), which further affects the penetration process for CO₂ [32]. H. Kuosa et al. [33] found that the chloride-bearing crystals could physically block the pores which contributed to a reduction of carbonation depth. The free chloride was hygroscopic adsorbing moisture into concrete, blocking the porosity of concrete, and retarding the carbonation reaction. Xu et al. [34] also considered that due to the filling of chloride crystal and the deposition of Friedel's salt, the invasion of chloride ions into concrete refined the pore structure and improved carbonation resistance of concrete.

On the contrary, some scholars believe that the chemical reaction between chloride salt and concrete, which generates Friedel's salt and expandable compound salt, consumes carbonizable substances in concrete, leads to the decomposition of part C-S-H, and degrades the compactability of recycled concrete, thus accelerating the carbonization reaction. Ye et al. [32] found that chloride ion

corrosion had a negative effect on carbonation resistance of concrete and the more serious the chloride ion corrosion, the worse carbonation resistance.

As aforementioned, it is not clear whether chloride penetration to some extent accelerates or retards carbonation corrosion. Therefore, more investigation is needed regarding the influence of chloride penetration on the carbonation of RAC and the corresponding carbonation mechanism, which has aroused our great interest.

1.5. Significance of the Research

The concise review presented in the previous subsections demonstrates that the research topic proposed in the present manuscript is worthy of investigation as most studies on carbonation resistance of RAC have focused on a single environmental factor, limited information is available on carbonation durability of RAC when subjected to chloride ion corrosion [35,36].

For concrete, chloride ion corrosion is the most common environmental factor, which causes corrosion of reinforcement in concrete and damages the concrete structures. The chemical reaction and physical adsorption of chloride ions with cement material affect the material composition and microstructure of concrete. The chloride ions that permeate into the concrete react directly with the unhydrated cement components, mainly $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ and $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_3$, to generate Friedel's salt and also can be replaced from the AFm ($3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaSO}_4\cdot 12\text{H}_2\text{O}$) in the form of ion exchange to generate Friedel's salt, which causes the change of concrete pore structure and affects the permeability of concrete. As explained in Section 1.4, the crystallization of chloride and the physical adsorption of chloride ions on concrete change the internal environment of concrete, which further affects the carbonation reaction of concrete.

China has a wide geographical area and a long coastline, and chloride ions are found in abundance in coastal soils and groundwater. In these areas, carbonization is accompanied by chloride attack, which may even further increase the risk of corrosion in reinforced concrete structures than the single carbonization deterioration process alone [37,38]. Whether the carbonation durability of existing concrete structures with exposure to chloride attack can meet the requirements during the service life? Whether the carbonization durability of RAC, which recycled aggregate are prepared from these concrete structures after their service life, can meet the requirements for structural concrete? Solutions to these problems depend on a deep understanding of the carbonation resistance of concrete in a chlorinated environment.

This paper presents a study on the carbonation resistance of two generations of 100% RAC with the effect of chloride ion corrosion. Specifically, NAC and RAC with two generations of 100% RCA were considered in the present study, with the aim of investigating whether concrete with significantly different coarse aggregate resulted in a different evolution of carbonation resistance induced by chloride attack and evaluating whether the carbonation resistance of multi-recycled coarse aggregate could meet the requirements of structural concrete. Conversely, the majority of studies available in the literature were based on general environmental conditions, with no considerations about chloride ion corrosion.

2. Materials and Methods

2.1. Materials

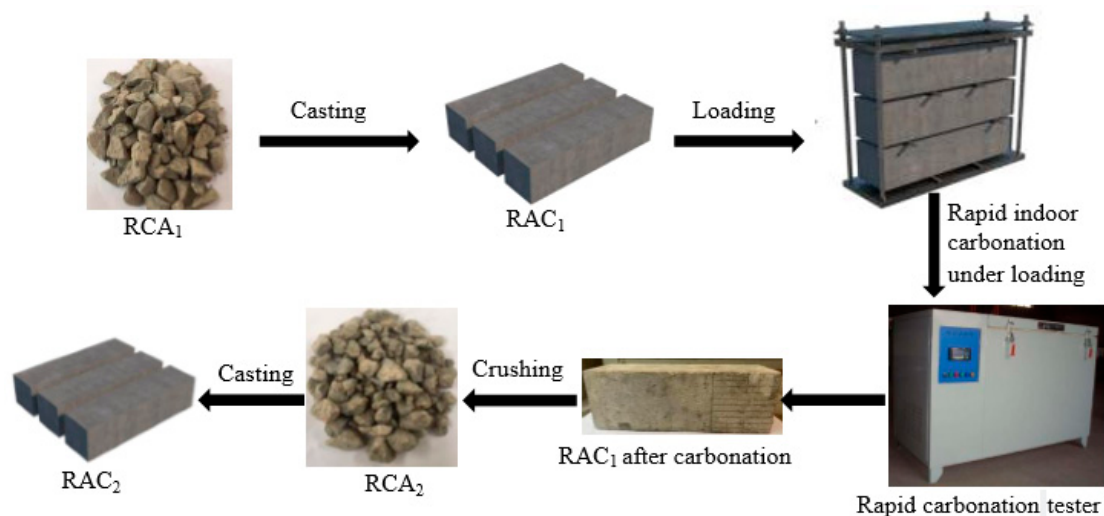
Four cementing materials, Portland cement (P•O 42.5R), slag, FA, and silica fume (SF) were used to prepare the RAC. Their chemical compositions are shown in Table 1.

Table 1. Chemical compositions of cement, slag, FA, and silica fume.

Chemical Composition (%)	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	MnO	K ₂ O	TiO ₂	LOI ¹
Cement	60.79	21.22	7.29	3.78	1.8	1.17	0.63	0.27	1.75
Slag	3.72	51.5	29.33	3.77	1.15	0.21	1.71	0.99	1.69
FA	25.68	31.92	16.80	1.41	7.93	0.51	0.89	0.79	0.69
SF	0.27	87.03	1.12	0.97	0.88	0.14	-	-	0.86
Cement	60.79	21.22	7.29	3.78	1.8	1.17	0.63	0.27	1.75

¹ LOI: Loss on ignition.

The simulated preparation of RCA was based on the equal degree of deterioration of concrete in indoor acceleration and natural environment. The same particle size distribution of natural and recycled aggregate and the same mixture slump was adopted to establish the comparison between different generations of RAC. The first-generation RCA (RCA₁) was provided by Yangzhou Huimin renewable resources co. LTD. and used to prepare the first-generation RAC (RAC₁). After curing for 28 days, the specimens of RAC₁ with 30% ultimate tensile stress were accelerated to carbonize for 17 days, which was equivalent to being carbonized for 50 years in the natural environment [39]. Then RAC₁ was crushed by two-stage crushing ratio technology and sieved to produce the second-generation RCA (RCA₂) with a particle size of 4.75–26.5 mm. The second-generation RAC (RAC₂) was prepared with 100% replacement of RCA₂. The target compressive strength of concrete was 40MPa. The preparation process of RAC₁ and RAC₂ was as shown in Figure 1.

**Figure 1.** Preparation flow diagram of two generations of RAC.

In addition, gravel was adopted as the NCA with a particle size of 4.75–26.5 mm and an apparent density of 2673 kg/m³. River sand was adopted as the natural fine aggregate with an apparent density of 2616 kg/m³ and a fineness modulus of 3.02.

2.2. Mixture Design

RAC with two generations of 100% RCA and NAC as a control group were prepared to investigate the effect of chloride ion corrosion on the carbonation durability. The total volume calculation method [40] was adopted in the proportion design of NAC and RAC, as shown in Table 2. Water consumption for RAC included additional water consumption, which was calculated according to the water absorption rate of RCA for 30 min.

It is well known that the w/c ratio has a great influence on the carbonation resistance of concrete. When the cement content remains unchanged, the internal porosity of concrete increases with the

increase of the w/c ratio, and a larger w/c ratio increases the amount of free water in concrete pores, which is conducive to carbonization reaction. Studies [5,13,22] show that the carbonation depth is not linearly proportional to the w/c ratio, but approximately exponential. Therefore, in this study, a fixed w/c ratio of 0.38 was used to limit the factors affecting carbonization resistance only to chloride attack and different generations of recycled coarse aggregate.

To improve the performance of concrete, polycarboxylic acid (PCA) and polypropylene fiber (PP) were used as an effective water reducer and a reinforcing agent, respectively.

Table 2. Mix proportions of NAC and two generations of RAC (kg/m³).

Concrete Types	Cement	NCA	Sand	RCA	Slag	FA	SF	PCA	PP	Water
NAC	293	1037	689	-	45	90	23	8.1	0.45	171
RAC ₁	293	-	689	1037	45	90	23	8.1	2.25	218
RAC ₂	293	-	689	1037	45	90	23	8.1	2.25	225

2.3. Measurements

The two-stage mixing method proposed by Tam et al. [41] was adopted to mix materials. According to the Chinese standard GB/T 50080-2016 [42], the mixed slump of NAC, RAC₁, and RAC₂ was tested to be 120 mm, 127 mm, and 130 mm, respectively. The mixed slump of three types of concrete was of small difference, which was convenient to establish the comparison between the different generations of RAC. The greater slump of RAC was due to the additional water in the mixing process, which was calculated according to the water absorption rate of RCA for 30 min. The experimental results show that the workability of the concrete with different generations of RCA is good. After curing for 28 days, three specimens with dimensions of 100 mm × 100 mm × 100 mm for each type of concrete were cast to measure the compressive strength. In addition, three specimens for each type of concrete with the same size were prepared for compressive strength test after 28-day carbonization.

In order to analyze the effect of chloride ion corrosion on carbonation resistance, three specimens with dimensions of 100 mm × 100 mm × 400 mm for each type of concrete were soaked in 10% NaCl solution for 30 days, 60 days, and 90 days respectively, and then carbonized for 28 days to test carbonation depth. In order to analyze the impact of carbonation on the compressive strength of specimens corroded by chloride ion, six specimens for each type of concrete, which had been soaked in chloride for 90 days with dimensions of 100 mm × 100 mm × 100 mm, were prepared for compressive strength test before and after 28-day carbonization.

The carbonization test was performed based on Chinese standards GB/T 50082-2009 [43]. In the carbonization process, specimens were placed in an accelerated carbonization test chamber with (20 ± 3)% carbon dioxide concentration, 20 ± 5 °C temperature, and (70 ± 5)% relative humidity.

The large pore porosity and total porosity of concrete can be obtained indirectly through the water loss rate of saturated concrete specimens under specific conditions, that is, the “evaporable water content method” [44]. The large pore porosity obtained by this method is corresponding to the pores with a diameter greater than 30 nm in the specimen [45]. Firstly, three specimens for each type of concrete with dimensions of 100 mm × 100 mm × 100 mm were determined to complete the vacuum saturation. After vacuum saturation, the surface water of the specimens was erased and the mass (M_0) of the specimens was weighed. Then the specimens were placed in a chamber with a relative humidity of 90%. When the water loss of the specimens tended to be flat, it was considered that the water diffusion reached an equilibrium state, and then the mass (M_1) of the specimens was weighed. Finally, the specimens were placed in a chamber at 105 °C and dried to a constant weight. After cooling, the mass (M_2) was weighed. The corresponding volume porosity P of concrete can be calculated by the following formula:

$$P = [(M_0 - M_i)\rho_c] / (M_0\rho_w) \times 100\% \quad (1)$$

where ρ_c is the density of concrete; ρ_w is the density of water; M_i is M_1 or M_2 . According to Formula (1), the large pore porosity P_1 of concrete was calculated by M_0 and M_1 . Similarly, the total porosity P_2 was calculated by M_0 and M_2 .

The physical properties of NCA and RCA were tested according to the Chinese standard GB/T 14685-2010 [46] and GB/T 25177-2010 [47], respectively. According to the difference of the heat attenuation coefficient and expansion coefficient between the aggregate and the attached mortar, the heat treatment method was adopted to remove the attached mortar from the surface of the recycled aggregate [48,49]. Firstly, the reclaimed coarse aggregates with the mass of m_i were weighed, generally about 250 g, and fully soaked in a water container for 2 h to make the water absorption of attached mortar tend to be saturated. Secondly, the aggregates were put into a heating furnace for heating at 500 °C for 2 h, and then immediately put into cold water. This operation was repeated several times to make the attached mortar loose to be easily scraped off with tweezers or knives. Finally, after removing all the attached mortar, the mass m_f of the remaining aggregates was weighed. The attached mortar content ϵ of RCA could be calculated by the following formula:

$$\epsilon = \frac{m_i - m_f}{m_i} \times 100\% \quad (2)$$

It should be noted that all the test data are the mean values obtained from parallel tests of three specimens.

3. Results

3.1. Natural and Recycled Coarse Aggregate

The microscopic morphology of NCA and multiple recycling RCA are displayed in Figure 2, and the physical properties of these aggregates are listed in Table 3. It was observed in Figure 2 that the NCA surface was smooth whereas the RCA one was rough and porous due to the old mortar attached to the surface. Mechanical damage to RCA was more pronounced, crystal particles were disordered, and there were more cracks and pores (Figure 2b,c). Particularly, when RCA had undergone multiple recycling, its properties were seriously degraded because of the increasing attached content.

The attached mortar content of RCA₁ to RCA₂ increased from 38.5% to 42.7%, displaying a rapid growth trend as shown in Figure 3a. A large amount of old mortar gathered on the surface of RCA, which intensified the roughness of the aggregate's surface as reported in previous studies [13,49]. In addition, the repetitive crushing processes caused damage to the aggregate structure and the appearance of a large number of micro-cracks inside and on the surface of RCA, which led to a sharp increase in the water absorption rate of RCA. The water absorption of RCA also exhibited a rapid growth with the increase of attached mortar content (Figure 3b). The average water absorption rate of the multiple recycling RCA was about four times as large as that of NCA. Similarly, the crushing index of RCA gradually increased with the increase of recycling number. There was a linear positive correlation between the crushing index and attached mortar content in Figure 3c. The crushing index of RCA₂ reached 17.1%, indicating that higher attached mortar results in a higher crushing index and lower strength of aggregate. However, the apparent density of RCA was much lower than NCA's, and it showed a rapid downward trend with the increase of attached mortar content (Figure 3d). In other words, the greater the recycling number, the smaller the apparent density of RCA. This is mainly because unlike NCA, which has a dense structure, a regular shape, and a smooth surface, RCA has more attached mortar and its structure is loose, porous, and cracking [50].

The recycling number is the factor to affect the deterioration degree of properties of RCA, but the attached mortar content is the key one, which determines the apparent density, water absorption, crushing index of RCA to a great extent.

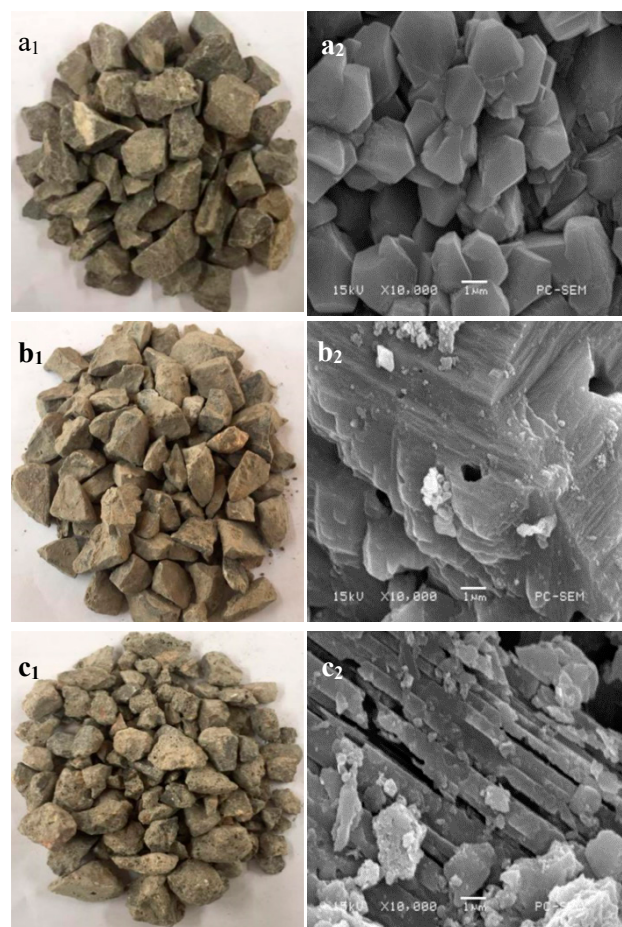


Figure 2. SEM images of coarse aggregate: (a₁) NCA, (a₂) Microstructure of NAC, (b₁) RCA₁, (b₂) Microstructure of RCA₁, (c₁) RCA₂, (c₂) Microstructure of RCA₂.

According to the physical properties indexes in Table 3, it could be concluded that RCA₁ is Class II and Grade M aggregate on the basis of Chinese standard GB/T 25177-2010 [47] and Japanese standard JIS A5023-2006 [51], respectively; RCA₂ is Class III and Grade L aggregate but all two generations of RCA meet the Type II aggregate in the US standard of RILEM [52]. Therefore, even though the quality of RCA prepared in this study decreases with the increase of recycling number, all of them still meet the requirements used for structural concretes.

Table 3. Physical properties of NCA and RCA.

Aggregate Types	Apparent Density (kg/m ³)	Water Absorption Rate for 30 min (%)	Crushing Index (%)	Attached Mortar Content (%)
NCA	2705	0.9	5.6	-
RCA ₁	2545	3.8	16.2	38.5
RCA ₂	2437	4.2	17.1	42.7

3.2. Carbonation Resistance

3.2.1. Carbonation Resistance without Effect of Chloride Ion Corrosion

With the increase of recycling cycles, it has been established that the carbonation resistance of RAC with 100% replacement of RCA decreases gradually. As expected, the same results are obtained in this study. The 28-day carbonation depth of specimens without chloride ion corrosion is shown in Figure 4. The carbonation depth of RAC was greater than that of NAC at the same carbonation time.

The average carbonation rate of NAC, RAC₁, and RAC₂ at 28 days was 0.05 mm/d, 0.07 mm/d, and 0.08 mm/d. Compared to NAC, the carbonation depth of RAC₁ and RAC₂ at 28 days was 1.46 times and 1.78 times. In addition, the carbonization depth of NAC, RAC₁, and RAC₂ increased with the increase of carbonization time, the carbonization depth of these three types of concrete at 28 days was about 2.36 times, 2.04 times, and 1.97 times of that at seven days, respectively.

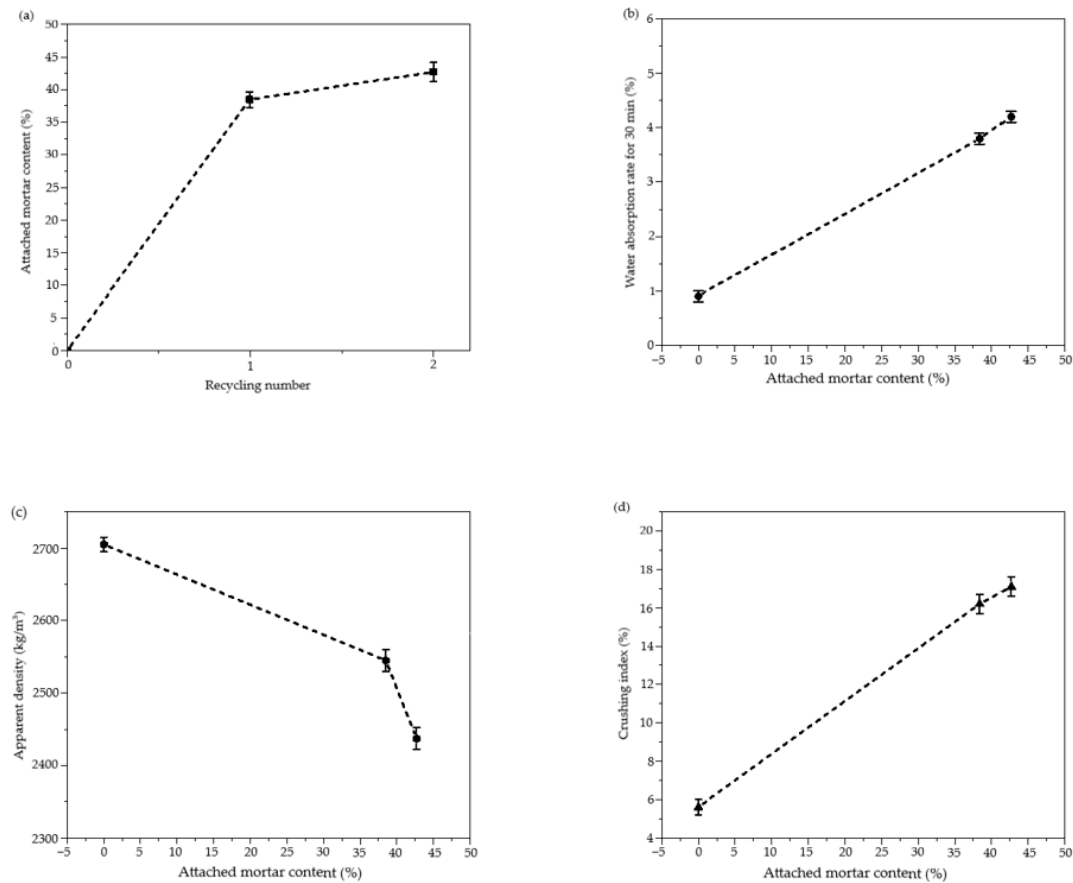


Figure 3. Evolutions of physical properties of coarse aggregate: (a) the change in attached mortar content with recycling number, (b–d) the change in physical properties with attached mortar content.

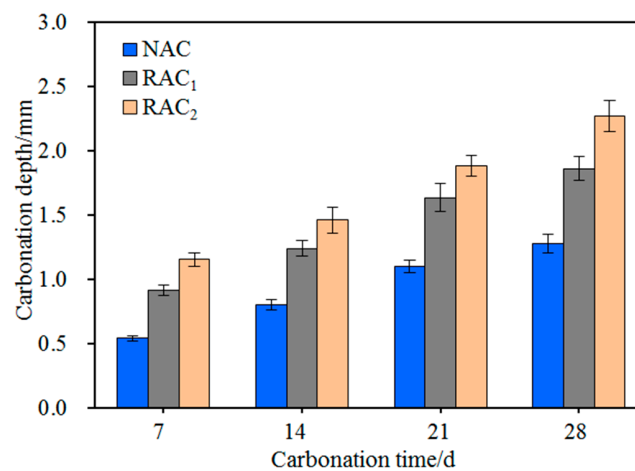


Figure 4. Carbonation depth of concrete at different carbonation time.

The results confirm that the properties of coarse aggregate have a great influence on the carbonation resistance of concrete. The fundamental reason for a reduction in the carbonation resistance of repeatedly RAC is the continuous accumulation of mechanical damage and the increase of attached mortar in the process of cyclic preparation of RCA, which leads to its high porosity and many micro-cracks. These defects of RCA facilitate the penetration of CO_2 into the concrete, resulting in accelerating the carbonation reaction of RAC and increasing the carbonation depth [48,49].

3.2.2. Carbonation Resistance with Effect of Chloride Ion Corrosion

Figure 5 lists the variation in the carbonation depth of RAC after chloride ion corrosion for different days. As listed in Figure 5, the carbonization depth of the specimens soaked in chloride salt was greater than that in the general environment (Figure 4), and the carbonation depth of RAC_2 was greatest at the same carbonation time. The average carbonation rate of RAC_1 at 28 days was 0.11 mm/d, 0.15 mm/d, and 0.17 mm/d when the soaking time was 30 days, 60 days, and 90 days, while the average carbonation rate of RAC_2 was 0.14 mm/d, 0.19 mm/d, and 0.22 mm/d, respectively. After 90 days of soaking, the carbonation rate of RAC_1 and RAC_2 at 28 days increased by 2.5 times and 2.7 times respectively, compared with the one without chloride ion corrosion. The results demonstrated that the negative effect of chloride ion corrosion on the carbonation resistance of concrete was dominant, and the negative effect was more serious as the recycling cycles increased, which was attributed to the properties of RCA. Chloride attack further increases the risk of carbonization corrosion of reinforced concrete structures.

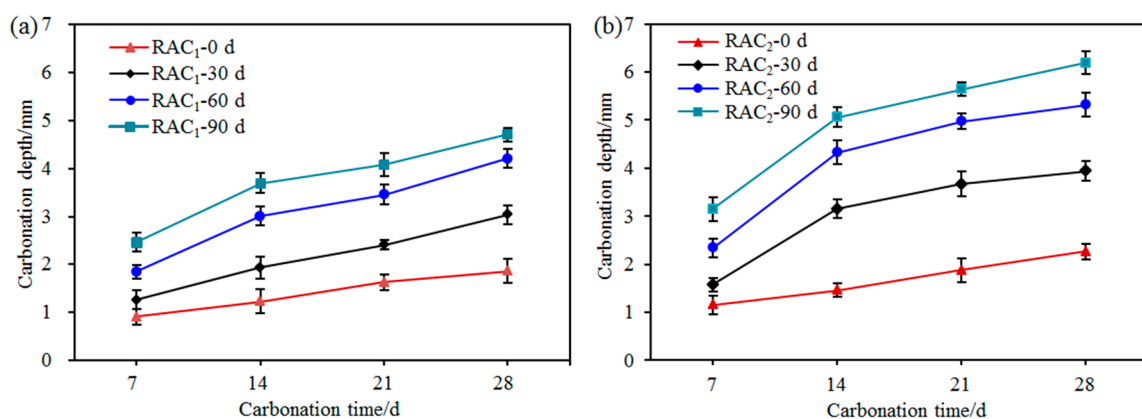


Figure 5. Carbonation depth of RAC with carbonation time after chloride ion corrosion for varying days: (a) carbonation depth of RAC_1 , (b) carbonation depth of RAC_2 .

Figure 6a₁–a₃ shows the microstructure of RAC_2 soaked in chloride for 0 days, 60 days, and 90 days, respectively, and Figure 6b₁–b₃ shows the corresponding microstructure of soaked specimens after carbonization for 28 days. As shown in Figure 6a₁, the RAC_2 surface was smooth with visible cracks whereas the cracks were obviously refined after soaking for 60 days (Figure 6a₂). After soaking for 90 days, many flocculent crystals precipitated on the surface of the structure, including chloride salts, Friedel's salts, and composite salts, which blurred the surface of RCA_2 (Figure 6a₃). It is because of the precipitation of chloride salts on the surface in the form of CaCl_2 crystals, gradually refining the pores and improving the carbonation resistance of RCA_2 to a certain extent. This is consistent with the viewpoints of Ramezani pour [53]. However, the chemical reaction of chloride ions with concrete generated Friedel's salt and compound salt, which consumed the hydroxyl in the mortar, led to partial C-S-H decomposition and the degradation of pore structure, thus reducing its carbonation resistance.

After 28-day carbonization, it was observed in Figure 6b₁ that the structural compactness of the specimens was improved and the surface pores decreased. Many carbonization reaction products were agglomerated and distributed on the surface of the structure (Figure 6b₂,b₃). This is due to the fact that CO_2 permeated into the concrete through the pores and reacted with the hydrate in the concrete to

produce CaCO_3 . Therefore, the RAC_2 surface was distributed with the chemical products generated by chloride ion corrosion and carbonization reaction, and the structure was more loose and fuzzy.

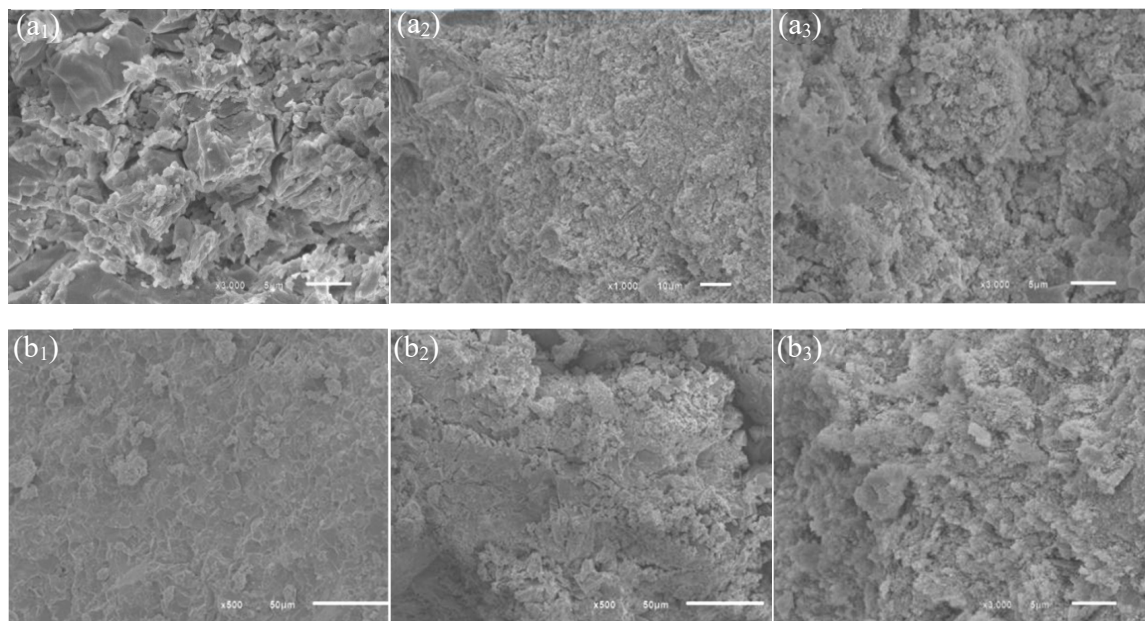


Figure 6. SEM images of RAC_2 . (top) Specimens corroded by chloride ion for varying days in the states of no carbonation: (a₁) 0 d; (a₂) 60 d; (a₃) 90 d. (bottom) Specimens corroded by chloride ion for varying days and 28-day carbonization: (b₁) 0 d; (b₂) 60 d; (b₃) 90 d.

In a word, chloride penetration involves a coupled physical-chemical interaction between environments and dynamic responses of concrete, which will change the internal environment of concrete and thus affect its carbonation resistance. The crystallization of chloride is beneficial to carbonation, but the chemical reaction between chloride and concrete is averse to carbonation. With respect to the RAC, the chloride attack obviously degraded the carbonation resistance.

3.3. Compressive Strength

Compressive strength results for all the series are presented in Figure 7. Figure 7a lists the compressive strength of concrete specimens without exposure to chloride ions. As expected, the compressive strength of RAC before carbonation was less than that of NAC and decreased with the increase of recycling cycles but compressive strength of concrete for each cycle can reach the target strength (40 MPa) at the curing age of 28 days. After 28-day carbonization, the compressive strength of NAC, RAC_1 , and RAC_2 increased slightly by 4.5%, 5%, and 6.5%, respectively. In other words, carbonation increases the compressive strength of concrete. Figure 7b shows the compressive strength of concrete specimens with exposure to chloride ions for 90 days. Before carbonation, the compressive strength of NAC, RAC_1 , and RAC_2 decreased by 5.0%, 6.5%, and 7.0% respectively in comparison with the corresponding one without chloride ion corrosion. This indicated that chloride ion corrosion had a dominant-negative effect on the compressive strength of concrete. After carbonation, the compressive strength of NAC, RAC_1 , and RAC_2 showed little change, only increasing by 2%, 2.7%, and 3.2%, respectively.

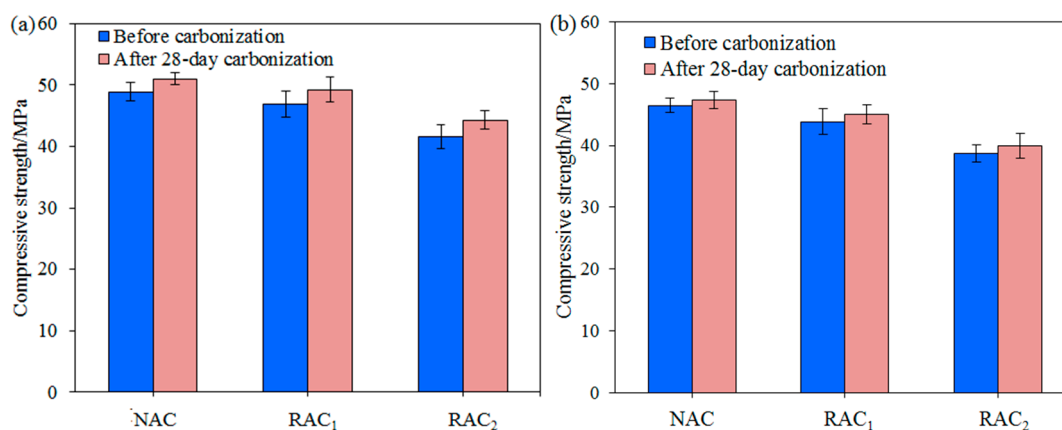


Figure 7. Compressive strength of concrete before and after carbonization: (a) without chloride ion corrosion; (b) chloride ion corrosion for 90 d.

This finding serves as a link between the results of the studies of Abed et al. [54] and Huda and Alam [1]; the former presented an increase in strength of RAC₂ by replacing up to 25% of NCA by RCA₂, whereas the latter presented a decrease in strength by replacing up to 20% of NCA by RCA₂. Abed et al. [54] considered that the increase in the strength of RAC was related to the strong bond between the rough RCA and the new mortar and the shape of the RCA and its rough texture enhanced the aggregate interlock, thus affecting the strength. In this research, the decrease in strength could be related to the stability of the corresponding RCA. As pointed out by Lotfi et al. [55], the presence of attached mortar of RCA weakened the interfacial transition zone of concrete. The attached mortar content of RCA increased with each cycle, and the characteristics of attached mortar, such as low density, large porosity, and low strength, decreased the stability of RAC. The negative effect of chloride ion corrosion on mortar strength was the same, which weakened the interface bond quality of new and old mortar. The findings we obtained from compressive strength with the increasing recycling cycles matched with the results of carbonation resistance, showed the general behavior of RAC.

It was clear that carbonization was beneficial for the compressive strength of concrete, especially RAC. It is because the CaCO₃ produced by the carbonization filled the concrete pores and increased the compactness of the concrete. For RAC, more attached mortar contributed to the production of more CaCO₃, thus it could be seen that the compressive strength increased the most after carbonization in Figure 7 but the strength gain of compressive strength was limited, especially when the specimens were subjected to chloride ion corrosion.

3.4. Concrete Porosity and Permeability

There are three types of pores in concrete, namely, gel pores (<10 nm), capillary pores (10–200 nm), and macro-pores (>200 nm). It is generally accepted that pores with a large pore size are more detrimental to the performance of concrete [56–58]. Wu [45] considered the pores with a pore diameter of 50 nm as harmful.

Figure 8 displays the changes in the porosity of three types of concrete. The total porosity and the large pore (>30 nm) to total pore volume ratio of concrete are shown in Figure 8a. It could be seen that the total porosity and the large pore to total pore volume ratio of concrete increased with the increase of recycling cycles. Compared to NAC, the total porosity of RAC₁ and RAC₂ was 1.28 times and 1.49 times, and the corresponding large pore to total pore volume ratio increased by 5.4% and 11.7%, respectively. This indicated that the compactness of concrete tended to deteriorate with the increase of recycling cycles. It should be noted that the high proportion of large pores is detrimental to the carbonization durability of concrete. There may be more pore solutions in the large pores, which are more convenient for CO₂ to dissolve into the liquid in these pores and carbonize with alkaline substances in the concrete.

Figure 8b shows the total porosity of concrete after chloride ion corrosion for 90 days. As shown in Figure 8b, the total porosity of NAC, RAC₁, and RAC₂ specimens soaked for 90 days increased by 9.6%, 11.2%, and 12.4%, respectively, indicating that the effect of chloride ion corrosion increased the total porosity of concrete. In general, it has been discussed previously that chloride penetration affects the performance of concrete in two ways. The chloride crystals block the internal pores of the concrete to a certain extent and decrease the porosity of concrete. However, chloride ions reacted with alkaline substances in the concrete to produce Friedel's salt and compound salts, which deteriorated the densification of the concrete's internal structure and increased the porosity. Ca(OH)₂ which dissolved in the pore solution was consumed in the reaction with chloride ions and continued to dissolve out from the cement matrix, thereby forming new pores and increasing the porosity of concrete. Most of these new pores were not closed, which was not conducive to the carbonation resistance of concrete.

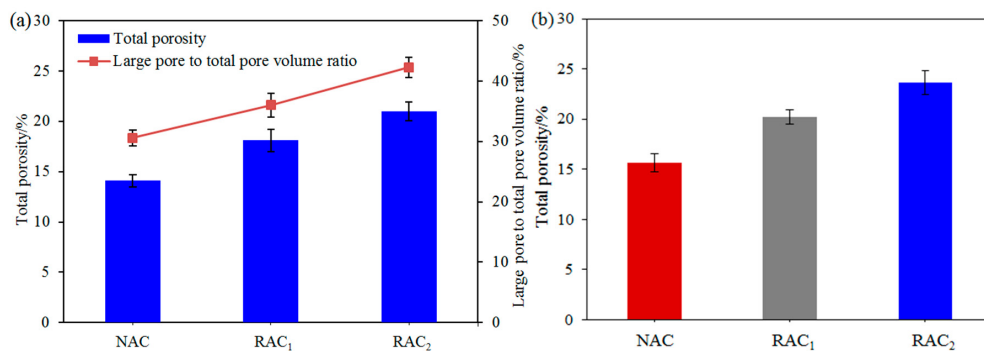


Figure 8. The changes in the porosity of concrete: (a) without chloride ion corrosion; (b) chloride ion corrosion for 90 d.

Many studies have confirmed that the porosity of concrete directly affects the permeability of concrete, further affecting the durability [59,60]. In order to further study the effect of porosity on the carbonation resistance of concrete, the relationship between total porosity and 28-day carbonation depth of three types of concrete is listed in Figure 9. The data in Figure 9 include the carbonation depth without chloride ion corrosion (namely, NAC, RAC₁, and RAC₂) and the one after chloride ion corrosion for 90 days (namely, NAC-90d, RAC₁-90d, and RAC₂-90d). As shown in Figure 9, the increase in the carbonation depth of concrete was closely related to the increase of porosity. The porosity of RAC was larger than that of NAC, and the porosity of concrete increased with the increase of recycling cycles, and the carbonation depth increased accordingly. Furthermore, the porosity of concrete increased after chloride ion corrosion, and the carbonation depth undoubtedly increased. In other words, the utilization of multi-recycled coarse aggregate and the effect of chloride attack increased the porosity of concrete and thus reduced the carbonation resistance.

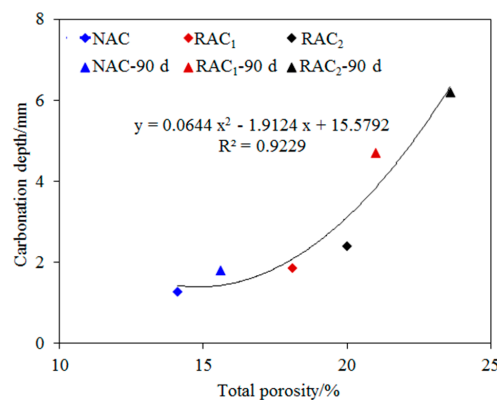


Figure 9. Relationship between total porosity and 28-day carbonation depth.

The following equation can be obtained by fitting the data in Figure 9.

$$y = 0.0644x^2 - 1.9124x + 15.5792 \quad (3)$$

where y is the carbonation depth of concrete, x is the total porosity. The fitting equation shows that the carbonation depth of concrete has a good correlation with the porosity, regardless of whether the concrete has been corroded by chloride ions or not. It is conceivable that the porosity is the main factor affecting the carbonation resistance of concrete and the deterioration of carbonation resistance can be effectively judged by measuring the total porosity of concrete.

In this study, the carbonation resistance of two generations of RAC with 100% RCA was worse than that of the corresponding NAC but compared with recycled concrete or even natural concrete in some other pieces of literature [13,15,22], it showed better carbonation resistance. This may be due to the mix ratio and aggregate source in this study when other experimental conditions are the same. The experiment is helpful to better understand the carbonation durability of multi-recycled coarse aggregate concrete subjected to chloride attack. Based on the Chinese standard for inspection and assessment of concrete durability, JGJ/T193-2009 [61], the carbonation durability of RAC₁ and RAC₂ with the effect of chloride ion corrosion can fully meet the requirements for structural concrete to be used for 50 years. According to the carbonation durability, the application of multi-recycled coarse aggregate in structural concrete under the chloride environment is feasible, only if some measures are taken to improve the concrete density and design a more reasonable concrete cover.

4. Conclusions

This paper presents a study on the carbonation resistance of two generations of 100% RAC with the effect of chloride ion corrosion. The variation trend of physical properties of RCA with the increasing recycling cycles was experimentally studied. Then, the carbonation resistance of three types of concrete, including NAC and two generations of RAC, were analyzed with exposure to chloride ion corrosion. For this purpose, the compressive strength and porosity of concrete before and after chloride attack were also tested. This research draws the following conclusions.

- The recycling number was the factor to affect the deterioration degree of properties of RCA, and the content of the attached mortar was the key one. Although the quality of RCA prepared in this study decreased with the increase of recycling number, all of them still can meet the requirements used for structural concrete.
- The carbonation depth of concrete specimens without chloride ion corrosion increased as the recycling cycles increased, and compared to NAC, the carbonation depth of RAC₁ and RAC₂ at 28 days increased 46% and 78% than that of NAC.
- Chloride ion corrosion negatively affected the carbonation resistance of RAC and the negative effect was more severe with the increase of corrosion time and recycling cycles. Chloride ion corrosion led to the increase of porosity of RAC, which was the fundamental reason for the carbonation resistance deterioration.
- The equation of concrete total porosity and carbonation depth was established, which could effectively judge the deterioration of carbonation resistance of RAC.
- Based on the Chinese standard JGJ/T193-2009, the carbonation durability of RAC₁ and RAC₂ with the effect of chloride ion corrosion can fully meet the requirements for structural concrete to be used for 50 years. According to the carbonation durability, the application of multi-recycled coarse aggregate in structural concrete under the chloride environment is feasible.

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