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Synergistic enhancement of sulfate resistance in recycled aggregate concrete through multi-pathway blocking via carbonation treatment and fly ash incorporation

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ABSTRACT

Due to the large amount of construction waste generated, the use of recycled aggregates from waste concrete as a substitute for natural aggregates in concrete production has become a hot research topic. However, the poor durability, particularly the inadequate sulfate resistance, limits the widespread use of recycled aggregate concrete. This study aims to investigate the synergistic effect of recycled aggregate carbonation and fly ash incorporation on the sulfate resistance of recycled aggregate concrete. In this work, a sulfate wet-dry cycle test was employed to assess the sulfate resistance of recycled aggregate concrete with varying carbonation pressures of the aggregate (0.1, 0.3, and 0.5 MPa) and fly ash contents (10 %, 20 %, and 30 %). Additionally, a novel experimental approach was applied to analyse the sulfate ion transport pathways in recycled aggregate concrete. The results indicate that both recycled aggregate carbonation and fly ash incorporation effectively enhance the sulfate resistance of recycled aggregate concrete. Carbonation treatment improves sulfate resistance in pathways involving old mortar, with an optimal pressure of 0.5 MPa. Fly ash enhances resistance in pathways involving new mortar, with an optimal content of 20 %. These methods show good compatibility. In sulfate wet-dry cycle test, the treated group (0.5 MPa carbonation and 20 % fly ash) exhibited a 146.9 % lower mass loss rate, a 2487 % higher relative compressive strength, and fewer harmful pores, similar to natural aggregate concrete. These findings confirm the effectiveness of multi-path synergistic blocking and provide important theoretical support for the engineering application of recycled aggregate concrete.

1. Introduction

With the rapid economic development and ongoing urbanization, a large quantity of construction materials is being applied to various types of buildings [1–3]. Due to natural disasters causing damage to the functional integrity of buildings or the need for renovation or demolition after the buildings reach the end of their service life, this process generates a substantial amount of construction and demolition waste, which exerts a significant environmental impact [4–10]. To improve the disposal and resource utilization of construction waste, many researchers have proposed the recycling of waste concrete into recycled aggregates, which can replace natural aggregates in the production of recycled aggregate concrete, thus achieving the resource utilization of recycled aggregates [11–16]. However, compared to natural aggregates, recycled aggregates exhibit lower strength and higher water absorption due to their more complex structure, particularly the loose surface and numerous microcracks in the old mortar. These characteristics severely affect the performance of recycled aggregate concrete [17]. Therefore, improving the performance of recycled concrete to ensure it meets the required functional performance has become an important research topic. This also presents a new challenge for concrete industry.

In the northwest and coastal regions of China, soils and groundwater

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contain high concentrations of sulfate, which can cause the corrosion of concrete structures during their service life, leading to a decline in structural performance and resulting in a series of structural safety issues [18–20]. In some regions, concrete structures are located in areas where water levels fluctuate, and the wet-dry cycles can accelerate sulfate corrosion. Under these conditions, the damage to the structure is more severe, significantly affecting its service life [21,22]. Additionally, recycled aggregates, due to their higher water absorption and porosity, create a more complex internal microstructure in recycled concrete produced by replacing natural aggregates. The increased number of transport channels for sulfate ions within the recycled concrete makes it easier to corrosion by harmful ions, thus reducing its durability and severely impacting its service life [23–27].

In recent years, numerous studies have shown that carbonation treatment of recycled aggregates significantly improves the durability of recycled aggregate concrete when used for its production. During the carbonation process, CO_2 reacts with $Ca(OH)_2$ in the old mortar on the surface of the recycled aggregates to form $CaCO_3$. Compared to $Ca(OH)_2$, $CaCO_3$ has a higher hardness and a larger solid volume, which leads to an increase in the density of the recycled aggregates. This process helps repair some microcracks, thereby enhancing the durability and mechanical properties of the recycled aggregate concrete [28–30]. Additionally, carbonation treatment of the recycled aggregate can enhance the interfacial transition zones (ITZs) in recycled aggregate concrete. It has been reported that carbonation treatment reduced the surface microporous content of the ITZ between new and old mortars by 67.2 %, decreased the ITZ thickness from 70 μm to 55 μm, and increased the elastic modulus of this ITZ from 14.14 GPa to 17.64 GPa [31]. Furthermore, carbonation treatment also resulted in a non-homogeneous pore structure distribution and improved the pore structure of recycled aggregate concrete [31]. Wu et al. [32] similarly reported that carbonation treatment increased the modulus and reduced the thickness of the ITZs in recycled aggregate concrete.

For the influence on sulfate resistance, it was reported that carbonation treatment improved the sulfate resistance of recycled aggregate concrete, as the $CaCO_3$ generated during carbonation filled some of the cracks and pores on the aggregate surface, reducing the penetration paths of sulfate ions [33,34]. Zhang et al. [35] explored the use of carbonation and sulfate-aluminate encapsulation for surface treatment of recycled aggregates and tested the sulfate resistance of the resulting recycled aggregate concrete. The study found that this treatment effectively blocked the infiltration of sulfate ions and structural degradation. However, although the sulfate resistance of the recycled aggregate concrete made from carbonated aggregates was improved compared to untreated recycled aggregates, it still lagged behind that of concrete made with natural aggregates [36,37].

The use of partial fly ash as a supplementary cementitious material has also been proven to enhance the durability of concrete [38,39]. Fly ash reacts with $Ca(OH)_2$ in concrete through a pozzolanic reaction, reducing the porosity of the concrete, thereby improving its durability [40–44]. Mei et al. [45] used 10 % and 20 % fly ash to replace cement and tested the mechanical properties and durability of recycled concrete. Their findings indicated that the pozzolanic reaction of fly ash with $Ca(OH)_2$ generated more calcium silicate hydrate gel, which filled the pores and reduced the penetration paths for sulfate ions. Somna et al. [46] replaced 20 %, 35 %, and 50 % of the cement by mass with fly ash to produce recycled aggregate concrete and tested its compressive strength, chloride ion penetration depth, and expansion under sulfate attack. The results showed that the recycled aggregate concrete with fly ash exhibited lower water permeability, restricting the penetration of sulfate ions and thereby enhancing its resistance to sulfate attack. Li et al. [47] tested the durability of recycled aggregate concrete under combined freeze-thaw cycles and sulfate attack by using both low and high dosages of fly ash as a cement replacement. Their experimental results demonstrated that low-dosage fly ash significantly improved the durability of recycled aggregate concrete under the combined effects of freeze-thaw cycles and sulfate attack. Although fly ash can improve the sulfate resistance of recycled aggregate concrete, the inherent defects of recycled aggregates still result in a durability that does not reach the level of natural aggregate concrete [48–50].

Based on the above, it is evident that neither recycled aggregate carbonation nor incorporation of fly ash alone can achieve the performance standards of natural aggregate concrete. Therefore, it is necessary to explore the compatibility between these two methods and investigate whether their simultaneous use can bring the sulfate resistance of recycled aggregate concrete closer to or meeting the standards of natural aggregate concrete. More importantly, compared to natural aggregates, recycled aggregates have a more complex structure which includes different materials and ITZs, leading to more pathways for sulfate transport in recycled aggregate concrete. However, there is limited literature addressing this field. Therefore, it is still unclear how sulfates specifically attack recycled aggregate concrete, and the internal reasons for the improvement in sulfate resistance of recycled aggregate concrete with the aggregate carbonation and fly ash incorporation (i.e., which part of the transport pathways has been specifically improved). This is crucial for exploring the compatibility of the two methods and further enhancing its performance in the future. In consequence, the objective of this study is to explore the effects of recycled aggregate carbonation (with varying carbonation parameters) and fly ash (at different dosages) on the macro and micro parameters of recycled aggregate concrete under sulfate attack, using a sulfate wet-dry cycle experiment. Additionally, this study aims to reveal the compatibility between the two modification methods. Furthermore, a novel approach was adopted to investigate and compare each sulfate transport path in recycled aggregate concrete, exploring the influence of recycled aggregate carbonation and fly ash incorporation on each sulfate attack pathway. This will help elucidate the mechanisms behind the macro tests and provide a theoretical basis for future research.

2. Materials and experiment design

The raw materials for the carbonated recycled aggregate concrete include Portland cement (P·O 42.5 R, in compliance with GB175–2023), fly ash, river sand, natural aggregate, recycled aggregate, and water. The fly ash was procured from Henan Borun Casting Materials Co., Ltd., and its chemical composition is presented in Table 1. The recycled aggregate was sourced from C30 concrete, which was laboratory-prepared using Portland cement, river sand, natural aggregate, and water. The concrete was cured under standard conditions (95 % relative humidity and 20 ± 2°C) for 28 days. The aggregate gradation employed in this study followed a ratio of 3:5:1 for particle sizes 5–10 mm, 10–15 mm, and 15–20 mm, respectively.

The aggregate and river sand (in a saturated surface dry condition) were initially mixed for 30 s, followed by the addition of cement and mixing for another 30 s. Water was then incorporated, and the mixture was blended for 120 s. After 1 day, the cast concrete samples were demoulded and cured under standard conditions (95 % humidity and 20 ± 2°C) for 28 days. A carbonization furnace (Fig. 1) was utilized for the carbonization process of the recycled aggregate. The recycled aggregate was carbonized for 24 h at different pressures (0.1, 0.3, and 0.5 MPa). The carbonation time of 24 h and carbonation pressures up to 0.5 MPa were selected in this study based on preliminary testing. The results indicate that although longer carbonation times and higher pressures continuously enhanced the carbonation degree, the rate of improvement significantly decreased when the carbonation time exceeded 24 h or the pressure exceeded 0.5 MPa. Therefore, these two

Table 1
The chemical component of fly ash.

Name	SiO_2	Al_2O_3	CaO	SO_3	Fe_2O_3	Cl^-
Content (%)	45.1	24.2	4.5	1.2	0.85	0.015

Fig. 1. High pressure carbonation furnace.

parameters were chosen for the present study.

A total of 9 groups (comprising 297 samples) were investigated, with varying substitution rates of recycled aggregate (0 % and 100 %), carbonation pressures of recycled aggregate (0.1, 0.3, and 0.5 MPa), and fly ash content (10 %, 20 %, and 30 %), as shown in Table 2. For clarification, NAC refers to natural aggregate concrete, RAC refers to recycled aggregate concrete, CRAC-0.3 refers to carbonated recycled aggregate concrete with a carbonation pressure of 0.3 MPa, FRAC-20 % refers to recycled aggregate concrete with 20 % fly ash, and FCRAC-20 % refers to carbonated recycled aggregate concrete with 20 % fly ash. It is important to note that the carbonation pressure was maintained at 0.5 MPa for all FCRAC groups.

3. Experimental techniques

3.1. Aggregate properties

Apparent density is defined as the ratio of the aggregate's mass to its apparent volume, where the apparent volume is the sum of the solid volume and the volume of closed pores. Water absorption refers to the change in mass of the aggregate from an absolutely dry condition to a saturated surface dry condition. The crushing index represents the aggregate's ability to resist crushing, serving as an indirect indicator of its corresponding strength. All of these properties were tested in accordance with the standards outlined in JGJ 52–2006. Additionally, a ZEISS Sigma 360 Scanning Electron Microscope (SEM) was employed to visually examine the microstructure of the aggregates both before and after the carbonation treatment.

3.2. Compressive strength

The compressive strength test was conducted according to GB/T 50081–2019, using non-standard cubic specimens with a side length of 100 mm. Three specimens were tested as a group, and the average value was taken and multiplied by the conversion factor of 0.95. The testing instrument used was the DYE-2000B compression machine.

3.3. Sulfate wet-dry cycle test

The concrete sulfate wet-dry cycle test was conducted using the HC-LSB fully automatic concrete sulfate wet-dry cycle testing machine (manufactured by Hangzhou Guanli Intelligent Technology Co., Ltd., as shown in Fig. 2), in accordance with the GB/T 50082–2004 standard. The test utilized 100 mm × 100 mm × 100 mm cubes and a 5 % Na_2SO_4 solution. Given the relatively low durability of recycled aggregate concrete, the wet-dry cycle count was stopped at 120, with cycles at 15, 30, 60, 90, and 120 cycles. Each wet-dry cycle was continuous, and the procedure for the cycle is outlined in Fig. 3, with each cycle lasting 24 h. In accordance with the standard, the distance between specimens and the sidewalls, as well as between individual specimens, was maintained at greater than 20 mm.

3.3.1. Mass loss rate

The mass loss rate refers to the percentage of weight reduction in the sample after n wet-dry cycles, indicating the extent of concrete degradation due to sulfate attack. Eq. (1) shows the calculation method, where w is mass loss rate, w_0 is the initial weight of the specimens, w_t is the weight of the specimens after n wet-dry cycles. The weight here refers to the weight of the specimens after drying at 80°C for 48 hrs.

$$\omega = \frac{w_0 - w_t}{w_0} \times 100\% \tag{1}$$

3.3.2. Relative compressive strength

Relative compressive strength refers to the ratio of the specimen's strength after n wet-dry cycles to its initial strength. Eq. (2) shows the calculation method, where K_f is the relative compressive strength, f_{cn} is the compressive strength after n wet-dry cycles, f_{c0} is the initial compressive strength. The compressive strength test was based on GB/T

Fig. 2. HC-LSB fully automatic concrete sulfate wet-dry cycle testing machine.

Table 2
Mix design (unit: kg/m^3).

No.	Cement	Fly ash	River sand	Nature aggregate	Recycled aggregate	Carbonated recycled aggregate	Water
NAC	430	0	562	1043	0	0	215
RAC	430	0	562	0	1043	0	215
CRAC−0.1	430	0	562	0	0	1043	215
CRAC−0.3	430	0	562	0	0	1043	215
CRAC−0.5	430	0	562	0	0	1043	215
FRAC−20 %	344	86	562	0	1043	0	215
FCRAC−10 %	387	43	562	0	0	1043	215
FCRAC−20 %	344	86	562	0	0	1043	215
FCRAC−30 %	301	129	562	0	0	1043	215

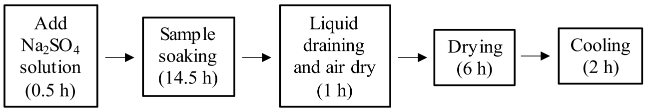

Fig. 3. Procedure of the concrete sulfate wet-dry cycle.

50081–2002. For each group, three 100 mm × 100 mm × 100 mm cubes were tested by the universal testing machine. The final result was the average outcomes of these three samples multiply by 0.95 (volume conversion factor).

$$K_f = \frac{f_{cn}}{f_{c0}} \times 100\% \quad (2)$$

3.3.3. Sulfate ion concentration

Barium sulfate weight method (based on GB/T 176–2017) was used to investigate the sulfate ion concentration at different depths (0–4 mm, 4–8 mm, 8–12 mm, 12–16 mm, 16–20 mm, and 20–24 mm) of the sample. The principle of this method is to use barium chloride to replace sulfate ions in the solution, forming barium sulfate precipitate. The general procedure is shown in Fig. 4. Eq. (3) shows the calculation method, where w_{SO4} is the sulfate ion concentration, m_0 is the dry weight of the sample, m_1 is the dry weight of $BaSO_4$ precipitate, 0.412 is the conversion ratio from $BaSO_4$ to SO_4^{-}.

$$\omega_{SO_4} = \frac{m_1 \times 0.412}{m_0} \times 100\% \quad (3)$$

The sample preparation method is illustrated in Fig. 5. First, a 50 mm × 50 mm × 50 mm cube was cut from the sample. Next, the cubic specimen was sliced into 4 mm thick sections, progressively cut from the outer surface to the interior. Finally, the slices were cut in a gradient manner, based on the desired analysis depth, ensuring that the distances from the three faces most susceptible to erosion to the concrete surface were uniform.

3.3.4. Microscopic properties

Thermogravimetric Analysis (TGA): Netzsch STA449F3 was used for TGA to investigate the compositional changes of the sample before and after different wet-dry cycle counts. The heating rate was 15 °C/min, and the temperature range was 20–800°C. The sample size was 50 mm× 50 mm× 8 mm, taken from the surface center of the 100 mm× 100 mm× 100 mm cubic specimen.

Mercury Intrusion Porosimetry (MIP): AutoPore IV9510 MIP was used to analysed the pore structure change of the sample during wet-dry cycles. The instrument parameters are as follows: pressure range of 0.2–33000 psi, pore range of 800 μm to 5 nm, and a test environment temperature of 23°C. The sample size was 50 mm× 50 mm× 50 mm, taken from the surface center of the 100 mm× 100 mm× 100 mm cubic specimen.

3.4. Sulfate ion transport experiment

To investigate and compare the sulfate ion transport through different pathways, including new mortar (NM), old mortar (OM), the interfacial transition zone (ITZ) between new and old mortar (NM-OM), the ITZ between old mortar and old aggregate (OM-A), and the ITZ between old aggregate and new mortar (A-NM), a novel analytical method was employed in this study. The experiment was adapted from

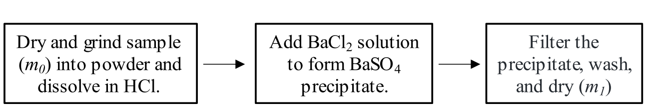

Fig. 4. The procedure of barium sulfate weight method to analyse sulfate ion concentration.

the capillary water absorption test, where pure water was replaced with a 5 % Na_2SO_4 solution. The underlying principle was to leverage the capillary water absorption properties to facilitate sulfate ion transport, which subsequently undergoes chemical reactions leading to material erosion. The erosion process was tracked by monitoring the concentration of sulfate ions. The schematic and actual images of the specimens are shown in Fig. 6. In the sample preparation procedure, mortar (OM) was firstly casted on the left side of the granite aggregate (A). After 28 days of standard curing, specimens intended for carbonation treatment were placed in the high pressure carbonation furnace under a pressure of 0.5 MPa for 24 h. Following carbonation, new mortar (NM) was casted on both sides of the specimen (the control group specimens were not subjected to carbonation treatment prior to casting). Finally, the specimens were cured under standard condition for 28 days before testing.

As shown in Fig. 7, the specimens were positioned on a mesh framework, with the bottom immersed in a 5 % Na2SO4 solution to a depth of 10 mm. Throughout the experiment, the solution was continuously replenished to maintain the specimens in this immersed state.

This experiment primarily aimed to determine the path of sulfate attack based on the concentration of sulfate ions. Higher concentrations of sulfate ions increase the likelihood of reactions that form gypsum and ettringite, leading to greater expansion and more severe damage to the concrete. As shown in Fig. 8, the samples were vertically cut and taken out five thin specimens, which were taken from the NM part, OM part, the interface between NM and OM (i.e., NM-OM), the interface between OM and A (i.e., OM-A), and the interface between A and NM (i.e., A-NM). Each research specimen was divided into four sections for testing, with analysis conducted from the top to the bottom of the specimen.

The innovation of this experiment lies in the fact that each sample contains all the transport pathways of the recycled aggregate concrete mentioned above. Therefore, during the experiment, the external experimental conditions of each transport channel were completely consistent, ensuring the comparability of the test results. In addition, this experiment employed the capillary pore adsorption transport method (conformed to reality), providing an equal track for each transport pathway. This not only made the experimental results more intuitive but also offered an innovative reference for other durability studies of recycled aggregate concrete (such as chloride ion erosion).

4. Results

4.1. Aggregate properties

Table 3 presents the properties of natural aggregate, recycled aggregate, and carbonated recycled aggregate. Compared to natural aggregate, recycled aggregate exhibited slightly lower density, significantly higher water absorption, and a higher crushing index. Moreover, carbonation treatment of recycled aggregate resulted in an increase in density (up to 1.3 %), a decrease in water absorption (up to 17.7 %), and a reduction in the crushing index (up to 18 %). Notably, a carbonation pressure of 0.5 MPa was identified as optimal in this study. However, it is important to emphasize that despite the improvements in the properties of recycled aggregate through carbonation treatment, natural aggregate still demonstrated superior overall properties.

Fig. 9 presents the SEM results of the recycled aggregates with and without carbonation treatment (RA and CRA-0.5). The RA exhibited relatively wide cracks and a loose structure, which were attributed to the mechanical extrusion and impact forces during the preparation of recycled aggregates. In contrast, the CRA-0.5 demonstrated significantly narrower cracks, with a substantial amount of carbonation products adhering around these cracks, effectively filling the gaps. Additionally, numerous granular particles are observed to agglomerate on the surface of the aggregates, filling the pores and contributing to a denser overall structure compared to the non-carbonated aggregates.

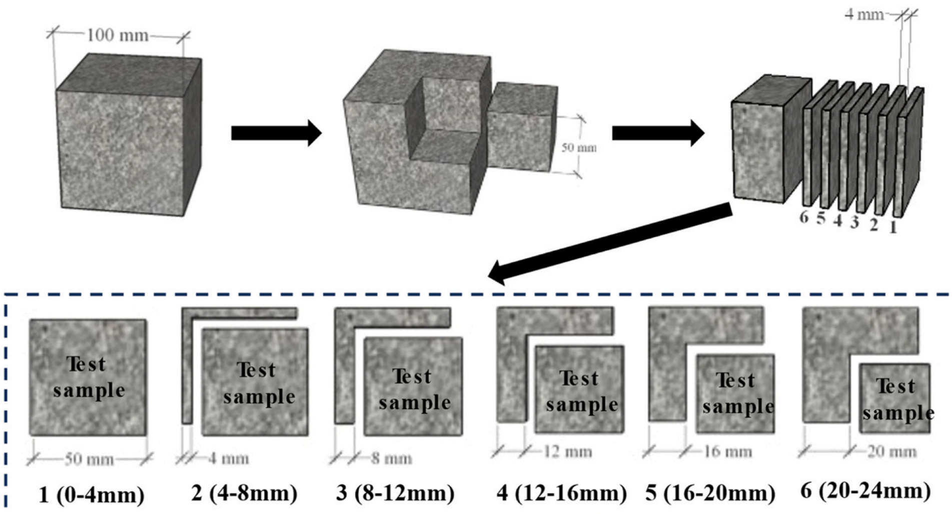

Fig. 5. Sample preparation method for sulfate ion concentration test.

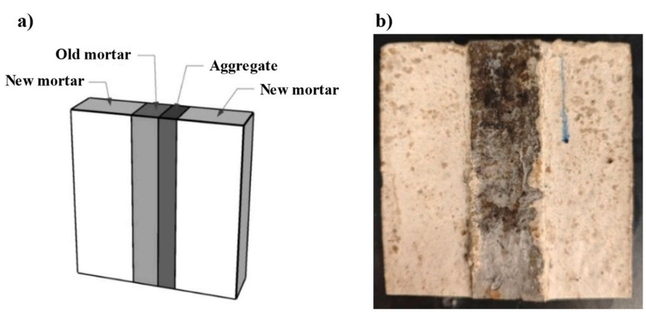

Fig. 6. The structure of the specimen: a) schematic image; and b) actual image.

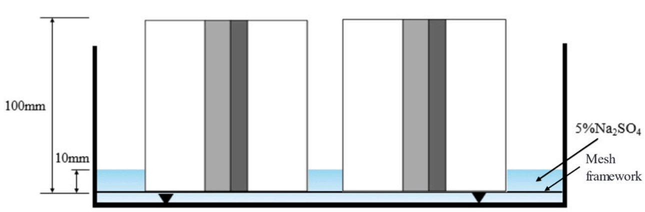

Fig. 7. Schematic of the sulfate ion transport experiment.

4.2. *Compressive strength*

Table 4 presents the compressive strengths of the samples at both 28-day and 148-day standard curing ages. It is evident that NAC exhibited the highest compressive strength. In comparison to RAC, the FRAC-20 % sample demonstrated a reduction in compressive strength by 13.1 % and 3.4 % at the 28-day and 148-day curing ages, respectively. Moreover, with the exception of NAC, CRAC-0.5 exhibited the highest compressive strength at 148 days, which was 19.3 % greater than that of RAC.

4.3. *Sulfate wet-dry cycle test*

4.3.1. *Mass loss rate*

As previously discussed, during the sulfate erosion process, the formation of ettringite crystals can lead to expansion, cracking, and spalling of concrete, ultimately resulting in the failure of the concrete structure. Fig. 10 illustrates the surface morphology of the samples after 120 wet-dry cycles. The surface of NAC appears the most intact, with almost no visible cracks, indicating the best resistance to sulfate erosion. In contrast, the worst surface morphology is observed in the recycled aggregate concrete samples without carbonation treatment (i.e., RAC and FRAC-20 %), which exhibit numerous cracks and even significant spalling. Furthermore, both the carbonation treatment of recycled aggregate and the incorporation of fly ash contribute to a reduction in surface cracks. The optimal carbonation pressure and fly ash content were found to be 0.5 MPa and 20 %, respectively.

During the sulfate erosion process, spalling contributed to the mass loss of the samples. Table 5 shows the Mass loss rate values of the samples in sulfate wet-dry cycle test, while Fig. 11 and Fig. 12 were based on the results in Table 5 for better analysis. Fig. 11 presents the mass loss rates of the samples, considering the effect of carbonation treatment on recycled aggregates. Initially, all samples experienced an increase in mass during the first 15–30 cycles, followed by a gradual decrease in mass as the number of wet-dry cycles increased. The NAC exhibited almost no mass loss, whereas the RAC showed the largest mass change. Notably, the carbonation treatment of the recycled aggregate (i.e., CRAC-0.1, CRAC-0.3, and CRAC-0.5) reduced the mass loss rate, with the optimal carbonation pressure being 0.5 MPa. After 120 wet-dry cycles, the mass loss rate of CRAC-0.5 was reduced by 76.6 % compared to RAC. Fig. 12 illustrates the mass loss rates of the samples considering the influence of fly ash. The combined use of fly ash and carbonation treatment of the recycled aggregate significantly reduced the mass loss. The optimal fly ash content was found to be 20 %. Compared to RAC, the mass loss rate of FCRAC-20 % after 120 wet-dry cycles decreased from 9.73 % to −4.56 %, by 146.9 %.

4.3.2. *Relative compressive strength*

Fig. 13 illustrates the relative compressive strength of the samples, considering the influence of carbonation treatment on recycled aggregate. A lower value indicates a higher strength loss. It is evident that the relative compressive strength of all samples decreased as the number of wet-dry cycles increased. Notably, the NAC exhibited the lowest relative compressive strength, while the RAC demonstrated the highest relative strength loss. Additionally, carbonation treatment of recycled aggregate improved the ability to resist strength loss under sulfate ion erosion, with the optimal carbonation pressure identified as 0.5 MPa. After 120 wet-dry cycles, the relative compressive strength of CRAC-0.5 increased by 1504 % compared to RAC. Fig. 14 depicts the relative compressive strength of the samples considering the influence of fly ash. The combination of fly ash and carbonation treatment of the recycled aggregate significantly enhanced the relative compressive strength of the samples, even approaching that of the natural aggregate concrete samples. The optimal fly ash content was determined to be 20 %. Compared to RAC,

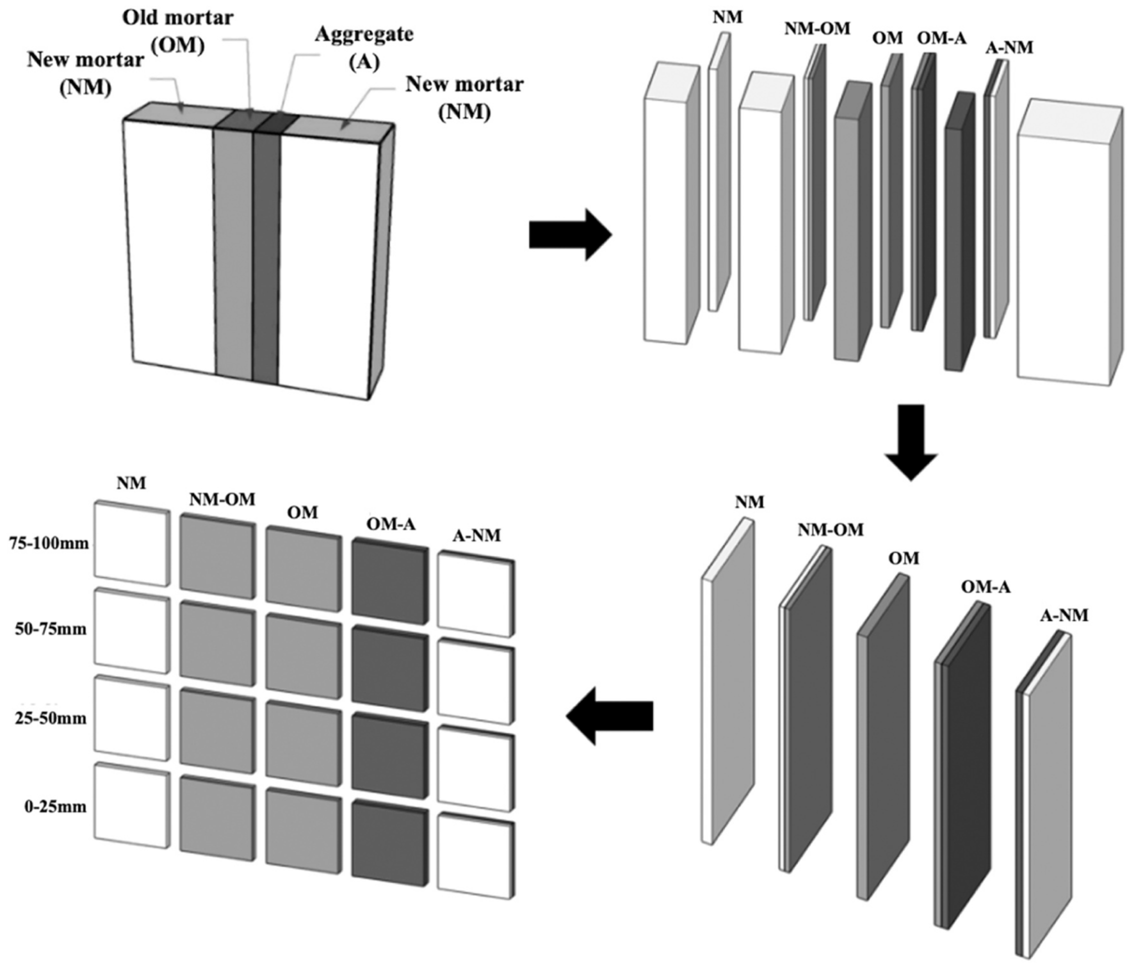

Fig. 8. Schematic of sample preparation for sulfate ion concentration analysis.

Table 3
Apparent density, water absorption, and crushing index of the aggregate.

Name	Apparent density (kg/m^3)	SD	Water absorption (%)	SD	Crushing index (%)	SD
NA	2719	5.68	0.43 %	0.08 %	11.67 %	0.21 %
RA	2669	4.33	6.90 %	0.07 %	17.85 %	0.21 %
CRA−0.1	2687	5.23	6.21 %	0.07 %	16.30 %	0.22 %
CRA−0.3	2697	1.99	6.02 %	0.08 %	15.37 %	0.13 %
CRA−0.5	2703	2.87	5.69 %	0.04 %	14.63 %	0.17 %

Note: NA is natural aggregate, RA is recycled aggregate, CRA-0.3 is the carbonated recycled aggregate under 0.3 MPa carbonation pressure.

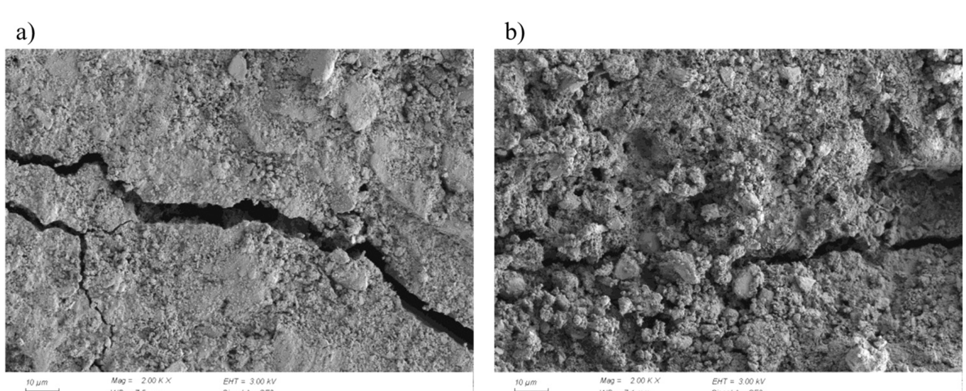

Fig. 9. SEM analysis of a) RA; and b) CRA-0.5.

Table 4
Compressive strength of the samples under different standard curing ages.

Group No.	28-day (MPa)	SD	148-day (MPa)	SD
NAC	36.9	1.02	49.71	2.3
RAC	32.87	0.73	41.72	0.47
FRAC−20 %	28.57	0.31	40.31	0.79
CRAC−0.1	34.6	0.61	43.04	1.76
CRAC−0.3	34.85	0.43	44.7	1.86
CRAC−0.5	31.88	0.57	47.5	1.3
FCRAC−10 %	34.86	0.57	43.49	0.62
FCRAC−20 %	30.94	0.31	43.13	0.38
FCRAC−30 %	29.93	0.22	41.34	0.94

the relative compressive strength of FCRAC-20 % after 120 wet-dry cycles increased by 2487 %. Please note that the raw data for the compressive strength of each sample group under different wet-dry cycles and curing ages is provided in the supplementary materials.

4.3.3. *Sulfate ion concentration*

Due to significant expansion and cracking of the samples after 60 wet-dry cycles, which hindered slicing, this section focuses on the samples after 30 wet-dry cycles. Fig. 15 illustrates the sulfate ion concentration at varying depths of the samples, considering the influence of carbonation treatment of recycled aggregate. It is evident that sulfate ion concentration decreased with increasing sample depth. Furthermore, NAC exhibited the lowest sulfate ion concentrations, while RAC displayed the highest concentrations. Additionally, carbonation treatment of recycled aggregate resulted in a reduction in sulfate ion concentration across the samples, with the optimal carbonation pressure determined to be 0.5 MPa. After 30 wet-dry cycles at a 24 mm depth, the sulfate ion concentration of CRAC-0.5 was reduced by 30.6 % compared to RAC. Fig. 16 presents the sulfate ion concentration at varying depths of the samples, considering the influence of fly ash. The combined use of fly ash and carbonation treatment of recycled aggregate (FCRAC-20 %) significantly reduced the sulfate ion concentration, approaching values observed in natural aggregate concrete samples (NAC). The optimal fly ash content was found to be 20 %. After 30 wet-dry cycles at a 24 mm depth, the sulfate ion concentration of FCRAC-20 % decreased by 97.3 % compared to RAC.

4.3.4. *Microscopic properties*

Fig. 17 presents the results of TGA. In the TGA, the mass loss observed between 30 and 200°C was primarily due to the dehydration of $CaSO_4$ (110–160°C) and AFt (85–120°C). The mass loss observed between 400 and 500°C was mainly attributed to the decomposition of $Ca(OH)_2$. The temperature range for the decomposition of $CaCO_3$, which releases CO_2, was found to be between 550°C and 800°C. The main products of sulfate corrosion in concrete were $CaSO_4$ and Aft, and thus

Fig. 10. Surface morphology of the samples after 120 wet-dry cycles.

Table 5
Mass loss rate values of the samples in sulfate wet-dry cycle test.

Wet-dry cycles	15	30	60	90	120
NAC	−1.78 %	−2.92 %	−2.78 %	−2.71 %	−3.31 %
SD	0.12 %	0.04 %	0.12 %	0.05 %	0.03 %
RAC	−3.46 %	−5.82 %	−3.42 %	2.61 %	9.73 %
SD	0.13 %	0.11 %	0.10 %	0.52 %	0.56 %
CRAC−0.1	−1.72 %	−2.33 %	−0.40 %	1.66 %	6.54 %
SD	0.02 %	0.49 %	0.11 %	0.61 %	0.35 %
CRAC−0.3	−1.26 %	−1.12 %	−0.29 %	1.46 %	4.55 %
SD	0.12 %	0.14 %	0.08 %	0.38 %	0.42 %
CRAC−0.5	−1.19 %	−1.66 %	0.79 %	1.23 %	2.27 %
SD	0.25 %	0.11 %	0.39 %	0.34 %	0.35 %
FRAC−20 %	−2.63 %	−2.81 %	−1.33 %	3.28 %	7.48 %
SD	0.18 %	0.06 %	0.29 %	0.27 %	0.61 %
FCRAC−10 %	−1.54 %	−2.65 %	−2.96 %	−5.23 %	−3.03 %
SD	0.14 %	0.18 %	0.33 %	0.41 %	0.42 %
FCRAC−20 %	−2.21 %	−2.24 %	−2.86 %	−3.86 %	−4.56 %
SD	0.16 %	0.16 %	0.34 %	0.28 %	0.30 %
FCRAC−30 %	−2.62 %	−2.42 %	−1.92 %	−0.41 %	−2.16 %
SD	0.13 %	0.07 %	0.20 %	0.31 %	0.21 %

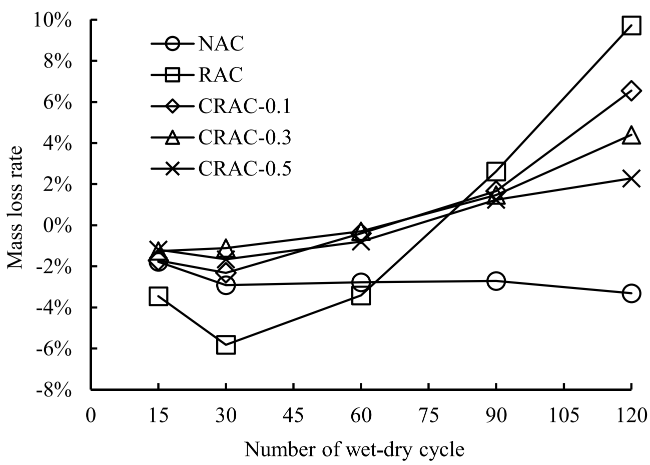

Fig. 11. Influence of recycled aggregate carbonation pressure on mass loss rate in sulfate wet-dry cycle test.

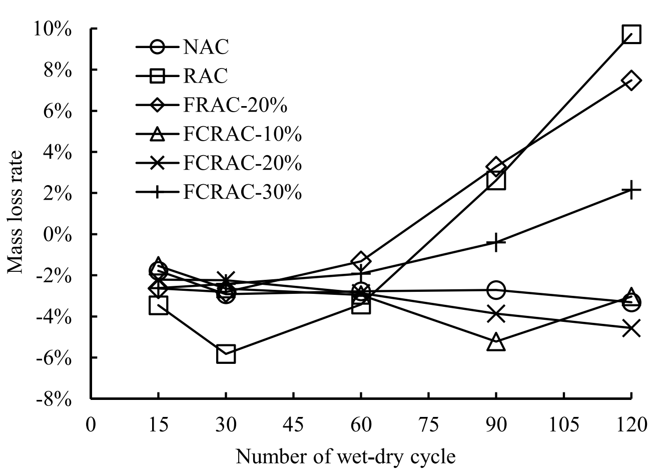

Fig. 12. Influence of fly ash on mass loss rate in sulfate wet-dry cycle test.

these two compounds were the focus of the TGA study in this paper. To achieve more accurate quantification of $CaSO_4$ and AFt formation, the mass loss contributions from other hydrated components at their respective characteristic temperatures were subtracted. This was done by using the mass loss of non-eroded samples within the same

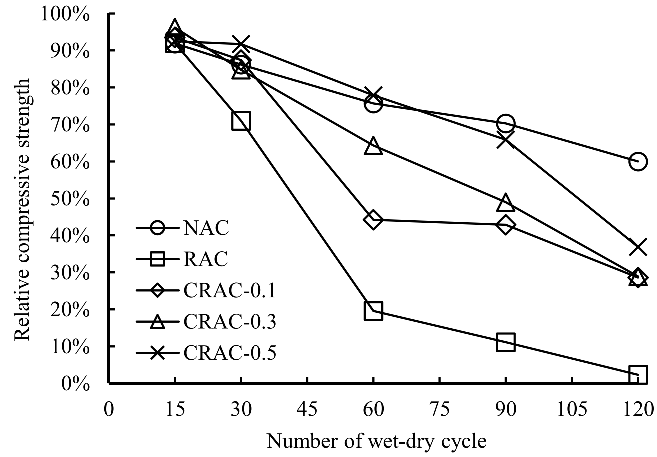

Fig. 13. Influence of recycled aggregate carbonation pressure on relative compressive strength in sulfate wet-dry cycle test.

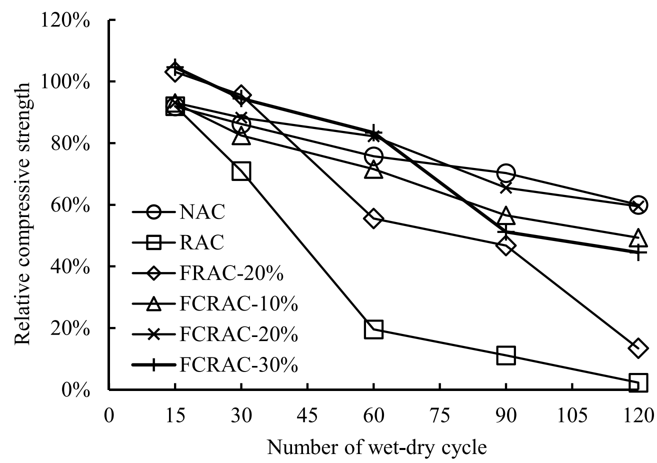

Fig. 14. Influence of fly ash on relative compressive strength in sulfate wet-dry cycle test.

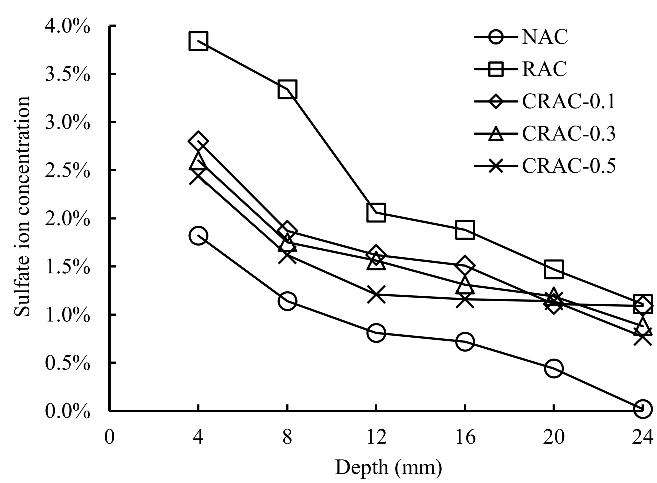

Fig. 15. Influence of recycled aggregate carbonation pressure on sulfate ion concentration in sulfate wet-dry cycle test (30 cycles).

temperature range as a reference baseline. Based on the results, the percentage of $CaSO_4$ and AFt follows the order: RAC > FRAC-20 % > CRAC-0.5 > FCRAC-20 %.

Fig. 18 and Fig. 19 present the MIP results for the samples subjected

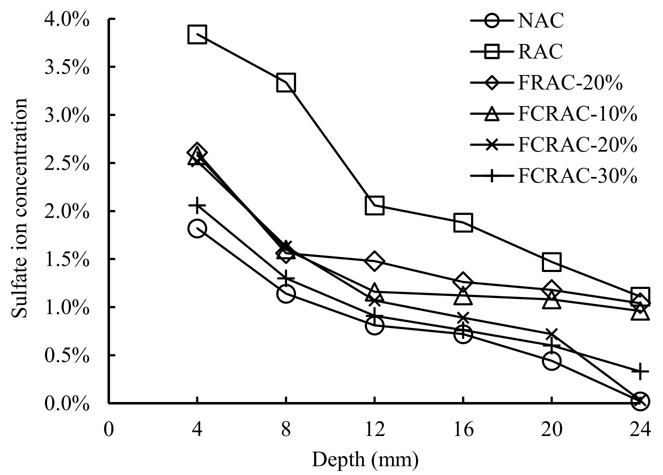

Fig. 16. Influence of fly ash on sulfate ion concentration in sulfate wet-dry cycle test (30 cycles).

to 0 and 30 wet-dry cycles, respectively. Based on the influence of pore size on concrete, pores can be categorized into four groups: harmless pores (d < 20 nm), slightly harmful pores (20 nm ≤ d ≤ 50 nm), harmful pores (50 nm ≤ d ≤ 200 nm), and highly harmful pores (d > 200 nm). The presence of more harmful and highly harmful pores indicates a more porous and less dense concrete structure. As shown in Fig. 18, the volume of harmful and highly harmful pores in the non-eroded samples follows the order: RAC > FRAC-20 % > CRAC-0.5 > FCRAC-20 %. Fig. 19 b) illustrates that the sulfate wet-dry cycles caused the pore structure of the concrete to become more complex, with each group displaying more than one peak, typically 2–3 peaks per group. Additionally, the sulfate wet-dry cycles increased the pore volume across all groups, as shown in Fig. 19 a). The volume of harmful and highly harmful pores in the eroded samples (after 30 wet-dry cycles) also follows the same order: RAC > FRAC-20 % > CRAC-0.5 > FCRAC-20 %.

4.4. Sulfate ion transport experiment

Fig. 20 and Fig. 21 illustrate the sulfate ion concentration in samples at different depths (0–25 mm, 25–50 mm, 50–75 mm, and 75–100 mm) after 15-day and 30-day testing periods, respectively. It is evident that the sulfate ion concentration increased with the testing duration (from 15 to 30 days), with similar trends observed between the 15-day and 30-day results. The RAC exhibited the highest sulfate ion concentration, while the FCRAC-20 % showed the lowest concentration. Additionally, among all groups, the OM region consistently displayed the lowest sulfate ion concentration. For RAC, the A-NM region had the highest sulfate ion concentration, followed by the NM-OM and NM regions. In comparison to RAC, the CRAC-0.5 showed particularly lower sulfate ion concentrations in the NM-OM, OM, and OM-A regions, although it still

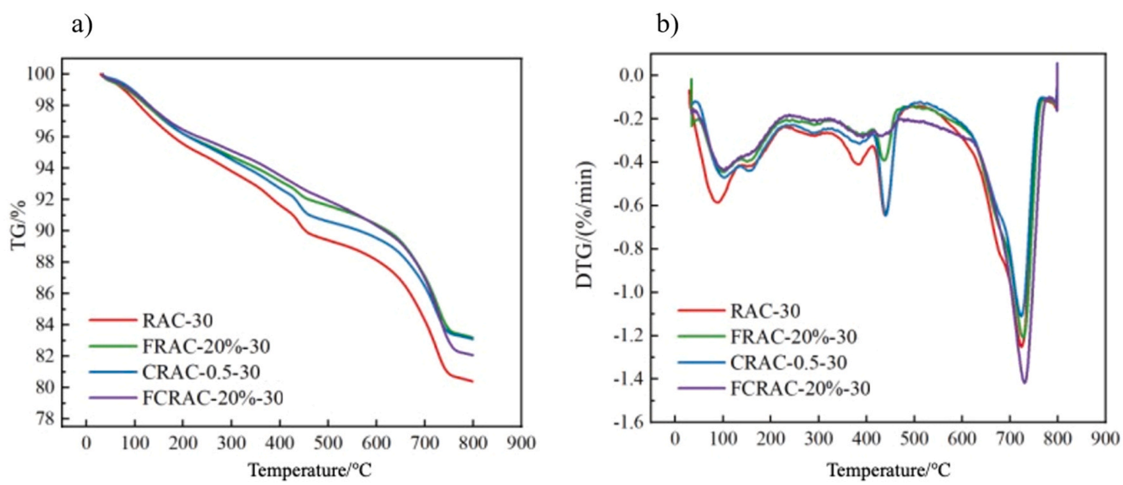

Fig. 17. Results of the a) TG; and b) DTG for samples under 30 wet-dry cycles.

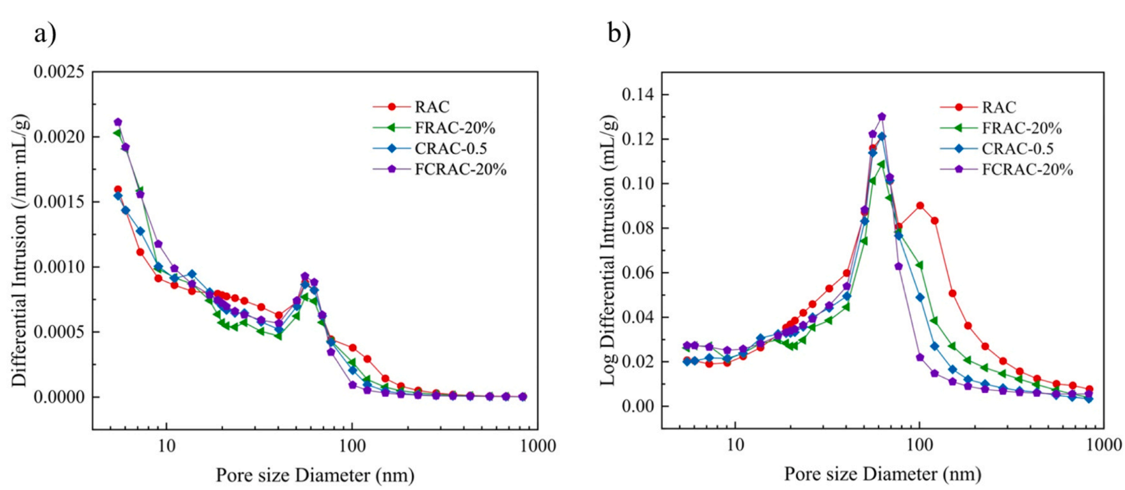

Fig. 18. MIP results for the samples under 0 wet-dry cycle (28-day curing): a) integral curve; and b) differential curve.

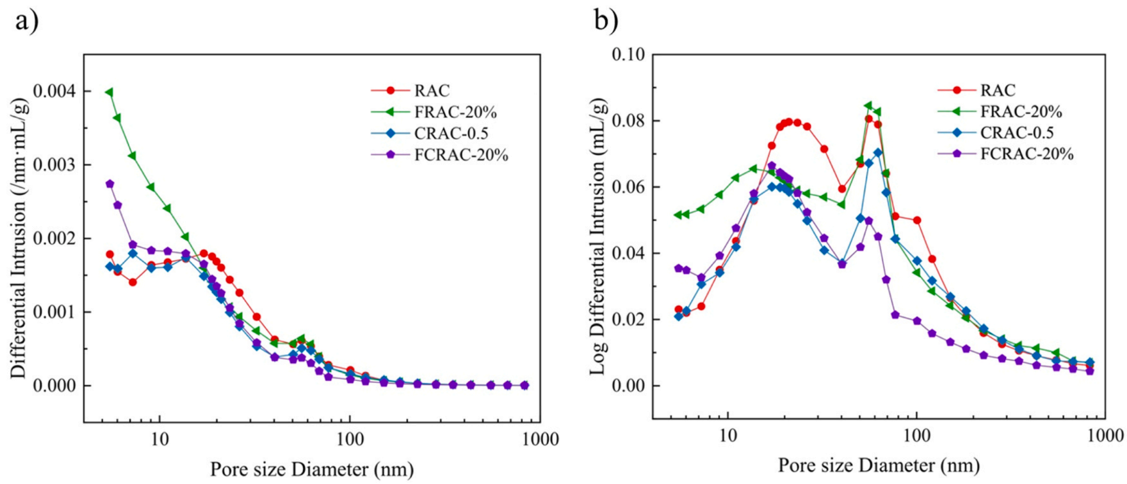

Fig. 19. MIP results for the samples under 30 wet-dry cycles: a) integral curve; and b) differential curve.

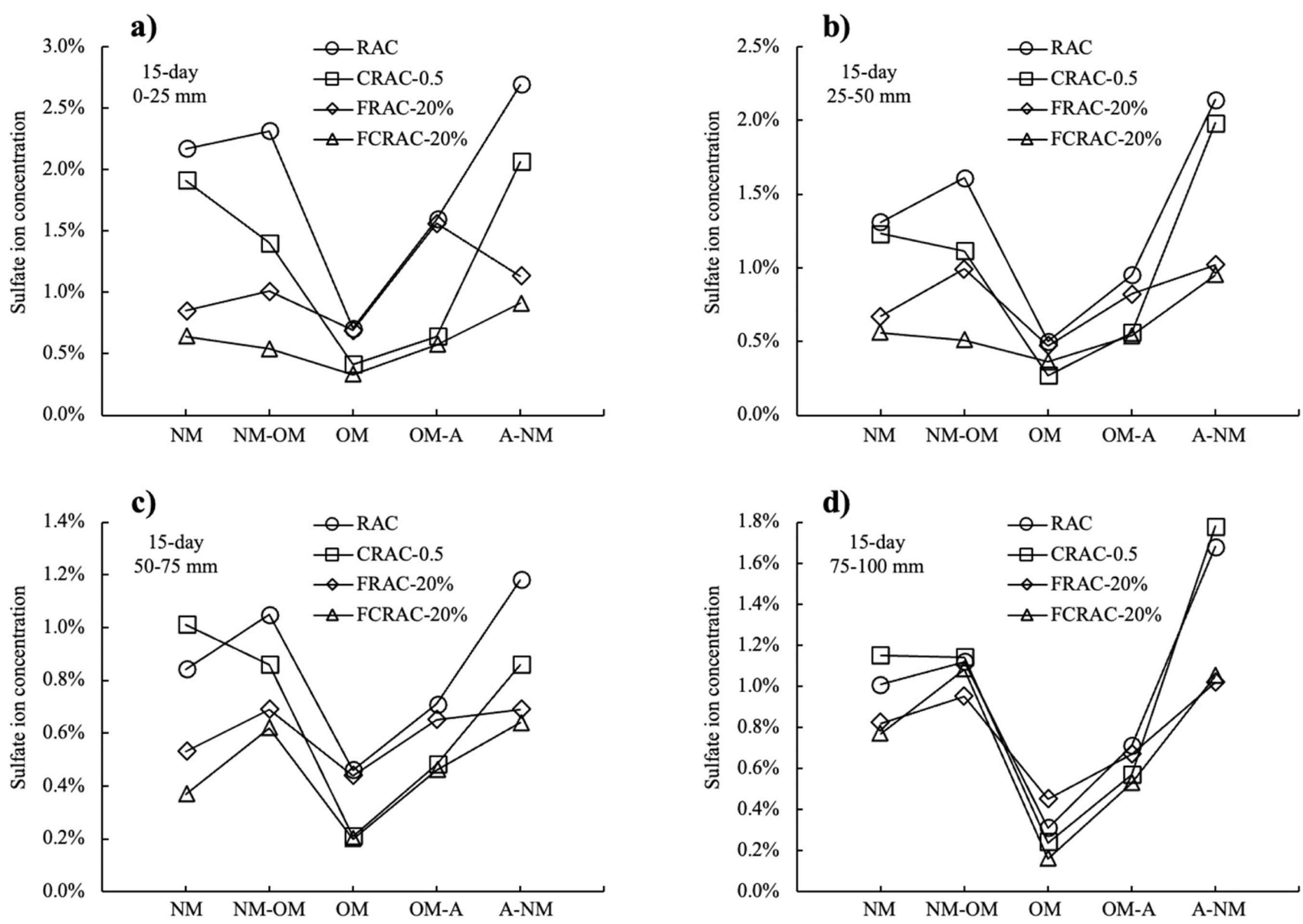

Fig. 20. The sulfate ion concentration of samples with different depths after 15-day test: a) 0–25 mm; b) 25–50 mm; c) 50–75 mm; and d) 75–100 mm.

exhibited high concentrations in the NM and A-NM regions. Similarly, the FRAC-20 % demonstrated lower sulfate ion concentrations in the NM, NM-OM, and A-NM regions, with similar values in the OM and OM-A regions. Furthermore, the FCRAC-20 % exhibited lower sulfate ion concentrations across all regions compared to RAC.

It is important to note that in the sulfate ion transport test, the sulfate ions migrated from the bottom (0 mm depth) to the top (100 mm depth). In theory, the sulfate ion concentration should decrease with increasing depth. However, the samples at the 75–100 mm depth exhibited higher sulfate ion concentrations than those at the 50–75 mm depth. This can be attributed to the limited height of the samples used in this study, which caused sulfate ions to accumulate near the top of the sample. Additionally, water evaporation from the top further contributed to the increased sulfate ion concentration. Therefore, it is recommended that samples with greater height be used with vapor sealing in future studies to better capture the sulfate ion distribution.

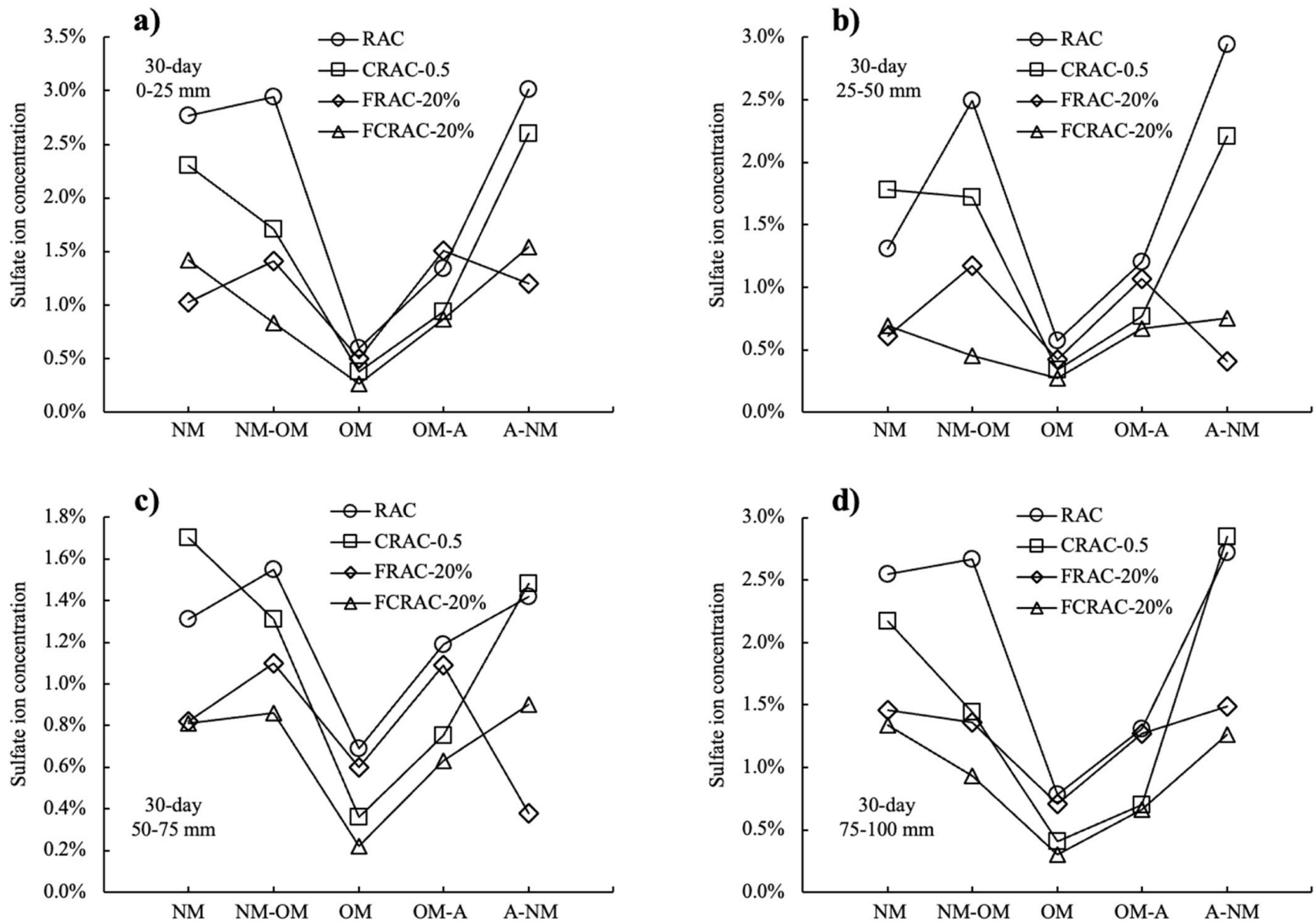

Fig. 21. The sulfate ion concentration of samples with different depths after 30-day test: a) 0–25 mm; b) 25–50 mm; c) 50–75 mm; and d) 75–100 mm.

5. Discussion

5.1. Compressive strength

Based on Table 4, it is evident that recycled aggregates negatively affected the compressive strength of concrete, primarily due to the inferior mechanical properties of the aggregates (as shown in Table 3). Carbonation treatment, especially at higher pressures, significantly enhanced the compressive strength of recycled aggregate concrete, with an improvement rate of 19.3 %. Li et al. [51] also observed that carbonation treatment of recycled aggregates increased the compressive strength of concrete by up to 22.5 %. However, the incorporation of fly ash had an adverse effect on the compressive strength of the specimens. While strength improved over time, a notable reduction in early strength (28-day compressive strength) was observed, with a decrease rate of 13.1 %. This can be attributed to the relatively slow pozzolanic reaction of fly ash, which exhibited low early activity and was unable to form sufficient hydration products to fill the structure in a timely manner. Additionally, the partial replacement of cement led to a reduction in the overall amount of early hydration products, resulting in delayed strength development. Similar findings were reported by Yun et al. [52]. Overall, combining the carbonation treatment of recycled aggregate (positive impact) with the incorporation of fly ash (negative impact) did not result in a significant impact on compressive strength of the recycled aggregate concrete.

5.2. Sulfate ion transport of recycled aggregate concrete

The transport pathways and speed of sulfate ions determined the ability of recycled concrete to resist sulfate attack. As mentioned before, the sulfate ions could transport though 5 ways in recycled aggregate concrete: new mortar, old mortar, ITZ of new mortar-old mortar, ITZ of old mortar-old aggregate, and ITZ of old aggregate-new mortar. For RAC (without carbonation treatment and fly ash), the sulfate ions mainly moved through the ITZs of new mortar-old mortar and old aggregate-new mortar, and then was new mortar (Fig. 20 and Fig. 21). It was because the new mortar had not fully hydrated yet, and its internal structure was not dense enough, providing a channel for the transport of sulfate ions. Furthermore, the high concentration of $Ca(OH)_2$ in new mortar could react with sulfate ions, and thus increase the erosion rate. Additionally, the ITZ formed between new mortar and other parts (old mortar and old aggregate) was even more porous, which accelerated the erosion rate of sulfate ions in this region. In contrast, old mortar had undergone a longer hydration period, resulting in a denser internal structure. As a result, compared to the aforementioned three parts, the transport of sulfate ions in old mortar and ITZ of old mortar-old aggregate was slower.

5.3. Influence of carbonation treatment and fly ash on sulfate ion transport of recycled aggregate concrete

Because of the micro cracks (caused by the crushing process of the old concrete) of the old mortar on recycled aggregate (Fig. 9a)), the water absorption and crushing index were higher than those of natural aggregate (Table 3). The carbonation treatment of recycled aggregate made CO_2 react with $Ca(OH)_2$ and product $CaCO_3$, which filled the micro cracks of the old mortar on recycled aggregate and made it denser (Fig. 9b)), and thus decreased the water absorption and crushing index of the recycled aggregate.

As mentioned before, the carbonation treatment consumed the $Ca(OH)_2$, which could easily react with sulfate ions. Furthermore, the carbonation product $CaCO_3$ was stable and hardly react with sulfate ions. In addition, carbonation treatment made the old mortar on the surface of the recycled aggregates denser, while also providing additional attachment points for the formation of the ITZs that includes the old mortar, making them more compact. Therefore, the more stable composition and denser structure of old mortar and the ITZs which contains the old mortar after carbonation treatment decreased the transport speed of sulfate ions through them. This is why the old mortar, ITZ of new mortar-old mortar, ITZ of old mortar-old aggregate of the samples after carbonation treatment showed lower sulfate ions concentration compared with those of the control samples (Fig. 20 and Fig. 21).

Although the carbonation treatment obviously increased recycled aggregate's properties, they were still worse than natural aggregate (Table 3), and thus the fly ash was used to further improve the durability of recycled aggregate concrete. Because of the small particle size, the addition of fly ash could effectively fill the pores between cement particles, reducing the porosity and pore size of the paste. This can be proved by the lower harmful and highly harmful pores' volume of the sample with 20 % fly ash compared to the control sample (Fig. 18). In addition, the pozzolanic effect of fly ash enabled it to continuously consume $Ca(OH)_2$ in recycled aggregate concrete over the long term, forming C-S-H gel. Since $Ca(OH)_2$ was more prone to reacting with sulfates, its consumption reduced the risk of sulfate attack. Meanwhile, the generated C-S-H gel continuously filled pores and optimized the gel system. Therefore, the addition of fly ash reduced the risk of sulfate attack in new mortar and the ITZs which contains the new mortar by both decreasing the sulfate reactants and filling the pores. This is why the new mortar, ITZ of new mortar-old mortar, ITZ of new mortar-old aggregate of the samples after carbonation treatment showed lower sulfate ions concentration compared with those of the control samples (Fig. 20 and Fig. 21).

In summary, the carbonation treatment of the recycled aggregates mainly improved the properties of the old mortar on the recycled aggregates, and thus decreased the sulfate ion transport speed through the parts which contain old mortar (i.e., old mortar, ITZ of new mortar-old mortar, ITZ of old mortar-old aggregate). The difference was that the addition of fly ash mainly improved the properties of new mortar, and thus decreased the sulfate ion transport speed through the parts which contain new mortar (i.e., new mortar, ITZ of new mortar-old mortar, ITZ of new mortar-old aggregate). Therefore, the couple effect of carbonation treatment and fly ash could decrease the sulfate ion transport speed through all the 5 ways, and thus improved the sulfate resistance of the recycled aggregate concrete.

5.4. Influence of carbonation treatment and fly ash on sulfate resistance of recycled aggregate concrete

As mentioned earlier, the carbonation treatment of recycled aggregates and the incorporation of fly ash improved and blocked different sulfate attack pathways to some extent through physical densification, chemical stabilization (consuming $Ca(OH)_2$ that was prone to sulfate reactions to form more stable substances), and ITZ optimization. This results in slower sulfate ion erosion rates and shallower erosion depths in sulfate wet-dry cycle tests. Compared to the control group, lower sulfate ion concentrations at the same depth effectively demonstrated this (Fig. 15 and Fig. 16). The slower erosion rate and shallower erosion depth mean that the reaction between the specimens and sulfates was slower, leading to lower $CaSO_4$ content (Fig. 17), which reduced the expansion effect and resulted in smaller volumes of harmful pores and highly harmful pores (Fig. 19). The occurrence of cracks and spalling during sulfate erosion was lighter (Fig. 10), and the mass change of the specimens was smaller (Fig. 11 and Fig. 12). At the same time, the impact on the compressive strength of the specimens was correspondingly smaller (Fig. 13 and Fig. 14). Kazmi et al. [33] and Ghafoori et al. [53] also stated that both the carbonation of recycled aggregate and the addition of fly ash can effectively moderate the mass loss and compressive strength decrease of the concrete samples when subjected to sulfate attack.

For the carbonation treatment of recycled aggregates, the higher the carbonation pressure, the better the carbonation effect. In this study, the optimal carbonation pressure was 0.5 MPa. However, for fly ash, a higher dosage did not necessarily lead to a greater improvement in the sulfate resistance of recycled aggregate concrete. The results in Section 4 indicate that a 20 % dosage was optimal. This was because an excessively high dosage of fly ash means a lower cement content, leading to a lower early-stage $Ca(OH)_2$ content that was overly consumed, which caused the pozzolanic reaction to stagnate. This resulted in a reduced C-S-H gel content from early hydration, which cannot fully fill the pores, leading to insufficient matrix densification and ultimately a deterioration of the sulfate resistance of recycled aggregate concrete. Similar results were also found by Gao et al. [27]. They stated that the optimal fly ash content was 16 % for improving sulfate resistance of recycled aggregate concrete. Comparing the samples with only carbonation treatment (CRAC-0.5) and only fly ash addition (FRAC-20 %), the results in Section 4 (Fig. 10 - Fig. 19) show that the carbonation treatment significantly improved the sulfate resistance of recycled aggregate concrete more than fly ash (with fewer surface cracks and spalling, smaller mass changes, less strength loss, lower sulfate ion concentration, less $CaSO_4$ content, and fewer harmful and highly harmful pores volumes). This was because although fly ash reduced the sulfate ion transport rate through physical densification, chemical stabilization, and ITZ optimization, it reduced the strength of the matrix (as the discussion in Section 5.1), making it more prone to cracking and spalling under the same degree of erosion. This limited its effectiveness in improving sulfate resistance.

As demonstrated earlier, the carbonation treatment of recycled aggregates mainly improved the old mortar on the surface of the recycled aggregates, thereby limiting the transport of sulfate ions in the areas containing old mortar (i.e., old mortar, ITZ of new mortar-old mortar, ITZ of old mortar-old aggregate). On the other hand, fly ash primarily improved the new mortar, thus limiting the transport of sulfate ions in the areas containing new mortar (i.e., new mortar, ITZ of new mortar-old mortar, ITZ of new mortar-old aggregate). Therefore, combining carbonation treatment with fly ash addition (FCRAC-20 %) simultaneously improved both the new and old mortars, thereby enhancing each sulfate ion transport channel and maximizing the sulfate resistance of recycled aggregate concrete. As seen in Fig. 10 - Fig. 19, FCRAC-20 % shows sulfate resistance performance very close to that of NAC (natural aggregate concrete), demonstrating the effectiveness and compatibility of both approaches.

6. Conclusion

In order to improve the durability of recycled aggregate concrete, this paper investigated the influence of carbonation treatment of recycled aggregate and fly ash addition on sulfate ion transport pathways and sulfates attack resistant ability of the recycled aggregate concrete. The conclusion is as follows:

1. The carbonation of recycled aggregates contributed to the improvement of the compressive strength of recycled aggregate concrete, with higher carbonation pressure yielding better enhancement effects. Although the addition of fly ash had little impact on the later strength of concrete, it did cause a certain degree of reduction in early strength. Overall, the combined use of recycled aggregate carbonation (positive impact) and fly ash (negative impact) did not have a significant effect on the compressive strength of recycled aggregate concrete.

2. The carbonation treatment, with 0.5 MPa as the optimal pressure, improved the old mortar on recycled aggregates by filling the micro crack and making it denser, and thus decreasing the water absorption and crushing index of the recycled aggregates. Furthermore, the carbonation treatment limited the transport of sulfate ions in the areas containing old mortar (i.e., old mortar, ITZ of new mortar-old mortar, ITZ of old mortar-old aggregate). For sulfate wet-dry cycle test under 120 cycles, compared with the control sample (RAC), the sample with 0.5 MPa carbonation treatment (CRAC-0.5) showed 76.6 % lower mass loss rate, 1504 % higher relative compressive strength, and less harmful and highly harmful pores volumes, indicating the better sulfate attack resistant ability.
3. The fly ash addition, with 20 % as the optimal content, filled the pores of new mortar, consumed $Ca(OH)_2$ that was prone to sulfate reactions to form more stable substances, and formed C-S-H gel to improve microstructure of mortar, and thus limiting the transport of sulfate ions in the areas containing new mortar of the recycled aggregate concrete (i.e., new mortar, ITZ of new mortar-old mortar, ITZ of new mortar-old aggregate). For sulfate wet-dry cycle test under 120 cycles, compared with the control sample (RAC), the sample with 20 % fly ash (FRAC-20 %) showed 22 % lower mass loss rate, 483 % higher relative compressive strength, and less harmful and highly harmful pores volumes, indicating the better sulfate attack resistant ability.
4. Compared with carbonation treatment (CRAC-0.5), the fly ash addition (FRAC-20 %) showed less improvement of the sulfate attack resistant ability of recycled aggregate concrete. It was because the fly ash reduced the strength of the matrix, making it more prone to cracking and spalling under the same degree of erosion, which limited its effectiveness in improving sulfate resistance.
5. The corporation of carbonation treatment and fly ash addition limited the transport of sulfate ions in all 5 pathways of the recycled aggregate concrete (i.e., new mortar, old mortar, ITZ of new mortar-old mortar, ITZ of new mortar-old aggregate, and ITZ of old mortar-old aggregate). For sulfate wet-dry cycle test under 120 cycles, compared with the control sample (RAC), the sample with corporation treatment (FCRAC-20 %) showed 146.9 % lower mass loss rate, 2487 % higher relative compressive strength, and lest harmful and highly harmful pores volumes, which were similar with those of the natural aggregate concrete (NAC). It proved the effectiveness and compatibility of both approaches.

In summary, under the synergistic modification strategy, the sulfate resistance of recycled aggregate concrete was superior to that of the groups with only carbonation treatment or only fly ash addition. Its performance reached a level close to that of natural aggregate concrete, validating its efficiency in blocking multi-path sulfate ion transport and enhancing the durability of recycled aggregate concrete. This provided important theoretical support for engineering applications. In the future, the taller specimen should be used with vapor sealing in the sulfate ion transport experiment to get more accurate results, which can be used for further sulfate attack modelling.

CRediT authorship contribution statement

Xuanrui Yu: Investigation, Data curation. **Jie Yu:** Writing – review & editing, Methodology. **Mingtao Zhang:** Writing – review & editing, Visualization, Software. **Changming Bu:** Supervision, Methodology, Conceptualization. **Jiang Chen:** Software, Investigation, Formal analysis, Data curation. **Jiehong Li:** Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Yang Yu:** Writing – review & editing, Supervision, Software, Methodology, Data curation. **Yi Sun:** Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.conbuildmat.2025.142685.

Data availability

Data will be made available on request.

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