Influence of carbonated recycled concrete aggregate on properties of cement mortar

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HIGHLIGHTS

- Carbonation treatment increased the physical properties of recycled concrete aggregate (RCA).
- Mortar made with carbonated RCA exhibited better workability than the uncarbonated RCA mortar.
- Carbonation of RCA improved both original ITZ and newly formed ITZ in the RCA and RCA mortar.

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ABSTRACT

This work investigated the effect of carbon dioxide treatment of recycled concrete aggregate (RCA) on the performance of RCA and RCA mortar. The results indicated that carbonation increased the apparent density, and reduced both water absorption and the crushing value of the RCA. The flowability and compressive strength of the RCA mortar were lower than those of natural sand mortar. However, the properties of mortar made with carbon dioxide treated RCA were very similar to those of natural sand mortar. Compared with the mortar made of un-carbonated RCA, the mortar made with carbonated RCA showed increased autogenous shrinkage, reduced drying shrinkage, water absorption, and chloride migration coefficient. Scanning electron microscope (SEM) examination on the interfacial transition zone (ITZ) in the RCA and RCA mortar found that carbonation treatment of RCA not only improved the original ITZ in the RCA, but also improved the newly formed ITZ in the RCA mortar.

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1. Introduction

With rapid development of economy and construction, many construction and demolition (C & D) wastes are generated and need to be treated. Due to increased landfill cost and demand of aggregates in construction, recycling and reuse of waste concrete are becoming more important for sustainable development [1,2].

Waste concretes can be used to produce recycled concrete aggregate (RCA) after crushing and sieving. However, the mechanical properties and durability of recycled aggregate concrete (RAC) were weaker than those of ordinary concrete, mainly due to the attached cement paste or the cracks formed during crushing [3–5]. Compared with ordinary concrete, the tensile and compressive strength of RAC were reduced by 40% and the drying shrinkage was increased by 60% [6,7]. Cracks were more likely to appear when concrete contained more than 50% RCA, because of the reduced tensile properties of the concrete [8]. Therefore, it is necessary to improve the properties of RCA for getting better quality recycled aggregate concrete.

Several methods for improving the properties of RCA have been proposed in literature and can be classified into two categories: (1) removing the attached mortar by ultrasonic cleaning method [9], ball-milling [10], heating the RCA and then rubbing [11], or pre-soaking the RCAs with HCl, H_2SO_4, H_3PO_4 [12] or with waterglass [13]. (2) Improving the quality of attached paste, such as surface-coating with pozzalanic materials [13,14] or polyvinyl alcohol emulsion [15]. However, each of these methods introduces some negative effects either on the material or the environment.

This work uses CO_2 to pretreat RCA for improving the properties of RCA. The principle of this idea is that CO_2 can react with the adhesived paste on the RCA to form $CaCO_3$ and silica gel. The solid volume of the adhesived paste is increased after carbonation,

which increased the density and reduced the porosity of RCA. Shi et al. [16] proposed pre-curing technology to enhance and to accelerate CO_2 curing of concrete. After appropriate pre-curing, the strength of concrete cured with CO_2 for 2–4 h was similar to that of the concrete cured under steam for 24 h. Additionally, concrete samples cured with CO_2 demonstrated lower porosity, water absorption, and shrinkage than concrete cured under steam. Shi et al. [17] also found that increasing CO_2 concentration and pressure would accelerate carbonation and increase strength development rate of concrete. However, if the CO_2 pressure was greater than 0.5 MPa, the CO_2 curing degree and compressive strength of the CO_2 cured concrete did not show significant differences.

Carbonation of RCA not only improved the properties of RCAs, but also reduced the greenhouse effect that caused by carbon dioxide emission. Using CO_2 to treat RCA can store CO_2, which reduces the greenhouse effect. The cement industry is one of the major sources of greenhouse gases in particular with carbon dioxide emissions, which contributes about 7% to these emissions [18]. The manufacture of 1 ton cement generates about 0.79 ton carbon dioxide [19]. If those carbon dioxide can be applied for treating the RCA, it can help the cement industry to save the expenses of treating the carbon dioxide emission. This is a laboratory study on the feasibility of using carbonation treatment for improving the properties of RCA.

This work aimed at studying compressive strength, shrinkage, drying shrinkage, water absorption, and chloride ion resistance of the carbonated RCA mortars. Findings from this work provide useful information for improving the properties of recycled concrete.

2. Materials and testing methods

2.1. Raw materials

Recycled gravel concrete aggregate (G-RCA) and recycled crushed stone concrete aggregate (C-RCA) were obtained from concrete beams with the compressive strength of 30 MPa and 50 MPa, respectively. The gravel from a Xiangjiang river was crushed and used as the reference sand (NS). Both G-RCA and C-RCA were sieved to the same gradation as the reference, as shown in Table 1. P.O. 42.5 ordinary Portland cement was used in this work and the chemical composition of the cement is shown in Table 2.

2.2. Carbonation treatment of RCAs

G-RCA and C-RCA were placed in a carbonation chamber at $T = 20 \pm 2\ ^{\circ}C$, $RH = 60 \pm 5\%$, and CO_2 concentration of $20 \pm 2\%$. After certain period of carbonation, the RCA was ground and spreaded evenly, and then sprayed with 1% alcohol phenolphthalein solution to differentiate the carbonated and un-carbonated portions. G-CI and C-CI stand for the carbonated recycled gravel concrete aggregate and recycled crushed stone concrete aggregate, respectively.

2.3. Mix proportions and sample preparations

The sand to cement ratio of 2.25 and water to cement ratio of 0.50 were used in preparing the mortar samples. Fresh mortar was cast in three different molds. Cube samples with the size of $40 \times 40 \times 40$ mm were prepared for compressive strength and water absorption measurements. Mortar bars with the size of $25 \times 25 \times 275$ mm were prepared for drying shrinkage measurement, while cylinders with the size of $\Phi100 \times 100$ mm were prepared for rapid chloride migration measurement. Fresh mortar was injected into corrugated plastic tubes of $\Phi20 \times 345$ mm for autogenous shrinkage measurement.

2.4. Testing methods

2.4.1. Physical properties of recycled concrete aggregate

Physical properties of the recycled concrete aggregate were measured in accordance with the Chinese standard JGJ/52-2006, which include density, water absorption, and crushing value.

Table 1
Gradation of the three types of aggregate.

Diameter(mm)	2.5	1.25	0.63	0.315	0.16
Cumulative residue on sieve (%)	0	15	50	85	100

Table 2
Chemical composition of ordinary Portland cement.

CaO	Al$_2$O$_3$	MgO	Fe$_2$O$_3$	SiO$_2$	SO$_3$
65.40	5.40	3.40	2.80	21.00	2.00

Apparent density. Sample was placed in an oven at 105 °C till constant mass, and then placed in laboratory condition ($T = 20$ °C, RH = 60%) for cooling. Put 50 g sample into a Le pycnometer which contained the volume of water V_1, and record the volume of water V_2 after placed the sample. The solid volume of sample equals to the volume change ($V_2 - V_1$) of the water in the pycnometer when the sample was placed in the pycnometer. The apparent density of the sample equals to the mass of sample divided by the solid volume of the sample ($V_2 - V_1$).

Water absorption. A 500 g oven dried sample was placed in a disk, which contains enough water to ensure the water level was 20 mm above the sample. The sample was taken out after 24 h and then dried under a fan which blows above the sample. A stick was stir on the sample to ensure the sample was uniformly dried until the saturated-surface-dry (SSD) condition was reached. A 500 g SSD sample was placed in an oven at 105 °C till m_o was reached. The water absorption w_{wa} can be determined using equation: $w_{wa} = (500 - m_o)/m_o \times 100\%$.

Crushing value. The oven dried RCA was sieved, and then a 300 g sample, which has a particular particle size, was placed into a steel mold. The mold was placed on a loading machine, loaded at the rate of 500 N/s till 25 kN, and retained for 5 s. The sample was sieved after the loading process, and the material which had the same particle size as the original sample was determined (m_i). The crushing value δ_i can be determined using equation: $\delta_i = (300 - m_i)/300 \times 100\%$.

2.4.2. Properties of fresh recycled aggregate mortar

(1) Flowability

The flowability of fresh mortar was measured using a cone with 60 mm height, 70 mm top diameter and 100 mm bottom diameter. The testing was conducted on a flow table in accordance with the Chinese standard GB/T2419-2005. The maximum diameter and the diameter perpendicular to the maximum diameter of the spread out fresh mortar were measured. The average of the two diameters was used as the flowability of the sample.

(2) Compressive strength

Fresh mortar mixtures were cast in the $40 \times 40 \times 40$ mm cubic molds. The samples were demolded after 24 h, and then cured in lime saturated water at 20 ± 2 °C till 3, 7, 28 and 90 days for compressive strength test.

(3) Autogenous shrinkage

Autogenous shrinkage of mortar was measured using a corrugated plastic mold with the size of $\Phi20 \times 345$ mm in accordance with ASTM C1698-09. An eddy current displacement sensor was installed on the steel frame after the fresh mortar sample was filled in the corrugated plastic mold and sealed with a coil. The other end of the sample was fixed by coil springs. Two samples were prepared for each mixture. The eddy current displacement sensor records the longitudinal deformation on the free ends. A multi-channel data acquisition instrument with the measurement range of 0–4 mm, resolution of 0.5 μm, accuracy of 0.05% was used for data collection. The samples were stores in a controlled room with a temperature of 20 °C and relative humidity of 50% during the test.

(4) Drying shrinkage

Three samples with the size of $75 \times 75 \times 275$ mm were prepared for the drying shrinkage measurement. The samples were demolded after 24 h of casting and then placed in a curing room at $T = 20 \pm 1$ °C and RH = $60 \pm 2\%$. The initial length of the sample was recorded after the curing period, and the length of the samples were measured at 1, 7, 14, 21, 28, 35, 42, 49 and 56 days. A vertical length comparator with the accuracy of 0.001 mm was used for calibration.

(5) Water absorption of mortar

The water absorption of mortar was measured in accordance with the Chinese standard JGJ/T70-2009. Samples were removed from the curing room at 28 days, and then dried in an oven at 105 °C for 48 h. The mass of the samples was measured after they reached room temperature, and then they were placed in a water bath which filled with water at 20 ± 2 °C for two days. The top surface of the samples was at least 20 mm below water. Samples were taken out from the water bath and wiped with a filter paper to remove the surface water and then weighed. The water absorption of the samples equals to the mass change of the sample before and after placed in the water bath.

(6) Rapid Chloride Migration (RCM) testing

The central portion of the $\Phi100 \times 100$ mm cylinders was cut into slices with the thickness of 50 ± 2 mm, and then cured in curing room at $T = 20 \pm 2$ °C and RH ⩾ 98% for 35 days. The finished surface of the sliced samples was exposed to chloride solution. After that, the specimens were cured in water for another 7 days. The excess water on the sample was wiped off with a brush. The thickness of the sliced samples was measured

using a caliper with the precision of ±0.1 mm. The SSD specimens were placed in a container for vacuum until 1–5 kPa, and then the container was filled with Ca(OH)$_2$ saturated solution to vacuum for one hour. Samples were kept in the container for 18 ± 2 h. The catholyte solution was 10% NaCl and the anolyte solution was NaOH with the concentration of 0.3 mol/L. Store the solutions at the temperature of 20–25 °C for at least 24 h before testing. Silver nitrate solution of 0.1 mol/L was sprayed on the samples as the indicator of Cl$^-$ penetration.

2.4.3. Microstructure of interfacial transition zone (ITZ)

Both un-carbonated and carbonated RCA and RCA mortar samples were placed in an oven at 105 °C till constant mass was reached. Scanning electron microscope (SEM) was utilized in this work to exam the microstructure of the adhesive paste of the pre-treated RCA samples, and the microstructure of the newly formed ITZ between the RCA and paste.

3. Results and discussion

3.1. Physical properties of aggregate

Physical properties of the RCA before and after carbonation are given in Table 3. The apparent density of the un-carbonated RCA was 6.3–7.8% lower than that of the NS, while that of the carbonated RCA was 1.9–2.6% lower than the NS. After carbonation, the absorption and crushing value of RCA were reduced by 22.6–28.3% and 7.6–9.6%, respectively. Those were attributed to the reactions of carbon dioxide with the hydration products of cement and unhydrate cement particles, which filled the capillary pores in hardened cement pastes.

3.2. Properties of recycled aggregate mortars

3.2.1. Flowability of fresh recycled aggregates mortar

Fig. 1 shows the flowability of the fresh mortars with different sands. The flowability of natural sand mortar was better than that of the recycled aggregates mortar, which agreed with findings from previous work [20]. This is because the attached cement paste in RCA had high absorption which increased the friction between aggregate and the fresh paste. However, the flowability of carbonated recycled aggregate mortar was better than that of the un-carbonated recycled aggregates mortar. This is because that carbonation reduced the water absorption of the recycled aggregate. The flowability of the C-RCA mortar was slightly higher than that of the G-RCA mortar which might be due to the lower original water-cement ratio and less porous original attached cement paste.

3.2.2. Compressive strength of the hardened recycled aggregate mortar

The compressive strength of hardened mortar with different sands is shown in Fig. 2. The 7-day, 28-day, and 90-day compressive strength of carbonated G-RCA mortar were 13%, 10%, and 18% higher than that of the un-carbonated G-RCA mortar. The 7-day, 28-day, and 90-day compressive strength of carbonated C-RCA mortar were 11%, 6%, and 8% higher than that of the un-carbonated C-RCA mortar. The compressive strength of carbonated
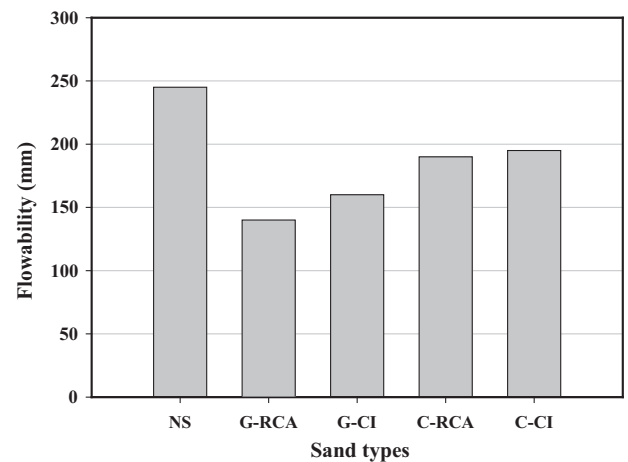
Table 3
Physical properties of aggregates.

Physical property	Un-carbonated aggregates			Carbonated aggregates	
	NS	G-RCAs	C-RCAs	G-CI	C-CI
Water absorption (%)	2.35	8.06	8.70	5.78	6.73
Apparent density (kg/m^3)	2.70	2.53	2.49	2.65	2.63
Crushing value (%)	–	18.6	17.1	16.9	15.8

Note. NS = natural sand; G-RCA = recycled gravel aggregate; C-RCA = recycled crushed stone aggregate; G-CI = carbonated recycled gravel aggregate; C-CI = carbonated recycled crushed stone aggregate.

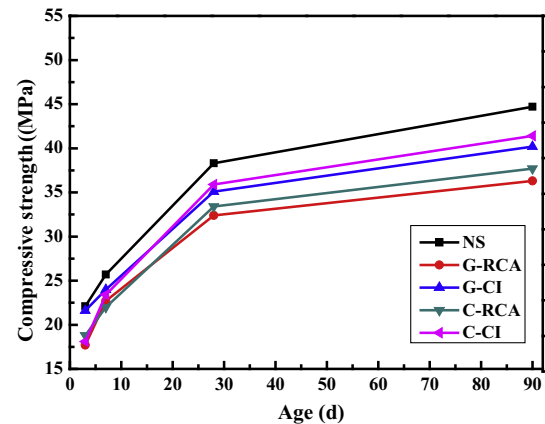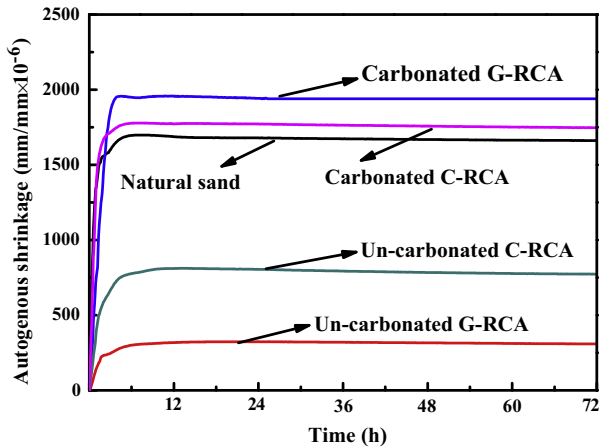**Fig. 1.** Flowability of fresh recycled aggregate mortars.

Fig. 2. Compressive strength of the hardened mortars with different sands.

recycled aggregate mortar was close to that of the natural sand mortar, which might related to the reduced water absorption and crushing value of the original attached cement paste [21].

The compressive strength of C-RCA mortar were higher than that of the G-RCA mortar, which is because the C-RCA was from the waste concrete has higher compressive strength than the G-RCA and that made the hardened mortar has higher compressive strength as well. However, the strength of the carbonated G-RCA mortar was close to that of the carbonated C-RCA mortar. This suggested that carbonation treatment was more effective to the recycled aggregate whose original attached cement paste had lower strength.

3.2.3. Autogenous shrinkage of recycled aggregate mortar

The autogenous shrinkage strain of mortar with different aggregate sand is shown in Fig. 3. The autogenous shrinkage strain of the recycled aggregate mortar was almost reached constant after 24 h. The autogenous shrinkage strain of the C-RCA mortar before carbonation was 774 µε at 3 days, which was increased by 126% after carbonation. Similar observation was noticed on the G-RCA mortar whose autogenous shrinkage strain was raised from 310 µε to 1940 µε (by 526%) after carbonation. Higher water absorption of the original attached paste resulted in lower autogenous shrinkage strain of the un-carbonated recycled aggregates. Because the Autogenous shrinkage was caused by the chemical reaction during cement hydration, and it was measured in a sealed condition which water can hardly evaporate. The absorbed water in the

Fig. 3. Autogenous shrinkage of mortars with different sands.

recycled aggregates can be released during cement hydration, which would reduce the effect to the internal humidity. Also, the pore diameter of the aggregates and cement paste affected the release of absorbed water. Therefore, denser carbonated recycled aggregates made the absorbed water more difficult to transport.

3.2.4. Drying shrinkage of recycled aggregate mortar

Concrete shrinkage cracking was caused by the volume change of concrete during hydration. Different from the autogenous shrinkage, the drying shrinkage of mortar increases with the volume of cement paste [22,23]. Many factors affect the drying shrinkage of concrete, and capillary porosity was reported affect the drying shrinkage most significantly [24–26]. The dry shrinkage of mortar samples prepared with different aggregate types is shown in Fig. 4. The drying shrinkage of RCA mortar was higher than the NS mortar, which might due to the original attached cement paste increased the total volume of cement paste in the RCA mortar. The attached paste in RCA includes more pores, and that results in water can easily get in and out during cement hydration. The results also indicated that the drying shrinkage of carbonated RCA mortar was lower than that of the un-carbonated RCA mortar. The drying shrinkage of the carbonated G-RCA and C-RCA mortar was reduced by 8% and 13% at 56 days. This suggested that carbonation reduced the porosity and water absorption of the attached cement paste, which reduced water evaporation and thus the drying shrinkage of the RCA mortar. The drying shrinkage of C-RCA mortar was lower than the G-RCA mortar. This may due to the denser original attached cement paste and the original ITZ.
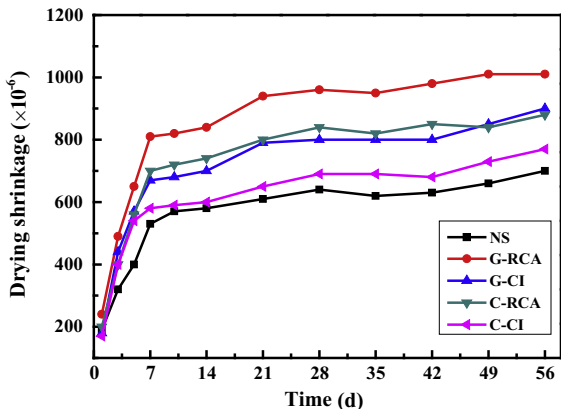
3.2.5. Water absorption of recycled aggregate mortar

Water absorption not only indicated the permeability of mortar samples, but also suggested the microstructure of the samples. The water absorption of mortar samples is shown in Fig. 5. Water absorption of the recycled aggregate mortar was higher than that of the natural sand mortar, which may due to the higher porosity and ITZ in the original attached pastes. However, it has been also noticed that carbonation on the recycled aggregates significantly reduced the water absorption of the mortar samples.

3.2.6. Chloride diffusion coefficient of recycled aggregate mortar

Table 4 presented the results of chloride diffusion coefficient from rapid chloride migration tests. The chloride diffusion coefficient of the G-CI mortar was 11 times lower than the G-RCA mortar, and the G-RCA mortar was about 20 times higher than the NS mortar. The chloride diffusion coefficient of R-CI mortar was 6 times lower than the R-RCA mortar, and the R-RCA mortar was 18 times lower than the NS mortar. The poor ITZ and high porosity of the recycled aggregates caused the mortar unable to resistant chloride effectively [27]. After carbonation treatment, density of the attached paste and the original ITZ were increased. Thus, the chloride diffusion resistance was increased as well.

3.2.7. Microstructure of interfacial transition zone (ITZ)

SEM images of the recycled aggregate surface are shown in Fig. 6. An denser ITZ was noticed in the carbonated recycled aggregate as shown in Fig. 6b, and the surface of the un-carbonated recycled aggregate was more porous (Fig. 6a). Also, the needle-like ettringite was found in Fig. 6a, and such crystal did not observed in the carbonated recycled aggregate. Both ettringite (AFt) and monosulfate (AFm) crystals are the hydration products of a typical Portland cement [28]. AFm was converted into AFt and aluminum oxide at pH of 11.6, and AFt was decomposed into sulfate ions and aluminum hydroxide ions at pH of 10.6 during carbonation [29]. The reaction products were less porous than the reactants after carbonation, which might contributed to the reduced water absorption of the mortars after carbonation.
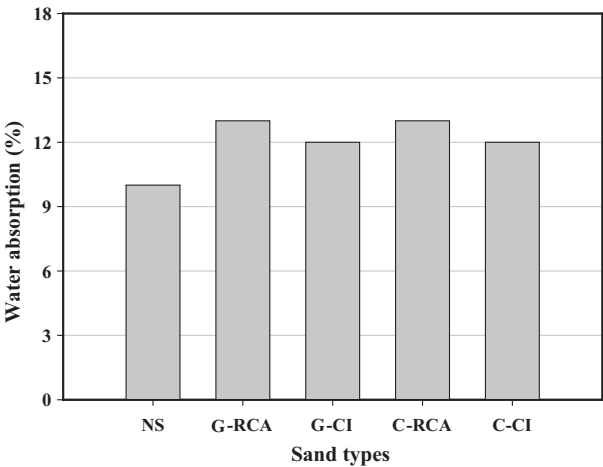

Fig. 5. Water absorption of mortars with different sands.

Fig. 4. Drying shrinkage of mortars with different sands.

Table 4
Chloride diffusion coefficient of mortars (10^{-12} m/s^2).

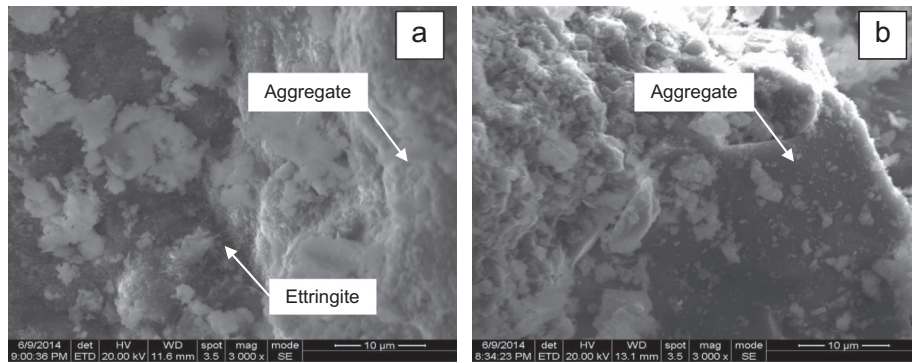| Sand-cement ratio	Un-carbonated mortar			Carbonated mortar	
	NS	G-RCA	C-RCA	G-CI	C-CI
2.25	1.3	18.8	14.7	1.7	2.1

Fig. 6. SEM images of recycled aggregate surfaces (a: un-carbonated RCA; b: carbonated RCA).
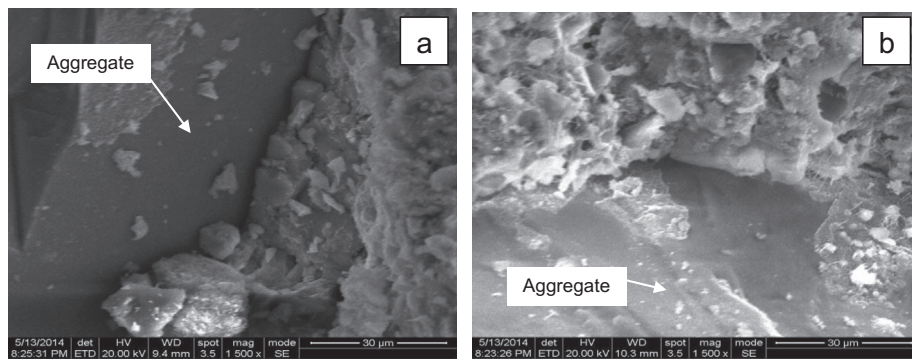

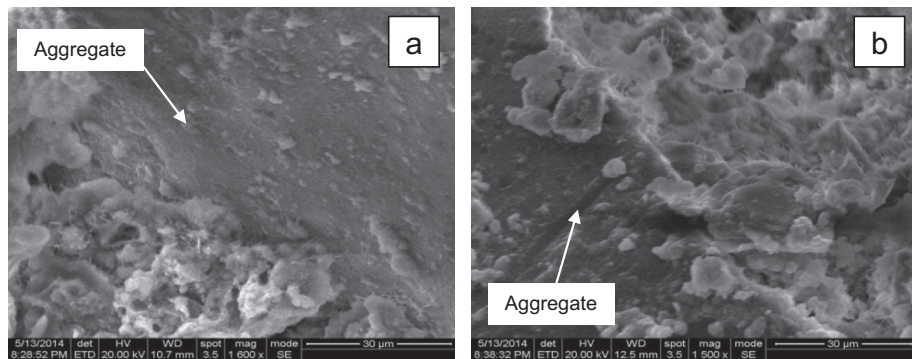**Fig. 7.** Typical interfacial morphology of natural sand mortar.

Fig. 8. Interfacial morphology of RCA mortars at 3 days (a: un-carbonated mortar; b: carbonated mortar).
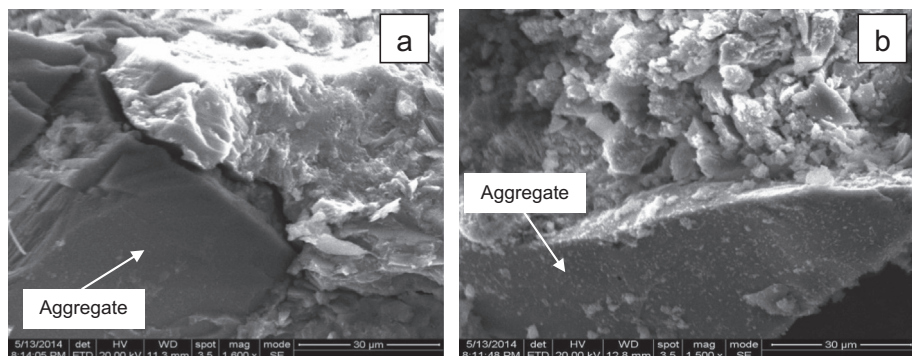

Fig. 9. Interfacial morphology of recycled gravel aggregate mortars at 28 days (a: un-carbonated mortar; b: carbonated mortar).

The ITZ between cement paste and aggregate is an important region in concrete. Typical microstructure of ITZ in a NS mortar is shown in Fig. 7. The microstructure of ITZ and cement paste were dense, and only a few large holes in both ITZ and the paste (Fig. 7a). The needle-like ettringite crystals were found in Fig. 7b.

The SEM images of carbonated and un-carbonated recycled aggregate mortar which cured for 3 days are shown in Fig. 8. The interface of un-carbonated recycled aggregate mainly consists of loose granular C-S-H and needle-like ettringite crystals. The region around the ITZ was more porous than that around natural sand–cement interface. The absorbed water by the attached cement paste resulted in a water film around the aggregate surface, which increased the local w/c. Thus, the porosity of the ITZ in RCA mortar was higher and less ettringite would form compared with the natural sand mortar [30]. Since carbonation reduced the water absorption of the RCA, the thickness of the water film was decreased and the ITZ of the carbonated RCA mortar was improved.

The interfaces of un-carbonated and carbonated G-RCA mortars at 28 days are shown in Fig. 9. It could found that the ITZ of the carbonated mortar was significantly denser than the un-carbonated mortar. The ITZ of the un-carbonated G-RCA mortar exhibited as large amount of loose hydrate products, and connected pores can be seen in the interface. The bonding between cement paste and aggregate was weak. The ITZ of the carbonated G-RCA was much denser (Fig. 9b), and large pores between the cement paste and aggregate can hardly found. The water absorption of RCA was reduced and the water film around the aggregate surface became thinner after carbonation treatment. Therefore, the local w/c of the interface was decreased, and the improved ITZ would increase the compressive strength and other mechanical properties [31]. Several reasons can contribute to the strengthened bonding between cement paste and RCA: (1) the surfaces of RCA were rougher and more angular than the natural aggregate; (2) the elastic modulus of RCA was closer to cement paste due to the original attached cement paste; (3) the microcracks in the attached cement paste absorbed cement particles from fresh cement paste, which filled the cracks and reduced the porosity of RCA.

4. Conclusion

From the experimental results and discussion above, the following conclusions can be drawn:

(1) Carbonation treatment enhanced the physical properties of RCA. The apparent density was increased by 4.7–5.6%, water absorption and crushing value were decreased by 7.6–9.6% and 22.6–28.3%.
(2) The flowability and compressive strength of RCA mortar were lower than that of the natural sand mortar. But, carbonation treatment increased the flowability and compressive strength of the RCA mortar. Both flowability and compressive strength of the carbonated RCA mortar were close to those of the natural sand mortar.
(3) Compared with the un-carbonated RCA mortar, the carbonated RCA mortar has higher autogenous shrinkage, lower drying shrinkage, water absorption, and chloride migration coefficient.
(4) SEM examination on the microstructure of the ITZ in the RCA and RCA mortar revealed that carbonation not only improved the original ITZ in RCA, but also increased the newly formed ITZ in the RCA mortar.

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