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The effects of various operational- and materials-oriented parameters on the carbonation performance of low-quality recycled concrete aggregate

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ABSTRACT

This study focuses on the examination of the parameters affecting the carbonation performance of low-quality recycled concrete aggregates (RCA) obtained from end-of-life buildings and having low mechanical and chemical properties. The accelerated carbonation process was implemented by using a lab-scale carbonation reactor, providing a dynamic carbonation process. In the scope of this study, as the main operational parameters, relative humidity (RH) (50-70-90%), medium pressure (1-2-3 bar), and temperature (50–90 °C) were determined. Carbonation processes were implemented on the low-quality RCAs with 5 different particle size ranges, including <0.85, 0.85–2.00, 2.00–4.75, 4.75–9.50, and 9.50–14.00 mm. Effects of carbonation durations (2-4-6-12-24-48-72-96-120 h) on the carbonation performance of RCAs were also investigated. Thermogravimetry was the test method used to quantify the CO_2 uptake rate of low-quality RCAs. In addition, the water absorption capacity of RCAs was determined to gain insight into precipitated calcium carbonate. To evaluate the microstructure of carbonation products, scanning electron microscopy (SEM) and X-ray diffraction (XRD) methods were used. Mortar specimens were prepared using uncarbonated and carbonated RCAs to assess the effects of the carbonation on the workability and mechanical properties of the RCAs-incorporated cementitious system. Results showed that all parameters tested were effective on the carbonation performance of the RCAs. Implementing carbonation with an optimal condition (70% RH, 1 bar, 90 °C, 48 h) resulted in an increase in the quality of low-quality RCAs in favor of increasing the compressive strength of the cement-based systems. Therefore, valuable upcycling of low-quality RCAs for the construction industry is possible in high-tech applications with accelerated carbonation under optimum conditions.

1. Introduction

Concrete is the most widely used building material in the construction industry worldwide, and Portland Cement (PC) and natural aggregate are the main ingredients used for its production. Globally, concrete production increases with a growing need for shelter/infrastructure/superstructure. Although the usage of concrete is beneficial regarding the quantity produced, and cost yet has an unde-

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niable environmental burden. Its detrimental effects can be mainly classified under two headings, namely PC production-related significant amount of CO_2 emission and overuse of natural sources (e.g., clinker, aggregate, gypsum, etc.). In total, cement production accounts for about 5–8% of global anthropogenic CO_2 emissions [1,2]. As the main aspect, concrete production requires different-size aggregates whose production process is also highly energy-intensive, creating significant amounts of waste next to the depletion of natural resources [3].

Negative impacts of concrete are coupled with insufficient longevity of traditional structures, having a high deterioration tendency under combined mechanical/environmental effects, demanding demolition/reconstruction activities, all of which create waste concrete/masonry/glass collectively termed as “Construction and Demolition Waste” (CDW). These wastes, which are stored in empty lands outside the city, have harmful effects such as emitting harmful substances, polluting the groundwater, and disrupting the relevant ecosystem [4]. CDW industry is one of the largest global solid waste producers and accounts for 30–40% of the total urban waste generation [5]. Worldwide, the amount of annually produced CDWs accounts for more than 10 billion tons [6]. Therefore, the generation of CDW can be regarded as a global issue requiring a relatively high demand for proper handling.

In general, CDW has a heterogenous composition and consists of different types of material, including concrete, ceramic, plastic, metal, glass, brick, and timber [7]. However, concrete waste has a large share of the CDWs, which accounts for 40–85% of the total amount of CDW, varying on the type of demolished structures [8]. Currently, a high portion of generated concrete waste is used for low-value-added applications, such as land fillings of road bases or non-structural filling. To valorize the waste concrete in high-value-added applications, researchers made great efforts to use concrete waste as a recycled concrete aggregate (RCA) in concrete production after several processes (e.g., crushing, sieving, etc.) [9–11]. However, there are some disadvantages to the use of RCA without any treatment in concrete mixture design due to mortar/paste residues on RCA leading to higher water absorption/abrasion/porosity, lower density, and more heterogeneity compared to natural aggregates [12–15]. Some strategies for weakening the link between the old mortar and the natural aggregate particles were reported [16,17], such as pre-wetting [18], mechanical abrasion [19], heating-abrasion [20], microwave heating [21], acid/sodium silicate solution-based treatments [12,22], and microbial-induced treatment [23]. Although these methods are successful for the enhancement of RCA’s properties, their acceptance in construction is still limited due to being time- and energy-intensive, impracticability on a large-scale and high cost. Therefore, an environmentally friendly, sustainable, and feasible method for the enhancement of RCA is an urgent need to eliminate the detrimental effects of both CDW generation and the environmental burden of concrete.

Carbonation can be one of the most effective and environmentally friendly methods for improving RCAs by refining pores via $CaCO_3$ precipitation [12,24,25]. Although carbonation can occur under all circumstances during the service life of concrete, the rate of natural carbonation is quite slower. To reduce the extended period of natural carbonation reactions, an effective and technically feasible carbonation process can be developed by arranging carbonation conditions [26]. Mineralization of RCA via accelerated carbonation provides RCA with improved density, lower porosity, water absorption capacity, higher strength, and resistance to external detrimental agents [12,27,28]. In the current literature, the efficiency of accelerated carbonation was enhanced by applying various arranged operational- and material-related parameters during the carbonation procedure. In this regard, the effects of humidity, pressure, temperature, carbonation duration, the particle size of RCA, CO_2 concentration, static/dynamic carbonation methodologies, and incorporation of carbonation accelerator agency on the efficiency of the accelerated carbonation process were some influential parameters tested in the literature [29–38]. However, the literature studies showed that there is no optimum accelerated carbonation condition valid for each type and size of RCAs. Depending on the parameters such as sources and particle size distribution of RCA, temperature, humidity, pressure, carbonation medium, etc., the optimal condition for each parameter range may differ under different carbonation parameters [39].

Increasing anthropogenic CO_2 emissions, generation of CDWs, and the need for natural sources are the main factors that lead authors to find solutions for managing CO_2 emissions and procurement of secondary raw materials via upcycling of CDW-based RCA. It is estimated that millions of buildings will be affected by the urban renewal law in Turkey. Also, large amounts of concrete waste having a compressive strength of less than 10 MPa will be released as a result of urban renewal. This low compressive strength belonging to those waste concrete from end-of-life buildings can be attributed to; (i) the high amounts of aggregate and the low quantity of cement in mix-design, (ii) inadequate hydration product because of improper curing, (iii) expired cement usage in the matrix (already hydrated cement during shelf), (iv) degradation and damage of the concrete during its service life because of external effects such as earthquake, weathering, etc. Besides that, the inadequacy of the regulation conditions in force at that time may have caused construction with low-quality concrete in the buildings. Due to all these reasons, obtained RCAs can consider as low-quality in terms of strength parameters since obtained RCAs compose of both original aggregate and residual mortar, which has low strength.

To solve tremendous amounts of generated CDW-based low-quality RCAs, which are not preferred to use in high-tech construction applications as a source, this study focused on the improvement of these RCAs by using accelerated carbonation. Thus, the comprehensive investigation of parameters affecting the efficiency of accelerated carbonation of low-quality fine and coarse RCAs from end-of-life buildings in favor of enhancing their performance properties and upcycling those low-quality RCAs in high-tech applications was aimed. In this study, carbonation performances of low-quality RCAs with different grain size distributions were investigated comprehensively considering different operational parameters. In this regard, relative humidity (50%-70%-90%), pressure (1, 2, 3 bar), grain size ($<$0.85, 0.85–2, 2–4.75, 4.75–9.5, 9.5–14 mm), temperature (50 °C and 90 °C), and curing duration (2 h, 4 h, 6 h, 12 h, 24 h, 48 h, 72 h, 96 h, and 120 h) were determined as variables for the carbonation process implemented with a rotary carbonation reactor designed by authors. It was aimed to increase the efficiency of carbonation procedures and enhance the properties of low-quality fine and coarse RCAs through accelerated carbonation. The carbonated RCAs were subjected to several tests, including thermogravimetry analysis (TG/DTG), water absorption capacity, scanning electron microscopy (SEM), and X-ray diffraction (XRD). After the characterization of the carbonation efficiency of the carbonated RCAs, the most promising carbonated RCAs were used in mortar production to evaluate the potential valorization of carbonated RCA in cement-based low carbon foot-print products by comparing the mortars with both uncarbonated RCAs and natural aggregates. Flow table and compressive strength tests were the methods carried out to assess RCAs performance in mortar mixtures.

2. Experimental program

2.1. Recycled concrete aggregate production

Low-quality end-of-life concrete rubbles were acquired from an Ankara urban transformation area in the form of various-sized pieces of rough fragments (Fig. 1). RCAs were obtained by crushing via jaw crusher and sieving by following the ASTM C136. To examine the effect of particle size on carbonation efficiency in detail, obtained RCAs were then classified according to particle size ranges of < 0.85 mm, 0.85–2.00 mm, 2.00–4.75 mm, 4.75–9.50 mm, and 9.50–14.00 mm. While determining the particle sizes based on ASTM C136, the easiness and common applicability of the process were considered. The physical appearances of the obtained RCAs with various particle sizes were presented in Fig. 1. As the demolished buildings were at the end of service life and constructed with expired specifications providing relatively low mechanical performance (with average compressive strength values of 8.7 MPa measured from the demolished buildings by using obtained core samples), the used RCAs can be regarded as low-quality. Low-quality RCA term can also be related to the inadequate amount of available compounds that have carbonation potential in the system, such as CH, CSH, and, etc because of the aforementioned reasons for low-strength concrete.

2.2. Accelerated carbonation procedure

To increase the speed of carbonation reactions of RCAs and to provide an effective process for permanently removing CO_2 from the atmosphere, a dynamic carbonation technique was followed in this study. In this regard, a lab-scale carbonation reactor designed by the authors was used to provide an accelerated carbonation process for low-quality RCAs. The reactor was designed to provide a reliable, controlled environment by arranging the relative humidity, pressure, temperature, and CO_2 concentration for the carbonation reaction. The details of the developed carbonation reactor are demonstrated in Fig. 2. In this reactor, the temperature was driven by the internal heaters and sensors, and temperature settings range from 20 ± 3 °C to 110 ± 3 °C. The relative humidity of the medium was controlled by a humidifier and can be operatable in a range of 30 ± 5% and 95 ± 5%. The CO_2 concentration of the reactor which has been chosen considering the CO_2 concentration of the emitted gas through the cement plants, run from 0 to 15 ± 2% via a concentrated CO_2 tank [40]. The pressure of the reaction medium is arranged with an air tank and can be set from 1 to 6 bar. The reactor has a cylindrical steel chamber with a capacity of approximately 25 l. However, the filling ratio of the reactor for effective process control is up to ~10%. In order to keep the CO_2 contact at a high level and to provide homogeneous carbonation, a mixer pro-

Fig. 1. Views of concrete rubbles and RCAs in different particle sizes.

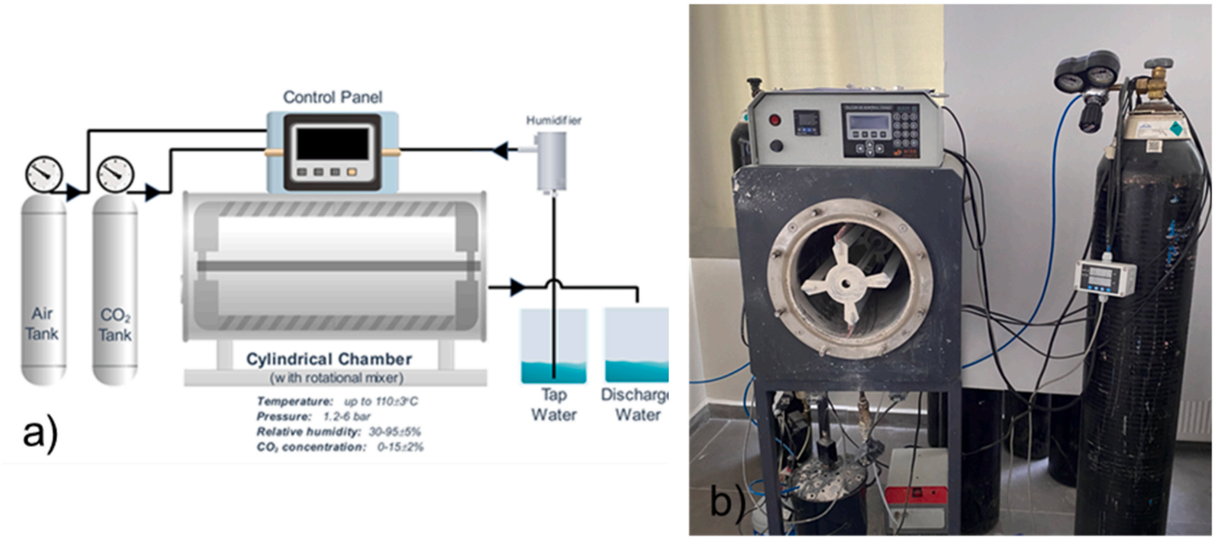

Fig. 2. a) Schematic view of carbonation chamber setup, b) the view of the carbonation chamber.

viding rotational mixing was designed for the carbonation reactor. The rotation speed of the mixer can be arranged in a range of 0–1 rpm. There is a control panel that stabilizes the interior of the chamber through sensors and adjusts the interior conditions.

In this study, to optimize the carbonation efficiency of RCAs, a series of carbonation processes were implemented on the low-quality RCAs under various operational conditions arranged by the pre-set parameters summarized in Fig. 3. Process parameters were designed comprehensively considering the industrial implementation and emitted flue gasses [40]. In total, four different cases were designed to investigate the effects of relative humidity, temperature, carbonation duration, pressure, and particle size of RCAs on the carbonation efficiency of the low-quality RCAs. When designing these four cases, it was aimed to prevent a sharp increase in the effort to figure out the optimum carbonation process with the rise of the number of independent variables. In this regard, in favor of simplifying the experimental program, the main effective parameters were treated as independent variables. The relevant parameter was investigated by fixing some other parameters that will be investigated in the next cases.

2.3. Specimen preparation

To reveal the performance of carbonated RCAs in the cement-based matrix, the mortar specimens were prepared with CEM I 42.5R Portland cement (PC), RCA (carbonated or uncarbonated), and tap water without any chemical admixtures. EN 196-1 standard was followed in the preparation of the mortar mixtures. The mixtures consisted of 450 g of PC, 225 g of tap water, and 1350 g of RCAs.

Test Methods.

To investigate the effects of various chemical carbonation processes implemented on the low-quality RCAs, several microstructural analyses were conducted on the carbonated and uncarbonated RCAs, including thermogravimetry (TG/DTG) analysis, scanning electron microscopy (SEM) analysis, and X-ray diffraction (XRD) analysis. Water absorption capacity test and compressive strength test were also performed for performance assessment.

2.3.1. Thermogravimetry analysis

Thermogravimetry (TG/DTG) analysis was performed to assess the chemical composition of carbonated and uncarbonated RCAs by focusing on the consumption of calcium hydroxide (CH) and the formation of different forms of calcium carbonate (CC). For the analysis, the RCAs were first crushed to obtain powder specimens and then dried in a laboratory-type oven at 40 °C. The RCA specimen with a weight of 10–30 mg was used for testing. In each analysis, at least three separate specimens were used. Analysis was carried out on the specimens by heating up to 1000 °C from 40 °C at the rate of 10 °C/min by using Shimadzu DTG-60H. Heat flow-temperature and weight-temperature data graphs were obtained and used to assess the efficiency of carbonation process parameters. The fluctuations observed in these graphs were used to determine the amounts of CH and CC in the sample. The weight loss observed in the temperature range of 375–450 °C (Δm375-450) indicates the decomposition of CH. In addition, $CaCO_3$ is characterized by weight loss in the temperature range of 600–890 °C(Δm600-890). In this temperature range, CO_2 in the CC structure dissolves and decomposes [41,42]. To calculate the CO_2 uptake rate of RCAs after the accelerated carbonation process, the differences between CC amount of carbonated and uncarbonated RCAs were considered [43].

2.3.2. Scanning electron microcopy analysis

A scanning electron microscopy (SEM) was used to examine morphological changes because of the carbonation process. The SEM examination was performed under vacuum conditions with a working distance of 10 mm. RCA samples were dried in a 40 °C oven before analysis. The morphologies of carbonation reaction products were observed in dried samples.

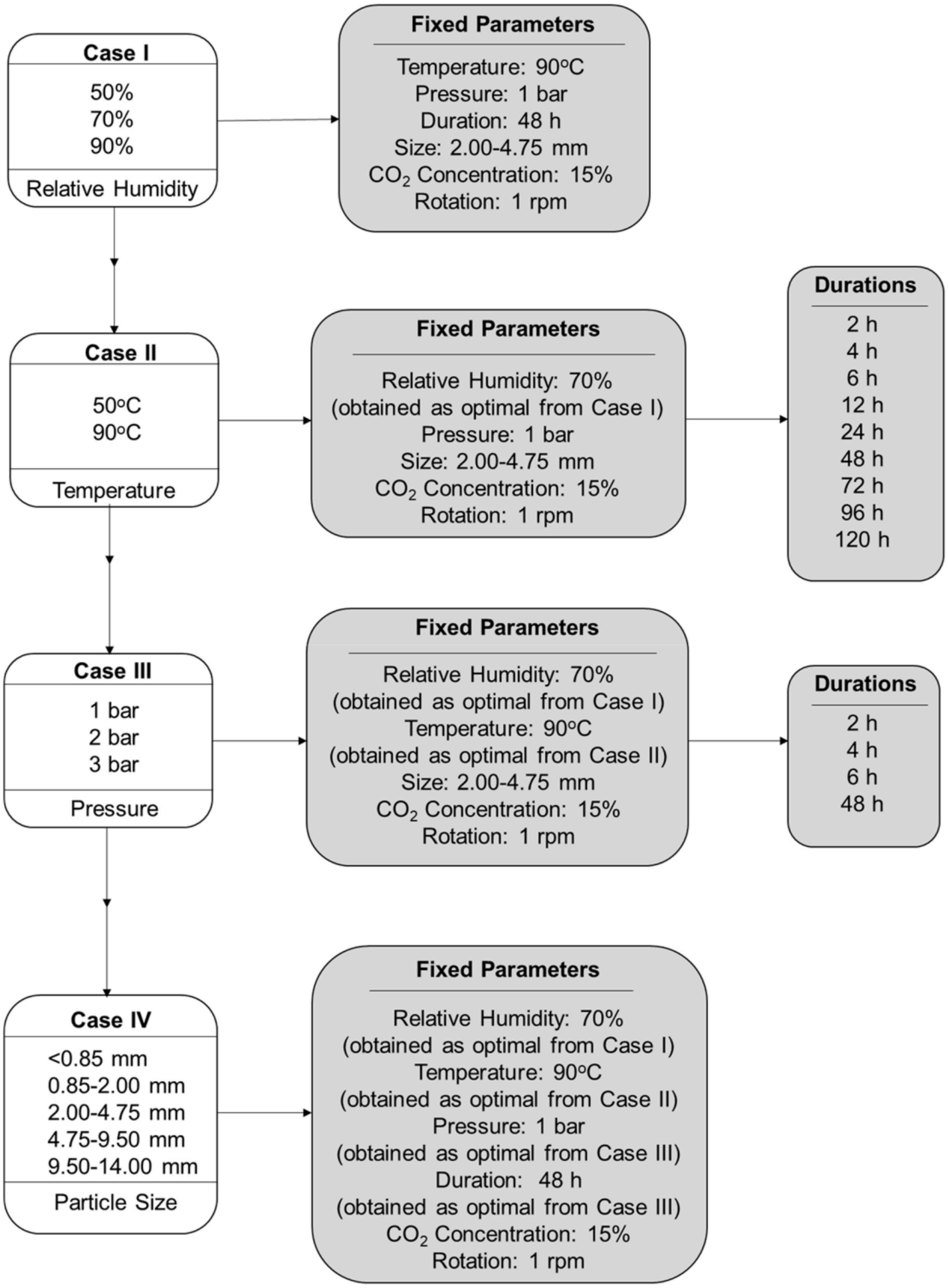

Fig. 3. Flowchart of parameters selected for different carbonation cases.

2.3.3. *X-ray diffraction analysis*

The crystalline structures of carbonated and uncarbonated RCAs were assessed by X-ray diffraction (XRD) analysis by focusing on the changes in crystalline peaks. Before analysis, materials were dried at 40 °C in a laboratory-type oven. The dried samples were then scanned between angles of 10–55° with an increase of 0.02°.

2.3.4. *Compressive strength test*

The compressive strength tests were performed on the 7-day and 28-day-old 50 mm-cubic specimens to evaluate the differences in the mechanical performance of mortar mixtures containing carbonated and uncarbonated RCAs. This test was conducted by using a compression device with a capacity of 100 kN in accordance with the ASTM C109 standard. A loading rate of 0.9 kN/s was used during testing. Six replicates were tested for each mixture and curing age, and the results were averaged.

2.3.5. Workability test

A workability test was performed on mortar specimens produced with uncarbonated RCAs and carbonated RCAs having the highest CO_2 uptake capacity by following the procedure given in ASTM C1437.

2.3.6. Water absorption test

Water absorption capacity test was carried out to assess the surface modification level of RCAs after the carbonation process. This experiment was conducted by following the procedure given in ASTM C128.

3. Experimental results

3.1. Effect of relative humidity

The carbonation efficiency of RCA significantly depends on the relative humidity of the carbonation medium as water is a vital player in the chemical reactions of carbonation and diffusion of CO_2 into the inner parts of RCA [28,44,45]. In the carbonation chamber, the solubility of reactants (e.g., CO_2) decreases at the lower RH because of the presence of limited solvents in the medium [39,46,47]. However, the diffusion of CO_2 into RCA is restricted at the extremely high RH due to the formation of a liquid layer on the pore surface (pore blockage by water presence), preventing the diffusion of CO_2 [29,48,49]. Considering these, optimal RH determination is crucial for the efficient carbonization of RCA. To clarify the effects of the RH on the carbonation efficiency of low-quality RCA carbonated under 15% CO_2 concentration, 90 °C temperature, and 1 bar pressure conditions, the obtained CO_2 uptake rates are given in Fig. 4. Results showed that the CO_2 uptake level increased with the increase in the RH up to 70%. This could be attributed to the increment of solvent materials in the reaction medium, enabling more carbonation reaction and CC precipitation [28,46]. The decrement in CO_2 uptake was observed with the increase in RH from 70% to 90%. The increment in humidity may cause lower permeation of CO_2 into the inner zones through cracks, leading to less precipitation of CC [29,48,49]. Considering the uncarbonated RCA, the CO_2 uptakes were recorded as 3.72%, 7.40%, and 6.36% for the RCA carbonated under the RH condition of 50%, 70%, and 90%, respectively. The water absorption capacity results of the uncarbonated and carbonated RCAs were also presented in Fig. 4. Similar trend was observed compared to TGA results for the varying RH level. Results showed that CC precipitation fills the cracks/pores of RCA, leading decrement in the water absorption capacity of carbonated RCAs [23]. The decrement in the water absorption of RCA considering the uncarbonated RCA was recorded as 17%, 32.7%, and 28.2% for the 50, 70, and 90% RH, respectively. According to the CO_2 uptake and water absorption results, the optimum RH level was obtained as the RH of 70% for the carbonation condition designed for this case, which is consistent with the literature studies [48,50]. However, although it was expected to obtain more CO_2 uptake and less water absorption capacity from the carbonated RCA under 50% RH compared to that under 90% RH, the opposite is the case for the low-quality RCA carbonated under the predefined condition of 15% CO_2 concentration, 90 °C temperature, and 1 bar pressure. The possible reason for obtaining such a result could be attributed to that high water content may benefit carbonation at elevated temperatures compared to the lower water content presence in the medium. Because in high temperatures, diffusion of CO_2 and the leaching of calcium ions accelerate, but the solubility of CO_2 and calcium ions in liquid decreases [51]. To overcome the decreased solubility because of elevated temperature, a high-water content in the medium can benefit the carbonation reactions. Therefore, RH level of 90% yielded better carbonation efficiency compared to the 50% RH, yet 70% RH provided the optimal water content that balances the solubility and diffusion process during the carbonation reactions.

3.2. Effect of pressure

In this study, 1, 2, and 3 bar (0.10–0.20-0.30 MPa) CO_2 and dry-air mixed gas pressure were examined throughout 48 h-duration. Fig. 5 depicts how the pressure affects RCA carbonation performance and water absorption capacity. When the pressure is increased, generally more CO_2 is dissolved in the pore solution [52], resulting in faster carbonation reactions [53,54]. However, there is an optimum pressure for exposing the RCA to CO_2, and excessive pressure can degrade the RCA's characteristics. This could be due to excessive pressure causing new cracks to develop in the attached old RCA mortar, resulting in significant porosity and water absorption of

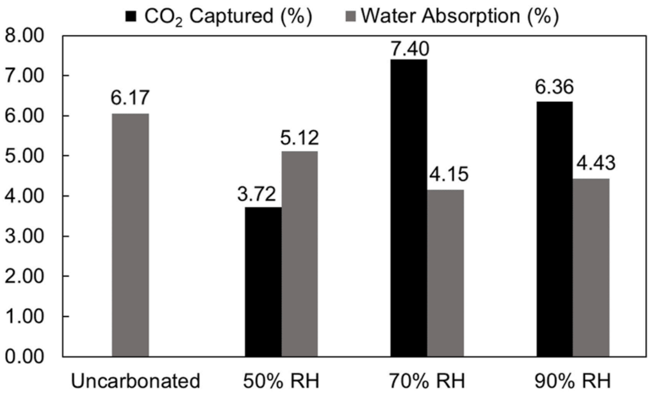

Fig. 4. Comparison of relative humidity conditions in terms of captured CO_2 and water absorption capacities.

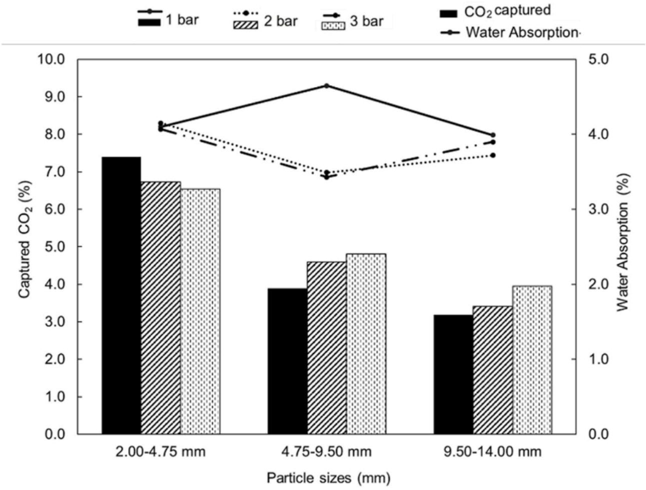

Fig. 5. CO_2 capture rates (represented in columns) and water absorption values (presented in lines) of three different particles size under 1,2 and 3 bar pressure for 48 h.

the RCA [55–57]. The formation of new cracks and new dust content with some pressure increase becomes even more effective for such low-quality RCAs.

As illustrated in the graph, the connection between CO_2 uptake and water absorption is not always linear. This relationship may be affected by parameters such as particle size and source of RCA [31,47,55]. Also, Fig. 5 shows that after 48 h of the carbonation process, the maximum CO_2 uptake performance was obtained from RCAs with the particle size range of 2.00–4.75 mm carbonated under 1 bar of pressure. On the other hand, the lowest CO_2 uptake was observed with the particle sizes range of 4.75–9.50 mm and 9.50–14.00 mm carbonated under 1 bar pressure. The CO_2 uptake rate reduced for the RCA having a particle size range of 2.00–4.75 mm when the pressure was increased from 1 bar to 2 bar. This decrease with pressure increase can be attributed to the fact that under high pressure, some vulnerable RCAs may be broken, and undesirable fine-grained materials may form and clog the fine pores, preventing CO_2 diffusion into deeper parts of RCAs. It can be stated that the pores in this size are much smaller than the pore structure in coarser RCA particles, which causes this effect to be negative in the size of 2.00–4.75 mm. On the other hand, it can be concluded that undesirable damage and formation of fine-grained materials due to high pressure create additional surface area for CO2 capture. However, the results confirm the conclusion that this surface already contains residual mortar whose carbonation is almost complete. Accordingly, with low pressure, remarkable CO_2 uptake performance can be expected in small-sized RCAs [30,58].

On the other hand, as the aggregate size increased (>4.75 mm), a slight increase in pressure resulted in both an increase in CO_2 capture rate and a decrease in water absorption capacity. For the RCAs with the particle size ranges of 4.75–9.50 mm and 9.50–14.00 mm, when the pressure was changed from 1 bar to 2 bar, approximately an 18% and 7% increase in CO_2 uptake rate and 26% and 3% decrease in water absorption capacity was observed, respectively.

Furthermore, at all three pressures, a high carbonation rate occurred in the first 6 h of carbonation, and the reduction in water absorption capacity of the samples exposed to 2 and 3 bar pressure was rapid in the first hours and slow in the subsequent hours (Fig. 6). Carbonation/the amount of captured CO_2 significantly increased over time, but a slower rate of increment was observed in the following periods. Additionally, the decrease in water absorption capacity in the following hours may be related to the rapid filling of surface voids and the slower absorption of CO_2 in the first few hours. It has been observed that RCAs with higher CO_2 uptake percentages

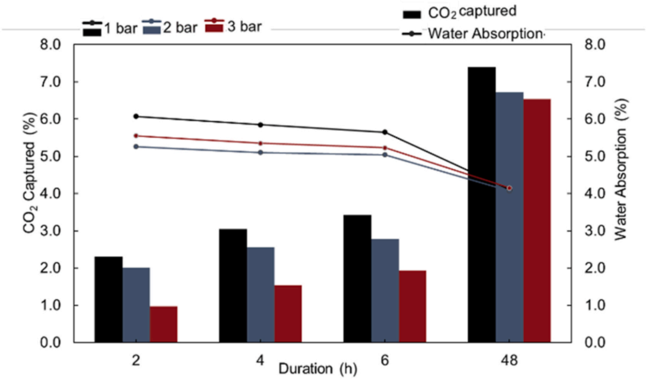

Fig. 6. Results of 2.00–4.75 mm size RCA for water absorption (lines) and CO_2 captured rate (columns).

also have higher water absorption values (Fig. 6). This is because the increased water absorption values of the RCA indicate a greater level of open porosity within the RCA that could allow CO_2 to penetrate the RCA. Even after most of the open porosity in the RCA is filled with free water, CO_2 still cannot freely enter the RCA [28]. In this case, CO_2 diffusion in the saturated RCA restricts the carbonation process of the interior of the RCA; this is also the non-linear relationship between the percentage of RCA carbonation and water absorption. Besides, the decrease in CO_2 uptake rate exhibited in Figs. 5–6 with increasing pressure can be attributed to the breaking of some brittle soft RCAs, under high pressure, as well as the development of undesirable fine-grained compounds and clogging of the pores [30]. While positive gains in water absorption and CO_2 uptake were noted with increasing pressure in the 4.75–9.50 mm and 9.50–14.00 mm grain sizes, it was observed that pressure had no noticeable effect on the carbonation process of fine-grained RCAs (< 4.75 mm).

Effect of Temperature and Duration:

The temperature is also one of the most critical factors affecting the carbonation efficiency of RCAs [51,59]. Therefore, in this study, the carbonation potential of RCAs was investigated considering the varied temperatures. In this regard, two different carbonation process temperatures, including 50 °C and 90 °C were performed. These carbonation processes were implemented with adjusted RCA particle size fraction (2.00–4.75 mm), CO_2 gas concentration (15%), pressure (1 bar), and relative humidity (70%) through different durations of carbonation treatment (2, 4, 6, 12, 24, 48, 72, 96 or 120 h). The CO_2 uptake rates and water absorption capacities of both uncarbonated RCAs and RCAs carbonated under different carbonation processes varied according to the temperature and carbonation treatment duration are shown in Fig. 7. As shown in Fig. 7, an increase in the carbonation process temperature from 50 °C to 90 °C favored the overall carbonation of RCA although the differences among them were not significant. Favoring effect of temperature was more evident for the specimens treated for up to 48 h. This favorable effect of the increased temperature on the carbonation rate can be attributed to the acceleration of diffusion of CO_2 and the migration of the materials, leaching of calcium ions, and thereby acceleration of carbonation reactions [29,31,51,60–63]. When the CO_2 uptake rates of RCAs carbonated during different carbonation treatment durations are evaluated from Fig. 7, it can be clearly seen that a longer carbonation process induced a higher CC precipitation rate, which means a higher CO_2 uptake level. This finding was also reported by several other researchers in the available literature [32,60,64–67] and can be attributed to the increase in the amount of carbonation reaction between CO_2 and relevant reagents available on the RCAs' surface owing to the increasing the specimens' exposure time to CO_2. The increase in the carbonation rate associated with the increased carbonation duration was more pronounced for up to the carbonation duration of 48 h. Namely, the carbonation reactions continued progressively at the RCAs' surfaces attached to the mortar residues in the first 48 h. After 48-h of the carbonation process, the CO_2 uptake became relatively stable, irrespective of the carbonation process temperature. This result was most probably due to the formation of a passivating layer of carbonation products on the cement-based mortar residues affecting diffusion phenomena of CO_2 as the carbonation progressed over time [58,60,68–71]. Products occurring from the carbonation process resulted in progressive densification of the micro-structure of the cement-based zones on the RCAs' surfaces and coating of them. This caused delaying the leaching of calcium ions and decrements in the rate of CO_2 diffusion and dissolution and undermining the specimens' exposure to CO_2. Thus, the carbonation reactions slowed down as a function of time yielding stabilization of CO_2 uptake over time. Based on this result, it can be concluded that it is necessary to determine the optimum CO_2 exposure time providing maximum penetration, interaction, and CO_2 absorption by cement paste particles attached on the RCAs.

When Fig. 7 are examined, it can be clearly seen that the overall pattern of the test results showing the water absorption capacities of carbonated RCAs was almost parallel to that of CO_2 uptake trend of them. As with the CO_2 uptake level, the water absorption capacities of RCAs were also fixed after a certain period of carbonation duration. Although this situation started from the 48th hour for the CO_2 uptake results, the stabilization in the water absorption capacities of RCAs started to occur from the 72nd hour, regardless of the carbonation process temperature. On the other hand, although higher CC precipitation rates were obtained from the RCAs carbonated under relatively higher carbonation treatment temperature level (90 °C), the water absorption capacities of these specimens were

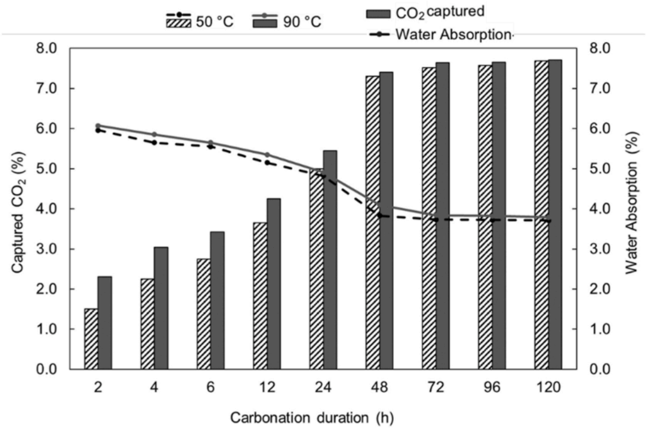

Fig. 7. The CO_2 captured rate (columns) and water absorption capacities (lines) of RCAs treated under different carbonation processes varied according to the temperature and carbonation treatment duration.

slightly higher compared to those of RCAs carbonated under the temperature of 50 °C. These results can be related to the form of the carbonation product occurring from the carbonation process. While more stable and denser polymorphs of the carbonation process is obtained at low temperatures, the formation of meta-stable forms of $CaCO_3$ can be induced under high temperatures [29,47,63,72–74]. Therefore, the higher water absorption capacities of the RCAs carbonated under the temperature of 90 °C can be related to their relatively higher content of meta-stable forms of $CaCO_3$.

3.3. *Effect of sizes of aggregates*

The particle size of RCAs is another parameter affecting the RCAs' CO_2 uptake rate during the accelerated carbonation process. Experimental results of this study also presented that the particle size of RCAs can influence the rate of calcium carbonate precipitation. According to CO_2 uptake results of RCAs with varying particle size ranges, a similar to normal distribution curve-shaped graph was observed (Fig. 8), meaning that in the results there is a diffraction point where the trend was reversed. This may be connected to the fact that RCA formed via the crushing process can vary the amount of residual mortar available on the RCAs due to different particle sizes. In this study, the RCAs were crushed and classified into five size fractions of <0.85 mm, 0.85–2.00 mm, 2.00–4.75 mm, 4.75–9.50 mm, and 9.50–14.00 mm. Among the particle sizes, 2.00–4.75 mm sized RCA stood out by having about 55.4%, 46.3%, 67.9%, and 60.3% higher CO_2 absorption than <0.85 mm, 0.85–2.00 mm, 4.75–9.50 mm, and 9.50–14.00 mm, respectively. The CO_2 uptake rates of RCAs exhibited an increase with the increase in the maximum particle size up to 2.00–4.75 mm, yet from 2.00 to 4.75 to 9.50–14.00 mm sized RCA showed a slight decrease in the carbonation performance [75]. The RCAs with particle size smaller than 2.00–4.75 led to more CO_2 uptake level compared to those with particle size higher than 2.00–4.75 mm [33,76].

As the particle size of RCAs decreases, the residual mortar content may increase, meaning the availability of more reactants for carbonation reactions. However, the finer particles may also contain unhydrated cement particles even in low-quality RCA that can produce hydration products because of water interaction [77]. This hydration reaction may cause the clumping of RCAs to each other, and so that prevents the diffusion of CO_2 [29,49,76]. Besides that, the RCAs with the finer size are prone to agglomerates in the presence of humidity, which is also supposed to be the main possible reason for the reduction in CO_2 uptake level from 2.00 to 4.75 mm to <0.85 mm. Compared to RCAs having a particle size range of <0.85 mm, a higher CO_2 uptake level was obtained from the RCAs with a particle size range of 0.85–2.00 mm. This could be attributed to the agglomeration degree of RCAs during the carbonation process, as seen in Fig. 9. On the other hand, RCAs larger than 4.75 mm showed a sharp decline in CO_2 uptake performance. This can be associated with the RCA's smaller specific surface area and fewer mortar residuals because of larger particles, thereby, less interaction capacity [33]. To measure the amount of residual mortar presence on the RCAs regarding the particle size, the proposed method, which is standardized guidelines provided in ASTM C88 [78] and ASTM C666 [79], was conducted by following the procedure given in a study [80] by using sodium sulfate solution. As seen from Table 1, the amount of residual mortar obtained at the end of the chemical and physical degradation process based on the aforementioned standards and method available on the RCAs with smaller particle size was higher than that with coarser particle sizes, which proves the statement expressed above. The precipitation of calcium carbonates at the surface of coarse RCAs may obstruct the CO_2 diffusion into inner zones through cracks after a certain degree of carbonation, which is also another reason for the low carbonation performance of coarse RCAs compared the fine RCAs which have a significantly higher surface area. Overall, the highest CO_2 uptake performance was recorded for the carbonated RCAs having a particle size range of 2.00–4.75 mm, meaning that a balance between the given opposing effects on carbonation was obtained for this particle size range.

4. Discussion

4.1. *X-ray diffraction (XRD) analysis*

The XRD patterns of the uncarbonated RCA and the carbonated RCA at temperatures of both 50 °C and 90 °C are presented in Fig. 10. During analysis, peaks corresponding to portlandite, calcite, aragonite, vaterite, calcium oxide, and monocalcium aluminate were

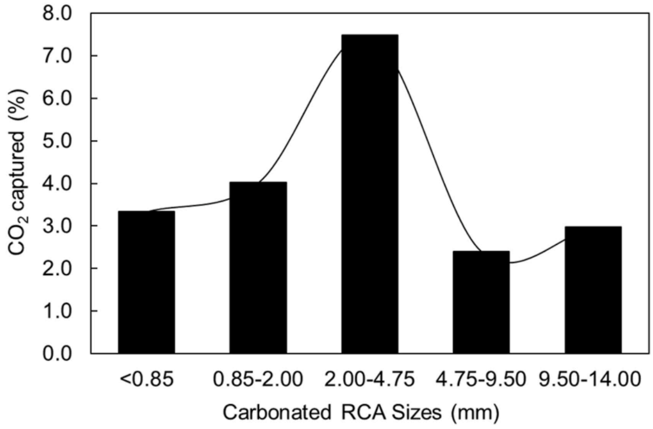

Fig. 8. CO_2 uptake percentages of carbonated RCAs-under 90 °C temperature, 70% RH and 1 bar pressure-with varying particle size.

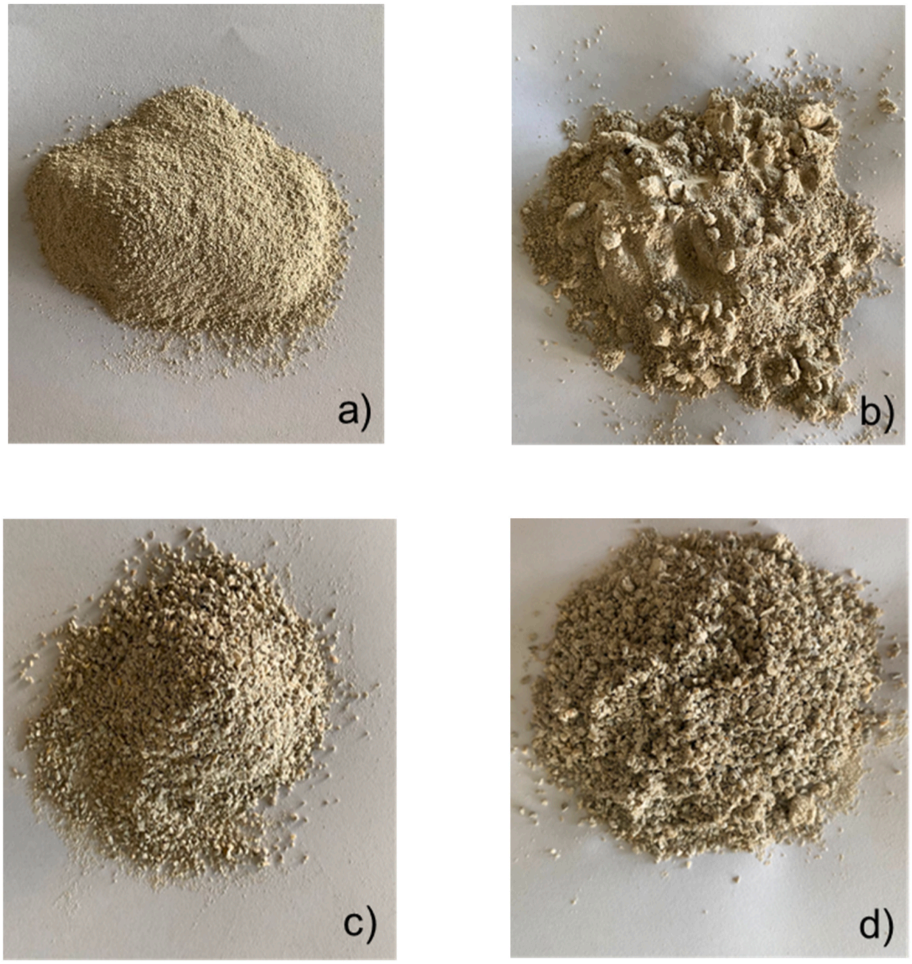

Fig. 9. Images of RCA; a) uncarbonated < 0.85 mm sized, b) carbonated and agglomerated < 0.85 mm sized, c) uncarbonated 0.85–2.00 mm sized, d) carbonated and agglomerated 0.85–2.00 mm sized.

Table 1
Residual mortar mass was obtained from 2.00 to 4.75 mm RCA and 4.75–9.50 mm sized RCA.

Sieve Opening (mm)	Retained Mass on Each Sieve	
	Coarse RCA	Fine RCA
4.75–9.50	159.6	–
2.00–4.75	39.1	123.7
0.85–2.00	28.2	41.0
< 0.85 (residual paste)	23.1	85.3
Initial Cumulative Retained Mass (g)	250	250
Residual paste (%)	9.24	34.1

detected. Intensified peaks of calcium carbonate in the form of calcite were observed for all tested specimens regardless of the accelerated carbonation process. The presence of calcite peaks for the uncarbonated specimen can be attributed to the original aggregate content of obtained RCAs. The natural carbonation process also can be another reason for the calcite peaks detected in XRD patterns of uncarbonated RCAs, as the used low-quality RCAs were obtained from the end-of-life structures served for years under the condition providing CO_2-exposing. The observation of calcite peaks in all RCAs prevented the evaluation of the carbonation potential of the RCAs in terms of calcite formation. The peaks representing crystal structures of portlandite, calcium oxide, and monocalcium aluminate recorded in the XRD pattern of uncarbonated RCA were not observed after the accelerated carbonation process regardless of temperature conditions because of the carbonation reactions [23,81,82–84]. While crystalline peaks belonging to different forms of calcium carbonate of aragonite and vaterite were detected for the RCAs carbonated under the temperature of 90 °C, only aragonite peaks were recorded for the XRD pattern of RCAs carbonated under the temperature of 50 °C. In the previous study of the authors, vaterite peaks were also not recorded for the RCAs carbonated under a different carbonation condition (in terms of CO_2 concentration, rela-

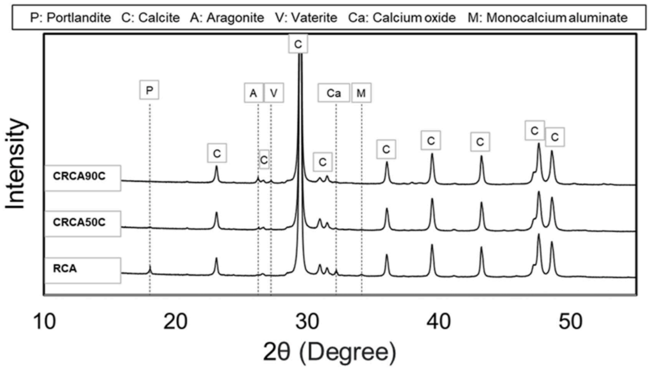

Fig. 10. XRD patterns of uncarbonated and carbonated RCAs under 1 bar,70% RH, 50 and 90°C.

tive humidity, duration, etc.) with a similar carbonation temperature of 50 °C [23]. These findings are supportive of the formation of varied forms of calcium carbonate regarding the temperature, which is also stated under the section regarding the effects of temperature [29,47,63,73,74]. Considering these results, it can be stated that high temperatures induce the formation of vaterite as a metastable form of calcium carbonates.

4.2. Scanning electron microscopy (SEM) analysis

SEM micrographs of RCA and carbonated RCA are shown in Fig. 11 to verify the forming of carbonation products on the surface of both treated and untreated RCA. For microstructural observation, due to its efficient CO_2 uptake results, 2.00–4.75 mm size, and RCA carbonated under 1 bar, 90 °C, 70% RH condition was selected. According to Fig. 11, on the surface of RCA, products of chemical carbonation reactions could be clearly observable.

Calcium carbonate was the primary carbonation product. The most stable phase of calcium carbonate is called calcite, which is often granular in texture and rhombohedral or cubic in form [85]. According to findings from a study [29], when RCA surfaces were treated with chemical carbonation, the surface of carbonated RCA became less porous. It was covered with visually-inspected granular-textured and cubic-shaped calcium carbonate crystals. Due to the existence of old mortar remains, the surface of the RCA was rough and irregular, as can be seen in Fig. 11a [86]. However, the RCA's EDX study findings also revealed that calcium carbonate was being produced on the surface, which may be related to either the chemical components of the RCA or calcite production as a result of CO_2 exposure over time [87]. On the other hand, the carbonated RCA surface's EDX results revealed higher calcite precipitation. The TG/DTG study reported in the previous section and the XRD analysis described in the previous section both supported the finding that this result was connected to the production of calcite on the carbonated RCA surface (Fig. 11b).

4.3. Compressive strength and workability

To investigate the effects of the inclusion of carbonated RCAs on the compressive strength and workability of cementitious system, mortar mixtures with constant water-to-cement ratio were produced with both 2.00–4.75 mm sized uncarbonated RCAs and RCAs carbonated under 70% RH, 1 bar, 90 °C for 48 h. While the average flow value of the mortar sample containing uncarbonated RCA was 115 mm, the average flow value of the sample containing carbonated RCA was 126 mm. The difference in the flow values of the cementitious systems was due to the carbonation resulting in the reduction in the presence of voids and permeability of the RCA, and therefore the decrement in the water absorption capacities of RCAs. (Pan et al., 2016). There are studies in which different w/c ratios are tested in mortar mixtures containing RCA. At different w/c ratios, increases in strength values can be seen due to the water absorption capacity of RCA. However, on the other hand, losses can also be observed in terms of durability [88]. Therefore, within the scope of this study, this parameter was kept constant, and only the carbonation effect was investigated as a preliminary.

The compressive strength results of uncarbonated RCA-incorporated mortar (M-URCA) and carbonated RCA-incorporated mortar (M-CRCA) were illustrated in Table 2. According to the results, the compressive strength of the specimens increased with the curing ages irrespective of the implementation of the carbonation process. When the results were examined by focusing on the effect of carbonation regarding compressive strength, it can be clearly seen from Table 2 that 7- and 28-day compressive strength of the mortar sample containing carbonated RCAs was higher than the that containing uncarbonated RCAs. These results prove that carbonation has an improvement effect on the old ITZ of the RCAs between aggregates and mortars. The mortar residues on the RCA surface with low density and high porous ITZ may cause low elastic modulus values and low strength values in mortars containing RCAs (Shi et al., 2018). Since ITZ is generally considered as the weakest region responsible for crack propagation, modification of the mortar residue on the RCAs and the old ITZ by carbonation results in the reduction of the void/cracked structure and cavity in these regions. This may yield more stable and robust RCAs. In this way, a significant increase in the strength of new mortars produced with carbonated RCA can be experienced [42,89]. The increase in strength can be attributed to the formation of a highly hexagonal crystalline calcite structure with carbonation, resulting in a denser ITZ [90].

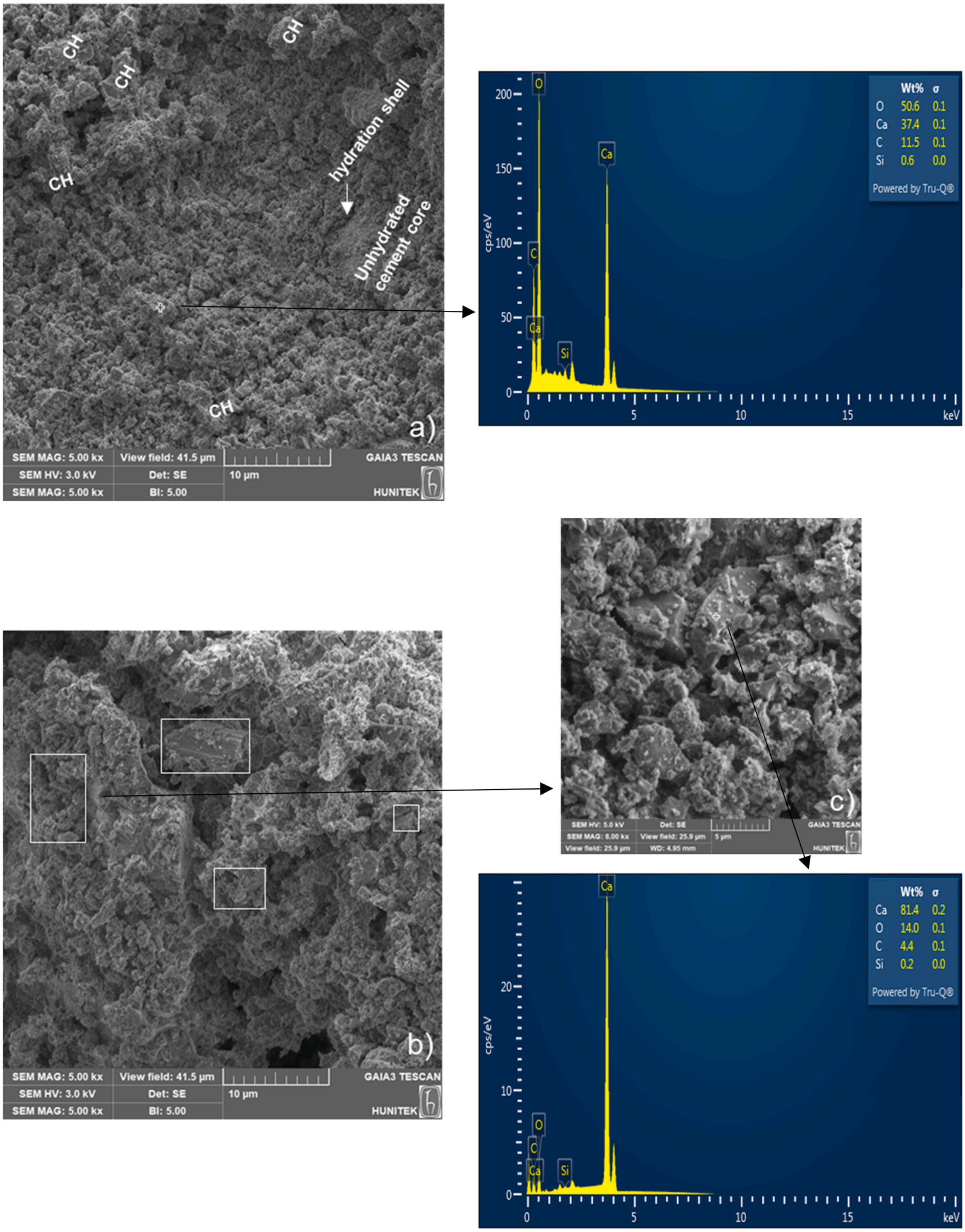

Fig. 11. SEM micrographs of (a) uncarbonated RCA, (b) carbonated RCA (squared area demonstrates extensive calcite precipitations).

5. Conclusion

This study investigates the effects of different carbonation process-related parameters including relative humidity, pressure, temperature, duration, and particle size of RCAs on the CO_2 uptake performance of low-quality RCAs obtained from end-of-life buildings. The following conclusions have been drawn from the experimental studies carried out within the scope of this research.

Table 2
Compressive strength test results of mortars containing uncarbonated RCAs or carbonated RCAs.

Mortar Samples	Compressive Strength (MPa)	
	7 days	28 days
M-URCA	23.1	32.2
M-CRCA	25.9	41.2

- Among the RH of 50%, 70%, and 90%, considering the CO_2 uptake level and water absorption capacity results, the RH level of 90% resulted in better carbonation efficiency compared to the 50% RH; however, 70% RH provided the optimal condition for carbonation.
- As the RCA size increases (>4.75 mm), the pressure increase is beneficial in terms of both CO_2 capture and water absorption capacity, but it does not provide a benefit in finer RCAs and even causes a decrease in CO_2 capture rate. In all sizes, there was a faster decrease in the water absorption capacity in the first hours of the curing period in 2 and 3 bar pressure environments, and this decrease slowed down in the following hours.
- An increase in the carbonation process temperature was found to be relatively favorable for the carbonation performance of the low-quality RCAs. Although calcite, aragonite, and vaterite were detected for the RCAs carbonated under 90 ˚C temperature, calcium carbonate in a form of vaterite was not observable in the uncarbonated RCAs and RCAs carbonated under 50 ˚C temperature.
- The carbonation performance of RCAs increased with the increase in carbonation duration. The carbonation reactions continued progressively in the first 48 h.
- Carbonated RCAs-incorporated mortar yielded higher workability performance compared to that containing uncarbonated RCAs. Owing to the carbonation, a significant increase in the compressive strength of mortars produced with carbonated RCA was also observed.
- The low-quality RCAs mentioned in this study have shown improvement in microstructural, physical, and mechanical aspects by studying the accelerated carbonation method comprehensively. Instead of being just a CDW-based waste, they can be actively used in the cementitious system and their adverse environmental effects can be reduced.

Author statement

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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.

Data availability

No data was used for the research described in the article.

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