

Article

Effect of the Concrete Slurry Waste Ratio on Supercritical CO₂ Sequestration

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Abstract: To prevent drastic climate changes due to global warming, it is necessary to transition to a carbon-neutral society by reducing greenhouse gas emissions in all industrial sectors. This study aimed to develop carbon utilization sequestration technology that uses the concrete slurry water generated during the production of concrete as a new CO₂ sink to reduce CO₂ emissions from the cement industry. This was achieved by performing supercritical CO₂ carbonation by varying the concrete slurry waste (CSW) ratio. The study's results confirmed that, according to the CSW ratio (5 to 25%), complete carbonation occurred within only 10 min of the reaction at 40 °C and 100 bar.

Keywords: concrete slurry water; concrete slurry waste; ready-mixed concrete; CO₂ sequestration; supercritical CO₂; net-zero

1. Introduction

Concrete is a major structural component of buildings and one of the most extensively used core materials [1–5]. The demand for concrete is expected to increase further owing to the increasing demand for social infrastructure and housing, continuous urbanization, and social development in developing countries [6–9].

Numerous studies have been conducted, and implementation strategies have been established worldwide to realize the 2050 carbon neutrality goals. Several initiatives have also been made in the cement industry to reduce greenhouse gas (GHG) emissions. However, global carbon emissions are increasing every year. In 10 major countries with high-GHG emissions (listed in Table 1), the carbon emissions per capita (as of 2021) are reported to exceed the global average (4.7 tons of CO₂ per capita) [10]. In South Korea, carbon emissions are also increasing every year. At the time of the Paris climate agreement, South Korea's GHG reduction target for 2030 was set at 37% compared with Business-as-Usual (BAU), but it was increased to 40% compared with 2018 considering the recent international trend toward GHG reduction. Accordingly, efforts have been expended in the cement industry to reduce CO₂ emissions, including the use of blended cement and fuel conversion to increase the thermal efficiency of kilns, but they are not sufficient to meet the reduction quota. CEMBREAU presented a carbon neutrality roadmap to achieving carbon neutrality in the cement industry. According to the roadmap, various carbon emission reduction measures were prepared from clinker to concrete levels, including the increased use of blended cement and fuel conversion to improve the thermal efficiency of kilns. However, these measures alone cannot achieve carbon neutrality; thus, it is essential to develop carbon capture, utilization, and storage (CCUS) technologies, such as mineral carbonation, to achieve considerable CO₂ reductions [11,12]. Figure 1 shows the carbon neutrality strategy of the cement industry presented by CEMBREAU.

Meanwhile, the production of ready-mixed concrete involves the generation of concrete slurry water because the returned/surplus concrete or the concrete attached to the truck agitator and batching plant mixer is washed [13–15]. CSW is mostly dehydrated cake produced with a filter press in a ready-mixed concrete plant after separating the fine and



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coarse aggregates in CSW. As CSW contains many unhydrated cement particles, studies on recycling CSW as a cementitious material are increasing every year [16–19]. Nevertheless, most of this research focuses on utilizing CSW as a filler or binder of cement matrices. However, as CSW contains a large amount of Ca^{2+} owing to unhydrated cement particles, it is expected to be highly applicable as a material for CO_2 sequestration [20,21].

Table 1. Carbon emissions per capita in major countries [10].

Country	CO_2 Emissions per Capita (tCO_2)	CO_2 Emissions (MtCO_2)
China	8.0	11,472.37
United States of America	15	5007
India	1.9	2710
Russian Federation	12	1756
Japan	8.6	1067
Iran	8.5	749
Germany	8.1	675
Saudi Arabia	19	672
Indonesia	2.3	619
South Korea	12	616

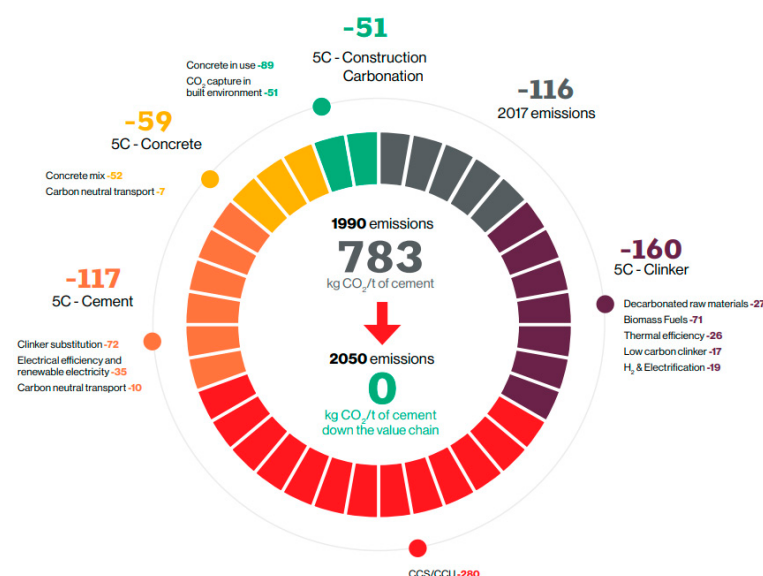


Figure 1. Carbon neutrality roadmap for the cement industry proposed by CEMBREAU [10].

Generally, the CO_2 mineral carbonation reaction at room temperature and atmospheric pressure is extremely slow and inefficient. Therefore, research on supercritical CO_2 mineral carbonation has grown to accelerate the reaction. According to previous studies on mineral carbonation using supercritical CO_2 , the carbonation efficiency increases with pressure even though there are no significant differences in the influences of temperature on the carbonation reaction in the supercritical state. In most studies, the temperature and pressure are set to be less than 80°C and 150 bar, respectively [22–25].

Therefore, in a previous study [26], mineral carbonation by supercritical CO_2 was performed for CSW, in which the CSW ratio was adjusted to 5% at a set temperature (40°C and 80°C) and pressure (100 and 150 bars) conditions based on previous studies. It was confirmed that complete carbonation occurred within only 10 min at 40°C and 100 bar.

However, it is essential to increase the proportion of CSW that contains a lot of Ca^{2+} to maximize the amount of CO_2 fixed in concrete slurry water. According to reports, Korea produces more than 30 million tons of concrete slurry water annually [27]. Therefore, it will be possible to sequester a significant amount of CO_2 generated from the cement industry if a supercritical CO_2 carbonation process capable of continuous processing in connection

with concrete slurry water in a ready-mixed concrete plant through CO₂ capture or bypass is developed, as shown in Figure 2.

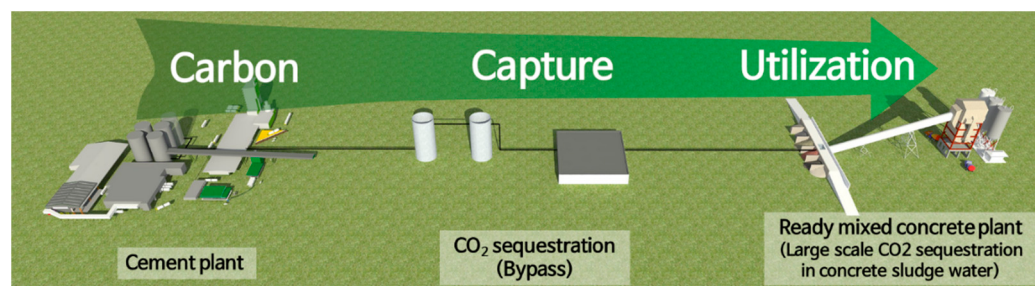


Figure 2. Schematic showing the sequestration of a large amount of CO₂ in concrete slurry water.

Therefore, in this study, the supercritical CO₂ carbonation reaction was performed for 10 min at 40 °C and 100 bar with CSW, in which the CSW ratio was adjusted to its maximum of 25% as part of enforced measures to reduce GHG emissions from the cement industry. The impact of the CSW ratio on supercritical CO₂ sequestration was also investigated using pH measurements, TG-DTA, and XRD analysis.

2. Experimental

2.1. Materials

CSW was collected from a ready-mixed concrete plant (company Y) located in Gyeonggi-do, Korea in the late afternoon, when the proportion of CSW was at its highest. Figure 3 shows the process of collecting CSW. For the CSW used in the experiment, supernatant water and CSW were separated to evenly adjust its ratio. The CSW was dried at 105 °C until a constant weight was reached. The dried CSW was then pulverized and adjusted using a No. 200 sieve (particle size $\leq 75 \mu\text{m}$). Dilution of the supernatant water and dried CSW to the target CSW ratios (5, 10, 15, 20, and 25%) was used to perform mineral carbonation based on the supercritical CO₂ reaction. Tables 2 and 3 show the chemical compositions of the supernatant water and CSW, respectively. As a result of measuring the chemical composition of CSW, it was determined that it can be sufficiently used as a CO₂ sequestration source, as it contains about 34% of the CaO component.

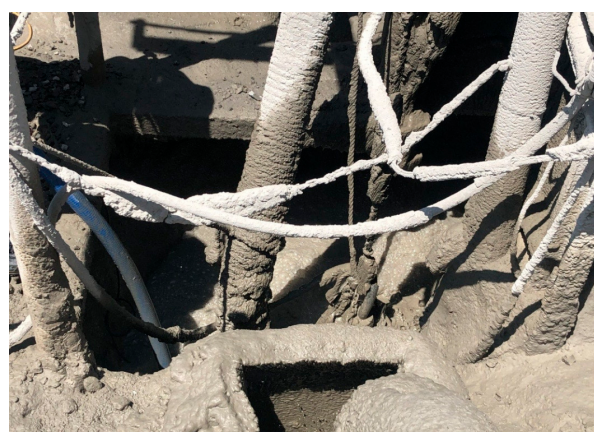


Figure 3. Concrete slurry water collection process.

Table 2. Chemical composition of supernatant water (obtained by ICP spectroscopy).

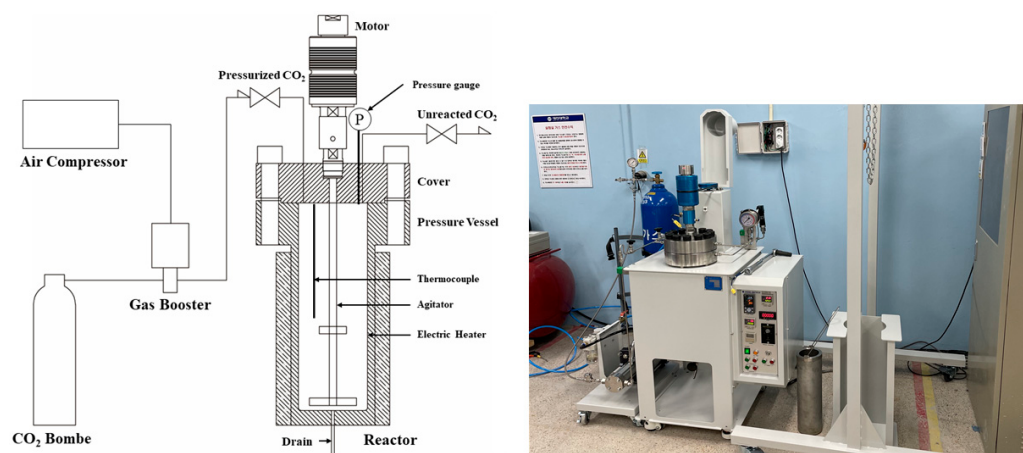
Chemical Composition (mg/L)				
Ca	Mg	Na	Fe	K
812	0	242	0	711

Table 3. Chemical composition of the concrete slurry waste (obtained by XRF spectroscopy).

Chemical Composition (wt.%)									
CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	K ₂ O	TiO ₂	Na ₂ O ₃	P ₂ O ₅
34.32	26.24	8.27	3.12	2.37	2.10	1.05	0.47	0.37	0.23

2.2. Supercritical CO₂ Reactor

Figure 4 shows a schematic of the supercritical CO₂ reactor used in this study. The supercritical CO₂ reactor consists of a gas booster (Maximator, Nordhausen, Germany) and a reactor (PHOS-ENTECH, Daejeon, Korea). A heating plate (used for temperature control) and an agitator are installed in the reactor, and a thermocouple and pressure gauge are used to measure the temperature and pressure, respectively. The gas booster is connected to the air compressor and is used to pressurize CO₂ gas into the reactor at a high pressure to maintain the supercritical CO₂ state. The maximum operating temperature and pressure of the supercritical CO₂ reactor are 80 °C and 200 bar, respectively, and the internal volume of the reactor was designed to be 4 L. The agitator's rotation speed can be adjusted to ≤400 revolutions per minute (rpm).

**Figure 4.** Schematic and photograph of the supercritical CO₂ reactor.

2.3. Supercritical CO₂ Carbonation

The carbonation test was conducted using supercritical CO₂ as follows:

The reactor was assembled after samples were added to it, in which the supernatant water and CSW had been diluted at different ratios (10, 15, 20, and 25%). When the interior of the reactor reached the target temperature of 40 °C, the electric heater was activated, and CO₂ was injected until the target pressure of 100 bar was reached. When the CO₂ inside the reactor reached the target pressure, the agitator was operated at 200 rpm, and accelerated carbonation was performed over a predetermined reaction time (10 min), while the temperature and pressure were maintained. After the predetermined reaction time, CO₂ was discharged, and the reactor was disassembled to recover the sample. The supernatant water and solid content in the sample were separated, and the solid content was dried at 105 °C until it reached a constant weight. The dried solid content was subjected to pH (Hanna Instruments HI2215, Woonsocket, RI, USA), SEM (Philips XL30 ESEM, Eindhoven, The Netherlands), XRD (Rigaku D/max 2200 + Ultima, Tokyo, Japan), and TG-DTA (NETZSCH STA2500 Regulas, Germany) analysis to quantitatively evaluate the degree of carbonation reaction.

2.4. Measurement of Mineralogical Property Changes

The pH, SEM, XRD, and TG-DTA measurements were performed for samples before and after the carbonation reaction with supercritical CO₂ to measure changes in the mineralogical properties of CSW.

The samples were diluted with distilled water at a 1:5 ratio before pH measurements were conducted.

TG-DTA measurements were conducted in the temperature range of up to 1000 °C at a rate of 10 °C/min in a nitrogen atmosphere to calculate the amount of CaCO₃ generated following the reaction.

3. Results and Discussion

3.1. PH Measurements

Figure 5 shows the pH measurement results before and after supercritical CO₂ carbonation. The specimen's pH was measured to be greater than 12 before the reaction, but regardless of the CSW ratio, it ranged from 9 to 9.5 after the reaction. Typically, during the cement hydration process, Ca(OH)₂ is produced and the pH is increased. Ca(OH)₂ is converted into CaCO₃ in a CO₂-containing environment that exists during the carbonation process, as shown in Equation (1), thus resulting in a pH reduction. In this study, it appears that the carbonation reaction also caused the pH of the concrete slurry water to decrease. The pH of high-purity CaCO₃ is known to be 9.4. It was determined that the conversion into CaCO₃ occurred because the pH of the reaction product after supercritical CO₂ carbonation ranged from 9.0 to 9.5.

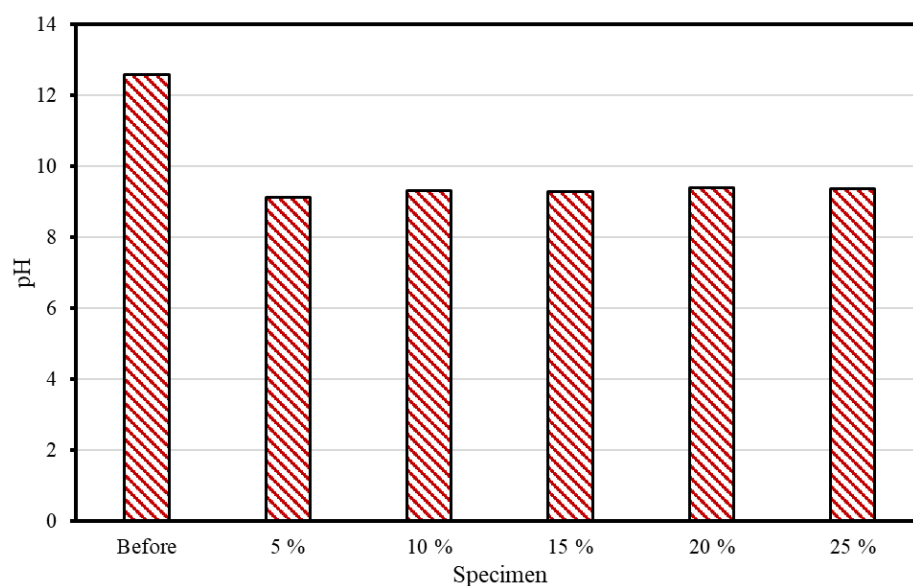


Figure 5. pH measurement outcomes.

3.2. XRD Results

Figure 6 shows the XRD measurements before and after supercritical CO₂ carbonation. In the XRD measurement results, the Ca(OH)₂ peak and a small amount of the calcite peak were detected from the solid sludge content before the reaction.

However, following the reaction, no Ca(OH)₂ peak was detected, and the calcite peak was dominant in conjunction with the small aragonite peak. Additionally, similar peaks were observed regardless of the CSW ratio. CaCO₃ is divided into aragonite, vaterite, and calcite depending on the crystal structure, and calcite is reported to be the most stable form [28]. Furthermore, a previous study reported that calcite is mainly formed when $\text{Ca}^{2+}/\text{CO}_3^{2-} \leq 1$. It was determined that calcite was also mainly generated as a reaction product in this study because a significant amount of CO_3^{2-} was generated at the supercritical CO₂ condition that caused the $\text{Ca}^{2+}/\text{CO}_3^{2-}$ ratio to decrease [24]. Meanwhile, quartz peaks could be confirmed both before and after the reaction. It was determined that this is due to the SiO₂ component caused by the fine powder of the aggregate mixed in concrete slurry water [29].

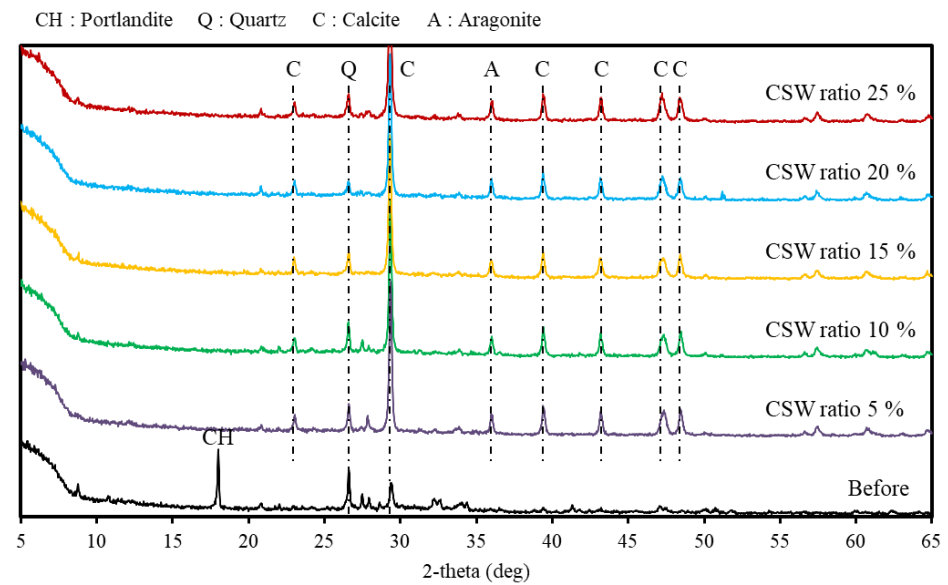


Figure 6. X-ray diffraction results.

3.3. TGA Results

Figure 7 shows the TG-DTA measurements before and after supercritical CO_2 carbonation. $\text{Ca}(\text{OH})_2$, the main hydrate of cement, thermally decomposes between 400°C and 550°C , and CaCO_3 , which is generated following the reaction with CO_2 , thermally decomposes between 600°C and 800°C . In the TGA results, small weight losses of $\text{Ca}(\text{OH})_2$ and CaCO_3 were observed in the sample before supercritical CO_2 carbonation. However, in the samples after supercritical CO_2 carbonation, the weight loss of $\text{Ca}(\text{OH})_2$ could not be detected, and only the weight loss of CaCO_3 was confirmed regardless of the CSW ratio.

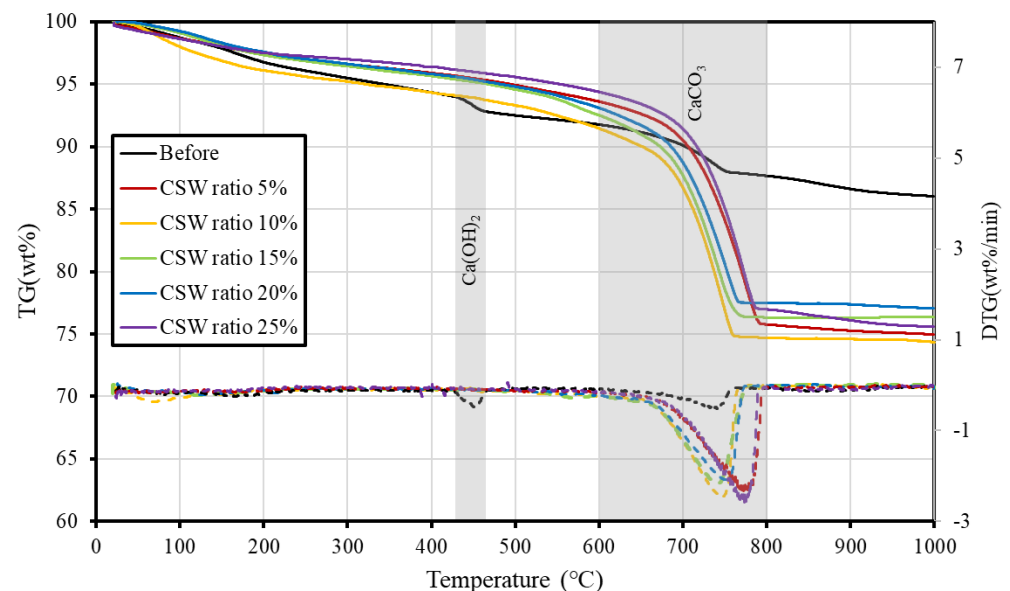


Figure 7. TG-DTG results (solid line: TG, dashed line: DTG).

In the typical carbonation reaction, the formation of CaCO_3 particles from the surface layer of particles reduces the carbonation rate in the typical carbonation reaction by inhibiting the dissolution of ions and the carbonation rate. However, according to previous studies [30,31], CaCO_3 that has been generated on the surface of particles after carbonation can be removed through agitation; this may improve the carbonation rate by accelerating ion diffusion. Because supercritical CO_2 is highly reactive and the agitation accelerates ionic

diffusion, it is determined that complete carbonation also occurred in this study within only 10 min of carbonation reaction for CSW ratios up to 25%.

3.4. Evaluation of CO₂ Sequestration

While there are many research reports on concrete carbonation using supercritical CO₂, most studies focused on the changes in the mechanical properties and microstructure attributed to the supercritical CO₂ carbonation of concrete [32–34]. Conversely, this study investigated a method for reducing industrial CO₂ emissions and realizing net-zero emissions by developing a carbon utilization and sequestration technology that utilizes the CSW generated during concrete production as a new CO₂ sink using the supercritical CO₂ reaction.

Typically, it is known that when accelerated carbonation is performed on Ca(OH)₂ or cement particles, a microcrystalline layer of CaCO₃ is formed on the particle surfaces; as the reaction proceeds, the densification of CaCO₃ particles in the surface layer causes the reaction to decrease gradually [22,35–38]. In this study, however, even when the CSW ratio was increased to 25%, complete carbonation occurred within only 10 min, and the reaction rate did not decrease. Previous studies [28,39] reported that when stirring is performed during the carbonation reaction, CaCO₃ formed on the surface layer is separated from the surface of Ca(OH)₂ particles, and the carbonation of Ca(OH)₂ is promoted. Moreover, supercritical CO₂ is known to be approximately 10 times more soluble in water than its dilution rate in natural carbonation conditions, and studies have reported that this can accelerate the carbonation reaction [40,41]. Accordingly, in this study, as stirring was performed during supercritical CO₂ carbonation, CaCO₃ particles were separated and the complete carbonation of Ca(OH)₂ occurred despite the increased CSW ratio due to the increase in CO₂ solubility within the CSW in supercritical CO₂ conditions.

Meanwhile, the most common method used to evaluate the amount of sequestered CO₂ through CCUS was the CaCO₃ weight loss ratio achieved based on TGA analysis. Using the amount of CO₂ produced by CaCO₃ pyrolysis in the temperature range of 600–800 °C, the CO₂ sequestration can be calculated by comparing the weight loss in this temperature range before and after the reaction; correspondingly, in the temperature range of 400–550 °C, the CO₂ sequestration can be calculated by using the amount of H₂O evaporation by Ca(OH)₂ pyrolysis. Additionally, the Ca(OH)₂ and CaCO₃ contents can be calculated using Equations (2) and (3) based on the TGA results.

$$Ca(OH)_2 \text{ experiment} = (\Delta 400 \sim 550 \text{ }^{\circ}\text{C}) \times \frac{MW_{Ca(OH)_2}}{MW_{H_2O}} \quad (2)$$

$$CaCO_3 \text{ experiment} = (\Delta 600 \sim 800 \text{ }^{\circ}\text{C}) \times \frac{MW_{CaCO_3}}{MW_{CO_2}} \quad (3)$$

As shown in Table 4, based on the TGA results in this study, an average weight loss of 15.67 wt% was calculated within the temperature range of 600–800 °C after supercritical CO₂ carbonation, and the weight loss before the reaction was 2.79 wt%. This indicates that the CO₂ sequestration ability of solid sludge is approximately 128.8 g per 1 kg. The theoretical CO₂ sequestration based on the CaO content of solid sludge (see Equation (4)) [12] is 130.7 g per 1 kg of solid sludge. The CO₂ sequestration calculated based on the experiment was almost identical to the theoretical at 128.8 g, thus indicating that complete carbonation occurred.

$$CO_2 \text{ theoretical} = CaO(\%) \times \frac{MW_{CO_2}}{MW_{CaCO_3}} \quad (4)$$

Table 4. Amounts of Ca(OH)_2 and CaCO_3 before and after supercritical CO_2 carbonation.

	400–550 °C	Amount of Ca(OH)_2	600–800 °C	Amount of CaCO_3
Before	1.1	4.57	2.79	7.04
CSW 5%	0	0	15.94	40.20
CSW10%	0	0	15.84	39.94
CSW 15%	0	0	15.12	38.13
CSW 20%	0	0	15.21	38.36
CSW 25%	0	0	16.23	40.93

4. Conclusions

The following conclusions were drawn according to the experimental results of this study.

1. When quantitative analysis (pH, XRD, and TGA) was conducted after performing the mineral carbonation of concrete slurry water by supercritical CO_2 , Ca(OH)_2 was detected before the carbonation reaction; however, this outcome was not confirmed, and only the presence of CaCO_3 was detected after the carbonation reaction regardless of the CSW ratio, thus confirming the occurrence of complete carbonation. It appears that complete carbonation occurred because supercritical CO_2 is highly reactive and agitation accelerates ionic diffusion. Future studies must quantitatively analyze the CO_2 sequestration of concrete slurry water to evaluate the possibility of reducing CO_2 emissions from the cement industry.
2. Future research will be conducted to develop a continuous process capable of supercritical CO_2 carbonation for CSW. By developing this technology, CO_2 captured in cement and other industries can be transported to ready-mixed concrete plants near bases and sequestered based on rapid supercritical CO_2 carbonation. Ultimately, the utilization of CO_2 generated in cement and other industries will greatly contribute to the effective utilization of waste in the ready-mixed concrete industry and carbon neutrality.

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