



Article Carbon Dioxide Capture and Product Characteristics Using Steel Slag in a Mineral Carbonation Plant

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Abstract: Carbon capture and storage (CCS) technology can reduce CO_2 emissions by 85 to 95% for power plants and kilns with high CO_2 emissions. Among CCS technologies, carbon dioxide capture using steel slag is a method of carbonating minerals by combining oxidized metals in the slag, such as CaO, MgO, and SiO₂, with CO₂. This study assessed the amount of CO₂ captured and the sequestration efficiency in operating a mineral carbonation plant with a CO₂ capture capacity of 5 tons/day by treating the exhaust gas from a municipal waste incinerator and identified the characteristics of the mineral carbonation products. As a result, the average concentration of CO_2 in the inflow and outflow gas during the reaction time was 10.0% and 1.1%, respectively, and the average CO_2 sequestration efficiency was 89.7%. This resulted in a conversion rate of CaO of > 90%. This study manifested that mineral carbonation products are more stable than steel slag as a construction material and are effective at sequestering CO_2 by forming chemically stable $CaCO_3$.

Keywords: carbon dioxide; steel slag; mineral carbonation; calcium oxide; calcium carbonate

1. Introduction

Global atmospheric carbon dioxide (CO₂) concentration has steadily increased due to the use of fossil fuels, leading to average annual CO_2 emissions from 11 Gt in the 1960s to 35 Gt in the 2010s [1]. The International Energy Agency (IEA) reported that global CO₂ emissions in 2021 were 36.3 gigatonnes (Gt), a 6% increase over 2020 [2,3]. The atmospheric CO_2 concentration hit the highest mark of 414.72 ppm in 2021 despite the economic recession from the COVID-19 pandemic [1]. Furthermore, continued increases in CO_2 can lead to acidification of the oceans, which is a growing concern for marine ecosystems [1]. The Intergovernmental Panel on Climate Change (IPCC) reported that to limit the global temperature increase to 1.5 °C, greenhouse gas emissions must be reduced by 43% (34–60%) by 2030 and 84% (73–98%) by 2050 compared to 2019, and CO₂ emissions must be reduced by 27% (11–46%) by 2030 and 52% (36–70%) by 2040 compared to 2019 [4,5]. Accordingly, the reduction in CO_2 emissions has been conducted globally by improving energy efficiency, promoting low-carbon fuels, increasing renewable energy, increasing forests, and using carbon capture and storage technologies [6,7]. Carbon capture and storage (CCS) technology can reduce CO₂ emissions by 85 to 95% for power plants and kilns with high CO_2 emissions [6–9]. CCS is a pivotal technology for reducing atmospheric CO_2 [7,10]. Among the various CCS technologies, CO_2 capture using steel slag is a method of carbonating minerals by reacting the Ca- and Mg-containing mineral phases in the slag with CO₂ to form carbonates [9,11,12]. As reported, steel slag is an industrial by-product (IBP) of the steelmaking process. It is primarily used in the cement industry and road construction [11,13]. Its method includes mixing it as an aggregate of asphalt and replacing



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). a part of the cement in the form of powder [14]. The main oxides of steel slag are CaO, Fe_2O_3 , and SiO_2 , and free CaO in CaO can influence the volume stability [14,15]. Various methods for controlling the expansion of steel slag are yarding or manual watering in the air, leading to the reaction of free CaO with water [14]. Therefore, steel slag through wet mineral carbonation can increase the volume stability by forming CaCO₃ from a stable state of free CaO.

Mineral carbonation is divided into direct and indirect carbonation depending on the method of CO_2 capture [11,16–19]. Direct carbonation is a dry gas-solid and wet aqueous method, while indirect carbonation is a pretreatment for the extraction of reactants followed by carbonation [11,20]. Direct carbonation is more straightforward and requires fewer chemicals than indirect carbonation, while wet direct carbonation reacts faster than dry direct carbonation [21,22]. These advantages have led many researchers to investigate wet direct carbonation using steel slag. For example, Rushendra Revathy et al. captured CO_2 using electric arc furnace slag under a 30 \pm 2 °C, six bar, liquid-to-solid ratio (L/S) of 10 mL/g, CO₂ gas concentration of 99.99%, and a reaction time of 3 h. They could capture 82 g of CO₂ per 1 kg of slag [23]. On the other hand, Baciocchi et al. [24] captured CO₂ using electric arc furnace slag under a 100 °C, 10 bar, L/S of 5 mL/g, CO₂ gas concentration of 100%, and a reaction time of 24 h, capturing 280 g of CO_2 per 1 kg of slag. Additionally, Baciochi et al. [24] reported that CO_2 was captured using two basic oxygen furnace slags under the same conditions. In total, 325 g and 403 g of CO₂ per kg of slag were captured from basic oxygen furnace slags 1 and 2 under the same conditions [22]. This resulted from their particle size difference based on the size distribution analysis (basic oxygen furnace slag 2 ($D_{90} = 50.2 \,\mu\text{m}$) versus basic oxygen furnace slag 2: $D_{90} = 208.0 \,\mu\text{m}$) [22]. It is well known that the smaller particle size can increase the area per unit weight, increasing the CO_2 sequestration rate. This manifests the significance of the particle size effect in mineral carbonation [25]. The factors (i.e., the size distribution of the slag, temperature, pressure, L/S, reaction time, and slag type) directly affect the amount of CO_2 capture. This study aims to provide primary data on the operation of a mineral carbonation plant to capture CO_2 in the emissions of a municipal waste incinerator by assessing the amount of CO_2 captured and the sequestration efficiency. In addition, the characteristics of the mineral carbonation products will be identified to suggest effective ways to utilize the products [9].

2. Process and Operation for Mineral Carbonation Plant

The mineral carbonation plant operated in this study is located at the Seongam Incinerator in Ulsan Metropolitan City, Republic of Korea. In 2021, Ulsan Metropolitan City had an area of 1062.3 km² and a population of 1,138,419, and was designated as a CO_2 Resource Special Regulatory Free Zone on 13 November 2020 [26]. Ulsan Metropolitan City, with many large-scale industrial complexes, GHG target management companies, and businesses subject to the GHG emissions trading scheme, has made excellent efforts to achieve carbon neutrality [26]. A schematic diagram of the mineral carbonation plant process operated in this study is shown in Figure 1. The plant with a CO_2 capture capacity of 5 tons/day has four reactors. As illustrated, Reactors A and B, and Reactors C and D are arranged vertically, and the slag mixture flows from Reactor A to Reactor D, and the exhaust gas from the incinerator flows from Reactor D to Reactor A. CO₂ measurements of the inflow and outflow gases were performed at the front end of Reactor D and the rear end of Reactor A. In our study, the pH values were measured at all four reactors, and the reaction was terminated from the reactor close to the gas inlet point. The mixture can be transferred to the carbonation product solution tank only via Reactor D. We designed the vertical arrangement to operate the plant without separate power using the gravity energy from the movement of the slag mixture from Reactor A to Reactor B and Reactor C to Reactor D. The operation of the mineral carbonation plant is initiated by mixing steel slag and tap water in a slag and water mixing tank. In this study, steel slag with a size of ~300 mesh is applied. The reactors are first filled with the mixture, then the exhaust gas from the incinerator is introduced into the reactors. The reaction can be stopped by

adjusting the pH to 6. It is noted that Tu et al. reported that the initial pH of the slurry showed a sharp decrease from 11 to 8.5 and stabilization at ~6.5 in the aqueous carbonation of steel slag [27,28] because carbonate ions dominate at a pH > 10.3 and bicarbonate ions dominate at a pH < 8.4 [27]. Equation (1) can represent its mechanisms [29]. Therefore, if carbonate ions are more abundant than bicarbonate ions, H⁺ also becomes more abundant. An increase in H⁺ leads to a decreased pH, increased dissolution of Ca²⁺, and accelerated mineral carbonate ions and exists in dissolved form, resulting in a decrease in the formation of CaCO₃ [31]. Equations (2)–(4) show mineral carbonation [29,32].

$$CO_2(aq) + H_2O \leftrightarrow H_2CO_3 \leftrightarrow HCO_3^- + H^+ \leftrightarrow CO_3^{2-} + 2H^+$$
 (1)

$$Ca(OH)_2(aq) \rightarrow Ca^{2+} + 2OH^-$$
⁽²⁾

$$Ca^{2+} + HCO_3^- \to CaCO_3 + H^+$$
(3)



 $Ca^{2+} + CO_3^{2-} \to CaCO_3 \tag{4}$

Figure 1. A schematic illustration of the process for the mineral carbonation plant.

Gu et al. used an aqueous solution of Ca(OH)₂ at 25 °C and 30 mM to determine the ratio of calcium to pH [33]. They reported that below pH = 12.6, Ca²⁺ dominated Ca(OH)₂ in proportion and that pH~12, Ca(OH)₂ was fully converted to Ca²⁺ [33]. They also observed decreased CaCO₃ and increased Ca²⁺ at pH < 6 [33]. Accordingly, it was determined to terminate the reaction of mineral carbonation at pH \geq 6 [34,35].

Figure 2 is a schematic diagram of the reactor of the mineral carbonation plant operated in this study. The reactor's capacity is 10 m³, and the gas phase is introduced from the bottom and discharged from the top. To improve the efficiency of the carbonation reaction, the gas phase is introduced into the liquid phase through bubblers, and 18 bubblers are

attached to each reactor. The steel slag mixture is introduced from the top and discharged from the bottom, and the mixture is circulated to increase the stirring efficiency. Table 1 shows the characteristics of the gas entering the reactor of the mineral carbonation plant. During the operation, the average flow rate and temperature of the reactor gas entering were about 1550 Nm³/hr and 90 °C, respectively, and the average concentration of CO₂ in the inflow gas and the inflow rate were 10% and 300 kg/h, respectively. In addition, to evaluate the CO₂ removal efficiency for the inflow gas, the CO₂ sequestration efficiency and rate were calculated using the flow rate and CO₂ concentration of the outflow gas. The CO₂ sequestration efficiency and rate can be calculated using Equations (5) and (6).

CO₂ sequestration efficiency (%) =
$$\left[1 - \left\{\frac{\frac{B}{(100-B)}}{\frac{A}{(100-A)}}\right\}\right] \times 100$$
 (5)

CO₂ sequestration rate (kg/hr) = {C × $\frac{A}{100}$ × $\frac{44.01}{22.4}$ } - {D × $\frac{B}{100}$ × $\frac{44.01}{22.4}$ } (6)

A = CO_2 concentration in inflow gas (%);

 $B = CO_2$ concentration in outflow gas (%);

C = Inflow gas rate (Nm^3/hr);

D = Outflow gas rate (Nm³/hr).

where A and B are the CO_2 concentrations entering Reactor D and leaving Reactor A, respectively, and Reactors C and D are the gas flow rates entering Reactor D and leaving Reactor A, respectively. In addition, 44.01 and 22.4 are 44.01 kg/kmole- CO_2 and 22.4 m³/kmole- CO_2 , respectively. In this study, it is noted that we also investigated the feasibility of fully recycling water.



Figure 2. (a) Side view and (b) top view of the reactor used in this study.

Table 1. Parameters for the inflow gas to the reactors in the mineral carbonation plant.

Inflow Gas Rate	Temperature of	CO ₂ Concentration in Inflow Gas (%)	Inflow CO ₂ Rate
(Nm ³ /hr)	Inflow Gas (°C)		(kg/hr)
1555	88.4	10.0	305

3. Materials and Methods

To characterize the steel slag and mineral carbonation products, particle size analysis (PSA), specific gravity, Brunauer–Emmet–Teller (BET), X-ray fluorescence analysis (XRF),

loss on drying (LOD), loss on ignition (LOI), free CaO, and thermogravimetric analysis (TGA) were performed. Two and nineteen samples of the steel slag and mineral carbonation products, respectively, were used to obtain reliable results. PSA and specific gravity were conducted to determine the changes in particle size and specific gravity due to the carbonation reaction of the steel slag. The measurement ranges of the particle size analyzer (LA-960, HORIBA, Irvine, CA, USA) was 0.01~5000 μm, the light source was a 650 nm laser diode with ~5.0 mW, and the detectors were silicon photodiodes. The specific gravity was measured using a specific gravity bottle. BET was used to measure the specific surface area to check the change of pores due to the carbonation reaction of the steel slag. The specific surface area meter (QUADRASORB evo, Anton Paar, Graz, Austria) used nitrogen as the analysis gas, and the bath temperature was 77.3 K. An XRF was performed to determine the elements of the steel slag and mineral carbonation products. A wavelength dispersive XRF spectrometer (S8 TIGER, BRUKER, Billerica, MA, USA) equipped with a rhodium (Rh) tube with a 75-µm Be window was used. LOD and LOI were conducted for the proximate analysis of the steel slag and mineral carbonation products according to the process of the mineral carbonation plant, and the LOD was measured via the weight change after 5 h at 105 °C and the LOI after 2 h at 1000 °C. Free CaO was tested to evaluate the stability of the mineral carbonation products from the carbonation reactions for use as construction materials. The analysis of free CaO was determined by titration with a hydrochloric acid solution. TGA was performed using a thermogravimetric analyzer (TGA 5500, TA Instruments, New Castle, DE, USA), and the content of CaCO₃ and CaO as CaCO₃ was estimated using the thermal decomposition amount of 500-800 °C under nitrogen conditions up to 900 °C at a heating rate of 20 °C/min. The content of CaO as CaCO₃ was calculated using Equation (7).

CaO as CaCO₃ (%) = (Loss of mass for 500–800
$$^{\circ}C(\%)/44.01) \times 56.08$$
 (7)

where 44.01 and 56.08 are 44.01 kg/kmol-CO₂ and 56.08 kg/kmol-CaO, respectively. We also estimated the conversion of CaO from the total CaO result of XRF, and the equation is shown in Equation (8).

Conversion rate (%) =
$$[{(A-B) - (C-D)}/(A-B)] \times 100$$
 (8)

A = Total CaO in steel slag (%); B = CaO as CaCO₃ in steel slag (%); C = Total CaO in mineral carbonation products (%); D = CaO as CaCO₃ in mineral carbonation products (%); A-B = Residue CaO in steel slag (%); C-D = Residue CaO in mineral carbonation products (%).

where A and B are the total CaO and CaO as $CaCO_3$ in the steel slag, respectively, and C and D are the total CaO and CaO as $CaCO_3$ in the mineral carbonation products, respectively. It is noted that this formula does not account for $CaSO_4$ [36–38]. Since the exhaust gases applied for CO_2 mineralization were obtained from the end of the telemonitoring system of the incinerator stack, we assumed that the effect of SO_x would be much less than that of CO_2 .

4. Results and Discussion

The CO₂ concentration variation and CO₂ sequestration efficiency of the inflow and outflow gases over time are summarized in Figure 3. As the mineral carbonation reaction occurred rapidly after interacting with the reactant gases, we collected the data after a 5 min stabilization time after the gas injection. The average concentration of CO₂ in the inflow and outflow gases during the reaction time was 10.0% and 1.1%, respectively, and the average CO₂ sequestration efficiency was 89.7%. Figure 4 shows the opening of the valve as a function of time, indicating the mixture's movement. Therefore, the mixture was transferred from Reactor D to the carbonation product solution tank at 50 min from

the start of the reaction, and the mixture was sequentially transferred to Reactor D from 58 min to 77 min. This showed that, from the initial reaction to the transfer from Reactor D to the carbonation product solution tank, the CO₂ sequestration efficiency was 95.8% in State 1 (reaction time 0–50 min) when four reactors were filled with the mixture, and 84.7% in State 2 (reaction time 58–77 min) when three reactors were filled with the mixture during the reaction. After 77 min, the unreacted mixture was transferred to Reactor A, and the mixture was transferred from Reactor D to the carbonation product solution tank. As a result, the CO₂ sequestration efficiency was 85.8% in State 3 (reaction time 77–113 min), with one reactor filled with the unreacted mixture and two reactors filled with the reacting mixture. Therefore, the CO₂ sequestration efficiency reached the highest in State 1, followed by States 3 and 2. Although the average value of CO₂ sequestration efficiency was not significantly different between State 2 and State 3, the time for CO₂ sequestration efficiency to drop to about 80% was about twice as long in State 3 as in State 2.



Figure 3. Variation of CO_2 concentration and CO_2 sequestration efficiency of the inflow and outflow gases as a function of time.



Figure 4. Valve positions for reactors as a function of time.

Table 2 shows the CO_2 sequestration efficiency, rate, and capacity at different Sates. The gas rate and CO_2 concentration in gas affect the CO_2 sequestration rate, as shown in Equation (6). The CO_2 sequestration rate in State 1 was the lowest, while the CO_2 sequestration tration capacity was the largest. Therefore, although there was no significant difference in CO_2 sequestration efficiency between State 2 and State 3, the CO_2 sequestration capacity and gram of CO_2 per kg of steel slag in State 2 became double that of State 3 in comparison. Additionally, it was found that State 3 exhibited higher grams of CO_2 per kg of steel slag than State 1, even if the unreacted mixture was in one reactor. We found that the more reactors that were filled with mixtures, the less the gas flow rate was loaded, consequently leading to less active contact with the reactants.

	Reaction	CO ₂ Sequestration			
State	Time (min)	Efficiency (%)	Rate (kg/h)	Capacity (kg)	g-CO ₂ /kg-Steel Slag
1	0–50	95.8	246.4	205.3	79.9
2	58-77	84.7	271.6	86.0	44.6
3	77–113	85.8	308.6	185.2	97.7

Table 2. The CO₂ sequestration efficiency, rate, and capacity at different States.

Table 3 shows the PSA, specific gravity, and BET of the steel slag and mineral carbonation products. The PSA analysis of the steel slag and mineral carbonation products confirmed that the median decreased from 31.7 μ m to 15.6 μ m and the mean increased slightly from 54.5 µm to 58.9 µm. Ho et al. conducted wet direct carbonation experiments using the dephosphorization slag. They reported that as the carbonation reaction time increased, the $CaCO_3$ particles became smaller via the collision of the initially large particles and the larger solidity ratio [39]. Therefore, it can be concluded that large $CaCO_3$ particles were initially formed via calcium ions from the steel slag, followed by collision processes as the reaction progressed, leading to smaller median particle sizes. However, we also observed that the mean particle size was increased, assuming that it may result from non-colliding CaCO₃ particles. In addition, the specific gravity of the mineral carbonation products decreased by about 0.2 compared to the steel slag due to the formation of calcium carbonate, which has a smaller specific gravity than steel slag. The decrease in specific gravity resulted in an increase in BET from $11.9 \text{ m}^2/\text{g}$ to $44.7 \text{ m}^2/\text{g}$. This means the mineral carbonation products have more pores than the steel slag. Chen et al. [20] characterized the carbonation of basic oxygen furnace slag and found that the density of carbonated basic oxygen furnace slag decreased from 3.13 g/cm³ to 2.66 g/cm³, and the Blaine Fineness increased from $672 \text{ m}^2/\text{kg}$ to $1020 \text{ m}^2/\text{kg}$, showing a similar trend to this study [40]. Figure 5 shows the characterization results for the XRF, LOD, and LOI of steel slag and mineral carbonation. The CaO content of steel slag was 44.6%, while that of the mineral carbonation products was 31%. It is considered that the LOD of the steel slag increased due to the wet carbonation reaction, and the LOI increased due to the formation of calcium carbonate. The formation of calcium carbonate can be confirmed via free CaO and TGA [20,40], and the free CaO and TGA results are shown in Table 4. The free CaO in the steel slag was reduced from 7.8% to 0.3% via the carbonation reaction, and the weight loss of TGA from 500 to 800 °C increased from 5.3% to 21.6%. These results are consistent with the study by Chen et al. [40]. It was reported that basic oxygen furnace slag with a content of 1.02% free CaO was not found after carbonation, and the TGA analysis showed that the DTG peak of calcium hydroxide (441 °C) disappeared and the DTG peak of calcium carbonate (775 °C) intensified with carbonation [40]. Therefore, it is anticipated that most of the free CaO and calcium hydroxide contained in the steel slag can be removed through the carbonation reaction. In addition, the conversion rate of CaO is >90%, according to XRF and TGA analyses. Wang reported that free CaO is the main cause of the volume expansion of steel slag; the reason for this is that when free CaO reacts with water to produce calcium hydroxide, the volume increases by about 90% [41]. Accordingly, we assumed that the mineral carbonation products are more stable than steel slag as a construction material and are effective at sequestering CO_2 by forming chemically stable calcium carbonate [42]. Therefore, when there is great concern about the concentration of CO_2 in the atmosphere, utilizing mineral

carbonation products as a construction material can bring great environmental benefits in terms of recycling steel slag and the sequestration of greenhouse gases [43].





Figure 5. XRF, LOD, and LOI analyses for (a) steel slag and (b) mineral carbonation products.

Table 4. Mass loss of TGA (500–800 °C), the content of free CaO, CaCO₃, CaO as CaCO₃, and residue CaO, and the conversion of CaO for steel slag and mineral carbonation products.

Category	Steel Slag	Mineral Carbonation Products
Mass loss of TGA (%)	5.3	21.6
Free CaO (%)	7.8	0.3
CaCO ₃ (%)	12.1	49.2
CaO as CaCO ₃ (%)	6.8	27.6
Residue CaO (%)	37.8	3.4
Conversion CaO (%)	-	90.9

5. Conclusions

This study assessed the amount of CO_2 captured and the sequestration efficiency of the carbonation plant to capture CO_2 in the emissions of a municipal waste incinerator. In addition, the characteristics of the mineral carbonation products were identified to suggest ways to utilize the products. In this study, the CO_2 sequestration efficiency reached 95.8% in State 1 (reaction time: 0–50 min) when four reactors were filled with the mixture, while it became 84.7% in State 2 (reaction time: 58–77 min) by filling three reactors with the mixture during the reaction. After 77 min, the unreacted mixture was transferred to Reactor A, and then, the mixture was transferred from Reactor D to the carbonation product solution tank. This resulted in a CO_2 sequestration efficiency of 85.8% in State 3 (reaction time: 77–113 min), with one reactor filled with the unreacted mixture and two reactors filled with the reacted mixture. We observed that the CO_2 sequestration rate in State 1 was the lowest, while its CO_2 sequestration capacity was the largest as the gas rate and CO_2 concentration in gas affect the CO_2 sequestration rate. Therefore, we found that State 3, with fewer filled reactors, received less load on the gas flow rate, leading to more active

contact between the gas reactant liquid. Then, consequently, the CO_2 sequestration rate and the carbon sequestration in grams of CO_2 per kg of steel slag were higher. We observed a conversion rate of CaO of >90%. Our plant-wide experimental findings suggested mineral carbonation products are more stable than steel slag as a construction material and are effective in sequestering CO₂ by forming chemically stable CaCO₃. Therefore, when there is great concern about the concentration of CO_2 in the atmosphere, utilizing mineral carbonation products as a construction material can bring significant environmental benefits in terms of recycling steel slag and the sequestration of greenhouse gases [44]. It is proposed that environmental exposures, such as the long-term heavy metal leaching from construction materials utilizing mineral carbonation products, should be evaluated, and more economic studies should be conducted based on this study [45]. Furthermore, studies on the effect of carbonation on heavy metal leaching from steel slags are also highly recommended [46]. For the proper utilization of mineral carbonation products, it is also desirable to accurately characterize all of the minerals of reactants and products under different operating conditions using X-ray diffraction (XRD) analysis [46], verifying the mineralogical reactions in detail. The outcomes can be applied to rationally designing futuristic experimental systems.

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