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Ca(II) Conversion Efficiency of Steel Slag Leachate via Enzyme Induced Carbonate Precipitation (EICP)

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ABSTRACT: Steel slag is a significant by-product of the steel industry, with an annual global production of approximately 190–280 million tons. To utilize steel slag as a construction material, it undergoes an aging process for about 3 months, during which water spraying is applied to remove the free-CaO component contained in the slag. However, residual free-CaO in aged steel slag can increase the pH of leachate and cause turbidity when used in construction applications, limiting its utilization despite its abundant availability. This study analyzed the efficiency of removing free-CaO from steel slag through Enzyme Induced Carbonate Precipitation (EICP) with respect to urea and urease concentrations. The steel slag used in the experiments was sieved to control the particle size to below 20 mm. The urea concentration was adjusted within a range of 0.5–1 M, considering the concentration of Ca^{2+} ions that could leach into the steel slag pores. To ensure economic feasibility, the urease concentration was set within a range of 35 and 50 g/L using soybean powder. To monitor the reaction process, pH measurements were taken at specific intervals to estimate Ca^{2+} ion concentrations. After reaction, the identification of the resulting product ($CaCO_3$) was performed using XRD, SEM, and EDS analyses. This study applied EICP during the aging process of steel slag to effectively mineralize free-CaO into $CaCO_3$, thereby improving the economic feasibility and addressing the challenges of steel slag utilization. Furthermore, it presents the optimal conditions for urea and urease to enhance process efficiency.

KEYWORDS: Steel slag, calcium carbonate mineralization, EICP, building materials, industrial waste recycling

1 INTRODUCTION

Steel slag is a representative by-product of the steel industry, and its production is increasing in parallel with the growing output of steel (US Geological Survey, 2025). It is generated during the refining process of hot metal, in which sulfur and phosphorus are removed. Due to its particle morphology and mechanical properties being similar to those of natural aggregates, various attempts have been made to utilize steel slag as an alternative construction material (Korea Iron & Steel Association, 2024, Euroslag, 2018). Notable examples include its use as road aggregates, backfill materials, and in some reported cases, as a raw material for sand compaction piles (SCP). However, the direct use of steel slag in construction materials without pre-treatment can exert adverse effects on the surrounding soil environment. Steel slag contains free-CaO, which reacts with water to form calcium hydroxide, subsequently dissociating into Ca^{2+} and OH^- ions, thereby increasing the alkalinity of the surrounding fluid. The alkaline fluid containing calcium hydroxide can promote the dissolution of atmospheric CO_2, leading to the generation of hydrogen ions and carbonate ions. The carbonate ions formed can further react with Ca^{2+} to precipitate calcium carbonate ($CaCO_3$), which may cause suspended fines and turbidity. Moreover, the formation of $Ca(OH)_2$ from the reaction between CaO and water is accompanied by volumetric expansion, which has been reported as a significant issue. Such problems can arise upon contact with rainwater or groundwater when untreated steel slag is used directly as a construction material. Consequently, steel slag generally undergoes an aging process before commercialization. A common aging method involves stockpiling the slag outdoors and spraying it with water to wash out the CaO content, which typically requires at least six months, thereby demanding considerable space and time (Engström et al. 2013, Liu et al. 2024). As a complementary approach, extensive research has also been conducted on inducing carbonation reactions and seawater-based aging approaches in steel slag by directly reacting it with CO_2 (Huijgen & Comans 2006; Huijgen et al. 2005; Li et al. 2020; Pan et al. 2017; Zhang et al. 2011).

In this study, the Enzyme-Induced Carbonate Precipitation (EICP) method, a bio-cementation technique, was applied to remove Ca components from steel slag. EICP is a process that can be employed to improve the strength and stiffness of soils by reacting carbonate ions (CO_3^{2-}), obtained from the decomposition of urea, with calcium ions (Ca^{2+}) to precipitate $CaCO_3$. The decomposition of urea is catalyzed by the enzyme urease to accelerate the reaction (Equation (1)-(2)). Since steel slag contains free-CaO, it provides a suitable source of Ca^{2+} for reaction with CO_3^{2-} , and recent experimental studies have successfully demonstrated the precipitation of $CaCO_3$ using this approach (Equation (3)-(4)). By removing free-CaO in steel slag through this process, its properties can be improved for safe and effective use as a construction material. Moreover, identifying the optimal EICP treatment conditions is essential for the efficient large-scale processing of steel slag. When EICP is applied to soil, the key factors influencing the reaction are the concentrations of calcium chloride, urea, and urease, with the urease-mediated urea hydrolysis rate being pH-dependent. In the case of steel slag treatment, Ca^{2+} is supplied through the reaction of free-CaO with water. Therefore, the concentrations of urea and urease become the main parameters. In this study, the EICP process was assumed to be implemented through water-spraying treatment, and two liquid-to-solid ratios (L/S), 1 and 5 mL/g, were applied to estimate the amount of urea solution required per unit mass of steel slag.

$$CO(NH_2)_2 + H_2O \xrightarrow{\text{urease}} 2NH_3 + CO_2 \quad (1)$$

$$CO_2 + H_2O \rightarrow 2H^+ + CO_3^{2-} \quad (2)$$

$$CaO + H_2O \rightarrow Ca^{2+} + 2OH^- \quad (3)$$

$$CO_3^{2-} + Ca^{2+} \rightarrow CaCO_3 \quad (4)$$

The optimal urea and urease concentrations for steel slag treatment were experimentally determined. The reacted slag was analyzed to confirm the formation of $CaCO_3$ using X-ray diffraction (XRD), scanning electron microscopy (SEM), and

Energy-Dispersive x-ray Spectroscopy (EDS). The quantity of precipitated $CaCO_3$ was measured to evaluate the CaO removal efficiency. The pH of the leachate was monitored over time during and after the reaction to track the reaction progress, and the environmental impact of the treated slag was assessed.

2 MATERIALS AND METHODS

2.1 *Basic Oxygen Furnace (BOF) steel slag*

This study utilized BOF steel slag obtained from POSCO (Pohang, Korea). The material was sieved to retain particles with diameters less than 4.75 mm, while excluding fines smaller than 0.075 mm. The specific gravity of the BOF steel slag was determined to be 3.3 in accordance with ASTM D854, consistent with the typical range reported for steel slag materials (Moon et al. 2002). The minimum and maximum void ratios were measured as 0.489 and 0.806, respectively, following the procedures specified in ASTM D4253 and ASTM D4254.

2.2 *Urea solution*

The urea solutions applied to the steel slag were prepared at target concentrations of 0.5 and 1 M using urea (reagent plus grade, Sigma-Aldrich). Urease was extracted from soybeans, and its concentration was controlled based on the mass of unheated soybean powder. The 0.5 M urea solution was prepared by mixing a 1 M urea solution with a 70 g/L soybean powder solution at a 1:1 ratio, sealing the mixture, and allowing it to react for 24 hours. Similarly, the 1 M urea solution was prepared by mixing a 1 M urea solution with a 100 g/L soybean powder solution at a 1:1 ratio, sealing the mixture, and allowing it to react for 48 hours.

These concentrations were determined through preliminary experiments designed to induce the EICP reaction among $CaCl_2$, urea, and urease. In these tests, urea solutions containing a fixed amount of $CaCl_2$, urea concentrations ranging from 0.5 M to 1 M, and soybean powder concentrations between 20 g/L and 50 g/L were reacted for up to 72 hours. The optimal conditions were identified as those in which all $CaCl_2$ reacted to form $CaCO_3$, and these conditions were subsequently applied to the EICP treatment of steel slag. To minimize the presence of soybean powder as an impurity during the reaction, the extracted urease solution was centrifuged at 2000 rpm for 15 minutes to precipitate and remove solids prior to use.

2.3 *EICP treatment procedure*

To evaluate the performance of EICP treatment in removing CaO from steel slag, urea solution was applied to the slag samples. The urea solution and steel slag were allowed to react for 72 hours in a sealed environment to prevent contact with external air. After the reaction, solids were collected using filter paper to confirm the formation of precipitated $CaCO_3$ and to quantify the amount produced. X-Ray Diffraction (XRD), scanning electron microscopy with Energy-Dispersive Spectroscopy (SEM-EDS), and acid-base titration using hydrochloric acid were conducted for this purpose. To simulate the leachate characteristics of EICP-treated steel slag, the reacted slag was immersed in distilled water at a liquid-to-solid (L/S) ratio of 5 mL/g after the 72-hour reaction period, and pH was continuously monitored to observe the dissolution behavior of residual CaO within the slag.

2.4 *Qualitative analysis of $CaCO_3$ formation*

2.4.1 *XRD analysis*

XRD (Ultima IV, Rigaku) analysis was conducted to confirm the formation of $CaCO_3$ in steel slag after EICP treatment. The XRD method identifies crystalline phases by determining the diffraction angles of x-rays, which are dependent on the atomic arrangement within the crystal structure of a material. The EICP-treated steel slag was dried at 80 °C for 72 hours, ground to a particle size smaller than 0.15 mm, and exposed to X-rays over an incidence angle range of 10–80 degree at a scanning speed of 2 °/min to obtain diffraction patterns. The PDF-2 database was used for pattern identification and phase analysis.

2.4.2 *SEM-EDS analysis*

To observe the crystals formed after the reaction, SEM (JSM-IT500HR, JEOL) imaging was performed. EDS (JSM-IT500HR, JEOL) analysis was also conducted to qualitatively determine the elemental composition of the crystals presumed to be $CaCO_3$ in the captured images. These techniques enabled the direct observation of the crystal morphology, size, and distribution patterns of the precipitated materials, as well as qualitative elemental analysis (Fitzgerald and Heinrich 1968). The samples were dried at 80 C for 72 hours, and to prevent charging during imaging, a platinum coating (~21 nm thick) was applied at a current of 10 mA for 300 seconds prior to observation. SEM imaging was conducted under an accelerating voltage of 10 kV.

3 RESULTS

3.1 *$CaCO_3$ formation*

3.1.1 *XRD analysis*

Figure 1 presents the XRD analysis results of EICP-treated steel slag along with the reference peak pattern of $CaCO_3$. In all four cases, diffraction patterns corresponding to $CaCO_3$ were observed, indicating that $CaCO_3$ precipitation occurred through the EICP treatment process. The most prominent peak near 25° corresponds to the diffraction pattern of SiO_2, a mineral commonly present in natural materials, including steel slag, which explains its high intensity. The extent of carbonation in steel slag is strongly correlated with particle size. The maximum particle size of the steel slag used in this study was 4.75 mm, with a D_{50} of 1.29 mm. Even if all free-CaO present in steel slag of this particle size range were to react, the resulting $CaCO_3$ would account for approximately 10% of the slag mass (Ma et al. 2024). Therefore, the stronger SiO_2 peaks observed in the XRD results after the reaction are a natural outcome.

3.1.2 *SEM-EDS analysis*

Figure 2a shows the surface morphology of steel slag after being treated with a 0.5 M urea solution at a L/S of 1 mL/g, followed by drying, observed at a magnification of 11,000×.

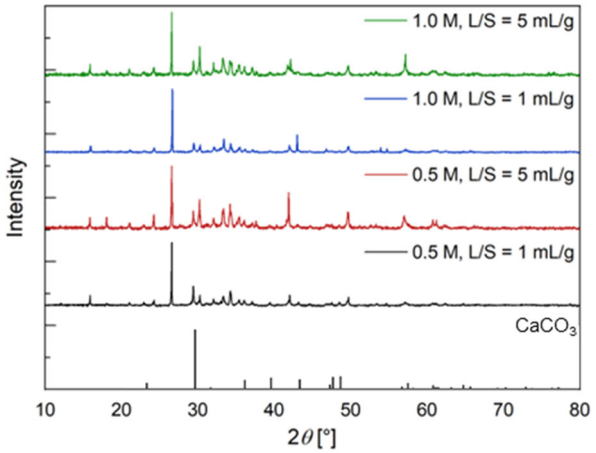

Figure 1. XRD patterns of EICP treated steel slag and reference peak data of calcium carbonate ($CaCO_3$)

Crystalline phases presumed to be $CaCO_3$ were observed on the steel slag surface. To analyze the composition of these crystals, EDS analysis was performed, and the analyzed spots (Spot 1–3) are indicated in the figure, with the corresponding results presented in Table 1. The primary constituent elements of the crystals were C, O, Ca, and Fe, and the relative proportions of C, O, and Ca were similar to the elemental ratio of $CaCO_3$ (1:3:1), indicating that the observed crystals can be identified as $CaCO_3$. The 10% Fe content observed at Spot 2 is attributed to the intrinsic Fe-bearing composition of steel slag.

EDS analysis detects elemental composition to a depth of approximately 1 μm from the sample surface. When the precipitated $CaCO_3$ layer is thinner than 1 μm, the elemental composition of the underlying slag can also be detected. Figures 2b-2d present SEM images, acquired under the same conditions as Figure 2a, for steel slag samples subjected to EICP treatment under different conditions, with the EDS analysis points indicated. In all conditions, $CaCO_3$ was observed to form as irregularly shaped crystals. In particular, when a 1 M urea solution was reacted with steel slag at an L/S ratio of 1 mL/g (Figure 2c), vaterite-type $CaCO_3$ crystals were also identified.

3.2 *Leachate pH evolution after EICP treatment*

To evaluate the efficiency of $CaCO_3$ formation induced by the EICP treatment, the treated steel slag was immersed in distilled water at a L/S of 5 mL/g, and the pH variation over time was monitored (Figure 3). The Ca^{2+} ions required for $CaCO_3$ precipitation originate from the free-CaO present in steel slag. CaO reacts with water to generate hydroxide ions, thereby rendering the solution alkaline. Consequently, it can be inferred that a higher amount of $CaCO_3$ formed during the EICP process would result in a lower pH when the treated steel slag subsequently reacts with distilled water. In addition, as steel slag is known to be a porous material, the leaching of CaO components occurs in a time-dependent manner. Therefore, pH variation over a period of 20 days was monitored to assess the extent to which the CaO content in the steel slag had been removed.

When the L/S was 5 mL/g, the initial pH was lower than that observed at an L/S of 1 mL/g. At the same urea concentration, a larger L/S provides a greater quantity of CO_3^{2-} ions available to react with the steel slag and a larger solution volume into which Ca^{2+} ions can leach, ultimately leading to greater $CaCO_3$ formation. A similar trend was observed when comparing the pH values after 20 days. Treatment with an 1 M urea solution resulted in a smaller pH increase over time compared to treatment with a 0.5 M urea solution. For the 0.5 M urea solution, pH increased in an approximately linear fashion over the 20-day period without reaching convergence. In contrast, treatment with a 1 M urea solution at an L/S of 5 mL/g resulted in the smallest 20-day pH increase (0.18), with a final pH of 9.59, which is the lowest among the four tested cases. For the 1 M urea solution at an L/S of 1 mL/g, the pH increased by 0.36 within the first two days, after which it remained nearly constant and exhibited a converging trend.

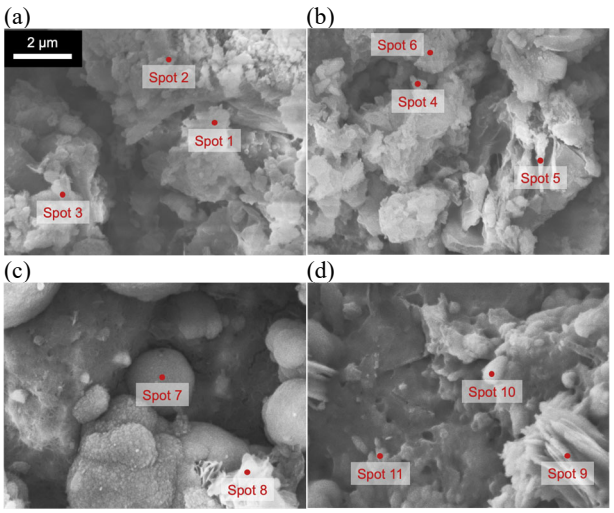

Figure 2. SEM image of EICP treated steel slag using urea solutions: (a) 0.5 M, L/S = 1 mL/g, (b) 0.5 M, L/S = 5 mL/g, (c) 1 M, L/S = 1 mL, and (d) 1 M, L/S = 5 mL/g

Table 1. EDS analysis result for spots highlighted in Figure 2

Spot no.	Atomic portion [%]								
	C	O	Mg	Al	Si	P	S	Ca	Fe
1	14.6	60.8	0.4	0.5	1.1	1.7	0.2	20.8	-
2	7.1	54.3	0.6	1.7	4.4	2.0	0.3	19.7	10.0
3	11.9	58.5	0.6	0.8	2.3	1.4	0.3	20.1	4.1
4	26.0	52.7	0.9	3.0	3.0	1.0	0.3	9.8	3.4
5	32.9	45.0	0.6	3.3	2.7	1.3	0.3	8.6	5.3
6	25.7	53.3	0.9	3.0	3.6	1.1	0.3	9.7	2.4
7	11.3	43.9	1.2	4.3	8.1	0.7	0.4	14.7	15.3
8	11.0	48.8	0.8	1.2	1.6	0.7	0.2	34.3	1.5
9	15.1	42.8	0.5	0.9	1.7	0.9	0.7	34.8	2.7
10	19.1	53.6	0.5	1.3	2.6	0.3	0.4	20.9	1.3
11	14.9	52.4	5.2	3.8	6.9	0.2	0.3	3.1	13.7

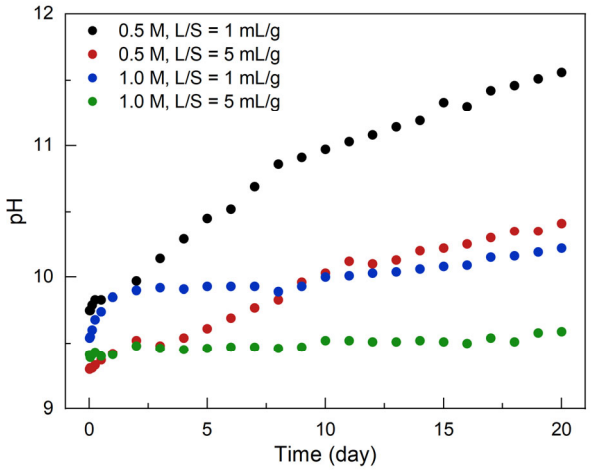

Figure 3. pH evolution of 72-hour EICP treated steel slag leachate

4 CONCLUSIONS

In this study, the EICP method, utilizing urease extracted from soybeans, was applied to BOF steel slag to promote the formation of $CaCO_3$. XRD, SEM-EDS, and acid titration analyses confirmed that the EICP treatment successfully precipitated $CaCO_3$ on the surface of the steel slag, indicating the effective utilization of Ca^{2+} ions derived from free-CaO.

The efficiency of Ca^{2+} removal via EICP was systematically evaluated with respect to urea concentration, urease concentration (controlled through soybean powder content), and L/S. The results demonstrated that higher urea concentrations and larger L/S led to improved treatment performance, with urea concentration identified as the more dominant parameter compared to L/S. High-concentration urea solutions not only enhanced the rate of $CaCO_3$ precipitation but also effectively reduced the residual alkalinity of the treated slag, as evidenced by lower pH values during post-treatment leaching tests.

These findings suggest that optimizing urea concentration is critical for maximizing the EICP treatment efficiency of steel slag, while an adequately large L/S can further enhance the overall carbonation performance. This approach offers a promising and environmentally friendly method for

accelerating the stabilization of steel slag for use as a construction material.

5 ACKNOWLEDGEMENTS

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