



# Article Application of Biomineralization Technology in the Stabilization of Electric Arc Furnace Reducing Slag

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Abstract: The unstable substances in steel slag are the main substances that affect its stability, which limits the large-scale resource utilization of steel slag. Most of the current methods for stabilizing electric arc furnace (EAF) slag are time-consuming and cannot be completely stabilized. In view of this, this study aimed to explore the feasibility of microbial-induced calcium carbonate precipitation (MICP) technology for stabilizing EAF reducing slag, and this was to be achieved by using the reaction between carbonate ions and free calcium oxide (f-CaO) in reducing slag to form a more stable calcium carbonate to achieve the purpose of stabilization. The test results showed that, when the EAF reducing slag aggregates (ERSAs) were immersed in a Sporosarcina pasteurii bacteria solution or water, the f-CaO contained in it would react such that stabilization was achieved. The titration test results showed that the f-CaO content of the ERSAs immersed in the bacterial solution and water decreased. The expansion test results of the ERSAs that were subjected to hydration showed that the seven-day expansion of ERSAs after biomineralization could meet the Taiwan regulation requirement of an expansion rate less than 0.5%. The thermogravimetric analysis showed that both the experimental group and the control group might contain calcium carbonate compounds. The results of the X-ray diffraction analysis showed that the CaCO<sub>3</sub> content in the ERSAs that were immersed in the bacterial solution was significantly higher than those that were immersed in water. Moreover, the compressive strength test results of concrete prepared with ERSAs showed that the compressive strength of the control group concrete began to decline after 28 days. In contrast, the experimental group concrete had a good stabilization effect, and there was no decline in compressive strength until 180 days. At 240 days, the surface cracks of the experimental group were particularly small, while the surface of the control group showed obvious cracks. These results confirmed that a mineralization reaction with S. pasteurii bacteria could be used as a stabilization technology for ERSAs.

**Keywords:** biomineralization; microbial-induced calcium carbonate precipitation; reducing slag; stabilization; free calcium oxide; calcium carbonate

## 1. Introduction

Electric arc furnace (EAF) steelmaking uses ferrous waste iron and steel scrap as raw materials [1]. It regenerates ferrous waste iron and steel scrap by smelting, and EAFs endow it with new value. Compared with traditional blast furnace–basic oxygen furnace (BF–BOF) steelmaking, EAF steelmaking is more environmentally friendly, especially in terms of energy demand, abiotic depletion, and global warming [2]. However, the EAF steelmaking process also produces many by-products, namely EAF slag. The EAF smelting process



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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/). must go through two stages: the oxidation period and the reduction period. Thus, EAF slag can be subdivided into "oxidizing slag" and "reducing slag" [3]. If these EAF slags are not used rationally, they must be stored. This will not only cause storage space problems, but will also cause environmental pollution and impacts; furthermore, it could even affect the normal production of steel. The cumulative production of EAF slag can have adverse effects on the environment [4,5]. To solve this environmental problem and to promote the sustainable development of human society, the recycling of EAF slag has become a hot research topic. Taiwan produces approximately 1.2 million metric tons of oxidizing slag and 0.46 million metric tons of reducing slag from EAF steelmaking plants annually [6]. These two types of EAF slag are declared as industrial waste in Taiwan, and they can be used as engineering or raw product materials after treatment. Their reuse management should follow the guidelines of Taiwan's "Ministry of Economic Affairs Reuse Types and Management Methods of Industrial Waste".

EAF slag is a rock-like material with mechanical properties that are suitable for use as aggregates in cement concrete production [7]. The use of EAF slags in concrete mixtures has attracted considerable attention over the past two decades [8–12]. There are many studies confirming that the reuse of EAF slag is a sustainable disposal method as a substitute for natural aggregates in infrastructure applications [13–15]. The biggest concern about the reuse of EAF slag is its poor volume stability because it contains a large amount of free calcium oxide (f-CaO) and free magnesium oxide (f-MgO) [16]. Furthermore, f-CaO has the characteristics of high activity, and it can easily react with water and carbon dioxide to decompose into calcium hydroxide (Ca(OH)<sub>2</sub>) and calcium carbonate (CaCO<sub>3</sub>) [17]. The conversion of f-CaO to calcium hydroxide causes a volume expansion of the EAF slag. However, EAF slag tends to be stable when the f-CaO is completely reacted with and digested, or when its content is low [18]. Accordingly, there is an opportunity for EAF slag aggregates (ERSAs) to replace general natural aggregates and to become a usable renewable resource, which can be achieved by stabilizing its dimensional stability and appearance problems.

Oxidizing slag has a high content of iron, a hard texture, and high specific gravity; in addition, it is a dark brown lump with stable physical and chemical properties [19]. Essentially, oxidizing slag has the characteristics of wear resistance and high hardness [8]. After proper treatment, the f-CaO content of oxidizing slag can be reduced, making it an ideal construction material [20] (for example, as aggregates for asphalt concrete [9,21,22]). Reducing slag contains less iron, more CaO and MgO, and is a gray-brown powder or block. Since reducing slag contains parts of free lime, it expands and disintegrates when exposed to water; thus, it must be matured to stabilize it. In addition, reducing slag is easily pulverized due to the volume changes caused by crystal phase transitions during storage. Compared with oxidizing slag, reducing slag has more materials involved in the manufacturing process, which makes its physical and chemical properties unstable, thus rendering its subsequent reuse difficult.

The attenuation or resolution of the volume expansion of EAF slag is commonly referred to as stabilization [13]. In other words, EAF slag must be properly stabilized first to dissolve the unstable components (such as f-CaO, f-MgO, and other expansive substances) to make it into stable calcium hydroxide, magnesium hydroxide, and stabilized products. In this way, EAF slag aggregates will not contribute to the soundness and safety problems of concrete structure surface bursting or structural cracking. A common method of stabilizing EAF slag is to expose it to the air for a period [13]. The efficiency of its stabilized product is affected by weather conditions, and it may take a long time to stabilize. Therefore, methods through which to accelerate the stabilization of EAF slag have been developed, including natural weathering, wind quenching, water quenching, gas quenching, afterheat self-degradation, steam aging, immediate treatment, modification, short slag flow, use of chemicals, hot pressing, and carbonization [23–26]. Nevertheless, there are large differences in the stability, uniformity, fineness, and activity of EAF slag that

are obtained using different stabilization techniques. Stabilization treatment by the natural or steam aging method requires a large stockpiling field, huge funds, and time.

Biomineralization is a process that occurs widely in nature, and it is defined as the process by which organisms produce minerals through metabolic activities related to environmental interactions [27,28]. During biomineralization, living organisms produce biominerals from biopolymers [29]. The microorganisms involved secrete one or more metabolites that react with ions or compounds in the environment, thereby contributing to the development of subsequent mineral grain changes into metabolite deposition [30]. There are three basic types of biomineralization, namely: (i) biologically controlled mineralization, (ii) biologically induced mineralization, and (iii) biologically influenced mineralization [31]. In the biologically controlled mineralization process, microorganisms can control the nucleation, composition, location, and morphology of biominerals [32]. In the biologically induced mineralization process, the microorganisms are not directly involved in the precipitation of biominerals, but the precipitation occurs because of interactions between the metabolic by-products of the bacteria and the ions existing in the environment [33]. In the biologically influenced mineralization process, an organic matrix and organic and/or inorganic compounds lead to the precipitation of biominerals without the necessity for the extracellular or intracellular biological activity of a living organism [34]. Biomineralization differs significantly from geological mineralization in that the crystallization of the inorganic phase is strictly controlled by the excretion of organic matter by the organism [35]. Therefore, biomineralization can often form well-ordered natural organic-inorganic composites with good structure. Microbial-induced calcium carbonate precipitation (MICP) is a type of biologically induced mineralization commonly found in nature [28]. In the past two decades, MICP technology has attracted widespread attention in academia and has been applied in geotechnical engineering, hydraulic engineering, geological engineering, environmental engineering, building materials, and other fields [33,36–45]. The most important input required for MICP is a calcium source [36]. In view of this, in seeking solutions for solid waste management, calcium-based solid waste can be considered a potential resource that can be made into value-added materials through MICP technology. Many researchers have used calcium-based solid waste as an alternative material for the manufacture of biomineralized materials [46-49]. The carbonation reaction of solid waste can fix CO<sub>2</sub>, eliminate the f-CaO and f-MgO in it, and generate stable insoluble carbonate [50–52]. Solid waste that can be used for carbonation mainly includes steel slag, carbide slag, waste concrete, cement kiln dust, waste incinerator dust, tailings produced in certain metal smelting processes, etc. [50–52].

The reducing slag is smelted at more than 1500 °C in the steelmaking process, and it is an inorganic aggregate. The low hydration activity and poor volume stability of reducing slag limit its wide application. Therefore, controlling or digesting f-CaO and f-MgO in reducing slag to keep the content within a controllable range with good volume stability is a prerequisite for utilizing reducing slag as a building material resource. In view of the problem of steel slag expansion, the main methods to suppress the volume expansion of steel slag at this stage are the natural aging method, mineral admixture method, high-temperature reconstruction method, carbonation modification method, etc. However, most of the current stabilization methods for EAF slag are time-consuming, costly, and not fully stable. Overall, the current stabilization methods for EAF slag are still time-consuming and cannot be completely stabilized. Only high-temperature and high-pressure steam treatment methods can quickly and effectively achieve the purpose of stabilization. However, this method requires expensive equipment and consumes energy, which is not in line with the United Nations' sustainable development goals.

There are currently twenty manufacturers in Taiwan's EAF steelmaking industry. The chemical composition of the produced reducing slag is mainly oxides and some f-CaO and f-MgO, with CaO and SiO<sub>2</sub> having the highest content, accounting for 35.3% to 54.9% and 16.57% to 34.83%, respectively. However, currently, only one manufacturer has completed the stabilization equipment for reducing slag, and other EAF steelmaking

manufacturers have no stabilization equipment. In view of this, this study aimed to explore the feasibility of using biomineralization technology to stabilize EAF reducing slag, and selected Sporosarcina pasteurii bacteria, which are environmentally friendly and easy to cultivate on a large scale, for the test. It mainly used the metabolic reaction of bacteria to quickly digest the f-CaO or f-MgO in the reducing slag to produce relatively stable calcium carbonate or magnesium carbonate salts. The new concept proposed in this study, using biomineralization to treat reducing slag, can provide an economical, simple, and fast method for stabilizing reducing slag, which can not only solve the problem of its volume stability, but can also absorb CO<sub>2</sub> [53]. In this study, ERSAs were subjected to biomineralization treatments for different periods of time. After proper treatment and stabilization by biomineralization, reducing slag can be reintroduced into the construction material cycle under the premise of volume stability to achieve the goal of resource recycling. In line with this, the aggregates before and after treatment were used to produce concrete, and their engineering properties and volume stability were measured. The results of this study confirm the feasibility of applying biomineralization to stabilize reducing slag, which has good economic, social, and environmental benefits. It can be inferred that the ERSAs stabilized by biomineralization have broad development prospects as a building material resource.

# 2. Materials and Methods

### 2.1. Experimental Program

In this study, the experimental group and the control group were planned. The reducing slag in the experimental group was treated in two different ways, namely, immersed in *S. pasteurii* bacteria solution and immersed in water. The bacterial liquid immersion method is the focus of this article, and the water immersion method is a common method to stabilize EAF slag. The reducing slag in the control group was exposed to the air without any stabilization treatment. This is also the more commonly used method for stabilizing EAF slag. The test items include the content of f-CaO and f-MgO in reducing slag, the potential expansion test by hydration, pH value test, thermogravimetric analysis (TGA), differential thermal analysis (DTA), and X-ray diffraction (XRD) analysis. The experimental variables of each test item include the stabilization method of reducing slag, immersion age, and the particle size of reducing slag. The particle size of reducing slag was divided into 1, 2, 5, and 10 mm, and the unscreened full particle size ranged between 1 and 10 mm. The range of each test variable and the designation of each sample are shown in Table 1. For example: 5B-3D refers to reducing slag with a particle size of 5 mm that was immersed in the bacterial solution for 3 days. In addition, unstabilized and stabilized reducing slag were used to prepare concrete, respectively, and their engineering properties and surface cracks were tested.

Test Variable	Variable Range and Sample Designation					
De chile et e	1–10 mm	1 mm	2	mm	5 mm	10 mm
Farticle size	A	1		2	5	10
Stabilization	Exposed to the air		Immersed in water		Immersed in a <i>S. pasteurii</i> bacteria solution	
method	N			W		В
Immersion age	1 day		2 days	3 da	ys	4 days
	1D		2D	3E	)	4D

Table 1. Test variables and sample designation.

The materials used in this test and their sources are described below:

- Reducing slag: This was produced in a local electric arc furnace steelmaking plant, and its appearance is shown in Figure 1. Among them, the reducing slag coarse aggregate had a water absorption rate of 10.7%, a specific gravity of 2.70, and the fineness modulus was 5.87. The reducing slag fine-grain material had a water absorption rate of 5.29%, a specific gravity of 2.44, and a fineness modulus of 2.97. The fineness modulus (FM) of reducing slag varies greatly. The maximum value can be close to the fine aggregate of ordinary concrete, about 2.64, while the minimum value is only 0.13. The chemical composition of reducing slag is shown in Table 2.
- Bacterial solution: This contained *Sporosarcina pasteurii*, and its optical density (OD<sub>600</sub>) value was 1.0, as shown in Figure 2 (provided by Moji Technology Co., Ltd., Chiayi City, Taiwan).
- Cement: This was a locally produced Type I Portland cement, with a specific gravity of 3.15, and its chemical composition is shown in Table 2.
- Superplasticizer: An R-550 produced by Taiwan Sika Company was used.

Table 2. The chemical composition of reducing slag and cement.

Chemical Composition (%)	Reducing Slag	Cement
Silicon dioxide, SiO <sub>2</sub>	30.34	20.49
Aluminum oxide, Al <sub>2</sub> O <sub>3</sub>	2.01	6.57
Iron oxide, $Fe_2O_3$	2.21	3.27
Calcium oxide, CaO	50.49	62.40
Magnesium oxide, MgO	10.06	1.91
Sulfur trioxide, $SO_3$	-	2.20
Free calcium oxide, f-CaO	-	1.03
Titanium dioxide, TiO <sub>2</sub>	0.15	-
Phosphorus Pentoxide, P <sub>2</sub> O <sub>5</sub>	0.54	-
Manganese oxide, MnO	0.10	-
Loss on ignition, LOI	0.56	1.57
Tricalcium silicate, $C_3S$	-	39.27
Dicalcium silicate, C <sub>2</sub> S	-	29.11
Tricalcium aluminate, C <sub>3</sub> A	-	11.75
Tetracalcium aluminoferrite, C <sub>4</sub> AF	-	9.96
Calcium sulfate dihydrate, CSH <sub>2</sub>		4.73



Figure 1. Appearance of fresh EAF reducing slag.



Figure 2. Bacterial solution containing Sporosarcina pasteurii.

The appearances of the ERSAs according to their stabilization method, immersion age, and particle size are shown in Figures 3 and 4, respectively.



Figure 3. Appearance of the ERSAs with a diameter of 2 mm.



Figure 4. Appearance of the ERSAs with a diameter of 10 mm.

# 2.3. Mix Proportions of the Concrete and Casting of Specimens

In this study, unstabilized and stabilized ERSAs were used to prepare the control group concrete and the experimental group concrete, respectively. Currently, the water-cement ratio of concrete mix commonly used in Taiwan's engineering industry is between 0.38 and 0.40. In addition, in order to easily compare the performance of the two groups of concrete, the same water-cement ratio was used for the two groups of concrete. Therefore, the water-cement ratios of the two groups of concrete were both 0.39, and their mixture proportion is shown in Table 3.

Table 3. Mixture proportions of the concrete.

Mix Designation	Cement (kg/m <sup>3</sup> )	Water (kg/m <sup>3</sup> )	Coarse Aggregate (kg/m <sup>3</sup> )	Fine Aggregate (kg/m <sup>3</sup> )	Superplasticizer (kg/m <sup>3</sup> )	Note
MN	508	197	865	721	10	Untreated raw ERSA
MB	508	197	865	721	10	ERSA treated by immersion in a solution of <i>S. pasteurii</i> bacteria
MW	508	197	865	721	10	ERSA treated by immersion in a water tank

The mixing and preparation procedures for each group of concrete specimens are as follows:

- (1) Sieve various ERSAs (stabilized and unstabilized) out of fine and coarse aggregates.
- (2) Put the cement and fine ERSAs into the forced single-shaft mixer and dry mix for 30 s until evenly mixed.
- (3) Pour water into the mixing drum and mix for 60 to 90 s to form a uniform cement mortar.

(4) Pour in the coarse ERSAs and mix for 90 to 120 s to make it a uniform fresh concrete.

After the concrete was mixed, the slump test was carried out first, and the rest was used for pouring the specimens. Subsequently, 45 cylindrical specimens of  $\varphi$ 100 mm × 200 mm and 27 cubic specimens of 50 mm × 50 mm × 50 mm were poured. Cylindrical specimens were poured into two layers, and each layer was compacted with vibrating rods; cubic specimens were compacted with rubber tamping rods and rubber mallets. After 24 h, the specimens were removed from the formwork and then cured in water. One day later, the specimens were taken out and maintained in the air. When the age of the specimen reached the planned age of the test, the compression test and surface observation were carried out.

#### 2.4. Test Equipment and Methods

The main equipment used in this research includes a Blue Wave double-hole aerator, frequency conversion electric soil compactor, electric hydraulic soil sampler, constant temperature heating water tank, high-energy vibration ball mill, digital titrator, magnetic stirrer, high-speed centrifuge, desktop pH meter, thermogravimetric analyzer, high-resolution X-ray diffractometer, SEM pre-processing gold plating machine, scanning electron microscope, etc. The methods of the main test items are introduced below.

### 2.4.1. Method of Test for Potential Expansion of Aggregates from Hydration Reactions

In this study, the potential expansion test of reducing slag by hydration was in accordance with CNS 15311 [54]. The aggregates were compacted into specimens at the optimal moisture content and loaded with load plates, as shown in Figure 5a. Then, they were immersed in water with a temperature of  $70 \pm 3$  °C for seven days (as shown in Figure 5b), and the feasibility of aggregate reuse was evaluated based on the amount of expansion.



**Figure 5.** Test for the potential expansion of aggregates from hydration reactions: (**a**) aggregates that filled in the specimen module; (**b**) specimens immersed in the water tank.

2.4.2. Determination of f-CaO in Steel Slag by EDTA Complexometric Titration

The f-CaO content test of reducing slag was based on the literature [55]. The test steps are listed below.

- (1) Take 20 g of dried aggregate and grind it to pass through a no. 100 sieve. Precisely weigh 0.200 g and add 25 mL of ethylene glycol solution, then heat for half an hour  $(150 \pm 5 \text{ °C})$ .
- (2) Use centrifugal filtration and wash thoroughly with cold water. The total filtrate is about 100 mL, and then placed in a 300 mL Erlenmeyer flask for analysis.
- (3) Measure about 0.3 g of vitamin C into a 300 mL Erlenmeyer flask and adjust the pH value to 12.5–13 with potassium hydroxide aqueous solution (30%). Add 5 mL of triethanolamine solution (1:1) as a masking agent, and then add calcium red indicator (about 0.1–0.2 g).

- (4) Finally, titrate with 0.02 M Ethylenediaminetetraacetic acid (EDTA). When the color changes from purple to blue, the endpoint of the titration is reached and the titer volume is recorded.
- 2.4.3. Method for Analyzing and Testing Content of f-MgO in Steel Slag

The f-MgO content test of reducing slag was also based on the literature [56]. The test steps are listed below.

- (1) Take 20 g of dry aggregate and grind it to pass through the no. 100 sieve. Accurately weigh 0.200 g, add 25 mL of ethylene glycol solution and 6 mL of iodine-ethanol solution, and then heat for one hour ( $170 \pm 5 \,^{\circ}$ C).
- (2) Use centrifugal filtration and wash thoroughly with cold water. The total filtrate is about 60 mL and then placed in a 300 mL Erlenmeyer flask for analysis.
- (3) Add two or three drops of hydrochloric acid (1:1) solution into a 300 mL Erlenmeyer flask to acidify the solution. Use 6 mL of sodium thiosulfate to remove excess iodineethanol solution until the solution is transparent and colorless. Then, add 5 mL of triethanolamine (1:1) solution, 10 mL of ammonium chloride–ammonia buffer solution with a pH of 10, and two or three drops of K-B indicator.
- (4) Titrate with 0.02 mol/L EDTA standard solution until the color of the solution changes from reddish-brown to blue-green. Record the volume of EDTA solution consumed and calculate the value of free magnesium oxide.

# 2.4.4. pH Value Test

The hydrogen ion concentration index (pH value) of the reducing slag sample was measured according to the method for measuring the hydrogen ion concentration index (pH value) of wastes announced by the Environmental Inspection Institute—Electrode Method (NIEA R208.04C) [57]. The test steps are listed below.

- (1) Calibrate the pH meter using two different standard solutions.
- (2) Take  $20 \pm 0.2$  g of the sample and place it in a centrifuge tube. Then, add 20 mL of reagent water and shake continuously to mix it evenly.
- (3) Place the centrifuge tube in the centrifuge and run for one minute. Afterward, the upper aqueous phase layer can be obtained, and the pH value of the aqueous phase layer can be measured.

# 2.4.5. TGA/DTA Test

This test method involves placing the sample under a heating instrument with controllable temperature (heating, cooling, and constant temperature), through which nitrogen gas is introduced and the temperature continues to rise. When the evaporation temperature and cracking temperature of a certain material component in the sample are reached, weight loss will occur, and the proportion of the sample components can be known. The test steps are listed below.

- (1) Take the reducing slag that passes the no. 100 sieve and dry it for subsequent testing.
- (2) Turn on the nitrogen cylinder and the instrument, place the crucible in the instrument to zero, and put 5 to 10 mg of the sample into the crucible.
- (3) Set the temperature parameters and increase the temperature from 30 °C to 800 °C at a rate of 20 °C/min. After rising to 800 °C, keep the temperature at 800 °C for half an hour. After that, the temperature is lowered to 150 °C at 50 °C/min to complete the test. The detailed temperature curve is shown in Figure 6.



Figure 6. Heating time curve of the TGA test.

# 2.4.6. SEM Observation and XRD Test

A scanning electron microscope was used to observe the surface morphology of reducing slag and the surface crystallization characteristics of the carbonation reaction product. By comparing the differences between different reducing slag samples, electron microscopic images were obtained to explore the physical and chemical properties of reducing slag. The reducing slag powder of the control group and experimental group were analyzed for microscopic appearance. First, the powder sample was placed on a glass slide and coat it with gold film. Afterward, electron images of the samples placed in the instrument were taken under the scanning electron microscope of the vacuum system to observe the morphology of calcium carbonate crystals. XRD was used to observe the content of calcite (CaCO<sub>3</sub>) produced by the biomineralization reaction. This test generates a pattern through single-wavelength X-ray diffraction measurement. When reflected on a specific crystal surface, the reflected light can only be received at a certain angle. Therefore, it is unique and can represent the lattice characteristics of a certain crystal. Afterward, the spectra are compared with the database, and the compounds of the sample are deduced. In addition, the intensity ratio of the spectra is used to estimate the approximate percentage of the compounds in the sample. The differences in reducing slag samples treated with different stabilization methods can be identified, as well as the differences in composition percentage. Moreover, the compressive strength of the concrete cylindrical specimens was tested according to ASTM C39 [58].

### 3. Results and Discussion

The content of f-CaO and f-MgO in the reducing slag was the main factor that caused its volume expansion [17,18,26]. In view of this, the content of f-CaO and f-MgO can be used as a benchmark to measure the stabilization degree of ERSAs. However, the precise values of f-CaO and f-MgO contents are not easy to measure. Therefore, this study also used other qualitative test methods as auxiliary evaluation methods. Based on the results of the various tests, the feasibility of applying biomineralization technology to the stabilization of ERSAs is discussed below.

#### 3.1. Results of the Potential Expansion Test of ERSAs from Hydration Reactions

The test results showed that, as the hydration time increased, the expansion rate of each group of aggregates also increased, as shown in Figure 7. The expansion rate of the raw ERSAs in the control group (Group N) reached 0.53% after being hydrated for seven days, thus exceeding the standard of less than 0.5% that is required in Taiwan's current regulations. In contrast, the expansion rate in the stabilized ERSAs was less than 0.5%. It can be seen from Table 4 that after being hydrated for seven days, the expansion rate in the ERSAs of the experimental group (Group B) treated by immersion in the bacterial solution was between 0.28% and 0.36%, which did not exceed the standard value. The expansion

rate of the ERSAs in Group B was 32.1% to 47.2% lower than that of a raw ERSA. The above data show that biomineralization has a significant effect on the stabilization of ERSAs.



Figure 7. Relationship between the ERSA expansion rate and hydration time.

Table 4. Results of the potential expansion of ERSAs under hydration.

Sample		Expansion	n Rate in th	e First 7 Da	ys after Im	mersion in	Water (%)		Specification
Designation	Day 0	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Value (<0.5%)
AB-1D	0	0.05	0.11	0.15	0.19	0.23	0.27	0.31	< 0.5
AB-2D	0	0.05	0.07	0.12	0.16	0.2	0.24	0.28	< 0.5
AB-3D	0	0.08	0.12	0.16	0.20	0.23	0.27	0.29	< 0.5
AB-4D	0	0.07	0.12	0.17	0.21	0.27	0.33	0.36	< 0.5
AN	0	0.06	0.13	0.21	0.28	0.37	0.45	0.53	>0.5

# 3.2. Results of the Free Calcium Oxide Titration Test of ERSAs

The results of the f-CaO titration test of the ERSAs are shown in Table 5. It can be seen from Table 5 that under different treatment methods and treatment times, the f-CaO content of ERSAs with different particle sizes had roughly the same change trend. The f-CaO content of the ERSAs in the control group (Group N) without stabilized treatment ranged from 3.36% to 3.95%. The f-CaO content of the ERSAs in Group B ranged from 2.46% to 3.50%. The f-CaO content of the ERSAs of the experimental group (Group W) that were treated by immersion in water ranged from 2.82% to 3.86%. When taking the immersion age of four days as an example, the f-CaO content of Group B decreased by 23.1–26.8%, while the f-CaO content of Group W decreased by 2.1–19.0%.

Table 5. Titration test results of the f-CaO content in ERSAs.

Sample		f-CaO Co	ontent (%)	
Designation	1 mm	2 mm	5 mm	10 mm
N	3.95	3.73	3.48	3.36
B-1D	3.50	3.11	2.86	2.91
B-2D	3.25	3.15	2.58	2.68
B-3D	3.28	3.09	2.66	2.49
B-4D	2.97	2.87	2.56	2.46
W-1D	3.86	3.66	2.84	2.91
W-2D	3.74	3.69	2.86	2.87
W-3D	3.81	3.75	2.82	3.00
W-4D	3.74	3.65	2.82	3.08

Overall, the f-CaO content of each group of ERSAs generally increased with the decrease in the particle size, as shown in Figure 8. This may be due to the small particle size of the aggregate, which increased the specific surface area, resulting in a greater precipitation of f-CaO. Compared with the control group, under the same particle size, the f-CaO content of Group B and Group W was lower. In particular when considering them under the same particle size, the f-CaO content of Group B was also lower than that of Group W. The research results of Wang et al. [17] showed that the stability of steel slag with a free CaO content of 4.96% is poor. Motz and Geiseler [59] proposed that when the f-CaO content is less than 4%, it can be used for asphalt pavement. Bohmer et al. [60] believed that when the f-CaO content in steel slag is greater than 7%, it should not be used as a construction aggregate. Accordingly, this result once again verified that biomineralization has a definite effect on the stabilization of ERSAs.



Figure 8. The f-CaO content of ERSAs with different particle sizes.

### 3.3. Results of the Free Magnesium Oxide Titration Test of ERSAs

The f-MgO titration test results of ERSAs are shown in Table 6. The f-MgO content of ERSAs in the control group ranged from 5.83% to 7.20%. The f-MgO content of the ERSAs of Group B ranged from 4.48% to 6.45%. The f-MgO content of the ERSAs of Group W ranged from 4.17% to 7.05%. Compared with the control group, under the same particle size, the f-MgO content of Group B and Group W was lower. However, under the same particle size, the f-MgO content of Group B was not significantly lower than that of Group W; in fact, it was even higher than that of Group W, as shown in Figure 9. This may be due to the obvious response of biomineralization metabolism to calcium and the limited response to f-MgO. Taking the immersion age of four days as an example, the f-MgO content of 7.68% did not exhibit poor stability. Accordingly, the results of this experiment proved that both biomineralization and water immersion can reduce the f-MgO content of reducing slag in order to achieve a stabilization effect.

Table 6. Titration test results of the f-MgO content in ERSAs.

Sample		f-MgO Co	ontent (%)	
Designation	1 mm	2 mm	5 mm	10 mm
N	7.20	7.06	7.08	5.83
B-1D	6.45	5.56	5.55	4.51
B-2D	6.34	5.43	6.08	5.46
B-3D	5.43	5.44	4.50	5.32

Sample		f-MgO Co	ontent (%)	
Designation	1 mm	2 mm	5 mm	10 mm
B-4D	5.68	5.46	4.48	5.36
W-1D	7.05	6.55	5.21	4.95
W-2D	6.97	6.02	5.81	4.17
W-3D	5.93	4.39	5.59	4.19
W-4D	5.46	4.28	5.25	4.36

Table 6. Cont.



Figure 9. The f-MgO content of ERSAs with different particle sizes.

# 3.4. Results of the pH Value Test of ERSAs

In the reducing slag production process, a large amount of carbon powder and limestone must be added as auxiliary materials to facilitate deoxidation and to generate scum in order to completely reduce iron. This results in reducing slag containing a large amount of f-CaO and f-MgO, which are alkaline. In this study, the pH value test was used to qualitatively analyze the content of f-CaO and f-MgO, and the test results are shown in Table 7. The pH value of the ERSAs of the control group without stabilization treatment was as high as 12.47. The pH value of the ERSAs of Group W was closer to the test value of the control group, ranging from 11.61 to 12.28. This is because the f-CaO in the reducing slag reacted with water to form  $Ca(OH)_2$  [17,18]. In contrast, the pH value of the ERSAs of Group B was much lower, ranging from about 10.65 to 12.19. The results of this experiment also proved that biomineralization technology could be used as a stabilizing treatment for reducing slag.

Table 7. Results of the pH value test of the ERSAs.

Sample		pH V	/alue	
Designation	1 mm	2 mm	5 mm	10 mm
Ν	12.47	12.47	12.47	12.47
B-1D	12.19	11.85	11.34	10.97
B-2D	11.97	11.13	11.18	11.17
B-3D	11.43	11.20	11.28	11.19
B-4D	11.32	10.68	10.65	11.47
W-1D	12.17	12.28	11.95	11.92
W-2D	11.85	11.89	11.65	11.80
W-3D	11.86	11.68	11.62	11.90
W-4D	11.94	11.62	11.61	11.81

## 3.5. Results of Thermogravimetric Analysis of ERSAs

In this study, TGA/DTA was used to observe the weight loss of the ERSAs in order to explore the differences in their chemical composition after different stabilization treatments. Thermal weight loss is usually produced by the phase change in hydrated substances. The weight loss at 650–800 °C was mainly related to carbonate materials [52,61–63]. Accordingly, the TGA results can be used to calculate the percentage of weight loss from 650 °C to 800 °C, and can then be used to evaluate the difference in the  $CaCO_3$  content among the groups. The TGA results showed that the trends of the thermogravimetric curves of each group were almost similar, as shown in Figures 10–12 (where the TG curve refers to weight loss and DTA refers to the loss rate). From these figures, it can be seen that the compound composition of the reducing slag did not result in a great difference due to immersion in bacterial solution or water. Therefore, the difference in CaCO<sub>3</sub> content can be analyzed from the weight loss data for the temperatures between 650 and 800 °C, as shown in Table 8. It can be seen from Table 8 that, in this temperature range, the CaCO<sub>3</sub> content of the control group was 1.16%, while the CaCO<sub>3</sub> content of Group B was the highest, reaching between 1.42% and 1.56%. This showed that Group B contained a higher content of CaCO<sub>3</sub>. In other words, the amount of f-CaO that had been stabilized into CaCO<sub>3</sub> in Group B was larger, indicating a higher degree of stabilization, as shown in Figure 13.



Figure 10. The TGA and DTA curves of sample AN. Negative values are shown in red.



Figure 11. The TGA and DTA curves of sample AW4D. Negative values are shown in red.



Figure 12. The TGA and DTA curves of sample AB4D. Negative values are shown in red.

Samula Designation	Weight Per	$C_{2}C_{2}$ $C_{2}$	
Sample Designation —	650 °C	800 °C	- CaCO <sub>3</sub> Content (%)
AN	92.94	91.78	1.16
AB1D	92.81	91.34	1.47
AB2D	92.85	91.43	1.42
AB3D	93.2	91.64	1.56
AB4D	93.23	91.78	1.45
AW1D	93.53	92.53	1.00
AW2D	92.74	91.57	1.17
AW3D	93.22	92.14	1.08
AW4D	93.48	92.45	1.03

Table 8. The CaCO<sub>3</sub> content in each group of samples.



Figure 13. Comparison of the CaCO<sub>3</sub> content in each group of samples.

# 3.6. Results of SEM Observation and XRD Analysis of ERSAs

The above test results showed that the f-CaO in the Group B and Group W samples was effectively reduced, and calcium carbonate was generated. In order to understand the crystal formation inside the sample, the ERSAs of Group B were observed with a scanning electron microscope. As shown in Figure 14a, needle-like or long rod-like structures were observed. In addition, the SEM observation results of Group W samples are shown in

Figure 14b. It can be observed from the figure that the crystal structure was in the form of rounded edges and stacked sheets. In order to confirm the crystal composition, an EDS test was carried out. The results of the EDS analysis showed that the elements contained in the sample were C (CaCO<sub>3</sub>), O (SiO<sub>2</sub>), Mg (MgO), Al (Al<sub>2</sub>O<sub>3</sub>, Si (SiO<sub>2</sub>), S (FeS<sub>2</sub>), and Ca (calcium silicate). Accordingly, it can be concluded that the crystals observed in the SEM images were calcium carbonate crystals. This can prove that these calcium carbonate crystals were generated by the digestion of f-CaO in the reducing slag by the carbonate ion produced by the metabolic reaction of the microbial bacterial liquid. In contrast, no calcium carbonate crystals were observed in the Group N sample, as shown in Figure 14c.



Figure 14. SEM images and EDS analysis results: (a) Group B, (b) Group W, and (c) Group N.

On the other hand, each group of ERSAs was dried, ground, and sieved, and then subjected to XRD analysis. The spectra produced by the diffraction of X-rays are shown in Figures 15–17. According to the database data, the composition components of each group of samples were compared, and the results are shown in Table 9. In the ERSAs of the control group, the presence of CaCO<sub>3</sub> was not detected. The ERSAs of Group W had a CaCO<sub>3</sub> content of 4.76%. In contrast, the CaCO<sub>3</sub> content of the ERSAs in Group B was 8.53%. This result showed that there was a great deal of f-CaO in the reducing slag that had reacted via microorganisms in order to produce more stable CaCO<sub>3</sub>. In other words, the aggregates of Group B had an obvious stabilizing effect after being immersed in the bacterial solution.



Figure 15. The XRD results of Group N.



Figure 16. The XRD results of Group W.



Figure 17. The XRD results of Group B.

Compound		Percentage of Ingredients (%)			
Compound	Molecular Formula –	Group N	Group W	Group B	
Calcio olivine	CaSiO <sub>4</sub>	30.12	28.16	21.48	
Spinel	MgAl <sub>2</sub> O	0	12.58	9.76	
Gehlenite	$Ca_2AI[AlSi_2O_7]$	8.44	8.63	11.16	
Merwinite	$Ca_3Mg(SiO_4)_2$	29.89	22.61	27.93	
Katoite	$Ca_3Al_2(SiO_4)_{3-X}(OH)_{4X}$	5.13	3.63	2.65	
Brucite	Mg(OH) <sub>2</sub>	4.99	5.02	5.21	
Portlandite	Ca(OH) <sub>2</sub>	2.40	1.56	2.47	
Cuspidine	$Ca_4(Si_2O_7)(OH)_2$	11.10	1.65	1.37	
Periclase	MgO	4.46	4.44	2.60	
Gypsum	$CaSO_4 \cdot 2H_2O$	0	1.38	1.29	
Fluorite	CaF <sub>2</sub>	0.13	0.15	0.02	
Grossular	Ca <sub>3</sub> Al <sub>2</sub> (SiO <sub>4</sub> ) <sub>0.69</sub> (OH) <sub>9.24</sub>	3.34	5.43	5.51	
Quartz	SiO <sub>2</sub>	0	0	0	
Calcite	CaCO <sub>3</sub>	0	4.76	8.53	

Table 9. The XRD analysis results of each group of samples.

#### 3.7. Properties of Concrete Produced with ERSAs

The above tests confirmed that the use of a *Sporosarcina pasteurii* bacteria solution can effectively stabilize ERSAs, and that immersion in water also has a certain stabilization effect. These results show an initial achievement in the goal of applying biomineralization technology to the stabilization of ERSAs. In view of this, the feasibility of reusing stabilized ERSAs in concrete was further explored, and they were compared with the control group (which used raw ERSAs). This was achieved mainly through the compression test to understand the strength development trend of the prepared concrete. In addition, long-term observations were made on the surface of each group of concrete specimens to understand whether there was ERSA expansion, as well as to understand the conditions that cause concrete cracks and spalling.

### 3.7.1. Compressive Strength of the Concrete Cylindrical Specimens

Sosa et al. [9] advised that due to the higher mechanical strength and roughness of EAF slag and an improved paste-aggregate interface transition zone, the compressive strength of concrete that is produced using ERSAs was superior to that of natural aggregate concrete. Therefore, the concrete mixed with stabilized and unstabilized reduced slag aggregates in this study could exhibit good compressive strength. In particular, the compressive strength of the control group (which used raw ERSAs) concrete was higher than that of the experimental group at an early age and at 28 days. The compressive strengths of each group of concrete specimens at different ages are shown in Figures 18–20, respectively. In addition, based on the compressive strength of each group of concrete at 28 days, the strength development curves are also shown in Figures 18–20, respectively. It can be seen from these figures that with the increase in the curing age of the specimens, the strength growth trends of each group of concrete were different. The compressive strength of the Group N concrete began to decrease after 28 days, and this was seemingly due to the expansion of the raw ERSAs. The strength growth percent of the Group W concrete was 102.4% at 90 days, but this decreased to 87.6% at 180 days. In contrast, the compressive strength of the Group B concrete continued to increase with age, whereby its growth percentage of compressive strength was 104.8% at the age of 90 days and 111.3% at the age of 180 days. These results showed that the ERSAs treated with MICP had a good stabilization effect, and thus, the phenomenon of concrete volume expansion did not occur.



Figure 18. Trend of the compressive strength of Group N concrete at different ages.



Figure 19. Trend of compressive strength of Group W concrete at different ages.



Figure 20. Trend of compressive strength of Group B concrete at different ages.

3.7.2. Long-Term Surface Observation of Concrete Cube Specimen

The results of the long-term surface observation of the concrete cube specimen (50 mm  $\times$  50 mm  $\times$  50 mm) prepared with ERSAs are shown in Figure 21. It can be seen from Figure 21 that at the age of 240 days, the specimens treated with different stabilization methods compared to those that were unstabilized showed different results. The difference between each test group can be further observed by using the crack observer to zoom in. Among them, there were cracks or blast holes clearly visible to the naked eye on the surface

of the Group N concrete specimens. Some micro-cracks also appeared on the surface of the Group W concrete specimens. In contrast, the specimens of the Group B concrete had the smallest cracks. Moreover, the Group B concrete could still maintain the integrity of its surface after 240 days of aging, and no expansion cracks or concrete bulging occurred. Wang et al. [17] advised that when f-CaO content was within 2.09%, steel slag could still maintain a good integrity for more than 4 years. However, when f-CaO content was 4.96%, the soundness of steel slag was poor, which led to the soundness-induced failure of the concrete. Based on this, it can be inferred that the stabilization treatment of ERSAs when using a *Sporosarcina pasteurii* bacterial solution is effective.

Concrete Group	At the age of one day	At the age of 240 days
Control group (Group N)		5 6 7 8 9 10 11
Experimental group (Group W)		
Experimental group (Group B)		

Figure 21. Long-term observation results of the surface of each group of concrete cube specimens.

## 3.8. Summary and Application of Test Results

This study experimentally evaluated the degree of stabilization of biomineralized ERSAs. The test results of the potential expansion rate due to hydration showed that the seven-day expansion amount of ERSAs does not exceed the value specified in Taiwan's Recycling Management Regulations. The pH test showed that the pH value of ERSAs decreased after biomineralization, and the f-CaO content decreased. The titration calcium oxide test also proved that the f-CaO content of ERSAs was greatly reduced. The thermogravimetric analysis and X-ray diffraction analysis tests also proved that a large amount of f-CaO in ERSAs was biomineralized to form calcium carbonate, and calcium carbonate crystals were observed using scanning electron microscope SEM. The above experiments preliminarily prove that the f-CaO content of ERSAs after biomineralization was reduced to the level of stabilization. The compressive strength test results of the concrete produced by biomineralized ERSAs showed that the compressive strength of the specimens did not decline due to expansion at 180 days, and there was no visible crack on the surface of the specimens at 240 days. This result also showed that the stabilization treatment of ERSAs using the biomineralization technology of *Sporosarcina pasteurii* bacteria is effective.

The *S. pasteurii* bacteria used in this study is commonly found in animals. This bacteria belongs to the beneficial bacteria organisms and does not harm the environment. In particular, it is easy to cultivate in large quantities, and the required expenses are not high. Therefore, biomineralization applied to reducing slag is a relatively simple and cheap stabilization method and is easily accepted by the steel industry. In particular, MICP is an innovative and sustainable engineering technology that contributes to the achievement of specific United Nations Sustainable Development Goals (SDGs) [64]; for example, SDG 11: Sustainable Cities and Communities, SDG 9: Industry, Innovation, and Infrastructure, SDG 12: Responsible Consumption and Production.

In this study, biomineralization technology was used to stabilize reducing slag, mainly to biomineralize f-CaO near the surface of reducing slag to generate calcium carbonate. This study has not explored the depth at which reducing slag mineralization can be achieved. This part is related to the size and distribution of pores inside the reducing slag. Therefore, more in-depth research is still needed.

#### 4. Conclusions

In this study, the feasibility of utilizing biomineralization technology for stabilizing ERSAs was rigorously investigated through a comprehensive experimental approach. The stabilization efficacy of ERSA was evaluated using expansion rates, f-CaO and f-MgO content, pH values, and CaCO3 content. The ERSAs stabilized by immersion in the bacterial solution had a potential expansion rate of approximately 0.28% to 0.36% after being hydrated for seven days, aligned with Taiwan's waste recycling management regulations. Notably, compared with the raw ERSAs, the reduction in expansion rate ranged from 32.1% to 47.2%. The f-CaO content reduction through bacterial solution immersion demonstrated the stabilization potential. pH values indicated successful biomineralization treatment. Furthermore, analyses confirmed the conversion of f-CaO into CaCO<sub>3</sub>, validating stabilization. On the other hand, concrete specimens incorporating stabilized and unstabilized ERSAs were tested, revealing crucial insights. Importantly, concrete incorporating MICP-treated ERSAs exhibited superior compressive strength and structural integrity, suppressing volume expansion issues. This study highlights biomineralization technology's potential to enhance the sustainability of construction materials by efficiently stabilizing waste aggregates for concrete production.

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