

Effect of Carbonated Magnesium Slag Powder on the Carbonation Curing Performance of Magnesium Slag

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Abstract

This study proposes a waste valorization strategy in which carbonated magnesium slag powder (CMSP), prepared by semi-dry carbonation, is reused in a magnesium slag carbonation curing system. The preferred powder preparation conditions were a liquid-to-solid (L/S) ratio of 0.20 and a carbonation time of 4 h. Under identical conditions of a compaction pressure of 10 MPa, a carbonation time of 2 h, and an L/S ratio of 0.08, replacing 10% of the untreated magnesium slag with CMSP increased the compressive strength to 103.06 MPa, representing a 41.3% improvement over the specimen without CMSP. Subsequent sequential single-factor experiments identified a combination of 10% CMSP replacement, an L/S ratio of 0.10, a compaction pressure of 8 MPa, and a carbonation time of 8 h, at which the compressive strength and CO₂ uptake reached 108.45 MPa and 15.5%, respectively. These latter values reflect the combined effects of CMSP incorporation and changes in processing conditions. X-ray diffraction, Fourier transform infrared spectroscopy, and scanning electron microscopy confirmed the presence of crystalline calcite in the carbonated products and revealed changes in local microstructure and carbonate-related signals after CMSP incorporation. The results provide a basis for reusing CMSP in carbonation-cured magnesium slag products.

Keywords

Magnesium Slag; Semi-dry Carbonation; Carbonated Magnesium Slag Powder; Compressive Strength.

1. Introduction

Magnesium slag (MS) is a solid waste generated during the production of metallic magnesium [1, 2]. The production of one tonne of metallic magnesium generates approximately 5.5–10 tonnes of MS [3] and is associated with substantial CO₂ emissions [4]. Developing routes for MS valorization is therefore important. Its chemical composition resembles that of cement [5, 6], suggesting its potential as a raw material for cement production. Ji et al. [7] investigated the use of MS as a mineral admixture to improve concrete performance. However, delayed hydration of residual reactive components, including free CaO (f-CaO) and MgO, can cause volume expansion and, in severe cases, matrix cracking, which limits the use of MS in building materials [8, 9]. Carbonation can improve volume stability [10, 11]. Ye et al. [12] investigated wet carbonation of MS. Wet carbonation nevertheless has drawbacks, including high energy demand, whereas the relatively low water demand of semi-dry carbonation offers potential for large-scale application. Existing studies have mainly examined carbonated MS in cement-based systems, with limited attention to reusing CMSP in the carbonation curing of MS itself. Whether CMSP improves the performance of MS products, the conditions under which such improvement occurs, and the dominant mechanisms remain unclear. Accordingly, this

study prepares CMSP by carbonation and reuses it in an MS carbonation curing system, with a focus on the effects of CMSP incorporation on CO₂ uptake and mechanical performance.

2. Experimental Methods

2.1 Raw Materials

The MS used in this study was obtained from Yulin, Shaanxi Province, China. Its chemical composition and mineral phases are presented in Table 1 and Figure 1(a), respectively. The major chemical constituents were CaO, SiO₂, MgO, and Fe₂O₃, and the principal mineral phases were β -C₂S, γ -C₂S, and MgO, where C₂S denotes dicalcium silicate. Figure 1(b) shows the particle size distribution of MS. The median particle diameter (D₅₀) was approximately 18.1 μ m, and the specific surface area was 809.6 m²/kg.

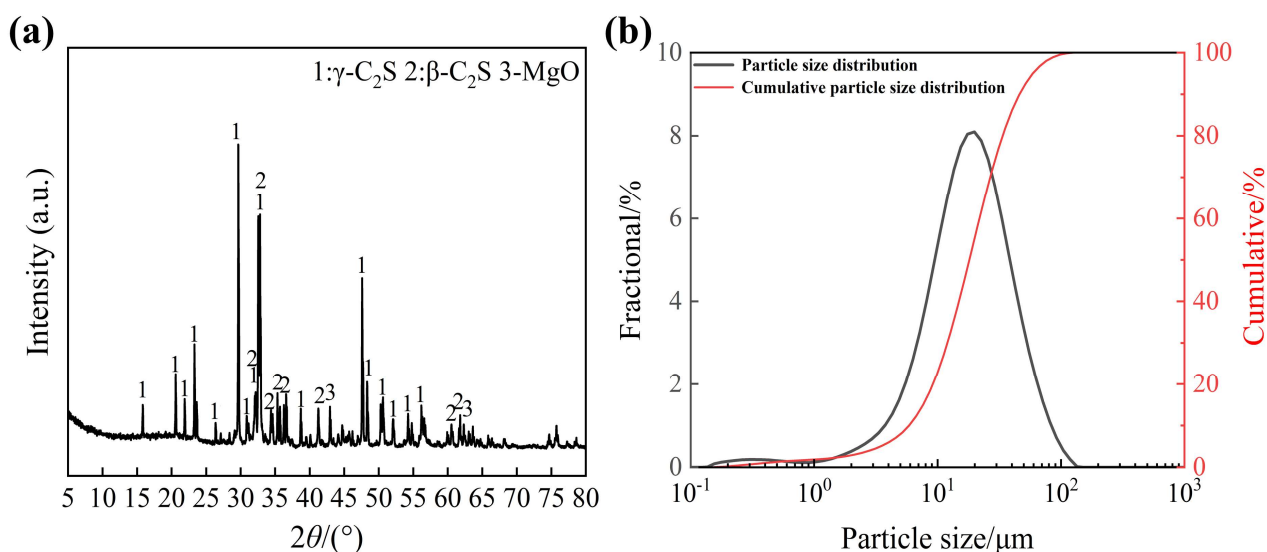


Figure 1. (a) XRD pattern and (b) particle size distribution of MS.

Table 1. Chemical composition of MS (%).

Material	CaO	SiO ₂	MgO	Fe ₂ O ₃	Al ₂ O ₃	Na ₂ O	SO ₃	MnO	Others
MS	58.07	32.99	3.85	2.97	1.21	0.262	0.238	0.0761	0.3339

2.2 Preparation of CMSP and Carbonation-Cured MS Specimens

MS was ball-milled for 10 min and dried in a forced-air oven at 105 °C for 24 h. The L/S ratio was defined as the mass ratio of water to the total solid materials. The dried MS was mixed with water for 5 min at L/S ratios of 0.08, 0.12, 0.16, 0.20, and 0.25. The mixtures were placed on a mesh screen, spread evenly, and transferred to a 75 L pressurized carbonation reactor. Carbonation was conducted at room temperature using 99% pure CO₂ at a pressure of 0.2 MPa for 1, 4, 8, or 12 h. During the L/S-ratio screening, the carbonation time was fixed at 1 h, whereas during the carbonation-time screening, the L/S ratio was fixed at 0.20. After carbonation, the resulting powder was dried at 50 °C to obtain CMSP. The CMSP replacement ratio was defined as the mass percentage of CMSP in the combined mass of CMSP and untreated MS, with the total solid mass kept constant by replacing untreated MS with an equal mass of CMSP. The resulting CMSP was then mixed with untreated MS and water for 5 min at replacement ratios of 0%, 3%, 6%, 10%, and 15%. Cubic specimens measuring 20 × 20 × 20 mm were prepared at L/S ratios of 0.08, 0.10, 0.12, and 0.16 and compaction pressures of 3, 5, 8, and 10 MPa. The specimens were subsequently carbonated at room temperature using 99% pure CO₂ at a pressure of 0.2 MPa for 2, 4, 8, or 16 h. In this study, CMSP refers specifically to the

powder obtained by semi-dry carbonation of MS and used to partially replace untreated MS in the preparation of carbonation-cured specimens.

A sequential single-factor experimental approach was adopted. The effect of CMSP replacement ratio was first evaluated at a fixed compaction pressure of 10 MPa, a carbonation time of 2 h, and an L/S ratio of 0.08. The effects of L/S ratio, compaction pressure, and carbonation time were then investigated sequentially, using the preferred conditions identified in the preceding stage. Within each stage, the effect of the variable under investigation was evaluated while the other processing parameters were held constant. Because the final processing conditions differed from those used for the initial specimen without CMSP, differences between these specimens represent the combined effects of CMSP incorporation and processing adjustments and do not quantify the independent contribution of CMSP. Specimens were prepared separately for each experimental stage; consequently, results from different stages were not compared directly.

2.3 Test Methods

2.3.1 CO₂ Uptake

For CMSP, the residual masses at 400 and 900 °C are denoted by m_{400} and m_{900} , respectively. CO₂ uptake associated with carbonate decomposition was calculated using Equation (1). For the carbonation-cured specimens, four specimens were prepared from the same batch of wet mixture for each group. Three were carbonated, while the fourth was subjected to the same handling conditions without carbonation and then dried at 50 °C to provide the uncarbonated dry-mass reference. The carbonated specimens were also dried at 50 °C for at least 48 h. CO₂ uptake was calculated from the difference between the dry masses using Equation (2), and the mean value for the three carbonated specimens was reported.

$$\text{CO}_2 \text{ uptake} = \frac{m_{400} - m_{900}}{m_{900}} \times 100\% \quad (1)$$

$$\text{CO}_2 \text{ uptake} = \frac{m_2 - m_1}{m_1} \times 100\% \quad (2)$$

In Equation (2), m_2 is the dry mass of a carbonated specimen, and m_1 is the dry mass of the uncarbonated reference specimen with the same formulation.

2.3.2 Compressive Strength

Compressive strength was measured using a compression testing machine (YNS-Y1000, China). The maximum compressive load at failure was recorded, and compressive strength was calculated using Equation (3). Considering the effect of the CO₂ diffusion path on carbonation uniformity, 20 mm × 20 mm × 20 mm cubic specimens were used. The loading rate was 1 mm/min. The resulting strengths were used to compare specimens tested under the same experimental conditions.

$$P = \frac{F}{S} \quad (3)$$

In Equation (3), P is the compressive strength (MPa), F is the maximum compressive load (N), and S is the loaded area of the specimen (mm²).

2.3.3 Physicochemical and Microstructural Characterization

The chemical composition of MS was determined by X-ray fluorescence (XRF; ARL Perform'X) at a tube voltage of 60 kV and a tube current of 120 mA. The mineral phases of CMSP and MS specimens were identified by X-ray diffraction (XRD; SmartLab) over a 2θ range of 5–80° at a scanning rate of 10°/min. Particle size distributions of CMSP and MS were measured using a laser particle size analyzer (Malvern 3000). Mass losses of carbonated samples were determined by

thermogravimetric analysis (STA8122) under nitrogen from 50 to 1000 °C at a heating rate of 10 °C/min. Changes in functional groups in CMSP and carbonated MS specimens were examined by Fourier transform infrared spectroscopy (FT-IR; BuckshP92FX) over the wavenumber range of 400–4000 cm⁻¹. Microstructural features of CMSP and MS specimens were observed by scanning electron microscopy (SEM; Merlin Compact). Elemental analysis of the carbonated MS specimens was performed by energy-dispersive X-ray spectroscopy (EDS; Oxford Instruments).

3. Results and Discussion

3.1 Phase Composition and Carbonation Extent of CMSP

3.1.1 XRD Analysis

Before carbonation, the principal crystalline phases in MS were β -C₂S, γ -C₂S, and MgO. As shown in Figure 2, the carbonated CMSP samples exhibited characteristic diffraction peaks of calcite, particularly at $2\theta \approx 29.40^\circ$, together with residual peaks of β -C₂S, γ -C₂S, and MgO. These observations indicate the formation of crystalline calcite during carbonation.

The relative diffraction peak intensities varied with the L/S ratio and carbonation time. Although these variations suggest changes in the crystalline phases under different preparation conditions, they do not directly quantify the extent of carbonation. Therefore, the effects of L/S ratio and carbonation time on carbonation performance were evaluated primarily using the CO₂ uptake results presented in Section 3.1.2. The influence of water availability and reaction time on carbonation has also been discussed in previous studies [13–15].

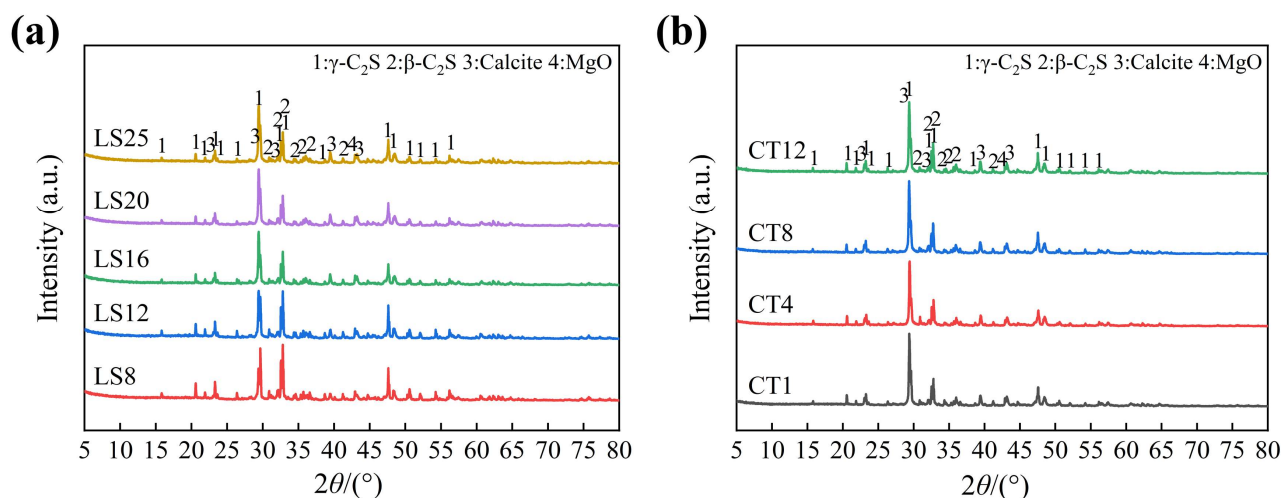


Figure 2. XRD patterns of CMSP prepared at different (a) L/S ratios and (b) carbonation times. LS8–LS25 denote L/S ratios of 0.08–0.25, and CT1–CT12 denote carbonation times of 1–12 h.

3.1.2 TG–DTG Analysis

Figure 3 presents the thermogravimetric and derivative thermogravimetric (TG–DTG) curves of CMSP prepared at different L/S ratios and carbonation times. The thermal decomposition process exhibited three main mass-loss stages. Mass loss up to 200 °C was mainly attributed to the removal of free and bound water [16]. Mass loss at 400–600 °C was attributed to the decomposition of amorphous calcium carbonate [17], whereas that at 600–900 °C was attributed to calcite decomposition [18].

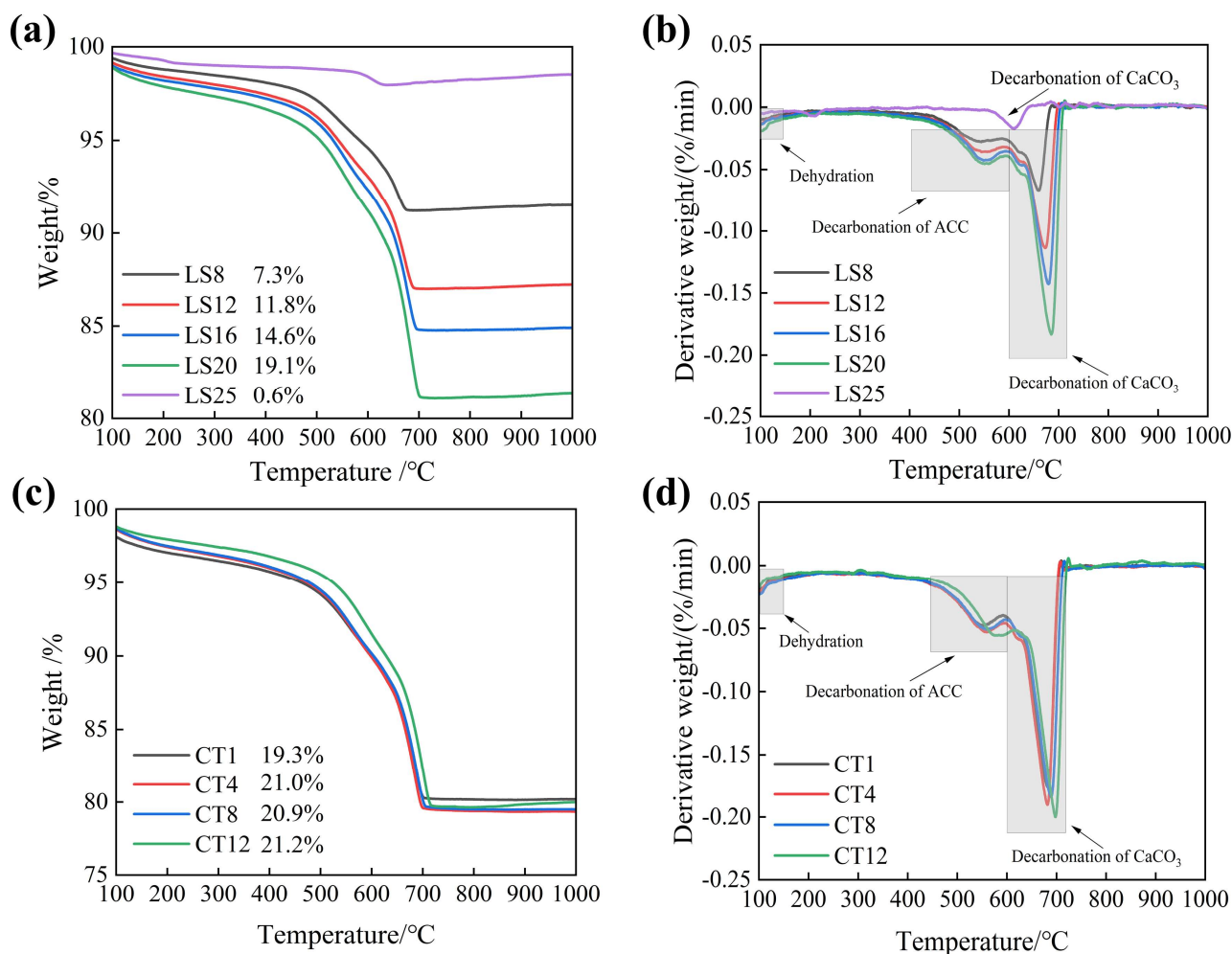


Figure 3. TG and DTG curves of CMSP at different L/S ratios (a, b) and carbonation times (c, d). LS and CT labels are defined in Figure 2; ACC denotes amorphous calcium carbonate.

Increasing the L/S ratio from 0.08 to 0.20 increased the CO₂ uptake of CMSP from 7.3% to 19.1%; a further increase to 0.25 reduced uptake to 0.6%. Excess water hindered CO₂ diffusion and limited the carbonation reaction [14]. Increasing the carbonation time from 1 to 4 h increased CO₂ uptake from 19.3% to 21.0%, after which uptake approached a plateau. Considering these results, an L/S ratio of 0.20 and a carbonation time of 4 h were selected as the preferred CMSP preparation conditions. CMSP prepared under these conditions was subsequently incorporated into MS specimens to investigate its effect on carbonation curing performance.

3.2 Effect of CMSP on the Carbonation Curing Performance of MS Specimens

3.2.1 CO₂ Uptake and Compressive Strength

The preferred CMSP replacement ratio was investigated at a fixed compaction pressure of 10 MPa, a carbonation time of 2 h, and an L/S ratio of 0.08. As shown in Figure 4(a, b), 10% CMSP replacement yielded a CO₂ uptake of 14.4% and a compressive strength of 103.06 MPa, representing increases of 11.6% and 41.3%, respectively, over the specimen without CMSP. At replacement ratios above 10%, CO₂ uptake approached a plateau, whereas strength decreased. This decrease may be associated with dilution of the carbonatable constituents of MS.

The effect of L/S ratio was then investigated at a CMSP replacement ratio of 10%, a compaction pressure of 10 MPa, and a carbonation time of 2 h. As shown in Figure 4(c, d), an L/S ratio of 0.10 yielded a CO₂ uptake of 14.8% and a compressive strength of 87.06 MPa, representing increases of 10.5% and 14.4%, respectively, over the specimen with an L/S ratio of 0.08 within this experimental

stage. Both CO₂ uptake and strength decreased at L/S ratios above 0.10, indicating a substantial influence of water content on specimen carbonation.

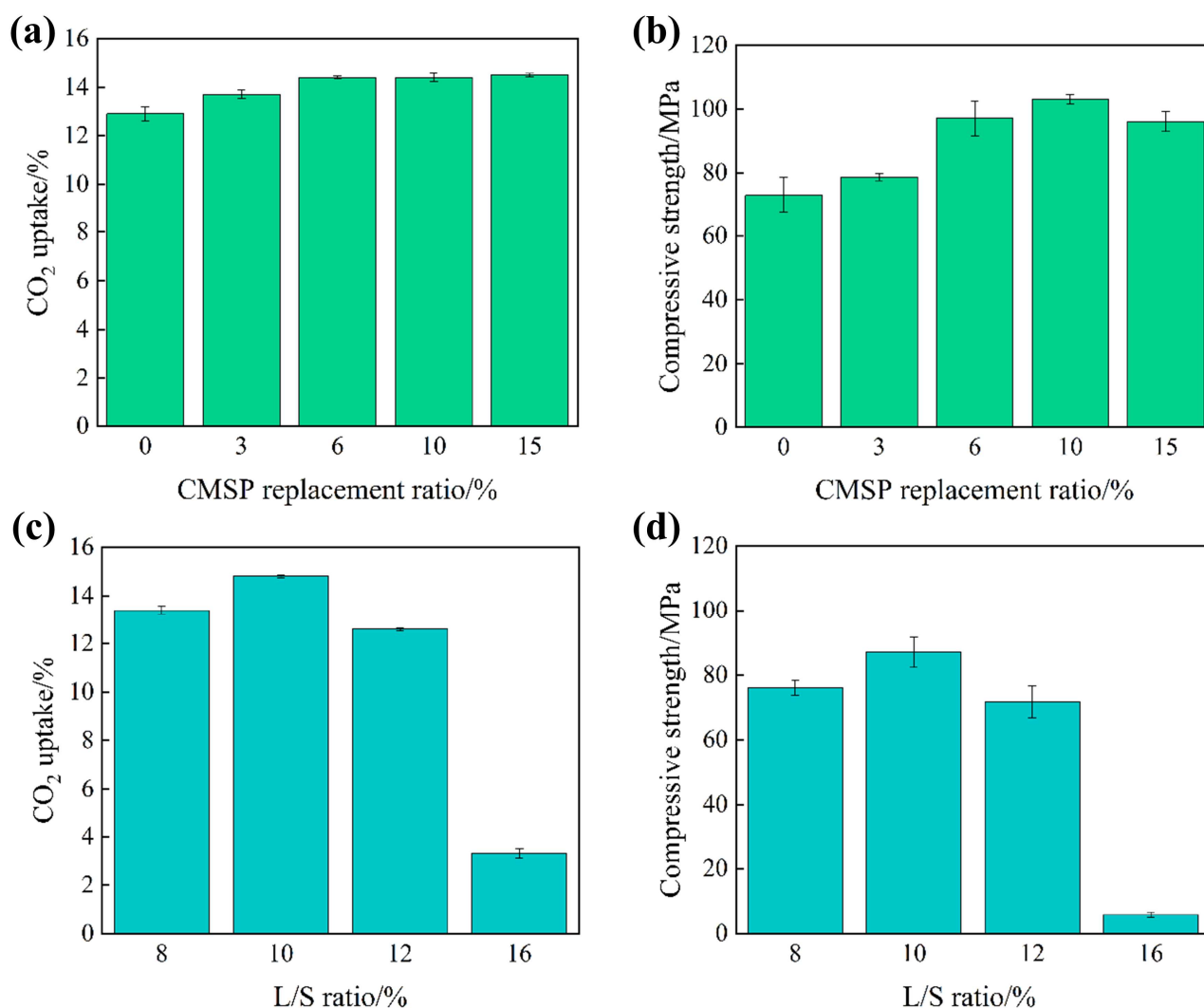


Figure 4. Effects of CMSP replacement ratio (a, b) and L/S ratio (c, d) on CO₂ uptake and compressive strength of MS specimens. The L/S ratios plotted as 8%, 10%, 12%, and 16% correspond to 0.08, 0.10, 0.12, and 0.16, respectively.

The preferred compaction pressure was investigated at a fixed CMSP replacement ratio of 10%, an L/S ratio of 0.10, and a carbonation time of 2 h. As shown in Figure 5(a, b), a compaction pressure of 8 MPa yielded a CO₂ uptake of 14.2% and a compressive strength of 100.48 MPa. Above 8 MPa, CO₂ uptake approached a plateau, while compressive strength decreased.

The effect of carbonation time was investigated at a fixed CMSP replacement ratio of 10%, an L/S ratio of 0.10, and a compaction pressure of 8 MPa. As shown in Figure 5(c, d), a carbonation time of 8 h yielded a CO₂ uptake of 15.5% and a compressive strength of 108.45 MPa. Both values remained relatively stable when the carbonation time exceeded 8 h.

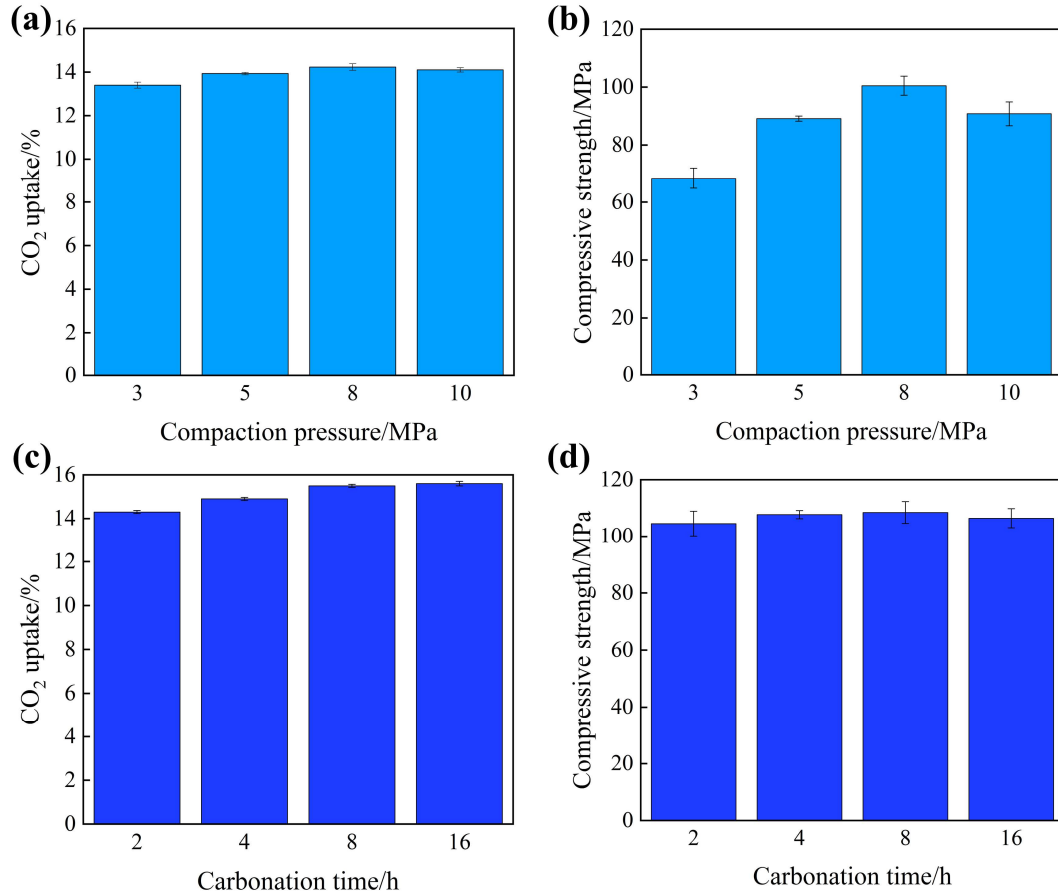


Figure 5. Effects of compaction pressure (a, b) and carbonation time (c, d) on CO₂ uptake and compressive strength of MS specimens.

3.2.2 XRD Analysis

Figure 6 presents the XRD patterns of carbonation-cured MS specimens prepared with different CMSP replacement ratios and carbonation times. The carbonated specimens exhibited diffraction peaks attributable to calcite, together with peaks of residual β -C₂S, γ -C₂S, and MgO. Variations in the relative diffraction peak intensities were observed among the specimens. No distinct diffraction peaks corresponding to crystalline MgCO₃ were detected. Magnesian calcite potentially formed during carbonation may have diffraction peaks overlapping those of calcite, making the two difficult to distinguish [19]. Therefore, the reaction pathway of MgO cannot be conclusively determined from the available XRD results.

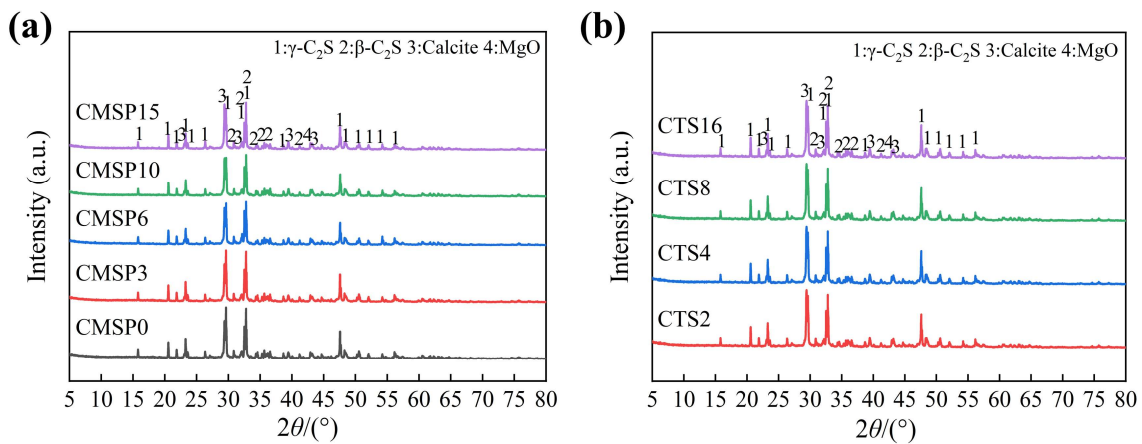


Figure 6. XRD patterns of carbonation-cured MS specimens with different (a) CMSP replacement ratios and (b) carbonation times. CMSP0–CMSP15 denote replacement ratios of 0–15%, and CTS2–CTS16 denote carbonation times of 2–16 h.

3.2.3 FT-IR Analysis

Figure 7 shows the FT-IR spectra of specimens prepared at different CMSP replacement ratios and carbonation times. The carbonated specimens exhibited calcite-related carbonate bands at 1420 cm^{-1} (asymmetric stretching), 875 cm^{-1} (out-of-plane bending), and 713 cm^{-1} (in-plane bending) [20]. The carbonate absorption band at 1420 cm^{-1} became more pronounced as CMSP replacement ratio and carbonation time increased.

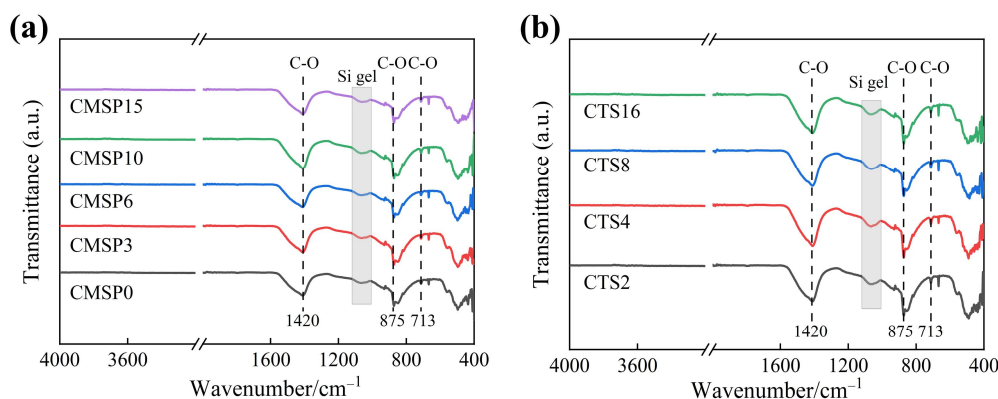


Figure 7. FT-IR spectra of MS specimens at different CMSP replacement ratios (a) and carbonation times (b). Sample labels are defined in Figure 6.

3.2.4 SEM Analysis

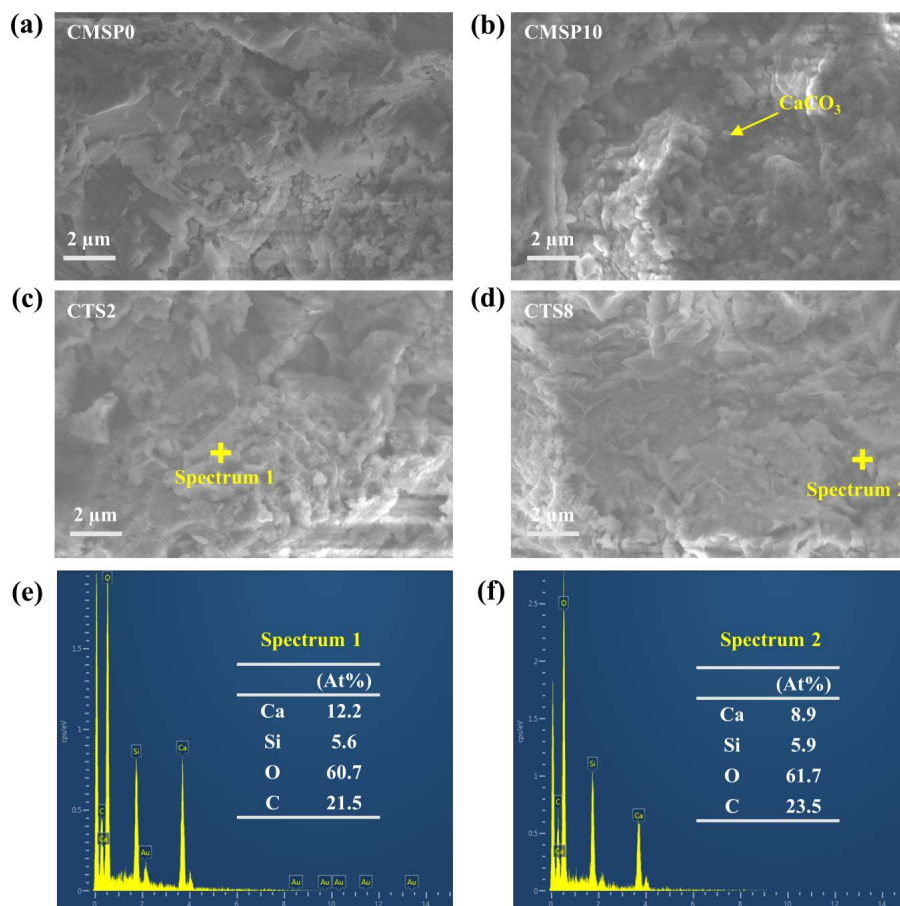


Figure 8. SEM images of MS specimens with 0% (a) and 10% (b) CMSP replacement and carbonation times of 2 h (c) and 8 h (d), together with the corresponding EDS spectra (e, f) of the marked locations in (c, d). Sample labels are defined in Figure 6; EDS denotes energy-dispersive X-ray spectroscopy.

Figure 8 presents the SEM images of specimens prepared at different CMSP replacement ratios and carbonation times. The specimen without CMSP exhibited a relatively loose structure. At 10% CMSP replacement, particle contacts were closer, consistent with the increase in compressive strength. With increasing carbonation time, the specimens became progressively denser, accompanied by a further increase in strength.

3.3 Microstructural Changes and Possible Roles of CMSP

The compressive strength results, together with the XRD, FT-IR, and SEM observations, suggest that CMSP incorporation may affect specimen performance by modifying particle packing and carbonate formation.

Changes in particle packing. The SEM observations showed closer particle contacts in local regions of the specimen with 10% CMSP replacement than in the specimen without CMSP, consistent with the observed increase in compressive strength.

In addition, carbonation products such as calcite can precipitate at particle contacts and bind particles together. Mo et al. [9] and Luo et al. [20] reported that this process can densify the matrix microstructure and improve mechanical performance.

Possible changes in carbonate formation. The XRD patterns confirmed the presence of crystalline calcite in the carbonated specimens, while the FT-IR spectra exhibited characteristic carbonate absorption bands.

The available XRD results cannot distinguish calcite initially present in the added CMSP from calcite newly formed during specimen carbonation. Because CMSP already contains calcite, and Mo et al. [9] reported that carbonation products can act as crystallization sites that promote aggregation and interconnection, it is hypothesized that CMSP particle surfaces may provide nucleation interfaces for subsequent carbonate formation.

Overall, CMSP incorporation was associated with changes in local microstructure, carbonate-related signals, and compressive strength. Quantitative characterization of pore structure evolution and carbonate nucleation is still needed.

4. Conclusion

(1) Carbonation conditions affected the phase composition and carbonation extent of CMSP. Increasing the L/S ratio from 0.08 to 0.20 increased the extent of carbonation, whereas a further increase introduced excess water that restricted CO₂ diffusion and reduced carbonation effectiveness. Increasing the carbonation time from 1 to 4 h increased the extent of carbonation, which subsequently approached a plateau. Based on the XRD and TG–DTG results, an L/S ratio of 0.20 and a carbonation time of 4 h were selected as the preferred CMSP preparation conditions.

(2) CMSP replacement ratio, specimen L/S ratio, compaction pressure, and carbonation time jointly affected CO₂ uptake and compressive strength. An appropriate CMSP replacement ratio may improve particle packing, whereas excessive replacement dilutes the MS constituents and reduces strength. An appropriate L/S ratio favors carbonation, while excessive water restricts CO₂ diffusion within the specimens and causes substantial reductions in strength and uptake. An appropriate compaction pressure improves specimen compactness; above 8 MPa, CO₂ uptake approached a plateau and strength decreased. Increasing the carbonation time to 8 h generally improved strength and CO₂ uptake, with both remaining relatively stable at longer times.

(3) Under identical processing conditions, 10% CMSP replacement produced a compressive strength of 103.06 MPa, 41.3% higher than that of the specimen without CMSP. Further adjustment of L/S ratio, compaction pressure, and carbonation time through sequential single-factor experiments yielded a compressive strength of 108.45 MPa and a CO₂ uptake of 15.5%. These results demonstrate the combined influence of CMSP incorporation and processing adjustments on specimen performance.

The contribution of this study is the preparation of CMSP by semi-dry carbonation and its reuse in carbonation-cured MS specimens, providing a route for valorizing MS within the same waste-derived material system.

Acknowledgments

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