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Design Optimization and Hydration Mechanism of a Composite Gypsum-Based Cementitious Material Made Entirely from Solid Waste Using Smelting Slag

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Abstract: The large-scale stockpiling of industrial solid waste poses serious environmental problems. Its co-utilization to produce low-carbon cementitious materials is an effective solution. In this study, desulfurization gypsum (DG), granulated slag (GS), copper slag (CS), and a calcium carbide slag–sodium silicate composite activator were used as raw materials to design a ternary, all-solid-waste-based cementitious material (SWCM). The Box-Behnken response surface method was used to optimize the mix proportions, with the 28-day linear expansion rate and the 7-day and 28-day compressive strengths as response variables. The optimal mix ratio was 18.4% DG, 2.1 GS/CS, and 1.6% activator, yielding a 28-day expansion rate of 0.40% and a compressive strength of 23.89 MPa, which meets the requirements for semi-rigid road base courses. The linear expansion rate increases with rising DG content, attributable to the extensive formation of ettringite. The compressive strength exhibits a threshold at 18% DG and a critical value at 2.1 GS/CS. Above these values, slag reactivity dominates, while below them, the micro-aggregate effect of CS becomes prominent. Microscopic analysis indicates that SWCM exhibits a gradual-to-accelerated pozzolanic reaction, with the main products being C-(A)-S-H gel and ettringite (AFt). The pore structure is regulated by the delayed crystallization of AFt. SWCM emits only 0.072 t CO₂/t, an 89.1% reduction compared to OPC, with carbon emissions per unit strength of 3.03 kg CO₂/MPa. This system is highly efficient and low-carbon, making it suitable for semi-rigid road base materials.

Keywords: industrial solid waste; solid waste based cementitious material; response surface methodology; optimal mix proportion; hydration mechanism; carbon emission reduction

0 Introduction

The rapid development of infrastructure and urbanization has led to the continuous accumulation of industrial solid wastes such as desulfurization gypsum, granulated slag, and copper slag. The disposal and utilization of these large-scale solid wastes pose significant environmental and economic challenges. Against the backdrop of sustainable development and carbon neutrality goals, the green recycling of industrial solid wastes as alternative cementitious materials is attracting increasing global attention. Solid waste-based cementitious materials (SWCM) offer significant advantages in terms of resource efficiency and energy consumption during production. Due to these benefits, they have garnered widespread attention in the road construction materials industry and in the transition to a low-carbon economy.

By utilizing the synergistic hydration effects among different solid wastes, cementitious materials with properties comparable to those of Portland cement can be produced [1]. In recent years, substantial progress has been made in this field. Zhang et al. [2] prepared a cementitious material using a quaternary system consisting of alkali slag, calcium carbide slag, granulated slag, and silica fume, which exhibited a 28-day compressive strength of 38.24 MPa. They identified C-S-H and C-(A)-S-H gels as the primary hydration products. Tian et al. [3] investigated the synergistic carbon sequestration performance and microstructure of steel slag-based cementitious materials and found that this system can achieve carbon sequestration while maintaining mechanical properties comparable to those of traditional cementitious materials. Zeng et al. [4] developed a low-carbon cementitious material based on gypsum slag and composed of multiple solid wastes, including an alkaline activator, fly ash, steel slag, and lithium slag. This cementitious material system exhibited a 28-day compressive strength of 49.8 MPa. Wang et al. [5] investigated the effect of coal

gangue on the properties of coal-based solid waste geopolymer grout materials and prepared a grout material with a 28-day compressive strength of 8.89 MPa. They found that its strength primarily originated from aluminate hydrates (Aft), C-S-H gel, and calcium aluminosilicate hydrate (C-(N)-A-S-H) gel. Shi et al. [6] prepared mine backfill materials using only solid waste raw materials such as red mud, calcium carbide slag, coal gangue, and ground granulated blast furnace slag. Their test specimens achieved a strength of 8.8 MPa at 28 days. Liao et al. [7] based on the principle of synergistic utilization of industrial solid waste, prepared an early-strength solid waste-based cementitious material (SSC) using white mud from the ammonia-soda process, granulated slag, steel slag, and desulfurization gypsum. This material achieved a compressive strength of 12.9 MPa at 1 day and 44.5 MPa at 28 days.

Current research on solid waste-based cementitious materials has primarily focused on their application in construction as substitutes for 42.5 or 32.5 Portland cement, or on the development of ultra-low-strength materials for uses such as grouting and mine backfilling. However, research on medium-strength grades (such as 22.5MPa or 27.5MPa), which are widely used in highway engineering, remain insufficient. Furthermore, regional variations in solid waste sources often result in poor reproducibility of established mix designs and preparation processes. Research specifically targeting local solid waste resources remains insufficient. Notably, current studies lack reports on ternary all-solid-waste systems based on granulated blast furnace slag, copper slag, and desulfurization gypsum, as well as those using a composite of calcium carbide slag and sodium silicate as an activator.

In this study, we selected the main local solid wastes—desulfurization gypsum (DG), granulated slag (GS), and copper slag (CS) as the primary raw materials. The alkali activator (AA) was prepared by mixing locally sourced calcium carbide slag with sodium silicate. Within the framework of the response surface method, the mixing ratios were optimized using a Box-Behnken design. The synergistic hydration mechanism of the DG-GS-CS ternary system was systematically investigated, with a focus on the formation of key hydration products and the evolution of microstructure. Furthermore, the carbon reduction potential of this system was quantitatively evaluated in comparison with ordinary Portland cement (OPC). This study aims to provide a theoretical foundation and practical guidance for the design and low-carbon application of SWCM specifically for semi-rigid road base courses.

1 Materials and Methods

1.1 Materials

As shown in Figure 1, this study selected three types of solid waste—granulated slag (GS), copper slag (CS), and desulfurization gypsum (DG) as the main raw materials. After processing and ball milling, the specific surface area of each type of solid waste exceeded 500 m²/kg.

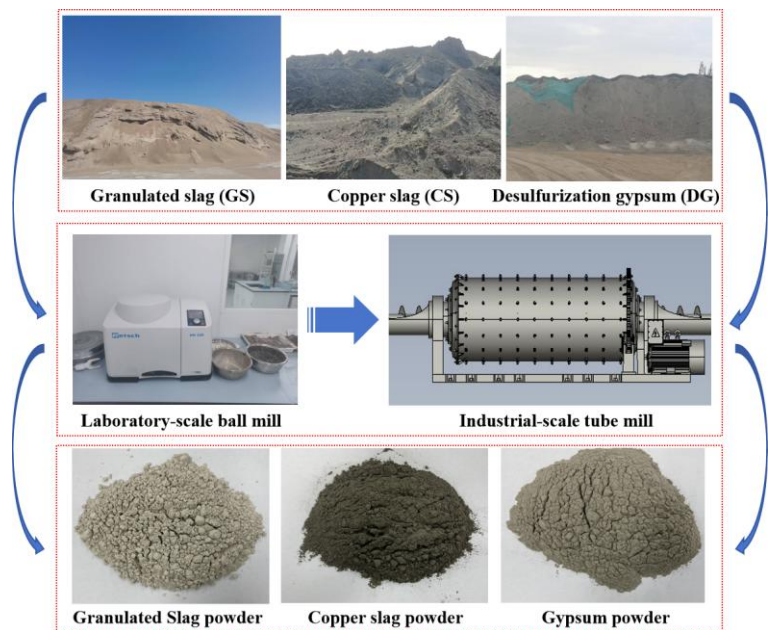


Figure 1. Processing and grinding procedures for solid waste raw materials

The chemical composition of three types of ground solid waste powders was analyzed using X-ray fluorescence spectroscopy (XRF). The phase composition was determined by X-ray diffraction (XRD), and microstructural characteristics were examined using a scanning electron microscope (SEM). The results are shown in Table 1, Figure 2 and Figure 3.

Table 1. XRF analysis results of solid waste materials

Solid Waste	SiO ₂	CaO	Al ₂ O ₃	MgO	SO ₃	K ₂ O	MnO	Fe ₂ O ₃	SrO
GS	37.080	32.051	13.911	9.496	2.862	0.871	0.631	0.302	0.065
CS	37.966	2.342	7.299	0.000	0.572	1.077	0.059	45.086	0.017
DG	2.202	47.090	1.465	0.983	47.236	0.150	0.014	0.424	0.048

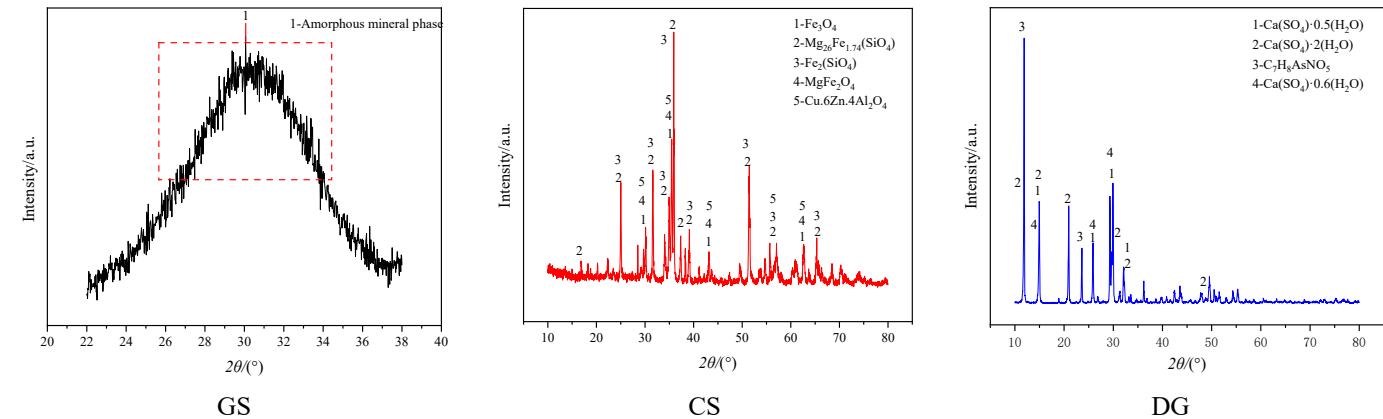


Figure 2. XRD analysis results of solid waste materials

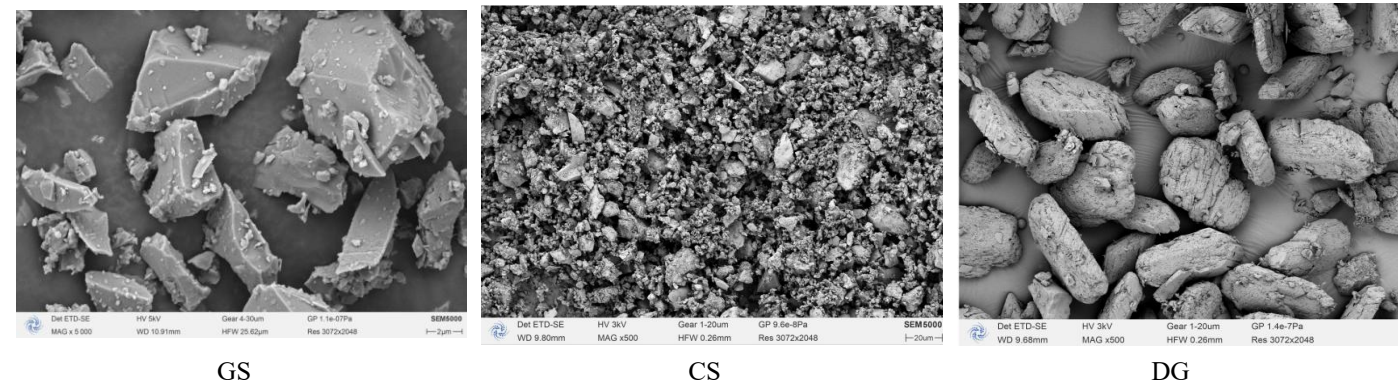


Figure 3. SEM micrographs of solid waste materials

GS, CS, and DG are all sourced from local metallurgical enterprises in Jiayuguan City, Gansu Province. Among these, the GS has a specific surface area of 550 m²/kg after grinding. The particles are irregularly angular in shape, with a dense, vitreous fracture. The main components are SiO₂, CaO, and Al₂O₃. The CS undergoes wet ball milling and magnetic separation, resulting in a specific surface area of 505 m²/kg. Its main components are FeO and SiO₂, with ferrosilicate (Fe₂SiO₄) and magnetite (Fe₃O₄) identified as the primary crystalline phases. The DG is a deep yellow powder with a free water content of less than 1%, composed primarily of CaO and SO₃. The main crystalline phases are CaSO₄·H₂O and CaCO₃, and the particles are lumpy with rounded edges. The alkaline activator (AA) was prepared by dry-mixing local calcium carbide slag and sodium silicate (modulus 1.2, SiO₂/Na₂O = 2.5) in a 6:4 ratio and grinding the mixture in a vibrating mill. It is primarily used to enhance the early reactivity of GS.

1.2 Response Surface Methodology

In this study, a three-factor, three-level experimental design was conducted using Design Expert 13.0 software. In this design, DG content was specified as factor X, the compound ratio of GS to CS was specified as factor Y, and activator content was specified as factor Z.

Table 2. Factors and levels of the Box-Behnken experimental design

Factor	Level		
	-1	0	1
DG(X) / %	10	20	30
GS / CS(Y)	3:1	2:1	1:1
AA(Z) / %	1	2	3

The 28-day linear expansion rate, 7-day compressive strength, and 28-day compressive strength were selected as response values and denoted as R1, R2, and R3, respectively. The 28-day linear expansion rate was chosen as the primary mix design parameter based on the anticipated application of this solid waste-based binder material in semi-rigid highway subbases. Its slight expansion characteristics help compensate for shrinkage, thereby reducing the risk of drying shrinkage cracking in the subbase[8]. At the same time, the 7-day and 28-day compressive strengths are key parameters for evaluating the material's mechanical properties and directly influence the feasibility of its practical engineering applications.

1.3 Testing and Characterization

(1) Experimental Testing: Cementitious mortar specimens measuring 40 mm × 40 mm × 160 mm were prepared. These specimens were subjected to standard curing (20 ± 2°C, relative humidity ≥ 90%), and their compressive strength was tested at 7 and 28 days. In addition, slurry specimens measuring 25 mm × 25 mm × 280 mm were prepared to test the linear expansion rate. Three days after standard curing, the mortar specimens were demolded and their initial lengths were recorded. They were then immersed in water for 28 days, and their final lengths were measured after 28 days. The linear expansion rate was calculated using the following formula (1):

$$\varepsilon = \frac{L_t - L_o}{L_o} \times 100\% \quad (1)$$

Where, L_t is the length of the specimen at age t (mm), and L_o is the initial length of the specimen (mm).

(2) Performance Characterization: The chemical composition of solid waste materials was analyzed using X-ray fluorescence (XRF, Shimadzu XRF-1800, Japan) with a Rh target (60 kV, 150 mA). The Rh target can detect elements ranging from Be to U. X-ray diffraction (XRD, Shimadzu XRD-7000, Japan) using Cu K α radiation (40 kV, 200 mA) was used to identify the mineral phase composition in the solid waste. Scanning was performed from 5° to 75° 2 θ at a rate of 2°/min, with the sample heated from 30°C to 1000°C at a heating rate of 10°C/min. The microstructural morphology of the solid waste raw materials and the solid waste cementitious materials was examined using a scanning electron microscope (SEM, JEOL JSM-IT800, Japan). Prior to imaging, the samples were sputter-gold-coated. The pore size distribution of the samples was tested using the mercury intrusion porosimetry (MIP) method (Micromeritics Autosorb iQ, USA). The samples were vacuum-degassed prior to testing. The intrusion pressure (0.1 Pa to 400 MPa) could detect pores with diameters ≥ 3 nm, which were calculated using the Washburn equation. An isothermal calorimeter (TA Instruments TAM Air, USA) was used to monitor the heat of hydration of the cementitious materials at 25.0 ± 0.1°C. The test utilized samples weighing 50 ± 1 g, with data recorded every minute.

2 Results and Analysis

2.1 Analysis of Experimental Results

Seventeen sets of solid waste cementitious material formulations were designed using the response surface method (RSM). The response test results for each formulation are shown in Table 3.

Table 3. Response Surface Design and Results of SWCM Proportions

X/%	Y	Z/%	R1/%	R2/MPa	R3/MPa
10	1	2	0.35	9.54	16.08
30	1	2	0.56	5.46	11.47

10	3	2	0.41	14.67	25.62
30	3	2	0.66	11.03	17.82
10	2	1	0.27	12.78	22.89
30	2	1	0.59	8.91	15.24
10	2	3	0.39	15.12	23.41
30	2	3	0.56	12.47	16.87
20	1	1	0.43	7.12	14.02
20	3	1	0.48	12.53	23.37
20	1	3	0.41	10.28	15.12
20	3	3	0.47	17.03	24.68
20	2	2	0.45	14.92	22.81
20	2	2	0.43	13.21	23.45
20	2	2	0.46	14.58	21.86
20	2	2	0.45	13.85	22.71
20	2	2	0.42	15.21	23.58

The experimental data were analyzed using Design Expert software. Based on the sum of squares, mean square error, and fitting error, different models were compared using nonlinear fitting, and a quadratic model was determined to be optimal. The specific regression models for the 28-day linear expansion rate, 7-day compressive strength, and 28-day compressive strength are given by Equations (2) through (4). Table 4 summarizes the corresponding analysis of variance (ANOVA).

$$R_1 = 0.442 + 0.1187X + 0.0337Y + 0.0075Z + 0.0100XY - 0.0375XZ + 0.0025YZ + 0.0290X^2 + 0.0240Y^2 - 0.0185Z^2 \tag{2}$$

$$R_2 = 14.35 - 1.78X + 2.86Y + 1.70Z + 0.11XY + 0.305XZ + 0.335YZ - 1.80X^2 - 2.38Y^2 - 0.2345Z^2 \tag{3}$$

$$R_3 = 22.88 - 3.32X + 4.35Y + 0.57Z - 0.7975XY + 0.2775XZ + 0.0525YZ - 2.41X^2 - 2.72Y^2 - 0.8647Z^2 \tag{4}$$

Table 4. Regression model data statistics of SWCM

Response	Source	Sum of Squares	df	Mean Square	F-value	p-value
R1	Model	0.1357	9	0.0151	31.94	<0.0001**
	Residual	0.0033	7	0.0005	-	-
	Lack of Fit	0.0022	3	0.0007	2.75	0.1769
R2	Model	154.93	9	17.21	34.42	< 0.0001**
	Residual	3.5	7	0.5001	-	-
	Lack of Fit	0.8336	3	0.2779	0.4168	0.751
R3	Model	309.66	9	34.41	70.14	< 0.0001**
	Residual	3.43	7	0.4905	-	-
	Lack of Fit	1.54	3	0.5148	1.09	0.4493

Note: ** denotes extreme significance.

The goodness-of-fit test (R2 test) was used to assess the significance of the model. To evaluate the significance of each factor in the model, the p-values were examined: a p-value < 0.05 indicates that the factor is significant, while a p-value < 0.01 indicates that the factor is highly significant [9–10]. As shown in Table 4, the p-values for all models were < 0.001, indicating that the regression was highly significant. At the same time, the lack of fit terms showed p-values > 0.05, indicating that they were not significant, which

further supports the good fit of the regression model. Therefore, the regression equations can be used in place of the actual data points to analyze the experimental results.

The R2 values for the 28-day linear expansion rate, 7-day compressive strength, and 28-day compressive strength models are 0.9762, 0.9779, and 0.9890, which confirms the reliability of the experimental methodology. Scatter plots (x, y) were generated to compare the predicted and measured values, as shown in Figure 4. The close agreement between the predicted and measured values verifies the good fitting performance of the models, demonstrating their utility for analyzing and predicting the properties of SWCM.

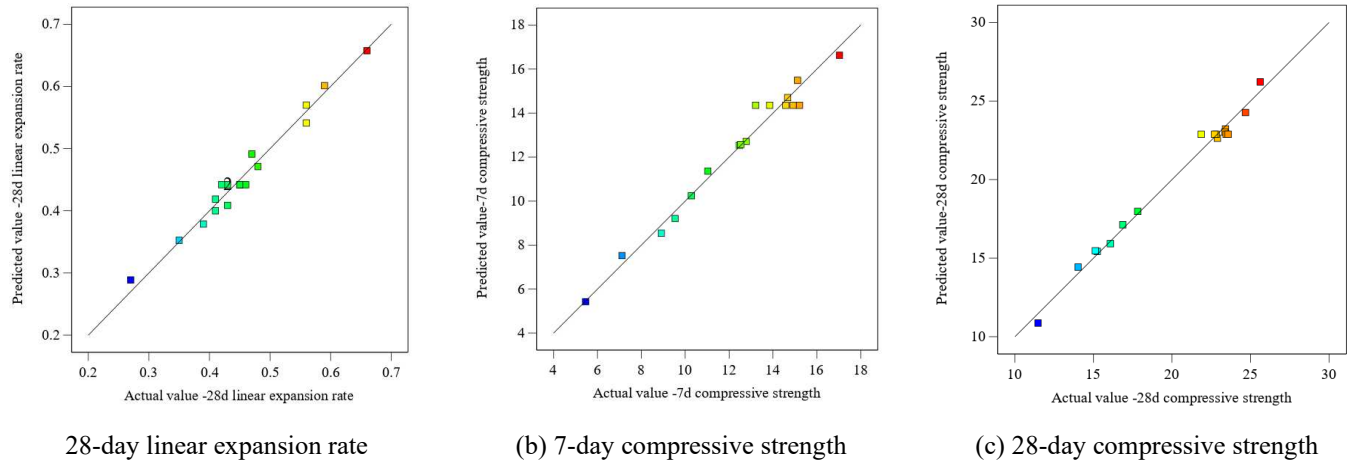


Figure 4. Comparison of Model-Predicted Values and Actual Measured Values: (a) 28-day Linear Expansion Rate, (b) 7-day Compressive Strength, (c) 28-day Compressive Strength

2.2 Influence of Factors and Their Interactions on Response Values

2.2.1 Linear Expansion Rate

A response surface plot of the SWCM linear expansion rate was generated based on a regression model to analyze the effects of individual factors and their interactions; the results are shown in Figure 5. The order of influence of these factors and their interactions on the linear expansion rate is as follows: $X > Y > XZ > X2 > Y2 > Z2$. Among these, factors X and Y, as well as the interaction term XZ, exhibited highly significant effects, while the quadratic terms X2, Y2, and Z2 exhibited significant effects.

Table 5. Variance analysis results of model regression

Re- sponse	Value	X	Y	Z	XY	XZ	YZ	X2	Y2	Z2
R1	F-value	238.94	19.3	0.9531	0.8472	11.91	0.053	7.5	5.14	3.05
	P-value	< 0.0001	0.0032	0.3615	0.388	0.0107	0.8246	0.029	0.0578	0.1241
R2	F-value	50.69	130.62	45.96	0.0968	0.7441	0.8977	27.26	47.67	0.463
	P-value	0.0002	< 0.0001	0.0003	0.7648	0.4169	0.375	0.0012	0.0002	0.5181
R3	F-value	180.31	308.61	5.3	5.19	0.628	0.0225	50.05	63.49	6.42
	P-value	< 0.0001	< 0.0001	0.0548	0.0569	0.4541	0.8851	0.0002	< 0.0001	0.039

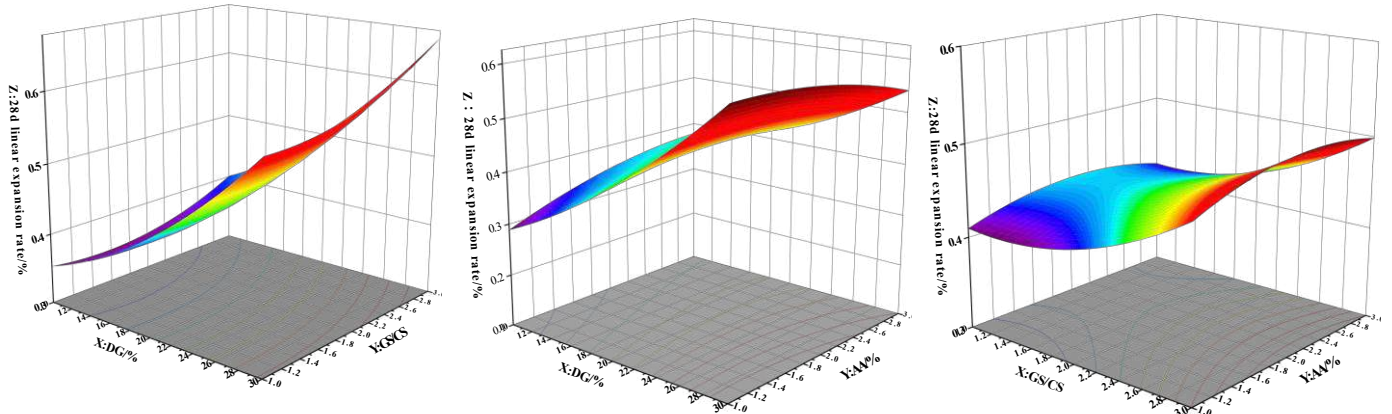


Figure 5. Response surface curves of the linear expansion rate to various factors

As shown in Figure 5, the linear expansion rate increases linearly with increasing DG content. This trend is primarily attributed to the release of SO_4^{2-} from DG, which reacts with Al_2O_3 in GS to form Aft—a crystallization process accompanied by approximately 120% volumetric expansion [11]. The GS/CS ratio exhibits a threshold effect: when the ratio exceeds 1.8, a higher GS content promotes Al^{3+} leaching. At this point, the inhibitory effect of $\text{Fe}^{2+}/\text{Fe}^{3+}$ in CS on Al^{3+} leaching reaches saturation, and the expansion rate rises significantly. When this ratio is below 1.8, Fe oxidation states preferentially occupy the Al^{3+} reaction sites, and the micro-aggregation effect of copper slag refines the pores and reduces stress concentration, resulting in no significant change in the expansion rate [12]. The effect of alkaline activators (AA) depends on the DG content. When DG is below 20%, AA primarily controls Aft formation by adjusting the pH. An AA content below 2% enhances GS dissolution and Aft nucleation by increasing the OH^- concentration [13]. However, when AA exceeds 2%, excess OH^- competes with SO_4^{2-} , inhibiting Aft formation and promoting the transformation to AFm, causing the expansion rate to first increase and then decrease. When the DG content exceeds 20%, SO_4^{2-} saturation causes Aft formation to become diffusion-controlled. The regulatory effect of the activator AA weakens, and the expansion rate tends to stabilize [14].

2.2.2 7-day Compressive Strength

A response surface plot of the 7-day compressive strength of SWCM was generated based on the regression model, as shown in Figure 6. The order of influence of the factors and their interactions on the 7-day compressive strength is: $Y > X > Z > Y^2 > X^2$. Among these, factors Y, X, and Z exhibited highly significant effects, while the remaining factors were not statistically significant.

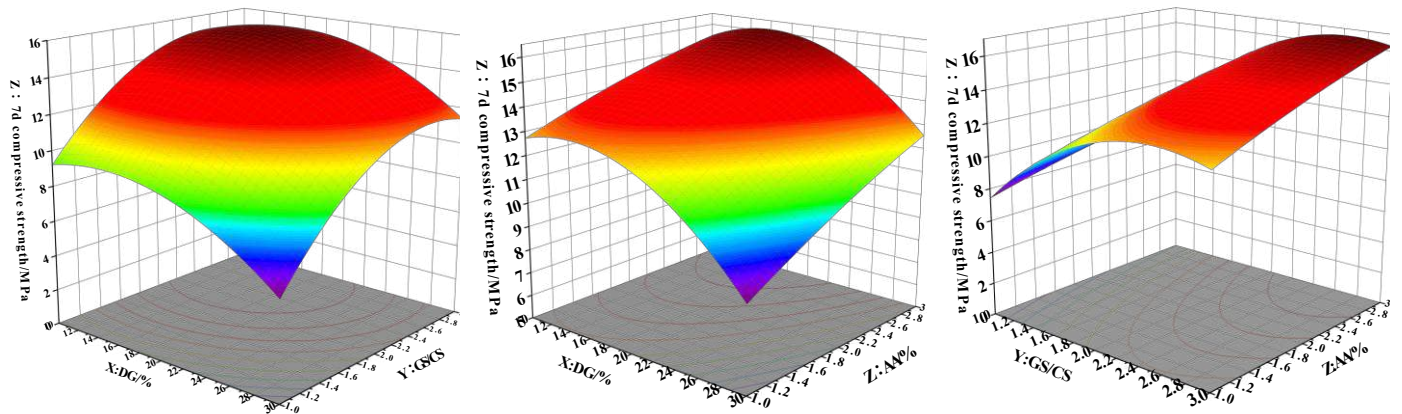


Figure 6. Response surface curves of the 7-day compressive strength to various factors

Figure 6 shows that there is an optimal threshold of 18% for the DG content. At levels below 18%, the SO_4^{2-} derived from DG promotes the interweaving of Aft and C-(A)-S-H, enhancing the strength of the matrix and the dissolution of GS. At levels above 18%, excess Aft leads to microcracks and weak gypsum interfaces, thereby reducing strength [15]. The GS/CS ratio exhibits a critical value of 2.1. A ratio > 2.1 enhances GS activity, promoting $\text{CaO} - \text{SiO}_2 - \text{Al}_2\text{O}_3$ dissociation and the co-formation of C-(A)-S-H and Aft. A ratio < 2.1 improves compaction through CS microaggregation but reduces strength due to insufficient Al^{3+} supply

and dilution of the GS component. Strength increases linearly with AA content, but the rate of increase depends on the DG content. When DG exceeds 18%, sufficient SO_4^{2-} allows AA to accelerate Al^{3+} dissolution, synergistically enhancing the Aft and C-(A)-S-H dual network. When the DG is below 18%, the strength gain is limited by SO_4^{2-} , and the strength improvement depends solely on C-(A)-S-H densification [16]. The independent effect of the activator AA is constrained by the consumption of OH^- by Fe^{2+} in the CS, and its contribution is far lower than that resulting from its interaction with gypsum.

2.2.3 28-day Compressive Strength

A response surface plot of the 28-day compressive strength of SWCM was generated based on the regression model, as shown in Figure 7. The order of influence of the various factors and their interactions on the 28-day compressive strength is: $Y > X > Y^2 > X^2 > Z^2 > Z > XY$. Among these, Y, X, Y^2 , and X^2 have a very significant effect on the 28-day strength, Z^2 , Z, and XY have a significant effect, and the other factors have no significant effect.

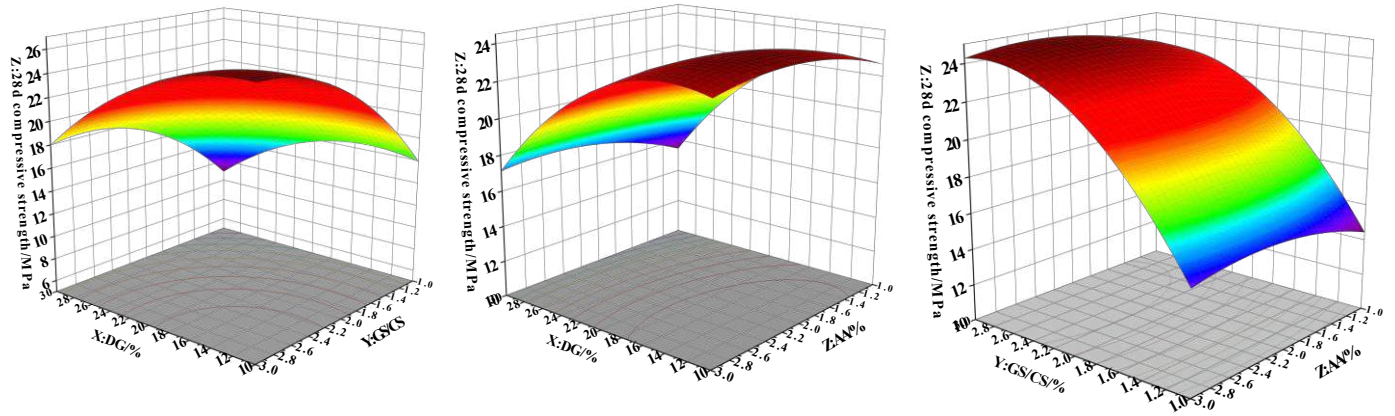


Figure 7. Response surface curves of the 28-day compressive strength to various factors

Regression analysis of Figure 7 indicates that the 28-day compressive strength is predominantly governed by the GS/CS ratio and DG content. The strength increases with the GS/CS ratio. Ratios greater than 2:1 promote the formation of a reinforced C-(A)-S-H and Aft skeleton via highly active GS, while ratios less than 2:1 yield insufficient gel development due to low CS activity. DG exhibits an optimal range of approximately 18%, where SO_4^{2-} enhances Aft and C-(A)-S-H interweaving. Excess SO_4^{2-} induces microcracks and weak interfaces. AA content weakly influences the later strength but significantly contributes under high DG conditions by adjusting the system alkalinity. Crucially, the significant $\text{DG} \times \text{GS/CS}$ interaction term highlights their synergistic optimization for long-term strength enhancement [17, 18].

2.3 Response Surface Result Optimization Verification

Through analysis of variance (ANOVA), response surface model validation, and the joint optimization of multiple factors and response values in SWCM, the results met the numerical requirements for all response values, thereby yielding the optimal formulation for the comprehensive performance of SWCM. The optimal mix design obtained through optimization is as follows: DG content of 18.4%, GS/CS ratio of 2.1, and AA of 1.6%, with GS content at 54.2% and CS content at 25.8%, as shown in Figure 8. Using the predicted values from the model as a baseline, this mix design was validated through multiple experimental tests. As clearly shown in Table 6, the measured compressive strengths at 7 and 28 days both exceeded the predicted values from the model, and the linear expansion rate performed well with an error margin of less than 5%. The measured values for the three response variables were essentially consistent with the predicted values, demonstrating that the response surface method is generally accurate in predicting SWCM mix ratios and that the model exhibits high precision.

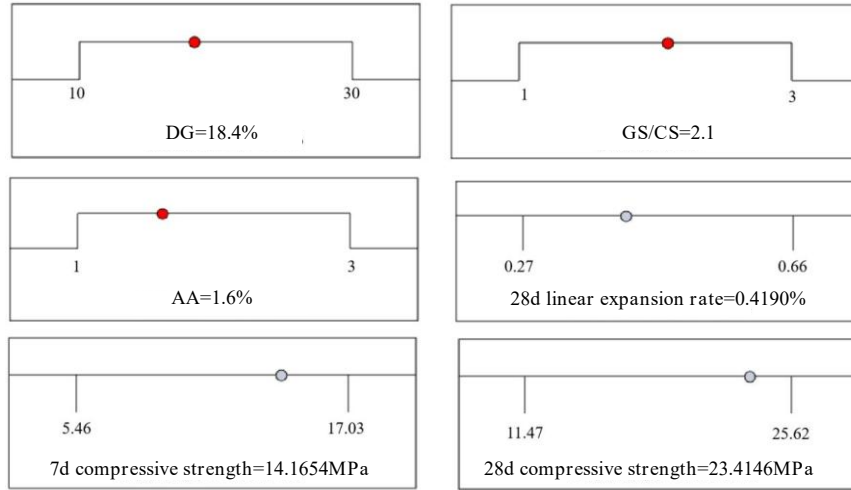


Figure 8. Optimization of the mix design for SWCM using the response surface method

Table 6. Comparison of optimized design and actual measurement results

Response	R1	R2	R3
Predictive value	0.419%	14.165 MPa	23.415 MPa
Actual value	0.402%	15.272 MPa	23.891 MPa
Actual error	-0.017%	1.107 MPa	0.396 MPa
Error ratio	-4.15%	7.81%	1.69%

3 Analysis of the Early Hydration Mechanism of SWCM

Using the optimal mix ratio for the solid waste cementitious material obtained through response surface optimization, slurry specimens were prepared and cured for 5 h, 8 h, 1 day, 2 days, 3 days, and 7 days. The early-age XRD hydration products, TG thermogravimetric analysis, SEM microstructural changes, pore changes, and hydration heat development patterns were analyzed. A comparison with Grade 42.5 ordinary Portland cement (OPC) was conducted to further elucidate the hydration reaction mechanisms of solid waste cementitious materials during the early-age period.

3.1 Analysis of Early Mineral Phases of SWCM

Based on quantitative phase analysis using XRD, this study revealed the fundamental differences and regulatory mechanisms of early-stage hydration products in OPC and SWCM, as shown in Figure 9 and Figure 10.

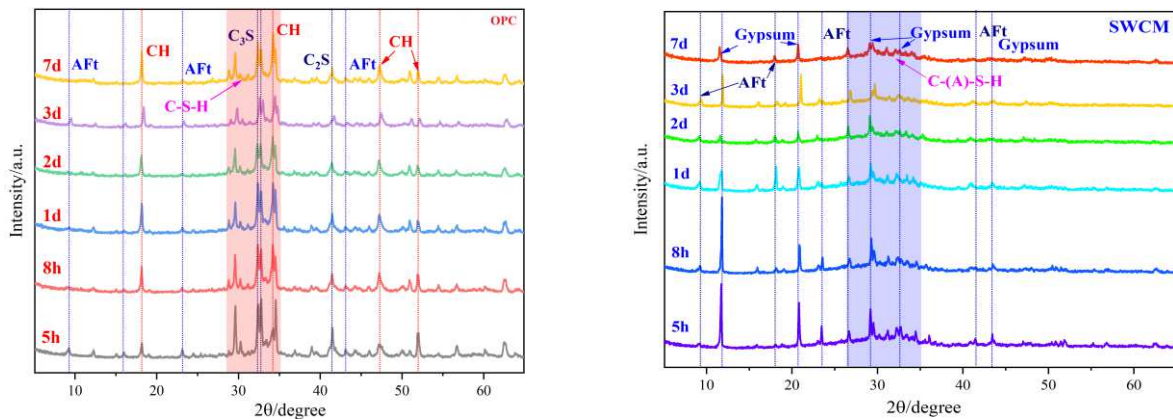


Figure 9. Early X-ray diffraction analysis of OPC and SWCM

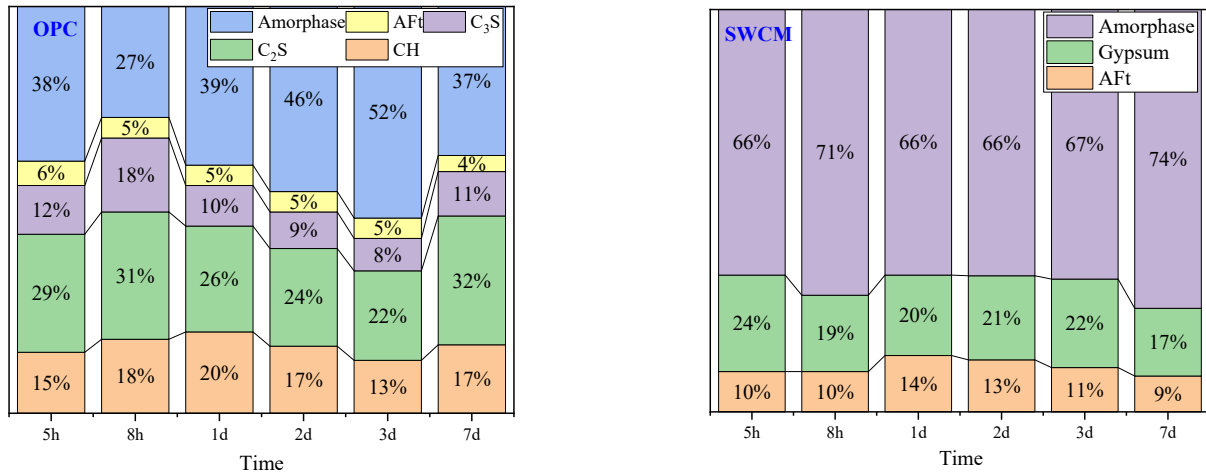


Figure 10. The quantitative analysis of XRD hydration products for OPC and SWCM

XRD test results indicate that there are significant differences in the early hydration mineral composition and phase evolution patterns between OPC and SWCM. In the early stages of OPC hydration, strong characteristic diffraction peaks of clinker C_3S and C_2S can be observed. As the hydration reaction progresses, the silicate clinker minerals continue to dissolve and be consumed, leading to the gradual formation of CH and aluminate. Quantitative analysis shows that the proportion of the amorphous phase in the OPC system increased from 38% at 5 hours of hydration to 52% at 3 days, clearly reflecting the continuous accumulation of C–S–H gel with age. By 7 days, the proportion of the amorphous phase had decreased slightly, while the content of CH crystals remained high. The overall hydration behavior is consistent with the hydration reaction pathway of traditional silicate cement [19].

For the SWCM system, stable gypsum diffraction peaks were observed throughout the entire hydration period from 5 h to 7 d. During this stage, the gypsum was not completely consumed and continued to participate in the system's hydration process. The amorphous phase content of SWCM remained consistently at a high level of 66%–74%, far exceeding that of OPC at the same age, corresponding to the formation of a large amount of aluminum-substituted C-(A)-S-H gel within the system. AFt precipitates rapidly during the early stages of hydration, but its content fluctuates minimally as the age increases, with no evidence of sustained proliferation. No distinct CH characteristic diffraction peaks were detected throughout the entire hydration phase. This indicates that the gypsum component consumes calcium ions within the system, effectively inhibiting the precipitation of calcium hydroxide crystals, altering the formation pathway of hydration products, and promoting the incorporation of aluminum ions into the gel structure to form C-(A)-S-H. The aforementioned characteristics of mineral phase evolution indicate that the gypsum-mediated hydration reaction alters the product system of traditional cement, reducing the formation of CH crystals and inducing the extensive formation of aluminum-rich hydration gels. This is also the core mineralogical mechanism underlying the early setting performance of SWCM [20].

3.2 Analysis of Early Thermogravimetric of SWCM

Based on the thermogravimetric (TG) method, the composition and evolution of the hydration products in SWCM were quantitatively characterized, as shown in Figure 11.

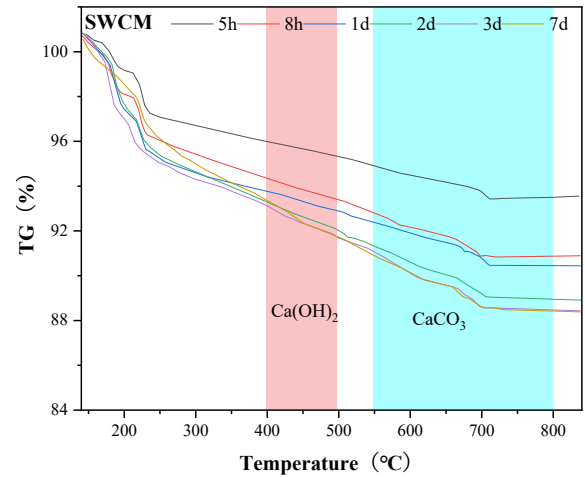
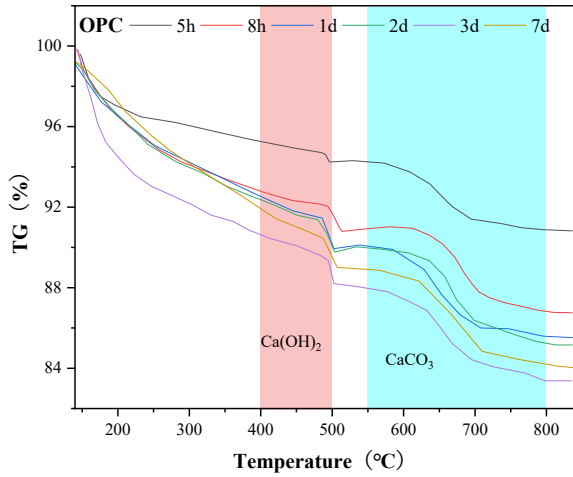


Figure 11. Results of early thermogravimetric analysis tests for OPC and SWCM

As shown in Figure 11, the 400–500 °C range on the OPC thermogravimetric curve corresponds to the characteristic decomposition range of calcium hydroxide. As the hydration age increased from 5 h to 7 d, the mass loss in this temperature range continued to increase, and the amount of CH generated rose steadily as hydration progressed. In the calcium carbonate decomposition range of 600–800 °C, mass loss also increases with age, indicating continuous carbonation of the hydration products and a gradual accumulation of calcium carbonate. Mass loss in the low-temperature range stems from the removal of bound water from C–S–H and ettringite. The low-temperature mass loss of OPC gradually increases with age, corresponding to the continuous formation of hydration products.

SWCM exhibits no distinct CH decomposition weight loss step in the 400–500 °C range. The curves show gradual changes across all ages, and no trend of increasing CH weight loss is observed with aging. This demonstrates that CH levels within the system are consistently maintained at extremely low levels. The gypsum component alters the form in which calcium exists, thereby inhibiting the precipitation of calcium hydroxide crystals. The loss of bound water in the low-temperature range of SWCM is generally higher than that of OPC, and the loss continues to increase with age, reflecting the continuous formation of hydrated products such as C-(A)-S-H gel and Aft. The carbonate loss observed in SWCM at 600–800 °C is attributable to two sources: first, surface carbonation resulting from the reaction of hydration products with CO₂ in the air during sample preparation and storage; second, the reaction of free Ca²⁺ within the system with dissolved CO₃²⁻ to form calcium carbonate. Since the carbonate content does not increase significantly with hydration age, this indicates that carbonation induced by subsequent exposure to air is the primary mechanism, rather than the result of primary hydration products. [21-23].

3.3 Analysis of Early Pore Characteristics of SWCM

Based on multiscale pore structure characterization using the mercury intrusion porosity method, this study systematically reveals the key differences and underlying mechanisms in the early-age pore structure evolution of OPC and SWCM. As shown in Figure 12 and Figure 13.

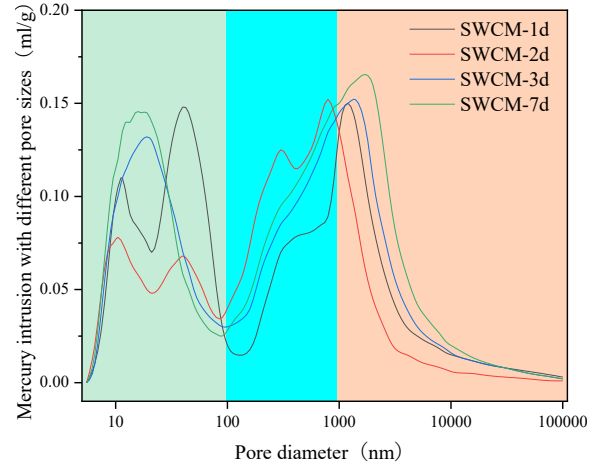
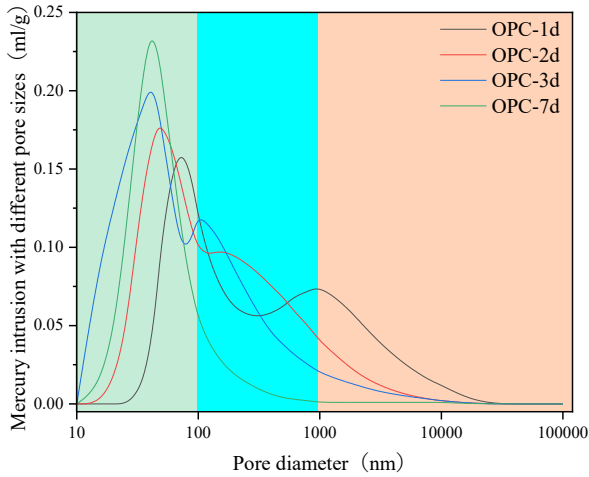


Figure 12. Results of early mercury compression tests for OPC and SWCM

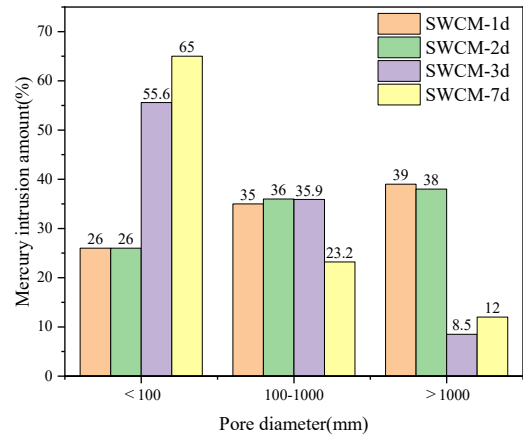
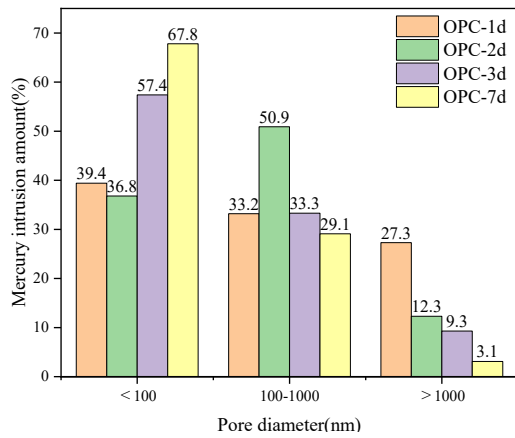


Figure 13. Statistical results of early pore distribution for OPC and SWCM

The pore structure evolution of OPC and SWCM shows essential differences, especially with the 100-1000 nm capillary pore domain as the key differentiation point:

(1) Microporosity (<100 nm): OPC accounted for 39.4% on day 1, decreased slightly to 36.8% on day 2, and rose to 67.8% on day 3 as hydration progressed. SWCM accounted for only 26.0% on day 1, remained at 26.0% on day 2, but jumped to 55.6% on day 3 and reached 65.0% on day 7. This trend of delayed surge indicates that the gel network in SWCM develops more slowly in the early stages, but the microporosity filling efficiency improves significantly once the volcanic ash reaction accelerates.

(2) Capillaries (100–1000 nm): In OPC, the proportion increased from 33.2% on day 1 to 50.9% on day 2, corresponding to rapid CH crystal precipitation leading to localized bridging and coarsening of pore sizes. In contrast, the proportion of capillaries in SWCM showed little change from day 1 to day 2. However, its differential curve exhibited a broad peak at approximately 800 nm on day 2, indicating that the delayed crystallization of Aft induced localized expansive stresses, causing the early C-S-H network to rupture. After day 3, the capillary fraction dropped to 35.9% and further decreased to 23.2% by day 7, as the structure re-densified.

(3) Macroporosity (>1000 nm): OPC showed a continuous decline from 27.3% to 3.1% at 7 days. SWCM reached as high as 39.0% at 1 day, remained at 38.0% at 2 days, dropped sharply to 8.5% at 3 days, but rebounded to 12.0% at 7 days. This rebound phenomenon is primarily attributed to the propagation of microcracks induced by localized expansive stresses from delayed Aft crystallization. During the accelerated hydration phase (3-7 days), concentrated growth of Aft crystals generated internal tensile stresses, causing partial disintegration of the gel matrix and reopening of initially filled macropores.

In summary, the OPC pore structure exhibits a monotonically refining trend, controlled by the stagnation of silicate hydration dominated by CH. The SWCM undergoes a dynamic evolution characterized by early-stage macroporosity development, a surge in capillary pores, and late-stage densification. This aligns with the gradual-to-accelerated hydration pattern revealed by XRD and TG

analyses, indicating that the crystalline expansion of Aft is a key factor regulating the stability of the SWCM pore structure [24, 25].

3.4 Analysis of Early Hydration Kinetics of SWCM

Isothermal calorimetry was used to reveal fundamental differences in the early hydration kinetics of OPC and SWCM. As shown in Figure 14.

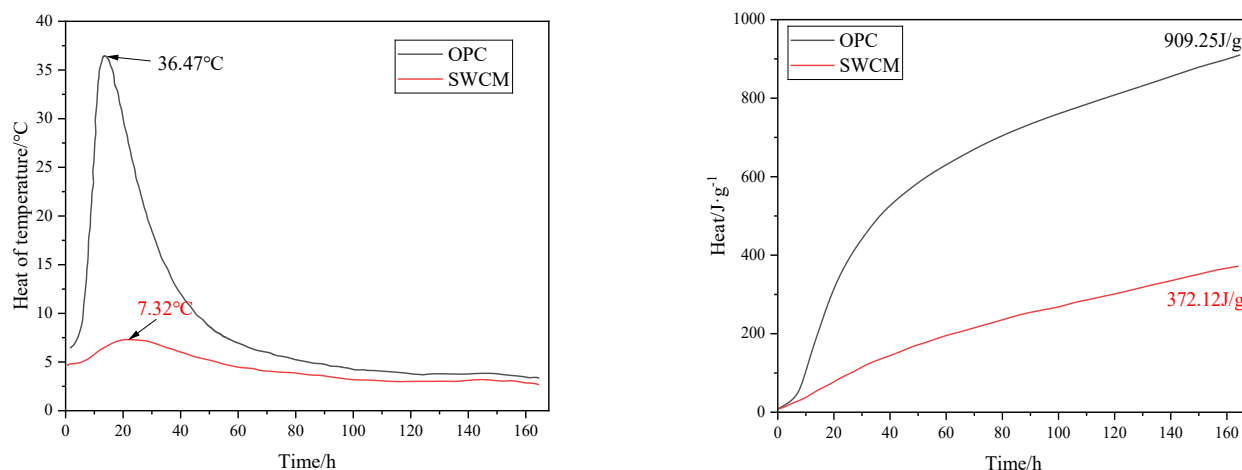


Figure 14. Test results of hydration heat of OPC and SWCM at early age

Based on hydration heat test data, the hydration heat release characteristics of OPC and SWCM were compared and analyzed. OPC undergoes rapid hydration in its early stages, with concentrated heat release. Within approximately 13 hours, the heat release rapidly increases to about 182 J/g, and after 30 hours, the cumulative heat release exceeds 400 J/g, with a total heat release of 910 J/g, indicating high early hydration activity and rapid temperature rise. The early heat release of SWCM is relatively slow, with only about 50% of the heat release of OPC at the same time, and a total heat release of about 372 J/g, significantly lower than OPC. The SWCM exothermic curve is smoother and the peak is delayed, indicating that its hydration reaction is slower and the exothermic process is more persistent. This is mainly due to the low activity of solid waste components, which inhibits the hydration process [26, 27].

3.5 Analysis of Early SEM Microstructural of SWCM

SEM images of OPC and SWCM taken at 5 hours, 1 day, and 7 days revealed fundamentally different hydration microstructures, as shown in Figure 15 and Figure 16.

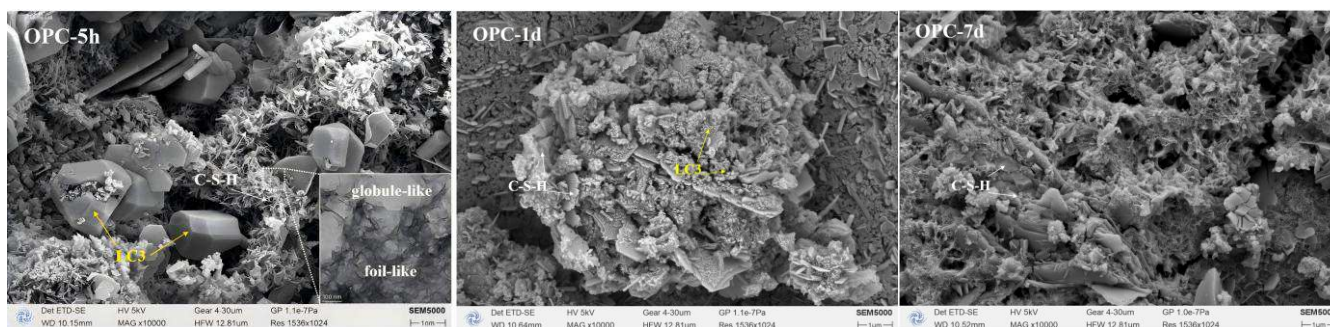


Figure 15. Microstructural evolution of hydration products of OPC with curing age

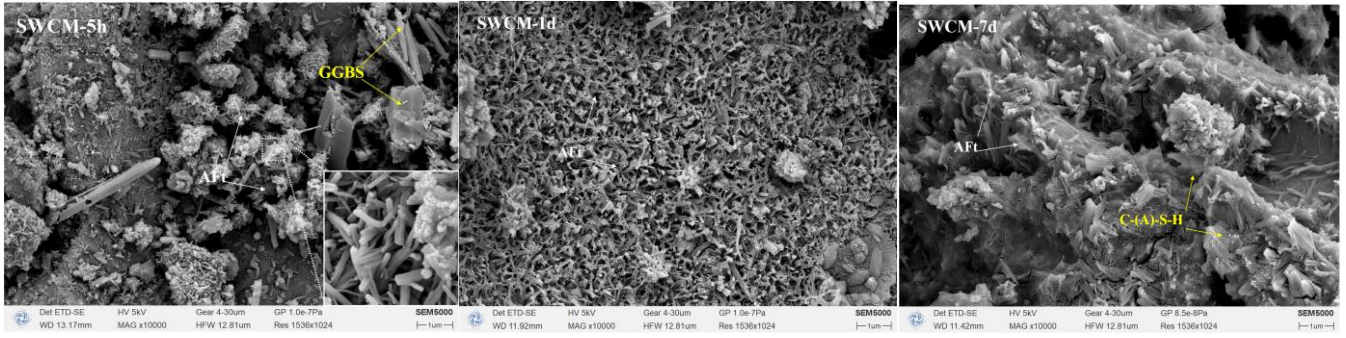


Figure 16. Microstructural evolution of hydration products of SWCM with curing age

For OPC (Figure 15), at 5 h, the matrix exhibited a morphology characterized by an interlacing of spherical particles and foil-like C–S–H gels, indicating rapid initial hydration of tricalcium silicate (C_3S) and dicalcium silicate (C_2S). This is consistent with the high exothermic heat released in the early stages and the formation of CH detected by XRD. At 1 day, the structure tended to be dense but remained porous, with a large number of CH platelets and fibrous C–S–H networks filling the pores. By 7 days, a well-developed C–S–H gel matrix had formed, with a dense and uniform structure, although some capillaries remained. This reflects the rapid precipitation of early OPC hydration products, followed by a transition to a diffusion-controlled, slow phase due to the barrier effect of the dense CH and C–S–H layers [28].

For SWCM (Figure 16), the evolution exhibited a gradual–accelerating pattern. At 5 h, the matrix was loosely packed, with a large number of unreacted GS particles and CS particles clearly visible. Only a small amount of hydration products was attached to the particle surfaces, confirming a delayed pozzolanic reaction due to the slow dissolution of the glass phase. At 1 day, a large number of fibrous AFt crystals appeared, and bridging particles formed a relatively dense network. At 7 days, the microstructure became more heterogeneous, with the formation of a continuous C–(A)–S–H gel matrix interwoven with AFt needle-like crystals. However, microcracks and enlarged pores were also visible, attributed to the delayed crystallization of AFt and the damage caused by its expansion stress to the early gel network. This dynamic competition between gel densification and AFt expansion stress determines the long-term performance of SWCM, representing a fundamental difference from CH-dominated OPC systems [29].

4 Analysis of Carbon Emissions

4.1 Carbon Emission Factors

To quantify the low-carbon benefits of SWCM, based on the life cycle assessment (LCA) method, the carbon emissions of the optimized proportion SWCM production process were systematically calculated and compared with OPC. Based on practical production considerations, the SWCM production process in this study mainly involves drying, grinding, and mixing solid waste materials. Therefore, the carbon emissions during the production process need to consider the energy consumption of drying, grinding, and proportional mixing of each solid waste material. OPC production mainly considers the energy consumption of limestone calcination and cement clinker grinding. The unit energy consumption and carbon emission factors for solid waste pretreatment are shown in Table 7.

Table 7. Unit energy consumption and carbon emission factors for solid waste pretreatment [30, 31]

Solid waste type	Moisture content	Grindability (specific surface area>500m ² /kg)	Drying energy consumption (kWh/t)	Grinding energy consumption (kWh/t)	Mixed energy consumption (kWh/t)	Carbon emission factor (kg CO ₂ /t)
DG	15%	easy to grind	25	35	5	63.35
GS	8%	difficult to grind	12	55	5	70.17

CS	12%	extremely difficult to grind	20	65	5	87.71
OPC clinker calcination	-	-	-	-	-	830
OPC clinker grinding	-	-	-	40	-	38.98

4.2 Carbon Emission Accounting Model

1) SWCM system

Carbon emissions are composed of solid waste pretreatment and mixed production:

$$CE_{SWCM} = \sum_{i=1}^n [M_i \times (E_{d,i} + E_{g,i} + E_{mix}) \times EF_e] \quad (5)$$

Where, CE_{SWCM} is the carbon emissions of SWCM products (kg CO₂/t); M_i is the mass ratio of the i-th solid waste in SWCM; $E_{d,i}$ is the drying energy consumption of the i-th type of solid waste (kWh/t); $E_{g,i}$ is the grinding energy consumption of the i-th type of solid waste (kWh/t); E_{mix} represents mixed energy consumption (kWh/t); EF_e is the carbon emission factor for electricity, taken as 0.9746 kg CO₂/kWh [32].

2) OPC system

Carbon emissions primarily stem from clinker calcination and grinding:

$$CE_{OPC} = EF_c \times C_c + C_c \times E_{g,c} \times EF_e + E_{other} \quad (6)$$

Where, CE_{OPC} is the carbon emissions of OPC products (kg CO₂/t); C_c is the proportion of clinker in OPC, taken as 75%; EF_c is the emission factor for clinker calcination; $E_{g,c}$ is the energy consumption of clinker grinding; E_{other} is the emission from other processes, taking 10 kg CO₂/t [33].

4.3 Carbon Emission Comparison Results

Table 8. Comparison of SWCM and OPC carbon emission intensity [33]

Material	Unit emissions (tCO ₂ /t)	28-day compressive strength (MPa)	Unit strength emissions (kg CO ₂ /MPa)	Reduction rate compared to OPC
OPC	0.6617	42.8	15.46	—
SWCM	0.0723	23.9	3.03	89.1%

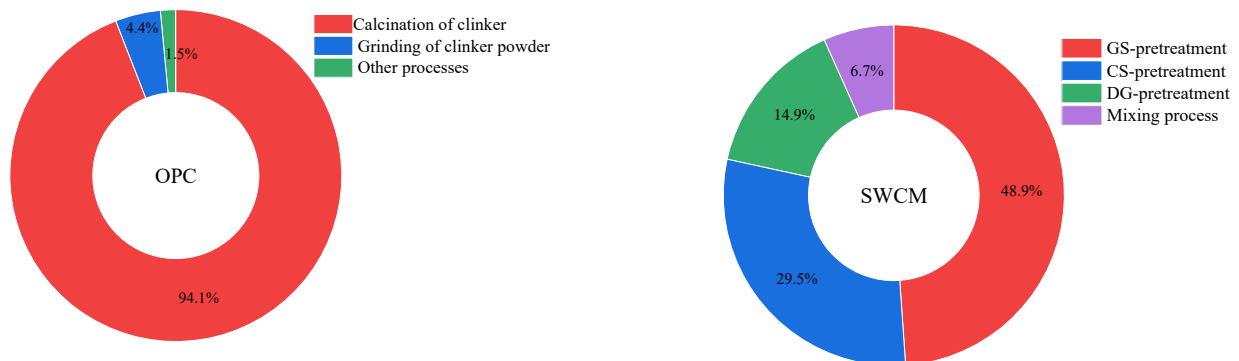


Figure 17. Comparison of Carbon Emission Composition in SWCM and OPC Production Processes

Based on the carbon emission accounting of the entire production process, this study shows that SWCM exhibits excellent low-carbon performance compared to OPC, as shown in Figure 17. The carbon emissions per unit product of SWCM are only 0.072t CO₂/t, a significant decrease of 89.1% compared to OPC (0.6617 t CO₂/t). Its carbon emissions mainly come from the energy consumption of solid waste pretreatment, among which the grinding process of GS and CS accounts for the highest proportion, and the mixed energy consumption accounts for 6.7%. Although the 28-day compressive strength of SWCM is lower than that of OPC, its unit strength carbon emissions are only 3.03 kg CO₂/MPa, far lower than OPC, demonstrating extremely high carbon efficiency. This is mainly due to SWCM completely avoiding the dominant high-temperature calcination process of clinker in OPC.

5 Conclusions

This study employed the RSM-BBD response surface methodology to design a fully solid waste-based cementitious material (SWCM) composed of GS, CS, DG, and AA (calcium carbide residue : sodium silicate = 6 : 4). Key indicators such as strength and linear expansion rate were analyzed, the early-age hydration reaction mechanism was revealed from a microscopic perspective, and carbon emission reduction potential was assessed. The following conclusions were drawn:

Using the response surface method, the optimal mix ratio for the SWCM was determined as follows: DG 18.4%, GS-to-CS ratio 2.1, and AA 1.6%. At this mix ratio, the 28-day linear expansion rate was 0.40%, and the 28-day compressive strength reached 23.89 MPa, meeting the requirements for volumetric stability and mechanical properties of semi-rigid road base courses.

(2) DG content and the GS/CS ratio are key factors in regulating the performance of SWCM. The linear expansion rate increases significantly with increasing DG content, primarily due to the extensive formation of AFt. The compressive strength reaches an optimal threshold at approximately 18% DG. There is a critical value of 2.1 for the GS/CS ratio: above this value, GS reactivity dominates strength development, while below this value, the filling and chemical inhibition effects of CS microfillers become more pronounced.

(3) OPC exhibits characteristics of a rapid-stagnation-type silicate reaction, with intense early-stage hydration but structural defects caused by the uneven deposition of CH crystals. In contrast, SWCM exhibits a progressive-accelerating pozzolanic reaction pattern. In the early stages, it gains a microporous advantage through the formation of nano-scale C-(A)-S-H gels. In the later stages, it is regulated by the delayed crystallization of AFt and the expansive stresses associated with its transformation into AFm, triggering a dynamic evolution characterized by a rapid increase followed by a decline in capillary pores. This process is the core mechanism determining the long-term performance and structural stability of SWCM.

(4) Carbon emissions calculations show that the carbon emissions per unit of SWCM production are 0.072t CO₂/t, which is 89.1% lower than those of OPC. These emissions primarily stem from the energy consumption associated with the grinding of GS and CS; carbon emissions per unit of strength are only 3.03 kg CO₂/MPa, far lower than the 15.46 kg CO₂/MPa for OPC, demonstrating excellent carbon efficiency and potential for low-carbon applications.

CRedit authorship contribution statement

Xu Wu: Writing-original draft, Visualization, Methodology, Investigation. Yang Yang: Writing-review & editing, Conceptualization. Jingzhuo Zhao: Writing-review & editing, Investigation. Taiping Huang: Writing-review & editing, Investigation. Xingzhen Zang: Investigation. Kaige Zhou: Investigation. Bo Li: Writing-review & editing, Conceptualization.

Data availability statement

All data that support the findings of this study are included in this manuscript and its Supplementary information files.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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