

Article

Mechanical Properties of Carbonation-Enhanced Alkali-Activated Slag-Solidified Shield Muck: Temperature–Humidity Coupling Effects

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Abstract: Efficient resource utilization of shield muck is critical for urban metro construction. This study investigates temperature–humidity coupling effects on the mechanical properties of carbonated alkali-activated slag-solidified shield muck through multi-scale analyses of compressive strength, pore structure, alkalinity, and microproperties. The results show that carbonation product filling in >1000 nm pores governs 28-day strength enhancement. Humidity regulates reactions via dual pathways. High humidity inhibits CO₂ diffusion, causing under-carbonation, and low humidity will lead to excessive carbonization under expansion stress, both increasing harmful porosity. Concurrently, low humidity depletes alkalinity, inhibiting C-A-S-H gel formation, whereas high humidity dilutes pore liquid alkalinity. Temperature modulates alkalinity through CO₂ diffusion, Ca²⁺ leaching, and evaporation—low temperatures preserve alkalinity, while high temperatures intensify surface carbonation and internal alkalinity loss. Synergistic temperature–humidity interactions drive calcite and C-A-S-H gel co-generation via CO₂ transport control, Ca²⁺ leaching optimization, and pore water-phase regulation, ultimately determining mechanical performance evolution.

Keywords: carbonation curing; conservation condition; alkali activated; shield muck; mechanical properties



Academic Editor: Valentino Paolo Berardi

Received: 22 April 2025

Revised: 16 May 2025

Accepted: 16 May 2025

Published: 20 May 2025

Citation: Wang, K.; Hu, S.; Li, Y.; Xi, Z.; Qian, J.; Yuan, B. Mechanical Properties of Carbonation-Enhanced Alkali-Activated Slag-Solidified Shield Muck: Temperature–Humidity Coupling Effects. *Appl. Sci.* **2025**, *15*, 5717. <https://doi.org/10.3390/app15105717>

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1. Introduction

Shield muck is a high-water-content, low-permeability fluid-plastic soil containing clay minerals and foaming agents. Due to its unique composition and physicochemical characteristics, conventional construction waste disposal and recycling methods cannot be fully extended to its treatment and resource utilization [1–4]. Based on the study by Jing et al. on the application of slag in ultrafine tailings [5], it was found that slag was employed in tailing remediation and resource utilization research, demonstrating favorable mechanical properties in practical applications. This indicates that slag contributes to the performance enhancement of solidified materials. Alkali-activated solidification technology employs alkaline activators to dissociate calcium aluminosilicate phases in industrial solid wastes, such as fly ash and slag, inducing depolymerization–reorganization reactions that form an interwoven C-(A)-S-H/N-A-S-H gel structure. By modulating parameters like activator modulus and alkali dosage, this technology can be precisely tailored to shield muck systems

with diverse mineralogical compositions, achieving the chemical immobilization of heavy metal ions while markedly enhancing the early-stage mechanical properties of solidified matrices [6–9]. However, engineering applications still face challenges such as sluggish strength development and suboptimal mechanical performance, which hinder compliance with modern underground construction demands for rapid shield muck processing and performance requirements of engineering materials derived from shield muck.

Carbonation curing technology, based on the carbon dioxide mineralization and sequestration principle, enhances material performance through controlled reactions between calcium/magnesium components and CO₂ to form carbonates (e.g., CaCO₃), achieving CO₂ immobilization [10–12]. The implementation process involves three stages: preprocessing, carbonation reaction, and post-curing [13–16]. Alkali-activated materials, though primarily composed of calcium aluminosilicate network structures, can be optimized through raw material modification (e.g., incorporation of high-calcium industrial solid wastes such as slag powder) to construct a composite system combining a siliceous matrix with supplementary calcium sources. This strategy effectively addresses the limited carbonation activity of conventional alkali-activated materials, thereby expanding their application potential in carbonation curing technologies [17–20].

The mechanism of temperature effects on carbonation reactions exhibits multifactorial coupling characteristics, making its comprehensive influence challenging to quantify through single-parameter evaluations [21–24]. From a reaction kinetics perspective, elevated temperatures significantly reduce Ca²⁺ solubility in pore solutions, directly weakening the driving force for carbonation. However, temperature increases also enhance CO₂ diffusion coefficients, facilitating its penetration through the formed carbonate surface layers into the matrix interior [25–27]. More complexly, temperature field variations reconfigure the moisture transport equilibrium in material–environment systems. Under constant humidity conditions, matrix water retention capacity diminishes with rising temperatures, while dynamic water content evolution concurrently influences CO₂ dissolution/permeation and governs ion migration rates, resulting in mutually constraining dual effects [28–31]. These multi-scale, multi-physics interactions necessitate experimental characterization to systematically resolve the net impact of temperature on carbonation rates.

The moisture distribution within pore systems exerts a dual regulatory mechanism on carbonation processes, governing the dynamic equilibrium of gas–liquid–solid phase reactions. As the inherent medium for carbonation, the liquid phase not only facilitates CO₂ dissolution/mass transfer and Ca²⁺ ion migration but also modulates the dissolution–precipitation equilibrium of minerals such as calcium hydroxide through pore solution chemistry [32–35]. However, excessive moisture induces pore blockage effects, creating diffusion barriers between solid product layers and the liquid phase that significantly reduce CO₂'s effective penetration depth [36,37]. When the pore saturation falls below the critical threshold, discrete liquid film structures fail to maintain the continuous-phase environment required for carbonate ionization. Conversely, excessive saturation diminishes gas-phase transport pathways [38–40]. More complexly, while water molecules enhance ion migration, their evaporation–condensation cycles restructure pore architecture, thereby influencing material strength development [41–43]. Previous studies have demonstrated that pore moisture distribution significantly influences material performance, with its spatial arrangement primarily determined by ambient humidity (excluding inherent initial water content). While extensive research has been conducted on humidity effects during carbonation processes, most investigations have focused on cement-based materials. In contrast, limited attention has been given to alkali-activated systems, particularly those utilizing shield machine muck as the primary raw material. This study, therefore, aims to

elucidate the impact of environmental humidity on carbonation curing effectiveness within alkali-activated shield muck systems.

This study employs slag micro-powder as the precursor for solidifying shield muck. A composite alkaline activator comprising sodium hydroxide and sodium silicate was utilized to activate precursor reactivity. The influence mechanisms of temperature and humidity on carbonation curing efficiency and the performance of alkali-activated slag-solidified shield muck were systematically investigated, with particular emphasis on exploring their governing laws on the material's mechanical properties. Through integrated microstructural analyses encompassing pore structure evolution, alkaline environment dynamics, and carbonation product formation, the mechanical performance enhancement mechanisms of carbonated alkali-activated slag-solidified shield muck under temperature–humidity coupling effects were elucidated. This study enhances the industrial value of shield muck recycling through the synergistic application of slag micropowder–alkali activation combined with carbonation curing technology. The approach accelerates the resource utilization process of slag micropowder while providing empirical insights into temperature–humidity behavior in carbonation-cured alkali-activated systems.

2. Materials and Methods

2.1. Raw Materials

2.1.1. Shield Muck

The shield muck used in this study was sourced from a shield tunnel section of Changsha Metro Line 7 in Hunan Province, China. According to ISO 11465:1993 [44] (gravimetric method for soil moisture determination), the initial moisture content of the raw shield muck measured $23.71 \pm 1.2\%$. Given the high and variable moisture content significantly affecting test results, the collected shield muck underwent pretreatment: dried to constant weight in a 105°C forced-air drying oven (China Shanghai Yiheng Scientific Instrument Co., Ltd., Shanghai, China), mechanically crushed using a jaw crusher (China Henan Red Star Mining Machinery Co., Ltd., Zhengzhou, China), sieved through a 2 mm square-aperture sieve, and then sealed for storage. Phase analysis of the shield muck was conducted using a MiniFlex 600 X-ray diffractometer (Cu target, 40 kV), as shown in Figure 1a, with the operational parameters set to a scanning range of $15\text{--}80^\circ$ and a scanning speed of $4^\circ/\text{min}$. The chemical composition of dried shield muck was analyzed by X-ray fluorescence spectroscopy (Table 1), revealing dominant components of SiO_2 (44.62%), Al_2O_3 (18.37%), and CaO (13.13%), collectively accounting for 76.12% of total content. The major mineral phases included calcite, quartz, muscovite, and kaolinite, consistent with component characteristics reported in previous shield muck studies [3].

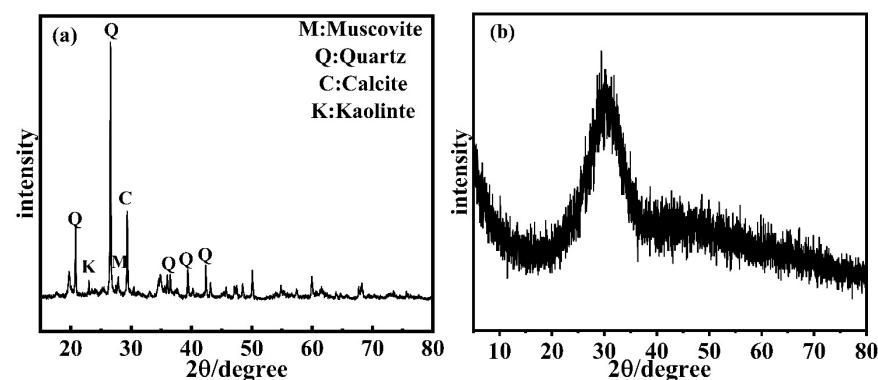


Figure 1. XRD patterns of (a) shield muck and (b) GGBFS powder.

Table 1. Chemical composition of raw materials (wt%).

Materials	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	TiO ₂	K ₂ O	Na ₂ O
Shield muck	13.84	48.18	14.10	3.90	0.01	3.42	0.65	2.55	0.45
GGBFS	41.32	30.64	15.04	0.41	2.68	6.24	2.31	0.40	0.41

2.1.2. Ground Granulated Blast Furnace Slag

The ground granulated blast furnace slag (GGBFS) used in this study was obtained from Xiangtan Iron and Steel Plant in Hunan Province, China. It exhibits a specific surface area of 426 m²/kg, total density of 2.92 g/cm³, and specific gravity of 2.54. The median particle size (D₅₀) measures 10.8 µm, with 78.4% of the particles below 20 µm. As shown in Table 1, the chemical composition of GGBFS primarily consists of SiO₂ (34.12%), CaO (38.65%), and Al₂O₃ (14.27%). The X-ray diffraction pattern (Figure 1b) demonstrates no distinct crystalline peaks within the 5–70° 2θ range, indicating high amorphous glass phase content that confirms its elevated pozzolanic reactivity.

2.1.3. Alkali Activator

The alkali activator was prepared using industrial-grade flake sodium hydroxide (purity ≥ 99.9%) and anhydrous sodium silicate powder with a modulus (SiO₂/Na₂O ratio) of 2.0. Deionized water was gradually added to the sodium hydroxide flakes at a solid-to-liquid ratio of 1:1 under continuous stirring until complete dissolution. The sodium silicate powder was subsequently blended with the sodium hydroxide stock solution. After adjusting the mixture to the target water content, homogenization was achieved through 30 min of mechanical agitation to form a homogeneous transparent solution. The prepared activator was sealed and aged for 24 h prior to experimental use.

2.2. Test Schemes

The experimental design of this study builds upon the existing literature on the carbonation curing of metakaolin-based geopolymer concrete. Carbonation-induced carbonate formation has been shown to refine the pore structure of metakaolin-based geopolymers by increasing the closed porosity and reducing the porosity ratio [10]. However, previous studies primarily implemented carbonation curing under vacuum pressurization conditions, with limited discussion on the temperature–humidity effects and impractical engineering applicability. This study, therefore, employs atmospheric-pressure carbonation curing in a controlled chamber to investigate its application in alkali-activated slag-solidified shield muck. Temperature (10–30 °C) and relative humidity (60–80%) were selected as key variables to assess their impacts on carbonation efficiency. As detailed in Table 2, the 24 h curing duration was designed to meet the critical early-age mechanical performance requirements in shield engineering, aligning with tailings matrix research protocols [5]. Laboratory observations further confirmed stabilized hydration and strength development trends within this timeframe.

As shown in Figure 2, alkali-activated slag-solidified shield muck specimens were fabricated via a cast molding process. Raw shield muck and slag micro-powder were dry-mixed for 5 min in a planetary mixer according to the mix proportions specified in Table 3, followed by 5 min of wet mixing after adding the alkaline activator (120 ± 5 rpm). The homogenized mixture was cast into 40 mm × 40 mm × 40 mm molds and subjected to vibratory compaction for 60 s. After demolding, the specimens were parafilm-sealed and pre-cured for 3 days under controlled conditions (20 ± 1 °C, 95 ± 2% RH). Carbonation-cured specimens were subsequently sealed and stored under standard curing conditions

(20 ± 1 °C, $95 \pm 2\%$ RH) for 28 days. The reference group (Ref) specimens underwent standard curing throughout (20 ± 2 °C, $95 \pm 2\%$ RH).

Table 2. Carbonation curing protocol for alkali-activated slag-solidified shield muck.

Group	Temperature (°C)	Relative Humidity (%RH)	CO ₂ Concentration (vol%)	Carbonation Duration (h)
G1	10	60	20	24
G2		70		
G3		80		
G4	20	60		
G5		70		
G6		80		
G7	30	60		
G8		70		
G9		80		

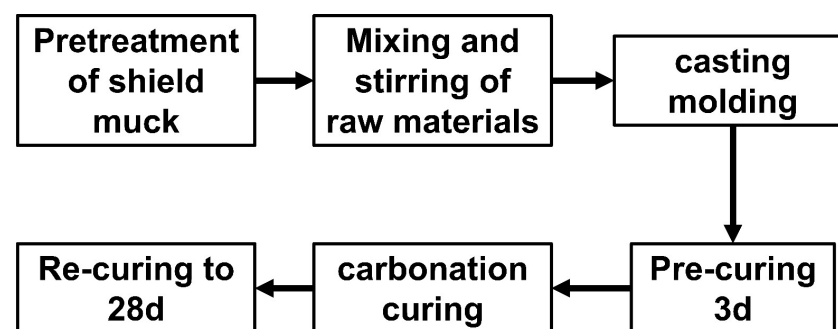


Figure 2. Testing procedures.

Table 3. Mix proportion of alkali-activated slag-solidified shield muck.

SM/g	GGBFS/g	NaOH/g	Na ₂ SiO ₃ /g	Water/g
1300	409.5	14.94	32.74	506

2.3. Experiment Test Method

2.3.1. Compressive Strength Testing

Compressive strength testing was conducted on specimens at 7-day and 28-day curing ages using a TYE-3000 universal testing machine. The reported values represent the mean of triplicate measurements, with all experimental procedures strictly following ASTM C39/C39M-21 [45] standard specifications.

2.3.2. Porosity of the Carbonated Zone

Pore structure characteristics were analyzed using an AutoPore IV 9500 mercury intrusion porosimeter (Micromeritics Instrument Ltd., Shanghai, China). Specimens at a 28-day curing age were dried to constant mass in a vacuum-drying oven at 50 °C, after which, the subsamples sectioned within a 3 mm depth from the exposed surface were subjected to porosimetry testing.

2.3.3. Pore Solution PH Analysis

Specimens at 28-day curing age were extracted from the curing chamber and dried to constant mass in a vacuum oven at 50 °C. Sequential powder samples were obtained along the carbonation depth direction through abrasive grinding at 2 mm intervals over a

total depth of 16 mm. The collected powders were sieved through a 0.08 mm square-hole sieve to remove coarse particles. Approximately 1 g of the sieved fines was dispersed in 10 g of deionized water and homogenized using magnetic stirring for 10 min. After 48 h of settling, the supernatant pH was measured with a calibrated pH meter, with the recorded value representing the pore solution pH at the midpoint of each sampled depth interval.

2.3.4. Qualitative Analysis of Carbonation Products

Powder samples obtained within 3 mm of depth from the exposed surface were crushed and sieved below an 80 μm particle size for phase analysis using a MiniFlex 600 X-ray diffractometer (Rigaku Corporation, Beijing, China). XRD patterns were acquired with a scanning range of $15\text{--}60^\circ 2\theta$ at a scan rate of $4^\circ/\text{min}$.

2.3.5. Quantitative Analysis of Carbonation Products

The decomposition temperature range of carbonation product calcium carbonate was identified as $500\text{--}900^\circ\text{C}$, with the mass loss percentage during calcination within this temperature regime corresponding to the specimen's carbon uptake rate [46]. Specimens at a 28-day curing age were vacuum-dried at 50°C for 72 h until reaching constant mass, with the initial mass recorded as m_0 . Subsequent calcination at 500°C for 1 h yielded mass m_{500} , followed by additional calcination at 900°C for 1 h to obtain m_{900} . The carbonation degree was calculated according to Equation (1).

$$C_a = \frac{m_{500} - m_{900}}{m_0} \quad (1)$$

2.3.6. Thermogravimetric

Powder samples were prepared by grinding subsamples sectioned within a 3 mm depth from the exposed surface to particle sizes below $80\ \mu\text{m}$. Thermogravimetric analysis was performed using a Netzsch STA 449 F3 analyzer (NETZSCH Scientific Instruments Trading Ltd., Shanghai, China) under a nitrogen atmosphere, with temperature programming from 35°C to 1000°C at a heating rate of $10^\circ\text{C}/\text{min}$.

2.3.7. Microstructural Morphology of Carbonation Zones

The samples were sectioned within 3 mm of the exposed surface at a 28-day curing age. Specimens with superior surface flatness were selected for observation. Prior to microscopic examination, a 5 nm-thick gold film was deposited via sputter coating to enhance surface conductivity. Microstructural characterization was performed using an FEI Quanta 450 FEG (Thermo Fisher Scientific, Shanghai, China) field-emission environmental scanning electron microscope operated at 15 kV accelerating voltage.

3. Results and Discussion

3.1. Compressive Strength and Pore Distribution

Figure 3 reveals the strength of the correlation between the specimen porosity ratio and the 28-day compressive strength. Pore structures were categorized into four classes based on pore size [47]: harmful pores ($>1000\ \text{nm}$), mesopores ($100\text{--}1000\ \text{nm}$), small pores ($10\text{--}100\ \text{nm}$), and gel pores ($<10\ \text{nm}$). As shown in Figure 3a, the harmful pore fraction exhibits a highly significant negative correlation with compressive strength (Pearson correlation coefficient $r = -0.97$, $|r| > 0.9$). In contrast, the mesopore fraction demonstrates a weaker positive correlation with strength ($r = 0.37$, $|r| < 0.9$) (Figure 3b). Neither small pores ($r = 0.12$, Figure 3c) nor gel pores ($r = 0.29$, Figure 3d) show statistically significant positive correlations with compressive strength, confirming that the volumetric fraction of harmful pores governs the 28-day compressive strength evolution in carbonation-cured

specimens. The weak linear correlation between mesopores, small pores, and gel pores may be due to the stronger linear correlation between strength and other factors, such as the degree of alkali-activated reaction and the uniformity of pore size distribution.

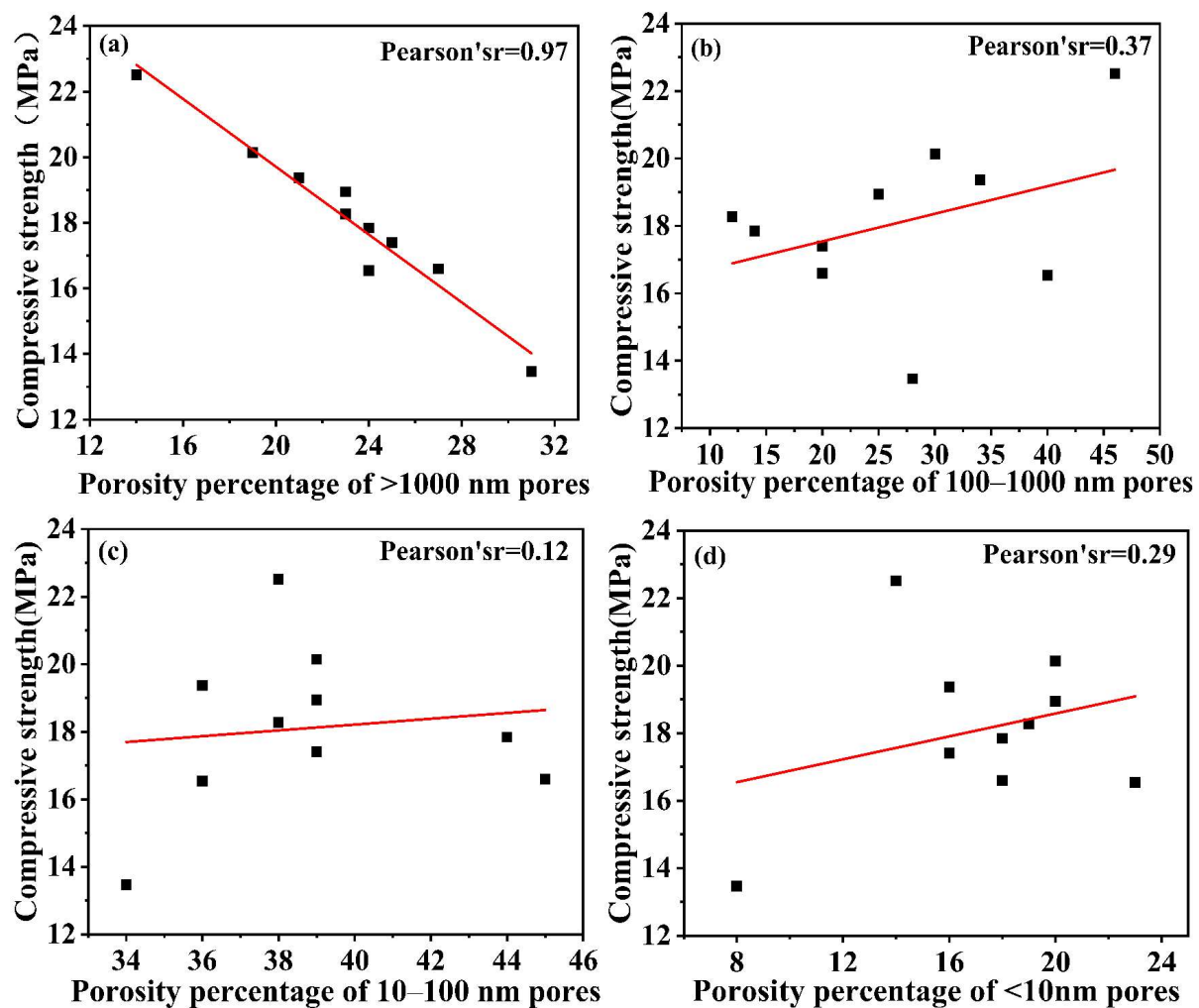


Figure 3. Relationship between 28-day compressive strength and pore distribution in specimens subjected to carbonation curing under varying temperature–humidity conditions: (a) harmful pores (>1000 nm), (b) mesopores (100–1000 nm), (c) small pores (10–100 nm), (d) gel pores (<10 nm).

Figure 4a–f systematically characterize the regulatory effects of relative humidity on the 28-day compressive strength and pore size distribution of carbonation-cured specimens under three temperature gradients (10 °C, 20 °C, and 30 °C). As the relative humidity increased from 60% to 80%, all temperature groups exhibited an initial strength increase, followed by a decrease. The 20 °C–70% RH group achieved the maximum strength of 22.51 MPa, representing a 36.1% enhancement compared to the standard-cured baseline strength (16.54 MPa). In contrast, the 30 °C–60% RH group reached only 13.47 MPa, showing an 18.6% reduction relative to the baseline. Notably, the 28-day strength evolution trend demonstrated a significant inverse correlation with a harmful pore fraction (>1000 nm), confirming that optimizing pore structure is the critical factor governing strength enhancement.

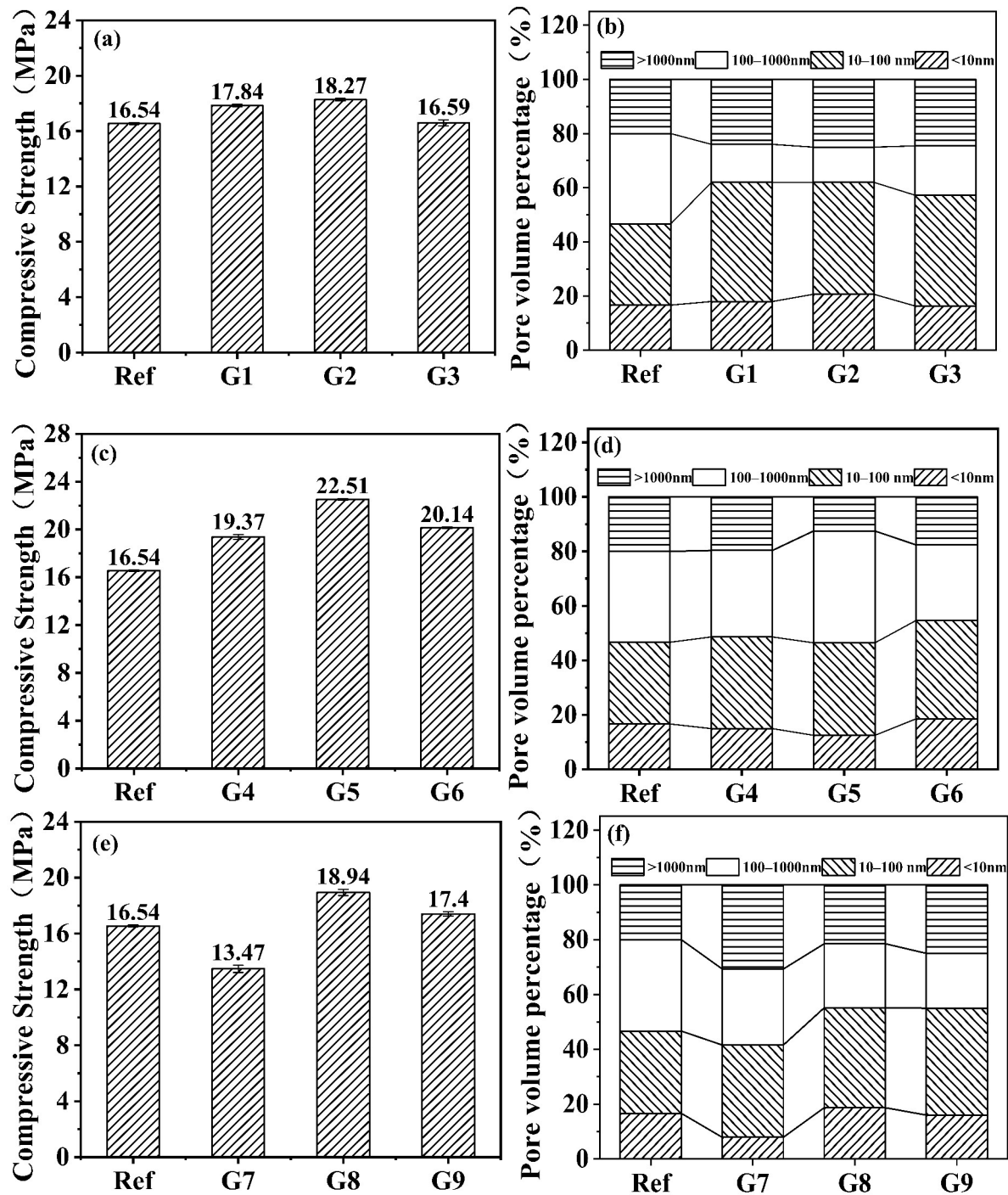


Figure 4. Effects of humidity on 28-day compressive strength and pore distribution after carbonation curing under varying temperatures: (a,b) 10 °C, (c,d) 20 °C, (e,f) 30 °C.

The experimental data indicate that moderate humidity elevation optimizes the carbonation reaction process. At 70% RH, a dynamic equilibrium between the CO₂ diffusion rate and the product deposition efficiency is achieved, enabling an effective filling of harmful pores (>1000 nm) by carbonation products. However, excessive humidity (>70% RH) increases pore water saturation, obstructs CO₂ transport pathways, and suppresses secondary hydration product formation, ultimately degrading the pore structure. On the contrary, low humidity conditions (60% RH) accelerate the diffusion of CO₂ but lead to excessive carbonation, generating expansive stresses that promote microcrack propagation, consequently leading to mechanical performance deterioration.

Figure 5a–f demonstrate the regulatory effects of temperature gradients on the 28-day compressive strength and the pore size distribution of carbonation-cured specimens under 60%, 70%, and 80% relative humidity (RH) conditions. As the temperature increased from 10 °C to 30 °C, all humidity groups exhibited a pronounced unimodal evolution pattern in both strength and harmful pore fraction (>1000 nm), which confirmed the threshold effect of temperature on the optimization of pore structure. Among these, the 20 °C–70% RH group achieved optimal performance, indicating that moderate temperature elevation accelerates Ca^{2+} leaching from alkali-activated precursors and enhances CO_2 mass transfer kinetics, thereby promoting a selective deposition of carbonation products within harmful pores (>1000 nm).

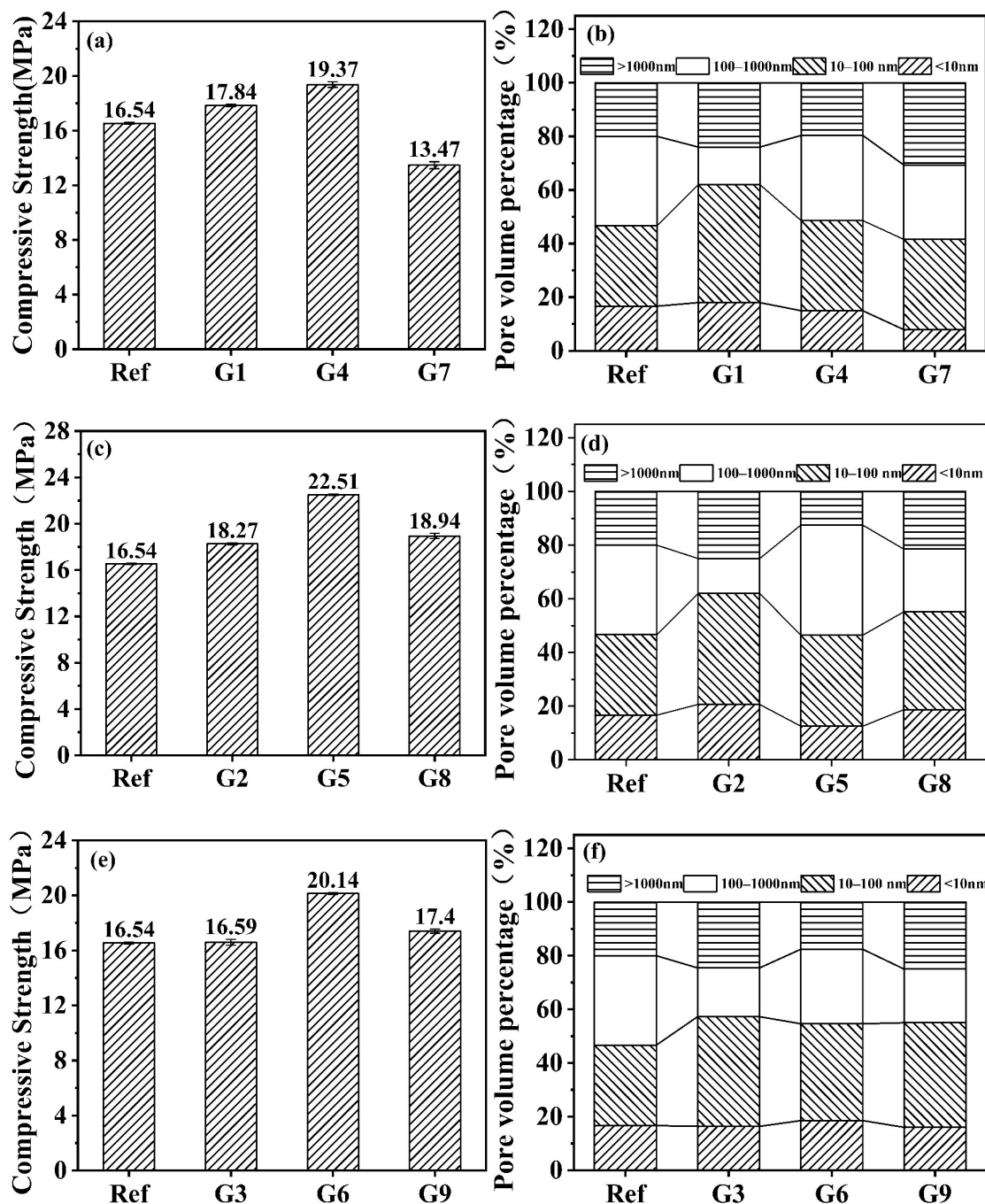


Figure 5. Effects of temperature on 28-day compressive strength after carbonation curing under varying humidity conditions: (a,b) 60% RH, (c,d) 70% RH, (e,f) 80% RH.

Notably, temperatures exceeding 20 °C induced a heterogeneous deposition of carbonation products, generating localized expansion stresses that coarsened pore structures. Conversely, at 10 °C, constrained Ca^{2+} dissolution and retarded CO_2 diffusion significantly suppressed carbonation progress, resulting in a diminished humidity sensitivity of harmful pore content (as evidenced by the negligible strength variations with humidity changes in Figure 4a).

3.2. Alkaline Environment Evolution

Figure 6a–c systematically reveal the dynamic influence patterns of humidity gradients on pore solution pH under varying temperature conditions. Under identical testing depths, specimens at 70% RH demonstrated the highest pore solution pH values and achieved dynamic equilibrium most rapidly, stabilizing within the 12.4–12.6 range. This phenomenon originates from humidity gradient-driven ion migration effects: under low humidity (60% RH), alkaline ions preferentially migrate toward the specimen surface along the humidity gradient, resulting in internal alkalinity reduction and suppressed C-A-S-H gel formation efficiency during the post-curing stages. Concurrently, surface alkaline ion accumulation accelerated carbonation reaction kinetics, thereby inducing instability in harmful pore structure filling processes and a consequent strength degradation. In contrast, high humidity (80% RH) environments triggered solution dilution effects through external moisture infiltration, significantly reducing internal pore liquid alkalinity and impairing C-A-S-H gel generation. Specifically, the humidity difference between the environment and the specimen induces the directional flow of a pore solution from the surface to the interior of the specimen. Water condensation on the specimen surface causes a dilution of the surface ions relative to the interior. This drives a diffusion transport of ions from the interior to the specimen surface and results in alkali depletion in the pore solution [48].

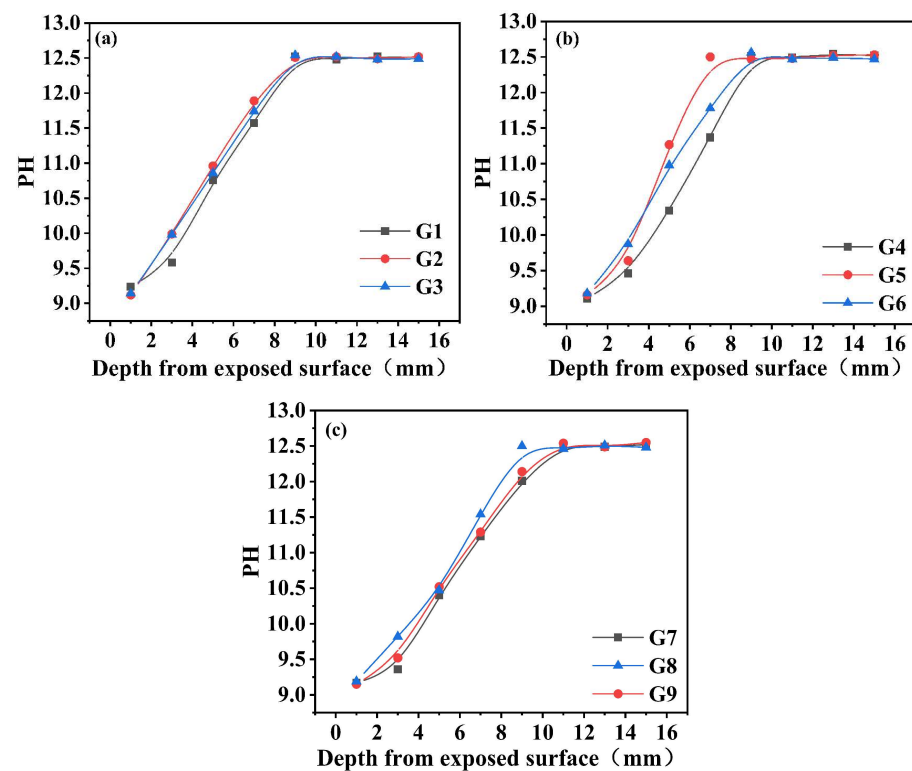


Figure 6. Humidity effects on pore solution pH at varying depths under different temperatures: (a) 10 °C, (b) 20 °C, (c) 30 °C.

Figure 7a–c illustrate the dynamic regulatory effects of temperature variations on pore solution pH under different humidity conditions. The experimental data revealed that pore solution alkalinity at equivalent depths peaked at 20 °C and minimized at 30 °C. This phenomenon is attributed to the densified carbonation product layer formed at 20 °C, which acts as a CO₂ diffusion barrier to preserve internal alkalinity. Conversely, low-temperature conditions (10 °C) suppressed CO₂ diffusivity, limited Ca²⁺ dissolution rates, and retarded pore water evaporation, collectively inducing alkaline ion sequestration that accumulated internal alkalinity. When the temperature increased to 30 °C, accelerated pore solution evaporation triggered a surface enrichment of the alkaline ions, intensifying the carbonation reactions and depleting the internal alkalinity, consequently impairing the hydration reaction efficiency during subsequent curing.

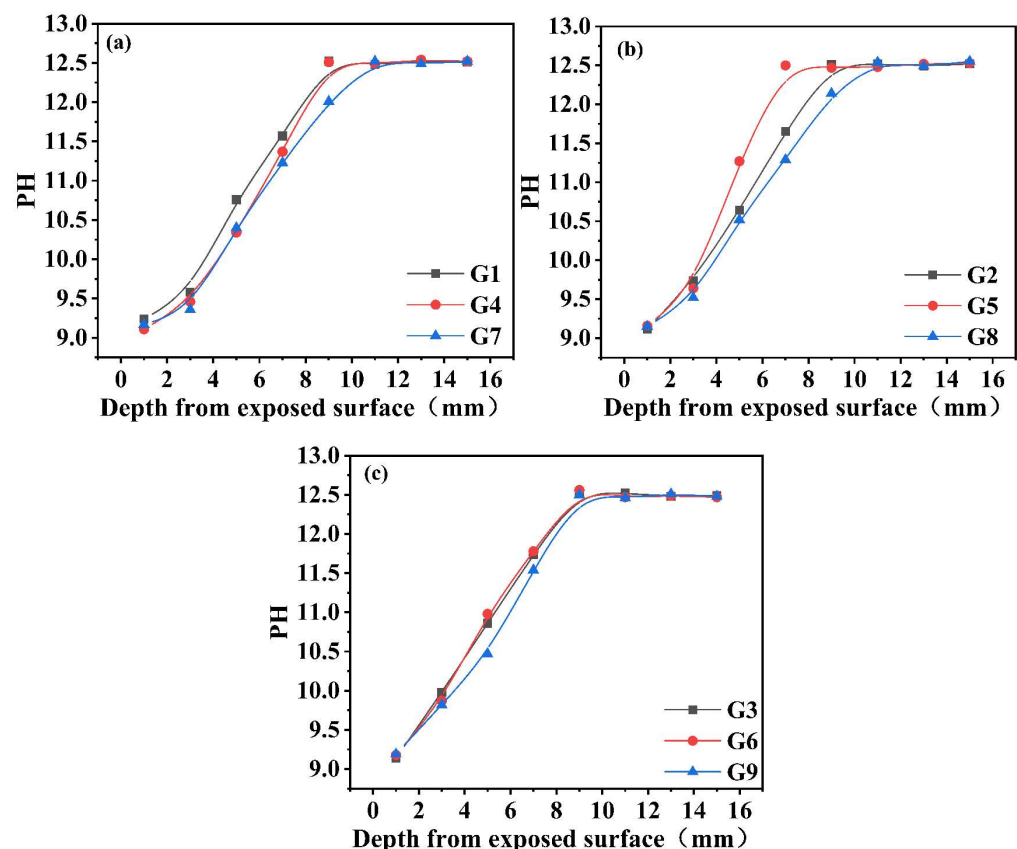


Figure 7. Effects of temperature on pore solution pH at varying depths under different humidity conditions: (a) 60% RH, (b) 70% RH, (c) 80% RH.

3.3. Carbonation Product Characterization

Figure 8a–f illustrate the evolution patterns of carbonation uptake rates and 28-day phase composition characteristics of carbonation-cured specimens under varying temperature–humidity conditions. As the relative humidity increased from 60% to 80%, the carbonation uptake rates exhibited a gradual decline, indicating limited humidity-dependent modulation of carbonation reactions. This behavior arises from humidity gradients induced by internal–external humidity differentials, which drive the formation of continuous water films at the specimen surfaces. These films physically inhibit CO₂ transport efficiency into internal pore networks through barrier effects. Concurrently, high-humidity environments intensify pore water blocking effects, confining carbonation reactions to surface regions and reducing kinetic rates.

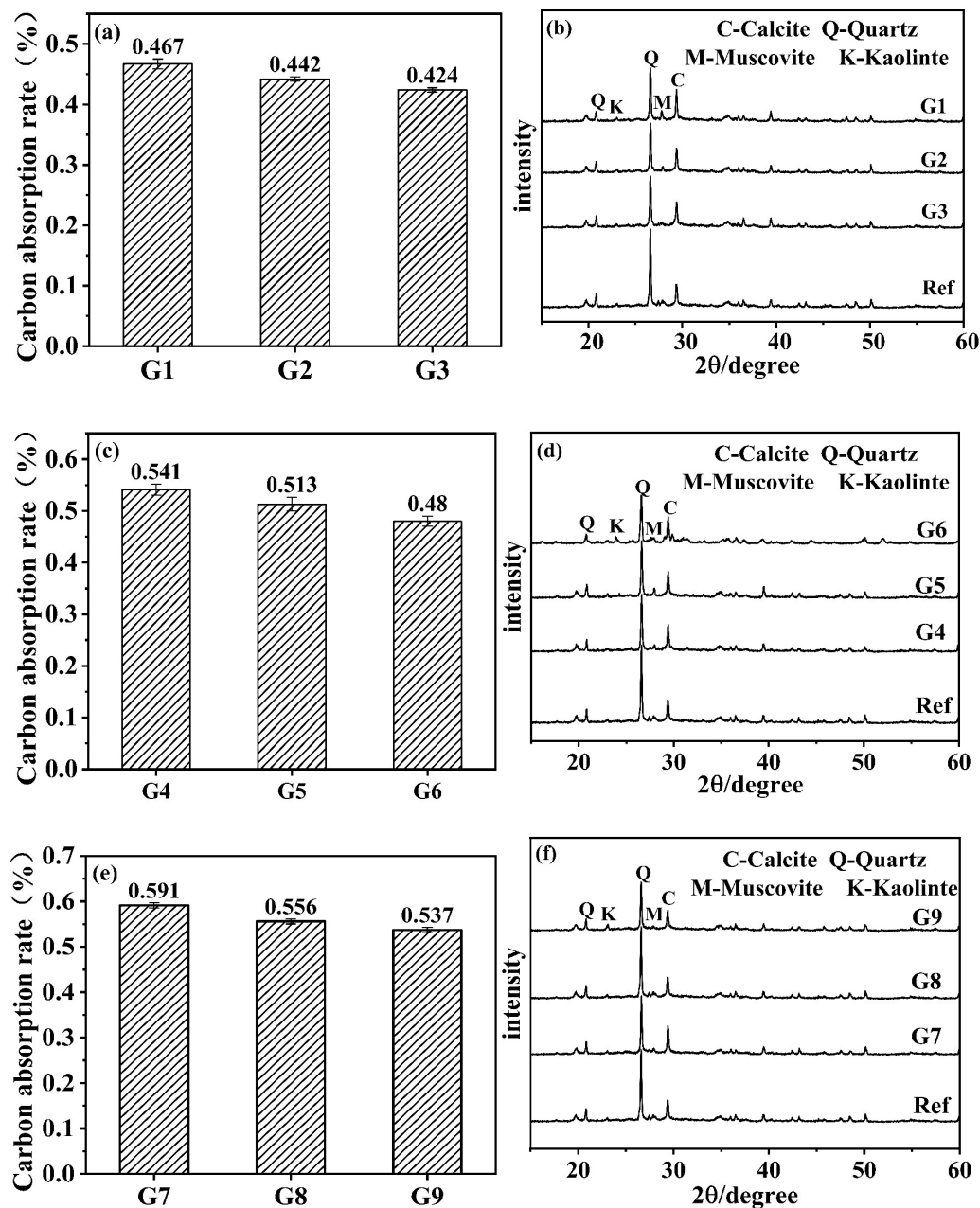


Figure 8. Effects of humidity on carbon uptake rate and 28-day phase composition under different temperatures: (a,b) 10 °C, (c,d) 20 °C, (e,f) 30 °C.

XRD phase analysis (tested on specimens at 3 mm depth from the exposed surface) shows that, compared with the standard curing group, the characteristic calcite (CaCO_3) diffraction peak intensity ($2\theta = 29.4^\circ$) in carbonation-cured specimens is significantly enhanced, confirming that calcite is the dominant carbonation product. This aligns with the findings of Eric R. McCaslin et al., whose study on alkali-activated slag materials identified the main factors controlling the decalcification degree of C-A-S-H gels and demonstrated that calcite is the sole crystalline calcium carbonate formed in carbonated alkali-activated slag pastes [49]. The higher the crystallinity of the crystal, the higher the intensity of the corresponding diffraction peak and the sharper the shape of the diffraction peak. With the increase in humidity, the diffraction peaks of calcite show a sharper morphology and intensity increase, indicating that its crystallinity is improved. This suggests that the continuous water film on the surface and the retarding effect of pore water compromise the crystallographic integrity of carbonation products. Lower crystallinity weakens the me-

chanical interlocking effect between calcite particles, leading to a reduced filling efficiency of harmful surface pores and diminished mechanical performance enhancement.

Figure 9a–f characterize the effects of temperature variations on carbon uptake rate and product composition in alkali-activated slag-solidified shield muck under different humidity conditions. The experimental data demonstrate a significant increasing trend in the carbon uptake rate with temperature elevation (10–30 °C), indicating enhanced carbonation reaction kinetics through thermal activation. This enhancement mechanism originates from the temperature-dependent acceleration of CO₂ diffusion coefficients, improved Ca²⁺ solubility, and evaporation-induced alkaline ion migration, which synergistically promote carbonation product formation.

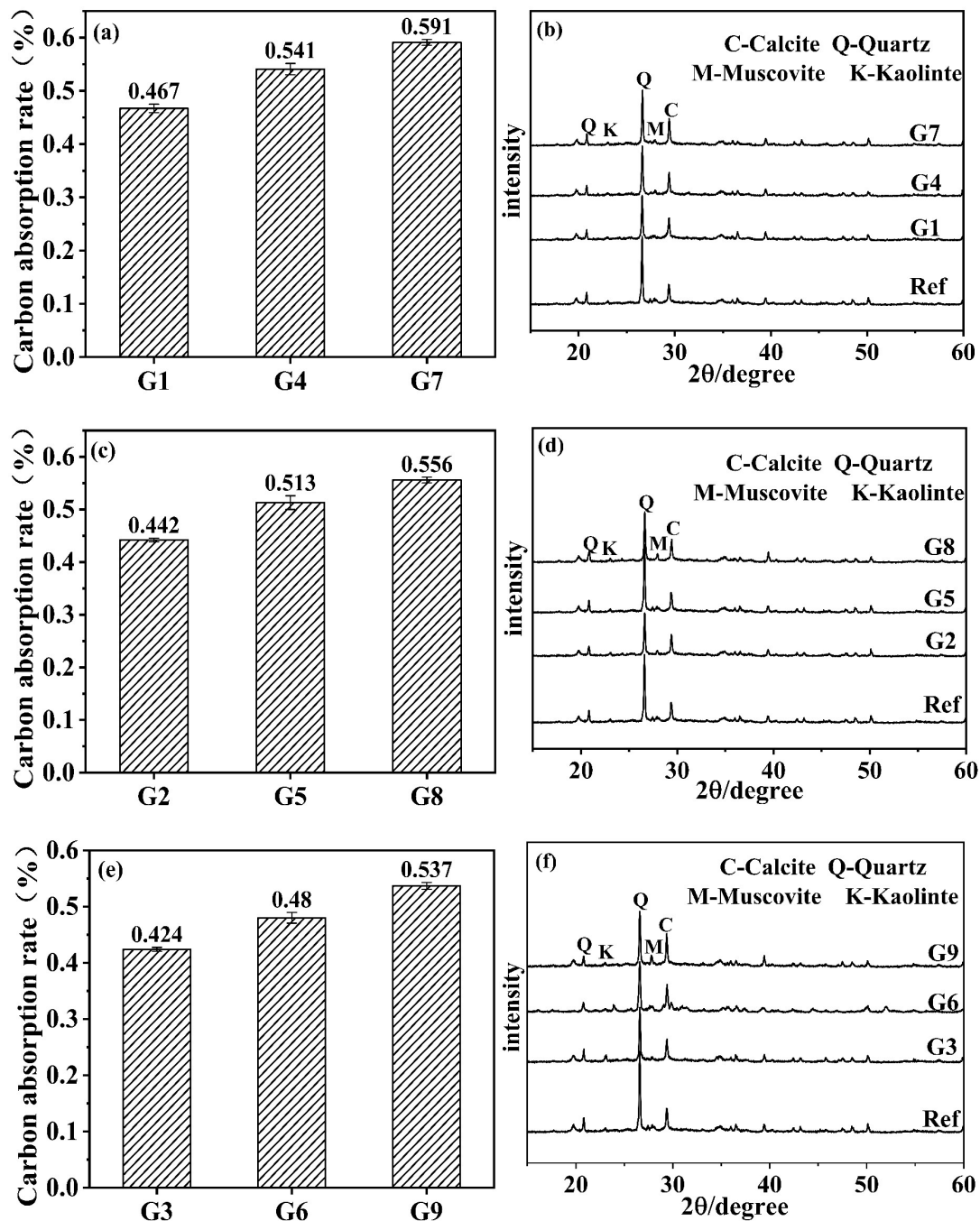


Figure 9. Effects of temperature on carbon uptake rate and 28-day phase composition under different humidity conditions: (a,b) 10 °C, (c,d) 20 °C, (e,f) 30 °C.

An XRD phase analysis demonstrated that the carbonation products across different temperature groups were predominantly composed of calcite-type calcium carbonate (CaCO_3). The characteristic diffraction peak intensity at $2\theta = 29.4^\circ$ increased with elevated temperatures, accompanied by sharpened peak morphology. These observations confirm that temperature elevation effectively enhances the crystallinity of carbonation products, thereby influencing the filling efficiency of harmful pores and ultimately achieving a targeted enhancement of the material's macroscopic mechanical properties.

3.4. Microscopic Test Analysis

Figure 10 illustrates the effects of temperature and humidity on the microstructure of carbonation-cured specimens. With an increasing temperature (Figure 10a,d,g), the nucleation and growth of calcite crystals around shield muck particles accelerate, effectively filling harmful pores. However, exceeding the critical temperature (Figure 10g) causes crystal coarsening, generating new pores and cracks [50]. As humidity rises (Figure 10g–i), the inhibited enrichment of alkaline ions on the surface reduces the crystal size, alleviating carbonation-induced volume expansion. Nevertheless, excessive humidity (Figure 10i) compromises the pore-filling capacity of calcite and diminishes structural densification. All experimental groups followed similar trends, with Group G5 (Figure 10e) demonstrating optimal calcite pore-filling effects due to its ideal temperature–humidity conditions.

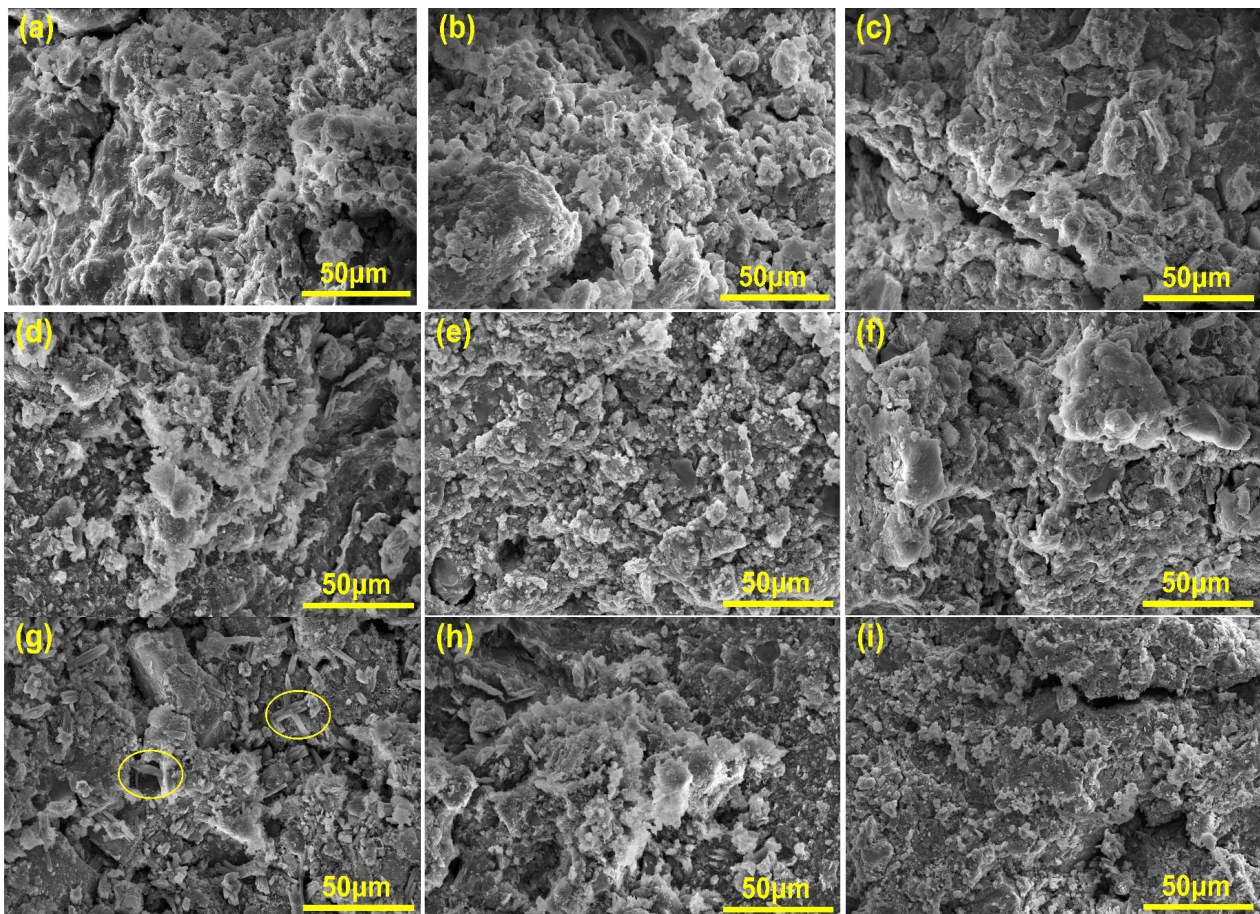


Figure 10. SEM images of (a) G1 (10 °C—60% RH), (b) G2 (10 °C—70% RH), (c) G3 (10 °C—80% RH), (d) G4 (20 °C—60% RH), (e) G5 (20 °C—70% RH), (f) G6 (20 °C—80% RH), (g) G7 (30 °C—60% RH), (h) G8 (30 °C—70% RH), (i) G9 (30 °C—80% RH). The circles represent calcite.

John L. Provis et al. proposed that carbonation in alkali-activated slag paste occurs directly within the C-A-S-H gel [51]. Additionally, based on the thermal analysis results from Shi et al.'s thermogravimetric tests on alkali-activated slag pastes, the differences in thermal decomposition behavior between G5 and G7 (exhibiting the largest performance variations in the study) were attributed to three characteristic stages: (1) structural water removal from the C-A-S-H gel in the 120–300 °C range, (2) C-A-S-H gel network depolymerization in the 250–600 °C range, and (3) calcite (CaCO_3) decomposition in the 650–900 °C range (Figure 11) [52]. The G7 specimen demonstrated more intense mass loss rates throughout the thermal degradation process, with a higher calcite characteristic decomposition peak intensity than G5, confirming that high-temperature and low-humidity conditions significantly promote carbonation product formation.

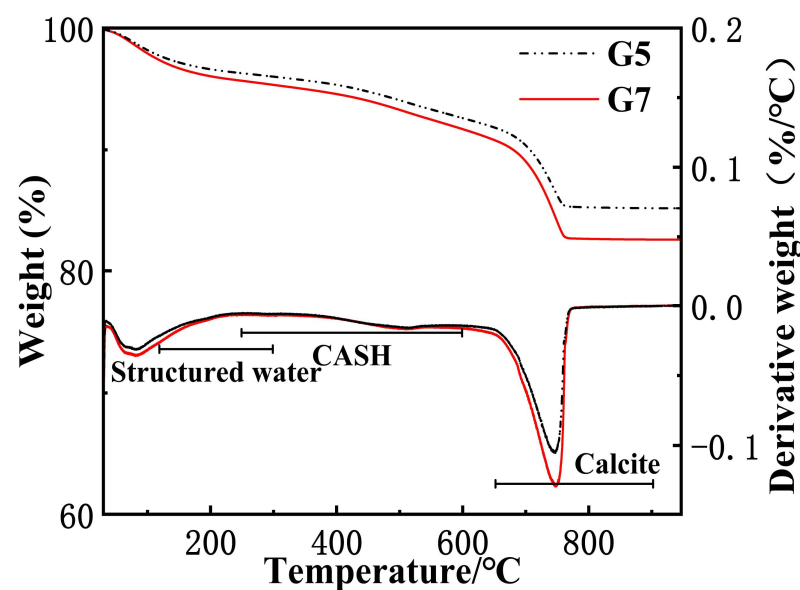


Figure 11. The TG/DTG curves of G5 (20 °C, 70% RH) and G7 (30 °C, 60% RH).

Quantitative analysis of the Table 4 data reveals higher mass loss percentages for G5 during both C-A-S-H structural water desorption and gel network decomposition stages, while demonstrating an inverse trend during calcite decomposition. This phenomenon is attributed to the densified calcite filling layer formed under 20 °C—70% RH conditions, which effectively inhibits CO_2 inward diffusion by refining harmful pore structures, thereby maintaining a stable alkaline environment for continuous C-A-S-H gel formation. Conversely, high-temperature and low-humidity conditions accelerate carbonation kinetics, where the crystallization-induced expansion stresses from excessive calcite generation cause pore coarsening. Concurrent carbonation-enhanced alkaline environment degradation and pore water migration-driven alkaline ion surface enrichment synergistically deplete internal alkalinity, ultimately suppressing C-A-S-H gel production during later curing stages.

Table 4. Thermogravimetric losses (%) for different temperature intervals.

Group	120–300 °C	250–600 °C	650–900 °C
G5	1.781	3.97	8.199
G7	1.599	3.685	10.927

Fabien Georget et al. utilized sulfur and alkali EDS mapping to delineate the carbonation front, explicitly identifying the interface between carbonated and non-carbonated

zones [53]. Based on these experimental findings, it is justified to extend their conceptual framework, as illustrated in Figure 12: the specimen's surface layer adjacent to the exposed face constitutes the fully carbonated zone; the region where pore solution pH has not reached dynamic equilibrium represents the active carbonation zone; and the area with stabilized pore solution pH corresponds to the non-carbonated zone. Within the fully carbonated zone, the significantly weakened alkaline environment fails to provide sufficient alkalinity for alkali-activation reactions, rendering mechanical performance predominantly dependent on calcite's pore-filling effects on harmful voids [54]. The strength of the active carbonation zone arises from the combined contributions of calcite-induced pore densification and the bonding effect of hydration-derived C-A-S-H gel on shield machine muck particles. The non-carbonated zone maintains a highly alkaline environment, where compressive strength correlates with hydration reaction progression [55].

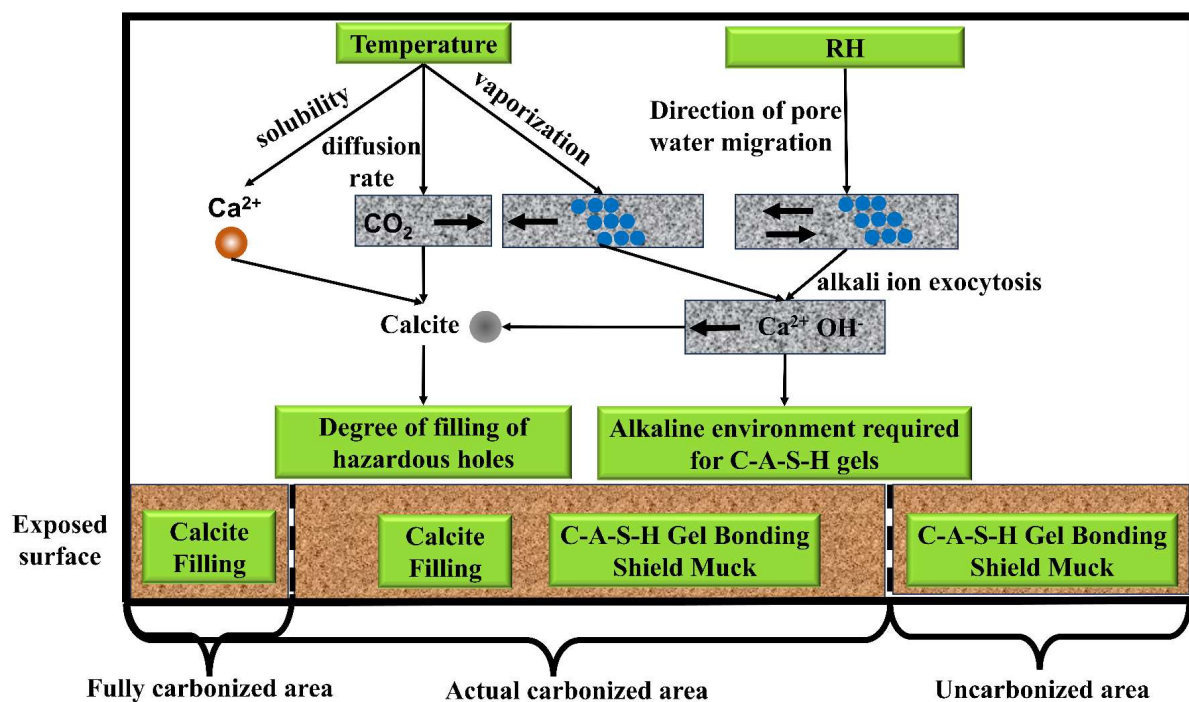


Figure 12. Mechanism of temperature and humidity effects on the mechanical properties of alkali-activated slag-solidified shield muck materials during carbonation curing.

The temperature field modulates the CO_2 diffusion rate, Ca^{2+} dissolution kinetics, and pore water evaporation process, thereby influencing calcite formation dynamics. It concurrently alters alkaline ion migration behaviors within pores and ultimately governs harmful pore-filling efficiency and the alkaline microenvironment required for subsequent C-A-S-H gel formation. The humidity field governs calcite's pore structure improvement effects by driving pore water migration under humidity gradients, while synergistically regulating alkaline ion transport to constrain the kinetics of later C-A-S-H gel generation. Calcite, exhibiting a strongly negative zeta-potential in alkaline solutions, generates repulsive forces and crystallization pressure during excessive formation, inducing volumetric expansion [50]. Thus, a strict control of temperature–humidity conditions during carbonation curing is essential to ensure calcite's positive microstructural contributions. The coupled temperature–humidity regulatory mechanism, governing both harmful pore filling efficiency and cementitious product formation environments, constitutes the critical control factor determining material macro-mechanical performance.

4. Conclusions

The experimental objectives of this study are to investigate the relationship between properties and pore structures in alkali-activated shield machine muck solidified bodies under carbonation curing at varying temperature–humidity conditions, the connection between pore solution alkalinity and temperature–humidity conditions, and the effects of temperature and humidity on carbonation and hydration products. Additionally, the mechanistic roles of temperature and humidity were summarized and analyzed:

- (1) The proportion of harmful pores (>1000 nm) exhibited a highly significant negative correlation with compressive strength, while pores <1000 nm showed a weaker positive correlation. Reducing harmful pore content constitutes the key to improving mechanical performance in alkali-activated slag shield machine muck solidified bodies;
- (2) The 28-day compressive strength demonstrated a distinct initial increase followed by a decrease across the tested temperature–humidity gradient range. The maximum strength occurred at 20 °C/70% RH, showing a 36.1% enhancement when compared with standard curing, fully confirming that controlled temperature–humidity conditions during carbonation curing can effectively improve mechanical properties of alkali-activated slag shield machine muck solidified bodies;
- (3) The temperature–humidity conditions during carbonation curing showed negligible but measurable effects on the alkaline environment of pore solutions. These effects primarily manifested through humidity-controlled pore solution dilution and ion migration processes, as well as temperature-regulated Ca^{2+} dissolution and pore solution evaporation;
- (4) The carbon absorption rates gradually increased with a rising temperature and decreasing humidity, indicating intensified carbonation reactions. The crystalline carbonation product was identified as calcite, while the hydration product remained the C-A-S-H gel phase. Under high-temperature/low-humidity conditions, an accelerated decomposition of C-A-S-H gel through carbonation promoted continuous growth and a coarsening of calcite crystals, leading to non-uniform deposition. This ineffective filling of harmful pores ultimately degraded mechanical performance.

Author Contributions: K.W.: Writing—original draft, Visualization, Software, Investigation, Data curation; S.H.: Writing—Investigation, review and editing; Y.L.: Writing—review and editing, Resources, Supervision, Funding acquisition; Z.X.: Conceptualization; J.Q.: Investigation; B.Y.: Writing—review and editing, Resources, Supervision, Funding acquisition. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the Natural Science Foundation of Hunan Province, China (Grant No. 2024JJ9079) and the National Key Research and Development Program of China (Grant No. 2022YFC3803402).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in the study are included in the article; further inquiries can be directed to the corresponding author.

Conflicts of Interest: Authors Y.L. and Z.X. were employed by the company China Construction Fifth Engineering Bureau Co., Ltd. (Changsha, China). The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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