

Article

Experimental Study on CO₂ Foamed Concrete Prepared from Alkali-Activated High-Fluidity Pipe-Jacking Spoil in Water-Rich Sandy Strata

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Abstract

Urban underground construction in water-rich sandy strata produces large quantities of high-fluidity pipe-jacking spoil whose high water content, residual conditioning agents and heavy metal contaminants make conventional dewatering and landfilling increasingly unsustainable under carbon peaking and neutrality targets. This study explores a low-carbon route that converts such spoil into CO₂ foamed concrete through a coupled alkali activation–CO₂ foaming process. Ground granulated blast furnace slag and fly ash are used as geopolymer precursors, while a CO₂-based aqueous foam is introduced as both a pore-forming phase and carbon source. Single-factor tests and an L16(4⁴) orthogonal design are conducted to quantify the effects of CO₂ concentration, foam volume fraction, geopolymer dosage and alkali activator content on fluidity, setting time and compressive strength. Scanning electron microscopy (SEM) is employed to examine pore structure, gel morphology, carbonate precipitation and the interfacial transition zone around spoil particles. The results identify an optimum mix window (CO₂ 60–80%, foam 70–80%, geopolymer ≈ 20% and alkali activator ≈ 10% of solids) that delivers a fluidity above 210 mm, 28-day strength exceeding 3.0 MPa and a uniform closed-pore network. A multi-scale mechanism is proposed in which physical foaming, chemical carbonation and spoil particle immobilization act synergistically to form a dense gas–solid–soil composite suitable for in situ backfilling.

Keywords: pipe-jacking spoil; CO₂ foamed concrete; alkali-activated materials; geopolymers; carbonation; microstructure; underground construction spoil recycling



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1. Introduction

The development and construction of urban underground space generate a large volume of construction spoil. The improper disposal of spoil not only leads to serious environmental problems, but the increasing scarcity of disposal sites also drives up project costs. Statistics indicate that more than 2 billion tonnes of construction and demolition waste are produced annually in China, among which construction spoil accounts for as much as 40–50% [1]. Traditional spoil disposal modes are expensive—typically 5–10% of the total project cost—and cause additional environmental burdens such as land occupation, dust pollution and groundwater contamination [2,3]. Unlike conventional spoil generated

by open-cut excavation, spoil produced by shield tunneling or pipe-jacking in water-rich sandy strata in the Yangtze River Delta usually has a very high moisture content (often exceeding 30%) and complex composition. It contains residual bentonite, polymer conditioning agents, heavy metals and organic pollutants used during shield or pipe-jacking construction. Therefore, spoil treatment requires not only on-site solid–liquid separation but also the removal or immobilization of harmful substances to avoid secondary pollution. Consequently, there is an urgent need to develop in situ, nearby and low-carbon resource utilization technologies for high-fluidity spoil to support sustainable urban development.

With increasingly stringent environmental regulations and continuously rising spoil disposal costs, the resource utilization of spoil has gradually become an important direction in the construction industry. The current reuse routes for spoil include spoil-based concrete, non-fired bricks, non-fired lightweight aggregates, filling materials and grouting materials. For spoil from shield and pipe-jacking construction, Zhang et al. reviewed solidification technologies based on sludge stabilization theories [4]; Wang et al. investigated the preparation and solidification mechanism of non-fired hollow bricks using spoil from silty strata [5]; Xu et al. studied the solidification of spoil from slate strata in earth pressure balance shield tunneling using alkali-activated slag [6]; Zhou et al. examined the efficient dewatering performance of liquefied dimethyl ether (DME) phase change on spoil from moderately weathered phyllite strata [7]; and Zhang et al. analyzed the dewatering behavior of sandy strata shield spoil using liquid DME–TTFP [8]. Depending on the sand, gravel and clay content, these methods generally require multiple unit operations—dewatering, separation, crushing, screening and solidification modification—which consume substantial energy and are difficult to scale to the huge spoil volumes generated by modern underground construction. They also struggle to match the high efficiency required on construction sites. Under the “dual-carbon” policy, transforming waste fluid spoil into low-carbon foamed concrete and forming a circular technical route of “treating waste with waste” represents a promising solution for spoil disposal [9]. However, two key technical bottlenecks must be overcome: (i) identifying a low-carbon binder that can largely replace Portland cement; and (ii) developing a robust foaming technology suitable for complex spoil systems.

Alkali-activated binders provide a feasible alternative to high-carbon Portland cement. Their core concept is to use alkaline activators (e.g., sodium silicate solution and sodium hydroxide) to activate the latent reactivity of industrial by-products such as ground granulated blast furnace slag (GGBS) and fly ash, forming inorganic polymer gels with a three-dimensional aluminosilicate network. Davidovits first proposed the concept of “geopolymers” in 1991 and systematically elucidated their polymerization mechanism and high temperature and chemical resistance properties. Duxson, Shi, Provis and others comprehensively reviewed the reaction mechanisms and microstructural evolution of alkali-activated materials (AAMs) and pointed out that their life cycle CO₂ emissions can be reduced by more than 70% compared with ordinary Portland cement [10–14].

Cement production is one of the largest industrial sources of anthropogenic CO₂ emissions worldwide, contributing approximately 8% of global anthropogenic CO₂ emissions [15]. The construction sector therefore faces considerable pressure to reduce carbon emissions. Monkman and Shao demonstrated that CO₂ curing can react with Ca(OH)₂ and C–S–H in cement-based materials to form CaCO₃, significantly enhancing early-age strength while sequestering CO₂ [16]. More recently, advanced concepts combining CO₂ and foaming technology have been proposed. CO₂ is used directly to generate foam within alkali-activated slag paste [17,18]; Namsone et al. confirmed the CO₂ uptake capacity of foamed concrete during carbonation [19], and Wei et al. reported a substantial CO₂ sequestration potential in alkali-activated slag/fly ash-based CO₂ foamed concretes, providing a promising route toward “negative-carbon” materials [20,21].

Foamed geopolymer (GP)/alkali-activated foam composites are lightweight porous inorganic materials produced by introducing gas bubbles into an alkali-activated aluminosilicate matrix. Due to their non-combustibility and the ability to tailor density and pore connectivity, foamed GP systems have been widely explored for thermal insulation, lightweight blocks/panels, void filling and backfilling and other low-to-moderate-strength applications. Their macroscopic performance is governed by density/porosity, pore size distribution and connectivity (open vs. closed pores) and the continuity of the geopolymer gel skeleton. In a fresh state, workability and foam stability (bubble coalescence/collapse) are critical because instability directly translates into non-uniform pore structure and degraded strength. Recent studies have emphasized that strength development, density control and functional performance (e.g., thermal conductivity and durability-related transport properties) must be considered simultaneously when designing foamed GP materials [22–24].

Although CO₂ foamed alkali-activated systems show great potential [25–27], nearly all existing studies are based on idealized laboratory raw materials, and there is a severe lack of research on real high-fluidity spoil with complex composition. In reality, fluid spoil is a multi-phase system containing water, soil, cementitious materials and gas. Its high moisture content, complex mineralogical and chemical composition and non-uniform particle size distribution are expected to strongly influence CO₂ dissolution and diffusion, bubble nucleation and stabilization, as well as the kinetics of carbonation reactions [28,29]. At present, the cooperative mechanism of CO₂ “foaming–stabilizing–strengthening–carbonation” in an alkali-activated fluid spoil system remains unclear. Systematically clarifying how CO₂ foaming behaves in such a complex and realistic system is therefore the key innovation point and primary focus of this work.

Despite the rapid development of foamed geopolymers and carbonation-based cementitious materials, the existing studies predominantly rely on well-controlled laboratory precursors and relatively stable pore-forming conditions. In contrast, high-fluidity pipe-jacking spoil from water-rich sandy strata is a heterogeneous, moisture-rich and chemically complex multi-phase system containing residual conditioning agents, which can profoundly alter CO₂ dissolution/diffusion, bubble nucleation and stability and the coupled kinetics of carbonation and alkali activation. Therefore, the coupled “CO₂ foaming–carbonation–alkali activation” mechanism in real spoil-based systems remains insufficiently understood. This study addresses this gap and makes three specific contributions: (i) proposing an alkali activation–CO₂ aqueous foam route to directly upcycle high-fluidity spoil into pumpable foamed backfill material without energy-intensive multi-step dewatering; (ii) establishing an experimentally validated parameter window for a workability–setting–strength balance using single-factor tests and an L16(4⁴) orthogonal array; and (iii) linking quantitative pore statistics and SEM evidence (closed-pore network, gel morphology, carbonate precipitation and ITZ densification around spoil particles) to macroscopic rheology and strength, thereby elucidating a composition–structure–property relationship for spoil-based CO₂ foamed alkali-activated composites.

2. Materials

2.1. High-Fluidity Spoil

The spoil used in this study was sampled from fluid spoil discharged during pipe-jacking construction in water-rich sandy strata. It is characterized by a high moisture content (typically $\geq 30\%$) and a complex mineralogical composition. The particle size distribution is dominated by fine sand and silt fractions, with a certain amount of clay-sized particles. In addition, residual additives from construction, such as bentonite and polymer conditioning agents, are present in the spoil as external admixtures.

2.2. Geopolymer System

The geopolymer system consists of fly ash, GGBS and a small amount of cement, which together act as the cementitious precursors that form an inorganic polymer gel with a three-dimensional aluminosilicate network under alkali activation.

(1) Fly ash

A Class I fly ash is used. Its main chemical components are SiO_2 (approximately 45.1%) and Al_2O_3 (approximately 36.8%), with minor amounts of CaO , MgO and other oxides. The spherical morphology of the ash particles helps improve the rheology of the paste. Fly ash possesses pozzolanic activity and can be depolymerized in an alkaline environment to participate in the formation of N–A–S–H-type gels.

(2) GGBS

An S95-grade slag is used, with major components of CaO (about 40%), SiO_2 (about 35%) and Al_2O_3 . It exhibits latent hydraulic reactivity. Under alkali activation, GGBS rapidly reacts to form C–(A)–S–H gels, providing the matrix with early-age strength.

(3) Cement

An ordinary Portland cement with strength grade 42.5 is used as an auxiliary binder. Its main mineral phases are C_3S , C_2S , C_3A and C_4AF . The small amount of cement helps regulate setting behavior and enhances the stability of the system at early ages.

2.3. CO_2 Foam System

The CO_2 foam system is composed of a physical foaming agent, a foam stabilizer and CO_2 gas. It is used to introduce uniform, closed pores into the paste, thereby improving the fluidity of the material and enabling partial CO_2 sequestration.

(1) Foaming agent

Sodium lauryl sulfate (K12 , $\text{C}_{12}\text{H}_{25}\text{SO}_4\text{Na}$), an anionic surfactant, is used as the foaming agent. It effectively reduces the surface tension of the liquid phase and promotes the generation and dispersion of CO_2 bubbles.

(2) Foam stabilizer

Hydroxypropyl methyl cellulose (HPMC), a non-ionic polymer, is employed as the foam stabilizer. By increasing the viscosity of the liquid phase and strengthening the interfacial film, it prevents bubble coalescence and rupture and improves foam stability.

(3) CO_2 gas

CO_2 gas with different purity levels (20%, 40%, 60%, 80% and 100%) is used. The CO_2 concentration is controlled to adjust the foaming efficiency and the extent of carbonation reactions.

2.4. Alkali Activator

The alkali activator is a blend of sodium hydroxide and sodium silicate solution, which is used to activate the aluminosilicate species in fly ash and slag and promote geopolymer gel formation.

(1) Sodium hydroxide: Analytical-grade solid NaOH with a purity of $\geq 96\%$ is used. It is dissolved in deionized water to prepare solutions of specified concentration, providing a high-alkalinity environment that facilitates the rupture of Si–O and Al–O bonds in the raw materials.

(2) Sodium silicate solution: An industrial-grade water glass ($\text{Na}_2\text{O} \cdot n\text{SiO}_2$) with a modulus of about 2.4 is adopted. Its SiO_2 and Na_2O contents are approximately 29.8% and 13.2%, respectively. When combined with NaOH , the overall alkalinity and silicate species concentration can be tuned, thereby influencing the polymerization rate and the development of the gel structure.

The main raw materials used in this study, including fly ash, GGBS, CO₂ gas cylinders, sodium hydroxide, fluid spoil and its particle-size distribution, are illustrated schematically in Figure 1. The chemical composition of the fly ash and the main parameters of the sodium silicate solution are summarized in Tables 1 and 2.

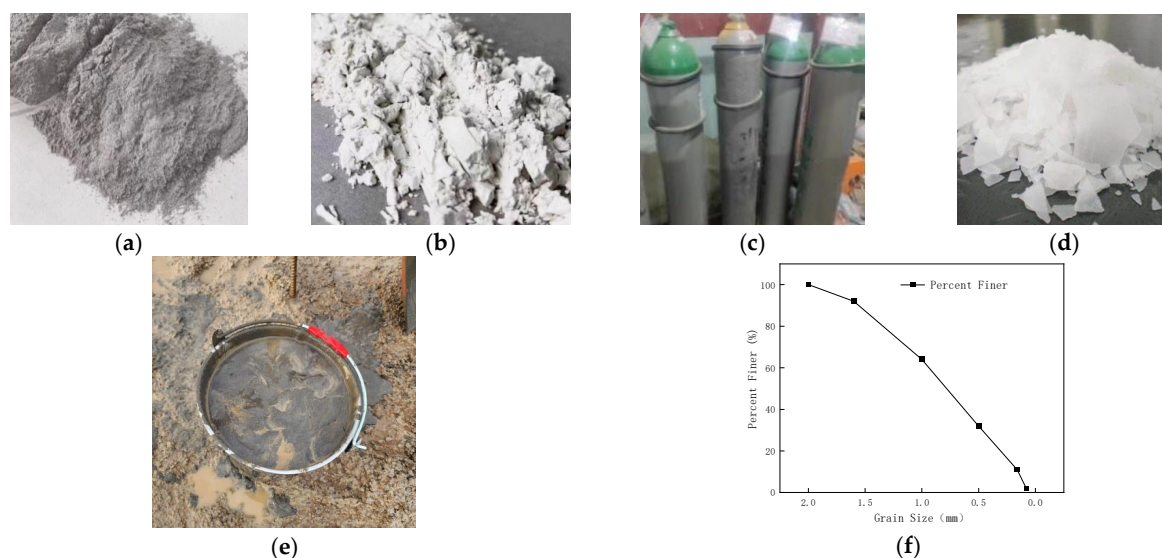


Figure 1. Raw materials. (a) Fly ash. (b) GGBS. (c) CO₂ cylinder. (d) Solid sodium hydroxide. (e) Flowable slag. (f) Soil particle size distribution.

Table 1. Chemical composition of fly ash.

Chemical Composition	SiO ₂	Al ₂ O ₃	MgO	CaO	SO ₃	Fe ₂ O ₃	Other
Content	45.1	36.8	7.74	5.3	1.2	0.85	3.01

Table 2. Parameters of water glass solution.

Type	Content of SiO ₂ /%	Content of Na ₂ O/%	Baumé Degree/(Be/20 °C)	Modulus/M
SP50	29.8	13.2	7.74	2.4

3. Test Plan

3.1. Mix Design of Geopolymer Gel Materials

Although a small amount of cement (50 g) is introduced into the geopolymer system in this study, its primary role is to adjust the setting time and improve early-age stability rather than to act as the main binder. The dominant cementitious phases are still C–A–S–H and N–A–S–H gels generated by alkali-activated slag and fly ash, which are responsible for strength development and microstructure formation. The cement content is much lower than that in conventional cement-based systems and does not compromise the core low-carbon objective of replacing Portland cement with geopolymers [30].

GGBS is a latent hydraulic material that rapidly forms C–A–S–H gels under alkali activation, providing early-age strength. Fly ash primarily participates in secondary pozzolanic reactions, improving the fluidity of the paste and contributing to long-term densification. By adjusting the relative contents of GGBS and fly ash, a balance between early strength and workability can be achieved. In this study, a single-factor experimental program is designed to investigate the influence of the GGBS-to-fly ash mass ratio on the workability and mechanical properties of alkali-activated CO₂ foamed concrete. The masses of the NaOH solution and sodium silicate solution are fixed at 100 g each, the cement dosage is

fixed at 50 g, the CO₂ concentration at 40 vol% and the foam volume fraction at 70%. The GGBS-to-fly ash mass ratio is set to 100:0, 75:25, 50:50, 25:75 and 0:100, corresponding to mix IDs D1–D5. This design aims to clarify the effects of different geopolymer precursor compositions on the reactivity, setting time and strength development of CO₂ foamed concretes. The mix proportions and test results for each group are summarized in Table 3. These results provide the baseline mix for subsequent multi-factor orthogonal experiments and are important for understanding the strength contribution and microstructural mechanisms of different geopolymer compositions.

Table 3. Different geopolymer mixture schemes.

ID	NaOH Solution/g	Water Glass Solution/g	Slag Powder/g	Fly Ash/g	Cement/g	CO ₂ Concentration/vol%	Foam Volume Content/%
D1	100	100	400	0	50	40	70
D2	100	100	300	100	50	40	70
D3	100	100	200	200	50	40	70
D4	100	100	100	300	50	40	70
D5	100	100	0	400	50	40	70

3.2. Mix Design of CO₂ Foamed Concrete

The factor levels in the orthogonal design were determined by combining (a) feasible operational ranges of the foam generator and CO₂ supply on construction sites, (b) preliminary screening tests to ensure foam stability and prevent segregation/bleeding in spoil-rich slurries and (c) literature-reported ranges for alkali-activated binders and foamed concretes. Specifically, the CO₂ concentration was set to 40–100% to cover the transition from insufficient carbonation/foaming driving force to potential over-carbonation and bubble coarsening; the foam volume fraction was set to 50–80% to span workable density and pore connectivity regimes for backfilling; the geopolymer dosage was set to 10–25% (by total solids) to balance binder sufficiency, cost and low-carbon substitution; and the alkali activator dosage was set to 5–20% to cover under-activation to excessive alkalinity/viscosity conditions.

Based on the single-factor results, an orthogonal experimental design is conducted to investigate the combined effects of CO₂ concentration, foam volume fraction, total geopolymer content and alkali activator dosage on the performance of spoil-based alkali-activated CO₂ foamed concrete. Four main control factors are considered: CO₂ concentration (A), foam volume fraction (B), geopolymer dosage (C) and alkali activator dosage (D). Each factor is assigned four levels: CO₂ concentrations of 40%, 60%, 80% and 100%; foam volume fractions of 50%, 60%, 70% and 80%; geopolymer dosages of 10%, 15%, 20% and 25% (by mass of total solids); and alkali activator dosages of 5%, 10%, 15% and 20%. An L16(4⁴) orthogonal array is adopted to systematically design the 16 combinations. The performance indices include 7-day and 28-day compressive strengths, fluidity and setting times. The detailed orthogonal mix design and test matrix are listed in Table 4.

Table 4. Orthogonal experiment scheme table.

ID	CO ₂ Concentration/vol%	Foam Volume Content/%	Geopolymer Content/%	Alkali Activator Content/%
1	40	50	10	5
2	40	60	15	10
3	40	70	20	15

Table 4. Cont.

ID	CO ₂ Concentration/vol%	Foam Volume Content/%	Geopolymer Content/%	Alkali Activator Content/%
4	40	80	25	20
5	60	50	15	15
6	60	60	10	20
7	60	70	25	5
8	60	80	20	10
9	80	50	20	20
10	80	60	25	15
11	80	70	10	10
12	80	80	15	5
13	100	50	25	10
14	100	60	20	5
15	100	70	15	20
16	100	80	10	15

4. Preparation Procedure

4.1. Preparation of Alkali Activator

Solid NaOH (analytical grade, purity $\geq 96\%$) is dissolved in deionized water in a polyethylene beaker to prepare a 10 mol/L concentrated solution. This process is highly exothermic, and the solution temperature can rise above 80 °C. The freshly prepared solution is sealed and allowed to stand for 24 h to cool to room temperature (20 ± 2 °C) and to stabilize its chemical activity, thereby avoiding the adverse effects of local over-concentration or overheating on the subsequent geopolymerization reactions.

After standing, the NaOH solution is blended with an industrial sodium silicate solution (modulus ≈ 2.4 , SiO₂ content 29.8%, Na₂O content 13.2%) at a mass ratio of 1:1. A mechanical stirrer is used to mix the solution at 400 rpm for 5 min until a homogeneous and slightly viscous composite alkali activator is obtained. The calculated final modulus (molar ratio of SiO₂ to Na₂O) of the composite activator is approximately 1.2.

4.2. Preparation of CO₂ Foam

First, K12 and HPMC are dissolved in deionized water to prepare a composite foaming solution with a K12 concentration of 1.5 wt% and an HPMC concentration of 0.2 wt%. The composite solution is then poured into a high-shear mechanical foaming device connected to CO₂ gas sources of different purities (20%, 40%, 60%, 80% and 100%). The gas flow rate is precisely controlled using a mass flow controller. Under an inlet pressure of 0.3 MPa and a shear rate of 1200 rpm, CO₂ foam with a wet density of 50 ± 5 kg/m³ is generated. To ensure stability and foaming efficiency, the prepared foam is used within 5 min.

4.3. Mixing Procedure

A planetary mortar mixer with a capacity of 5 L is used for mixing. The following sequence is strictly followed:

(1) Dry mixing: Pre-treated high-fluidity spoil (fixed mass of 400 g, moisture content $\geq 30\%$), GGBS, fly ash and 50 g of P.O 42.5 cement are added to the mixing bowl. The mixture is dry-mixed at a low speed (140 ± 5 rpm) for 60 s to ensure a preliminary homogeneity of the solid particles.

(2) Wet mixing with alkali activator: The pre-prepared composite alkali activator is then poured into the mixer. The mixture is stirred at a low speed (140 ± 5 rpm) for 120 s to disperse the viscous activator uniformly. The remaining mixing water is subsequently added, and the mixer speed is increased to high speed (285 ± 10 rpm) for 180 s to

fully activate the solid precursors and obtain a homogeneous geopolymer slurry with an adequate fluidity.

(3) Incorporation of CO₂ foam: The pre-measured volume of CO₂ foam is gently placed on top of the slurry. The mixer is restarted at a low speed (140 ± 5 rpm) and operated for a further 120 s to carefully fold the foam into the slurry. The final mixture is a uniform, creamy and highly flowable CO₂ foamed concrete without visible bleeding or large voids.

4.4. Casting and Curing

The fresh foamed mixture is cast into 40 mm × 40 mm × 160 mm three-compartment prismatic molds in a single layer and slightly overfilled to ensure complete filling. The molds are placed on a vibrating table operating at 50 Hz and vibrated for 15 s to remove large entrapped air bubbles and improve compaction. After vibration, the mold surfaces are immediately covered with plastic film to prevent moisture loss, and the specimens are stored at 20 ± 2 °C for 24 h. The specimens are then demolded, and the top surfaces are ground flat.

After demolding, the specimens are wrapped with plastic film and transferred to a standard curing chamber with a temperature of 20 ± 2 °C and relative humidity of at least 95%. They are cured to the target ages of 7 d and 28 d for subsequent macro-performance tests and microstructural analyses. The overall preparation procedure is illustrated schematically in Figures 2 and S1.

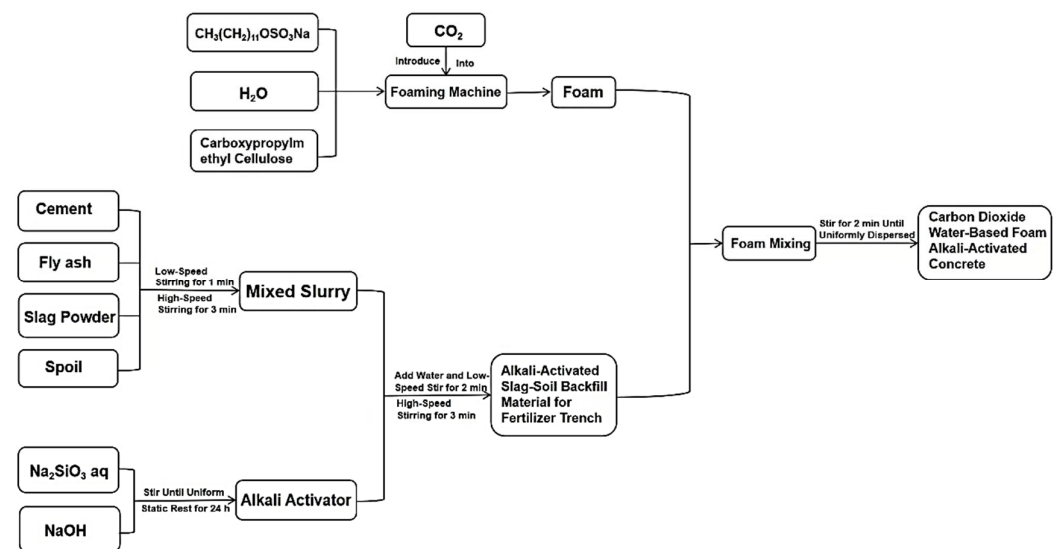


Figure 2. Preparation process flowchart.

5. Test Methods

5.1. Fluidity

The fluidity of the fresh mixtures is measured in accordance with the Chinese standard GB/T 2419-2022 “Test method for fluidity of cement mortar” [31], using a flow table. A clean, moistened truncated cone mold is placed at the center of the flow table and fixed. The mold is filled with fresh mixture in two layers; each layer is tamped 15 times with a tamping rod to ensure compaction and to expel trapped air. Excess material is then removed by drawing a straightedge across the top surface of the mold. The mold is immediately lifted vertically and smoothly. The table is dropped 25 times at a frequency of one drop per second. After the mixture has stopped flowing, the spread diameter is measured in two orthogonal directions with a Vernier caliper and the average value is taken as the fluidity of the mixture.

5.2. Compressive Strength

Compressive strength was determined in accordance with GB/T 17671-2021 using $40 \times 40 \times 160$ mm prismatic specimens. The loaded area for compression was 40×40 mm ($A = 1600 \text{ mm}^2$). A servo-controlled testing machine was used under [force-control] at a loading rate compliant with the standard (approximately 2.4 kN/s, equivalent to $\sim 1.5 \text{ MPa/s}$ for $A = 1600 \text{ mm}^2$) until failure. For each mixture, three specimens were tested and the results are reported as means $\pm$ standard deviation. Any outlier beyond $\pm 10\%$ of the mean was re-tested. The compressive strength f is calculated as follows:

$$f = F/A$$

where f is the compressive strength (Pa), F is the failure load (N) and A is the loaded area of the specimen ($40 \text{ mm} \times 40 \text{ mm}$).

5.3. Setting Time

The setting time of the mixtures is determined using a Vicat apparatus in accordance with the Chinese standard GB/T 1346-2022 “Test methods for water requirement of normal consistency, setting time and soundness of the Portland cements” [32]. After mixing, the paste is placed into the Vicat mold. Following compaction and levelling, the mold is immediately transferred to a standard curing box. The initial setting time is measured using the standard Vicat needle (mass 300 ± 1 g, needle diameter 1.13 ± 0.05 mm). Measurements start 30 min after the beginning of mixing and are repeated every 5 min. The time at which the needle penetrates the paste and stops at 4 ± 1 mm above the bottom plate is recorded as the initial setting time. For the final setting time, the mold is inverted and the final setting attachment (with an annular ring) is used. Measurements are taken every 15 min. The time at which only a shallow ring mark is left on the paste surface without penetration (i.e., indentation diameter of approximately 20 mm) is recorded as the final setting time.

5.4. SEM

To investigate the microstructural evolution mechanism, representative specimens with the optimum mix proportions (CO_2 concentration 60%, foam volume fraction 80%, geopolymer content 20%, alkali activator dosage 10%) are selected after 28 d of standard curing for SEM observations. Small blocks with dimensions of approximately $10 \text{ mm} \times 10 \text{ mm} \times 5 \text{ mm}$ are cut from the core region of the specimens using a precision saw. To terminate further hydration, the samples are immediately immersed in absolute ethanol for 7 d in sealed containers. They are then dried in a vacuum oven at 60 ± 5 °C to a constant mass. Fresh fracture surfaces are sputter-coated with a thin gold film to ensure electrical conductivity. SEM observations are carried out at appropriate accelerating voltages and working distances to examine the micro-morphology, pore structure, ITZ and distribution of the hydration and carbonation products.

The instruments used are shown in Figure S2.

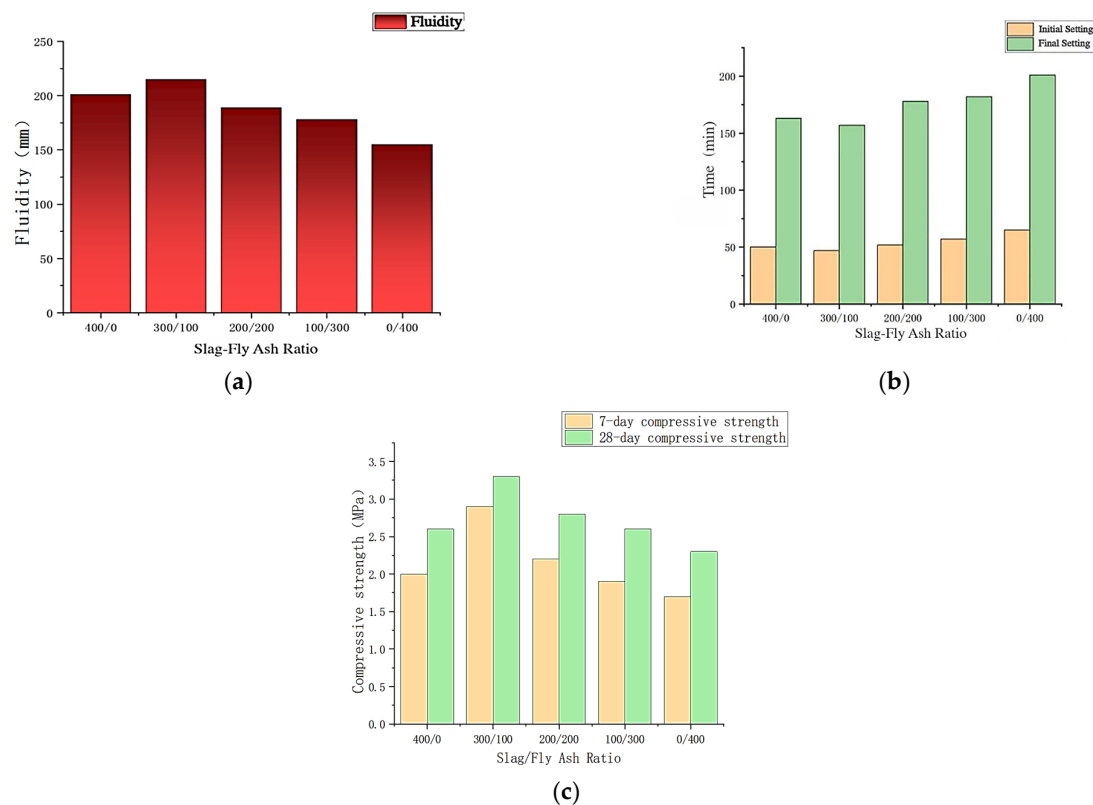
6. Results

6.1. Effect of Geopolymer Composition

To clarify the isolated influence of geopolymer precursor composition on material performance, other process parameters are kept constant and the effects of the GGBS-to-fly ash mass ratio on the macro-properties of alkali-activated CO_2 foamed concrete are systematically investigated. The experimental results are summarized in Table 5 and illustrated schematically in Figure 3.

Table 5. Experimental results of geopolymers with different proportions.

ID	Fluidity/mm	Initial Setting Time/min	Final Setting Time/min	7 d Compressive Strength/MPa	28 d Compressive Strength/MPa
D1	201	50	163	2.0	2.6
D2	215	47	157	2.9	3.3
D3	189	52	178	2.2	2.8
D4	178	57	182	1.9	2.6
D5	155	65	201	1.7	2.3

**Figure 3.** Changes in various factors under different geopolymer ratios. (a) Fluidity. (b) Setting time. (c) Compressive strength.

(1) Effect on compressive strength: As the fly ash content increases from 0% to 100%, both 7 d and 28 d compressive strengths first increase significantly and then gradually decrease. When the GGBS-to-fly ash mass ratio is 3:1 (mix D2), the strengths reach their maxima: 2.9 MPa at 7 d and 3.3 MPa at 28 d. Compared with the pure slag system (D1), the 7 d and 28 d strengths increase by 45% and 27%, respectively; relative to the pure fly ash system (D5), the increases are 71% and 43%. This indicates a pronounced synergistic strengthening effect when slag and fly ash are combined at an appropriate ratio (3:1).

The underlying mechanism is that a moderate amount of fly ash (25%) can be activated in the alkaline environment to produce N–A–S–H gels, which interweave with the C–A–S–H gels formed predominantly from slag, optimizing both the composition and spatial configuration of the gel products and thus enhancing strength. However, when the fly ash content exceeds 50%, the supply of Ca^{2+} ions from slag becomes insufficient, leading to a reduced rate and amount of C–A–S–H formation, which is crucial for early strength. Meanwhile, the reaction of fly ash is relatively slow and cannot compensate for the loss of strength in time. As a result, the overall matrix compactness is reduced and the strength decreases.

(2) Effect on fluidity: The fluidity of the fresh mixtures exhibits a non-linear decreasing trend with increasing fly ash content. When the fly ash content is 25% (D2), the best fluidity of 215 mm is obtained, which is slightly higher than that of the pure slag system (201 mm, D1), representing an increase of about 7%. However, when the fly ash content is further increased to 50% and above, the fluidity decreases markedly, from 215 mm (D2) to 155 mm (D5), a reduction of 28%.

This behavior can be attributed to a combination of effects. At the optimum ratio, the ball-bearing effect of the spherical fly ash particles dominates, reducing the internal friction between particles and improving rheology. When the fly ash content becomes too high, its large specific surface area leads to a greater adsorption of free water, and the micro-filler effect significantly increases the plastic viscosity of the paste. These detrimental effects outweigh the beneficial ball-bearing effect, resulting in a pronounced loss of fluidity.

(3) Effect on setting time: The test data clearly show that fly ash exhibits a marked retarding effect. As its content increases from 0% to 100%, the initial setting time is prolonged from 50 min (D1) to 65 min (D5), and the final setting time from 163 min to 201 min, corresponding to increases of 30% and 23%, respectively. Overall, increasing the fly ash content tends to prolong both the initial and final setting times; however, a slight reduction in the initial setting time is observed at 25% fly ash (D2), which may be associated with particle packing/nucleation effects, followed by a pronounced retardation at higher fly ash contents due to a reduced early-age reactivity.

The main reason lies in the difference in chemical reactivity between slag and fly ash. Slag possesses a latent hydraulic reactivity and can rapidly dissolve and form C–A–S–H gels under alkali activation, playing a dominant role in early setting. Fly ash, however, is a pozzolanic material with a more stable glassy structure; its dissolution and depolymerization rate in an alkaline solution is much slower than that of slag, and the formation of N–A–S–H gels is also sluggish. Therefore, increasing the fly ash content inevitably slows down the overall geopolymerization process and extends the setting time.

In summary, the mix D2 with a GGBS-to-fly ash mass ratio of 3:1 achieves an optimal balance between strength, workability and setting time. It offers the highest compressive strength while maintaining a favorable fluidity and acceptable setting behavior, and is therefore adopted as the baseline geopolymer composition for the subsequent orthogonal tests.

6.2. Effect of Orthogonal Mix Parameters

Building on the single-factor experiments, an L16(4⁴) orthogonal design is used to clarify the combined influences of CO₂ concentration (A), foam volume fraction (B), total geopolymer content (C) and alkali activator dosage (D) and to identify the optimum mix combination. The test results are summarized in Table 6, and the range analyses are presented in Tables 7–11 and Figures 4–9. The color of the line between points represents the concentration of CO₂, and the three axes of the three-dimensional spatial coordinate system represent the volume of foam, the total amount of geopolymer and the amount of alkali activator, respectively. The color of the points represents the experimental results.

Table 6. Results of the orthogonal experiment.

ID	Fluidity /mm	Initial Setting Time /min	Final Setting Time /min	7 d Compressive Strength/MPa	28 d Compressive Strength/MPa
1	211	41	112	1.7	2.1
2	207	42	123	2.3	2.8
3	204	49	147	2.4	2.9
4	195	54	160	2.5	3.1
5	221	62	192	2.3	2.8

Table 6. Cont.

ID	Fluidity /mm	Initial Setting Time /min	Final Setting Time /min	7 d Compressive Strength/MPa	28 d Compressive Strength/MPa
6	230	70	214	2.1	2.7
7	212	53	156	2.8	3.2
8	220	47	147	2.9	3.3
9	225	45	137	2.7	3.0
10	221	43	129	2.7	3.1
11	210	73	249	2.2	2.6
12	186	76	267	2.3	2.7
13	212	48	148	2.4	2.9
14	201	53	165	1.6	2.2
15	208	80	289	1.8	2.4
16	207	74	261	1.5	1.9

Table 7. Range analysis table of fluidity.

Factors	k1	k2	k3	k4	R
CO ₂ concentration	204.25	220.75	210.5	207	16.5
Foam volume content	217.25	214.75	208.5	202	15.25
Geopolymer content	214.5	205.5	212.5	210	9
Alkali activator content	202.5	212.25	213.25	214.5	12

Table 8. Range analysis table of 7 d compressive strength.

Factors	k1	k2	k3	k4	R
CO ₂ concentration	2.225	2.525	2.475	1.825	0.7
Foam volume content	2.275	2.175	2.3	2.3	0.125
Geopolymer content	1.875	2.175	2.4	2.6	0.725
Alkali activator content	2.1	2.45	2.225	2.275	0.35

Table 9. Range analysis table of 28 d compressive strength.

Factors	k1	k2	k3	k4	R
CO ₂ concentration	2.725	3	2.85	2.35	0.65
Foam volume content	2.7	2.7	2.775	2.75	0.075
Geopolymer content	2.325	2.675	2.85	3.075	0.75
Alkali activator content	2.55	2.9	2.675	2.8	0.35

Table 10. Range analysis of table initial setting time.

Factors	k1	k2	k3	k4	R
CO ₂ concentration	46.5	58	59.25	63.75	17.25
Foam volume content	49	52	63.75	62.75	14.75
Geopolymer content	64.5	65	48.5	49.5	16.5
Alkali activator content	55.75	52.5	57	62.25	9.75

Table 11. Range analysis table of final setting time.

Factors	k1	k2	k3	k4	R
CO ₂ concentration	135.5	177.25	195.5	215.75	80.25
Foam volume content	147.25	157.75	210.25	208.75	63
Geopolymer content	209	217.75	149	148.25	69.5
Alkali activator content	175	166.75	182.25	200	33.25

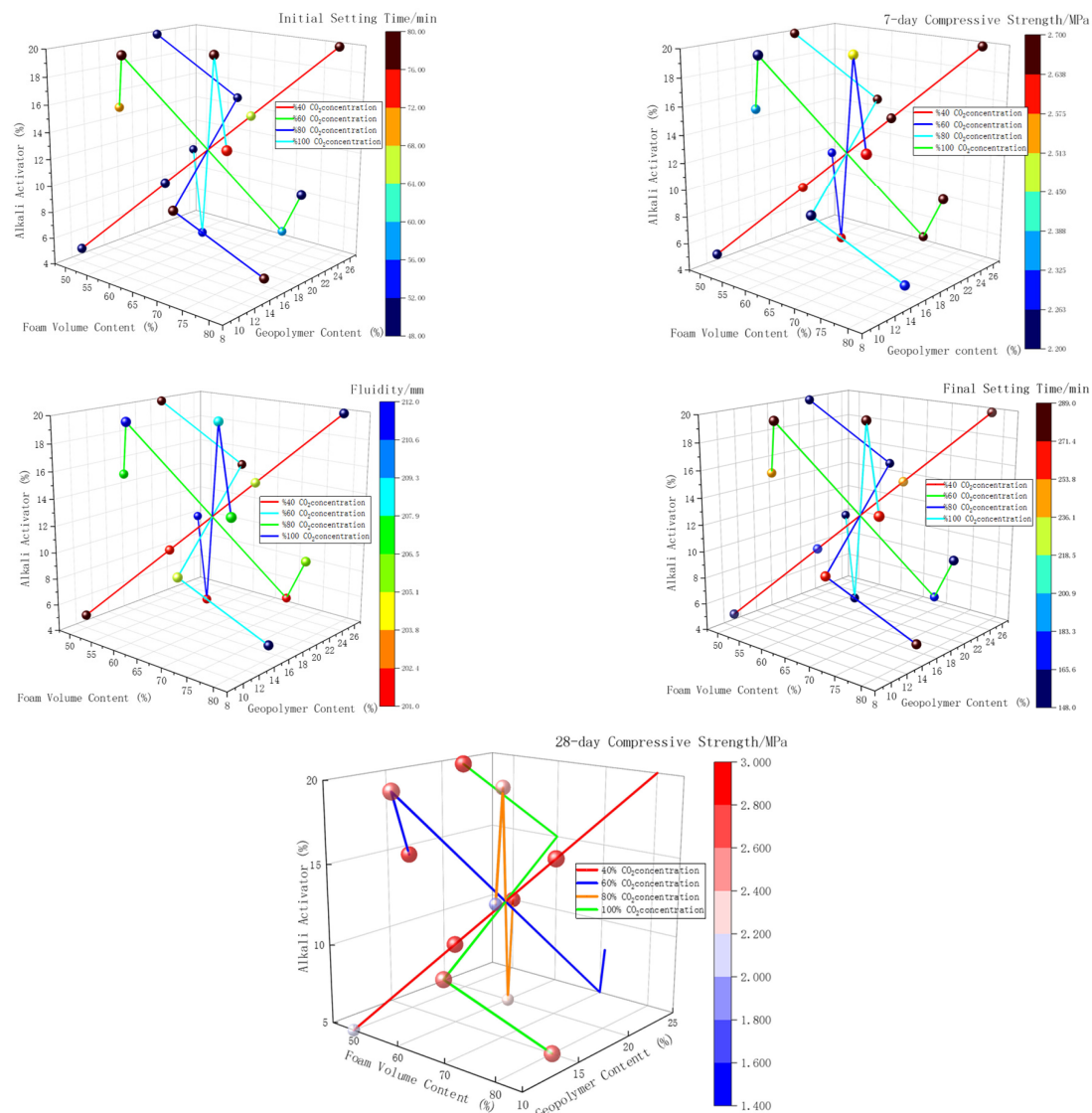


Figure 4. Three-dimensional visualization of the orthogonal experimental results.

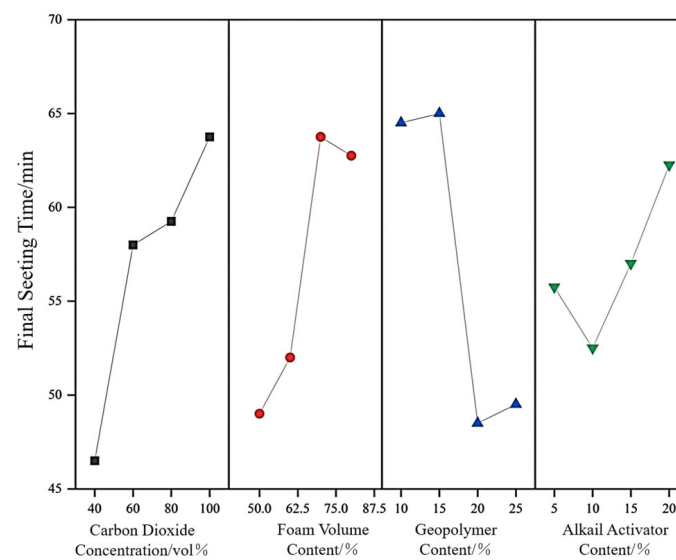


Figure 5. Range analysis of fluidity.

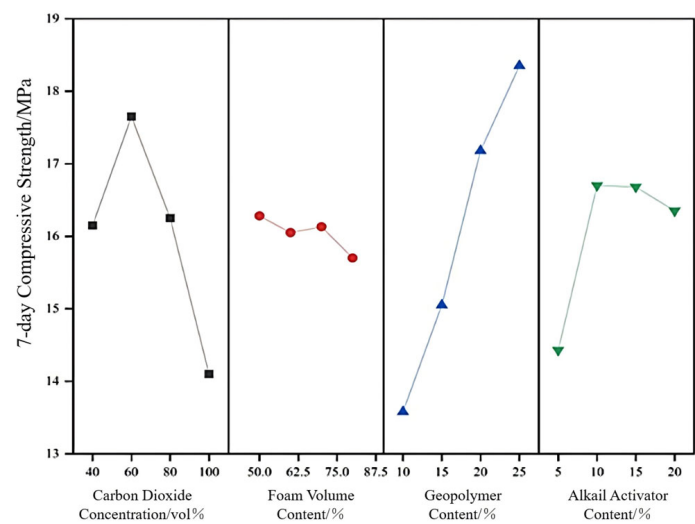


Figure 6. Range analysis of 7 d compressive strength range.

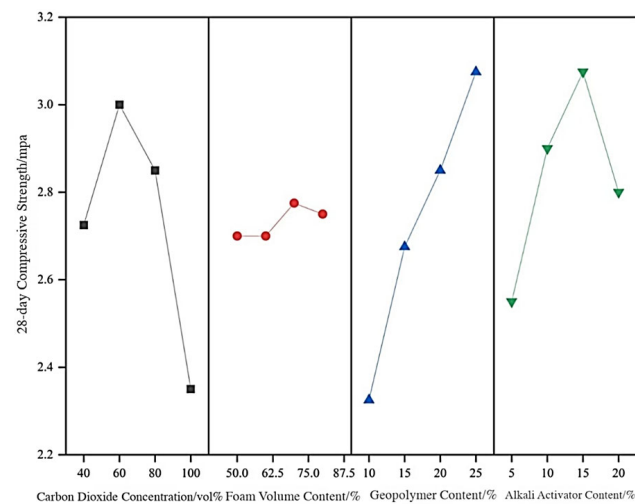


Figure 7. Range analysis of 28-day compressive strength.

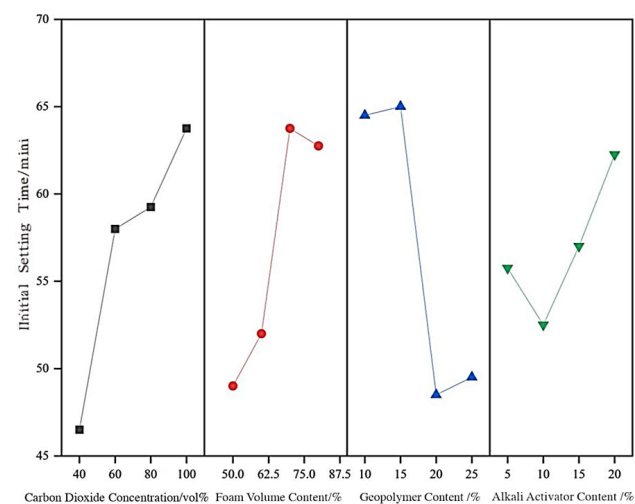


Figure 8. Range analysis of initial setting time.

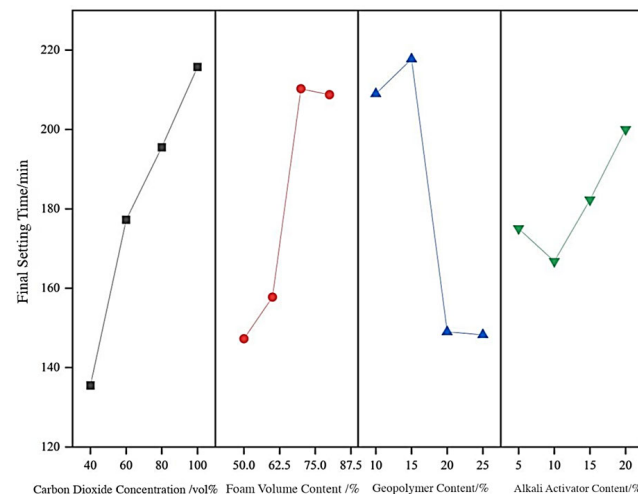


Figure 9. Range analysis of final setting time.

(1) Factor significance and influence patterns

1. Fluidity: According to the range analysis, the factors affecting fluidity can be ranked as $A (R = 16.5) > B (R = 15.25) > D (R = 12) > C (R = 9)$. This indicates that gas- and liquid-phase characteristics are the primary determinants of fluidity. A moderate CO_2 concentration of 60% ($k_2 = 220.75$ mm) promotes the formation of fine and stable bubbles, which provide a ball-bearing effect and significantly improve fluidity. The foam volume shows an optimum range (50–60%): when the foam content exceeds 70%, the excessive porosity weakens the paste skeleton and increases viscosity, leading to a decreased fluidity.

2. Compressive strength: For both 7 d and 28 d strengths, the factor ranking is $C > A > D > B$. Geopolymer dosage is the dominant parameter. When the geopolymer content is 20–25% ($k_3 = 2.4$ MPa; $k_4 = 2.6$ MPa for 7 d strength), a continuous and dense gel skeleton forms, providing the main structural strength. CO_2 concentration influences strength via carbonation: at 60% CO_2 ($k_2 = 2.525$ MPa at 7 d), carbonate microcrystals precipitate and fill pores, enhancing the strength. However, an excessively high CO_2 concentration (100%) coarsens the bubble structure and damages the skeleton, reducing the 7 d strength to 1.825 MPa.

3. Setting time: For both the initial and final setting times, the factor ranking is $A > C > B > D$. When the CO_2 concentration exceeds 80%, rapid carbonation consumes OH^- and significantly lowers the system alkalinity, thus slowing down the geopolymerization process and prolonging setting, with the longest final setting time reaching 215.75 min (k_4). An increase in the proportion of fly ash in the geopolymer also retards setting due to its lower reactivity.

(2) Interaction and optimum mix confirmation: A strong synergistic effect is observed between CO_2 concentration and geopolymer dosage. For example, in group 8 ($A_2B_3C_3D_2$), a moderate CO_2 concentration of 60% not only generates a uniform pore structure through physical foaming, but also triggers moderate carbonation, which cooperates with the 20% geopolymer to densify the matrix. As a result, a favorable balance of mechanical performance and workability is achieved, with an 28 d strength of 3.3 MPa and a fluidity of 220 mm.

Considering fluidity, strength and setting time comprehensively, the optimum mix combination is determined to be a CO_2 concentration of 60%, a foam volume fraction of 80%, a geopolymer content of 20% and an alkali activator dosage of 10%. This combination, corresponding to one of the orthogonal groups, exhibits the best overall performance and is therefore chosen for microstructural and mechanism studies.

Although formal inferential statistics (e.g., ANOVA or hypothesis testing) are not performed in this work, the repeatability and uncertainty of the experimental results were controlled and qualitatively evaluated to ensure the reliability of the observed trends. For each mixture, key fresh and hardened properties (fluidity, setting time and compressive strength) were measured using at least three replicate specimens/batches under the same curing and testing conditions, and the reported values represent the averaged results. The main sources of experimental variability are associated with (i) the inherent heterogeneity of pipe-jacking spoil, (ii) foam generation stability (bubble size distribution and drainage) and (iii) specimen preparation/compaction and end-surface conditions during compression testing. To minimize these effects, the spoil was homogenized prior to mixing, foam generation parameters were kept constant and the specimen preparation and curing procedures were strictly standardized for all groups. Importantly, the ranking of factor effects obtained from the L16(4⁴) orthogonal design is based on the consistent trend differences between factor levels across multiple groups rather than a single measurement, which improves the robustness for engineering-oriented parameter screening. Therefore, while the present orthogonal analysis primarily provides a practical factor ranking and an optimum window within the tested ranges, further work will incorporate more rigorous statistical inference and expanded repetitions to quantify significance and confidence intervals."

7. Discussion

7.1. Microstructural Evolution and Synergistic Mechanism

To elucidate the multi-scale role of CO₂ in alkali-activated spoil-based foamed concrete, SEM observations (Figure 10) and Image-Pro Plus analyses are carried out on specimens with the optimum mix after 28 d of curing. Combined with the pore size distribution (Figures 11 and 12) and microstructural data (Tables 12 and 13), the microstructural origin of the macroscopic performance is clarified. The histogram uses the maximum Feret diameter, whereas the average value in Table 13 refers to the equivalent circular diameter calculated from pore area.

The SEM images show that the internal structure of the specimens is dominated by uniformly distributed closed pores. Quantitative analysis using Image-Pro Plus reveals a porosity of about 23.50%, with most pores in the size range of 100–500 µm. The 200–300 µm interval has the highest frequency, accounting for approximately 42% of the pores. The pore area ranges from 2938.55 to 57,355.52 µm², with an average of 16,352.98 µm², and the average pore diameter is about 85.06 µm. This moderate porosity and pore size distribution indicate that CO₂ physical foaming has produced a stable three-dimensional bubble network. The uniform fine pores reduce internal friction and provide a pronounced ball-bearing effect, which explains the high fluidity (>210 mm) observed in the macroscopic tests.

At higher magnifications, C–A–S–H and N–A–S–H gels are observed to interweave and form a continuous network skeleton. Abundant nano- to micro-scale carbonate crystals are distributed within the gel network and along the pore walls. These crystals, generated by CO₂ carbonation, coexist intimately with the geopolymer gels, forming a "gel-carbonate" composite matrix. The fine carbonate crystals act as micro-fillers, effectively filling gel pores and ITZ defects and greatly improving matrix compactness. This is the fundamental reason why the optimum mix achieves a 28 d compressive strength of 3.3 MPa despite the relatively high porosity.

In particular, the SEM images show that originally loose spoil particles with smooth surfaces are completely wrapped and firmly bonded by the reaction products. This "spoil particle immobilization" transforms otherwise inert waste particles into effective reinforcing inclusions and eliminates potential weak interfaces. As a result, the interfacial structure of

the composite is optimized, and the macroscopic mechanical performance becomes more uniform and stable.

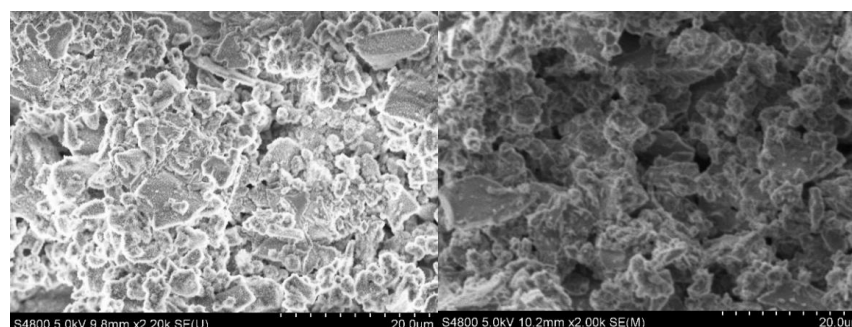


Figure 10. SEM microscopic morphology image.

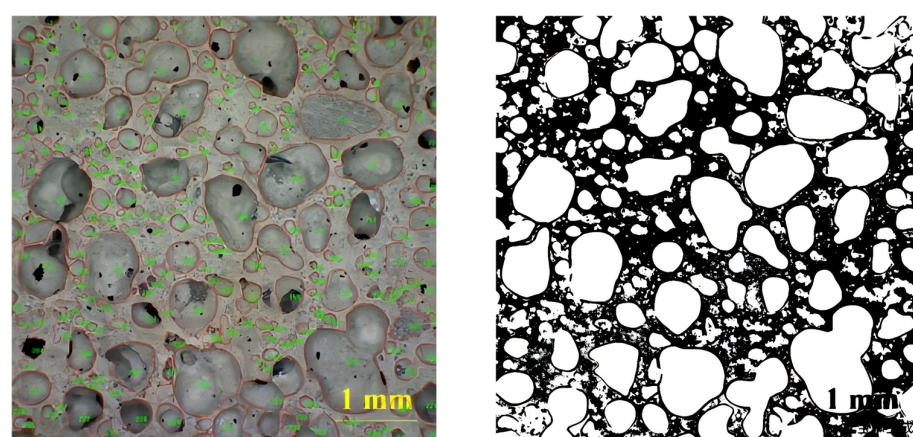


Figure 11. Image-Pro Plus microscopic analysis diagram.

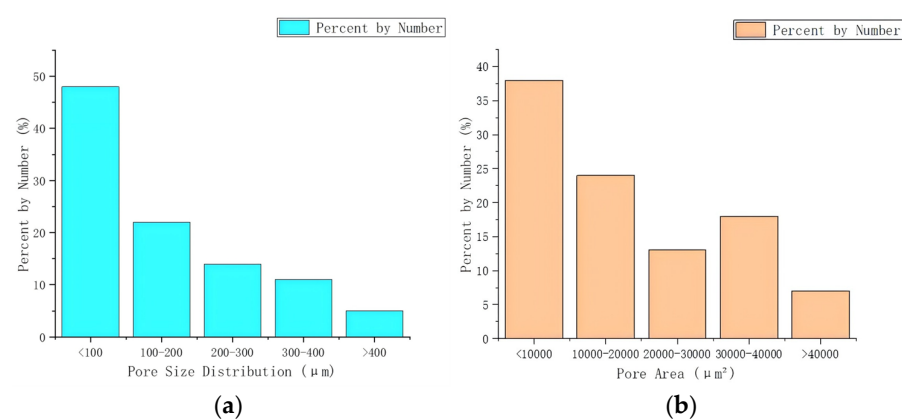


Figure 12. Quantitative statistics chart. (a) Pore size distribution. (b) Pore area distribution.

Table 12. Microscopic data analysis table.

Statistical Items	Pore Area/ μm^2	Maximum Diameter/ μm	Minimum Diameter/ μm	Pore Diameter/ μm	Circumference/ μm	Roundness
Minimum Value	2938.55	46.07	11.22	30.65	141.23	1
Maximum Value	57,355.52	649.65	246.50	487.14	1851.94	9.97
Range	51,016.96	606.57	225.28	450.48	1610.70	8.97

Table 13. Analysis of the mean values of microscopic data.

Statistical Items	Pore Area/ μm^2	Porosity/%	Average Pore Diameter/ μm	Average Perimeter/ μm	Average Roundness
Average Value	16,352.98	23.50%	85.06	879.55	1.43

Overall, CO_2 contributes to performance enhancement through multiple coupled pathways. Physical foaming creates a uniform pore structure that ensures a high fluidity; chemical carbonation generates nano-scale carbonates that work synergistically with geopolymer gels to construct a dense matrix; and the alkali-activated environment fixes spoil particles within the gel network. These coupled processes collectively form a stable “gas–solid–soil” composite structure, which is the core mechanism enabling the high performance and resource utilization in the proposed material.

The proposed multi-scale mechanism is herein framed as an evidence-linked interpretation anchored in the SEM observations and the macroscopic trends reported in Section 6.1. First, mixtures with a higher foam volume fraction exhibit a lower density and reduced compressive strength, which is consistent with the SEM-observed increase in pore population and the reduced continuity/thickness of the solid skeleton and pore walls; conversely, mixes showing more uniform and predominantly closed pores with continuous pore walls correspond to an improved strength retention at a comparable workability. Second, increasing the CO_2 concentration promotes the formation of fine carbonate precipitation and the partial infilling/lining of pore walls and inter-particle regions observed in SEM, which provides a microstructural basis for the observed enhancement in early-age stability and/or strength at comparable foam contents by densifying the matrix and reducing defects. Third, an increased geopolymer/activator dosage leads to more continuous gel products and a stronger bonding/encapsulation around spoil particles in SEM, which explains the improved setting stability and strength through particle immobilization and a more integrated load-bearing framework. Taken together, the SEM features (pore wall continuity, degree of infilling/densification and gel continuity at interfaces) provide a consistent microstructural rationale for the measured trade-off between flowability, setting behavior and strength across the tested parameter ranges, thereby strengthening the microstructure–property linkage.

7.2. Composition–Structure–Property Relationship Model

Based on the macro-performance tests and microstructural analyses, a composition–structure–property relationship model is proposed for alkali-activated high-fluidity spoil CO_2 foamed concrete, illustrating how key components govern microstructural formation and thereby determine macroscopic properties.

(1) Composition–structure relationships: GGBS and fly ash act as the core cementitious precursors. Their dosages and ratio directly control the quantity and type of gels formed. GGBS supplies Ca^{2+} and promotes C–A–S–H formation, dominating early strength, while fly ash mainly produces N–A–S–H and refines the structure at later ages. The CO_2 concentration and foam volume fraction, together with the foaming system, determine the porosity and pore size distribution through physical foaming and the amount and distribution of carbonates through chemical carbonation. The alkali activator dosage and modulus regulate the reaction kinetics and the rate of gel network formation, thereby controlling the development of matrix compactness and setting behavior.

(2) Structure–property relationships: The continuous geopolymer gel network and carbonate-filled ITZs constitute the load-bearing skeleton and directly determine the compressive strength. Uniform fine pores reduce internal friction and improve fluidity, while a closed-pore structure helps maintain durability. The rate of gel formation and carbonation,

together with ion transport, affects the evolution of the internal chemical environment and thus controls the setting process. Therefore, strength, fluidity and setting time are unified responses of the multi-scale microstructure.

(3) Synergistic optimization mechanism: The optimal performance is achieved when the contributions of all components reach a synergistic balance. Through rational mix design, the timing and spatial distribution of CO₂ physical foaming, chemical carbonation and geopolymer gel formation are coordinated, leading to a stable multi-scale structure composed of gas (closed pores), solid (dense gels and carbonates) and soil (immobilized spoil particles). The experimentally determined optimum window—a CO₂ concentration of 60–80%, foam volume fraction of 70–80%, geopolymer content of about 20% and alkali activator dosage of about 10%—is a manifestation of this synergy and yields balanced mechanical performance, workability and setting characteristics.

8. Conclusions

Focusing on the challenges of treating spoil from pipe-jacking in water-rich sandy strata—especially the difficulty of disposal and low resource utilization—this study proposes a novel collaborative treatment method based on alkali activation and CO₂ foaming and successfully prepares an alkali-activated high-fluidity spoil CO₂ foamed concrete with tunable properties. The main conclusions are as follows:

(1) A feasible technical route for the co-treatment and performance control of fluid spoil is established. Industrial solid wastes (GGBS and fly ash) are used as geopolymer precursors to build a gel skeleton via alkali activation, while CO₂ water-based foam is introduced for collaborative modification. The proposed route enables in situ resource utilization and the efficient solidification of high-moisture, complex-composition fluid spoil, providing a concrete technical scheme for “treating waste with waste” and transforming spoil into useful products.

(2) The influence laws of key mix parameters on macro-properties are clarified and an optimal mix window is identified. Single-factor and orthogonal tests show that geopolymer content is the primary factor controlling the compressive strength; the CO₂ concentration and foam volume fraction mainly govern fluidity, and both the alkali activator dosage and CO₂ concentration jointly affect the setting time. The optimum performance is achieved with a CO₂ concentration of 60–80%, foam volume fraction of 70–80%, total geopolymer content of about 20% and alkali activator dosage of about 10%, under which the material exhibits high fluidity (>210 mm), moderate strength (28 d strength > 3.0 MPa) and reasonable setting times.

(3) From a microstructural perspective, a multi-scale synergistic mechanism of CO₂ “physical foaming–chemical carbonation–spoil particle immobilization” is revealed. SEM observations show that CO₂ physical foaming produces uniform closed pores that enhance fluidity, while CO₂ carbonation generates nano-scale carbonate crystals that fill gel pores and ITZ defects, improving matrix compactness. Meanwhile, alkali-activated gels encapsulate and fix spoil particles, eliminating weak interfaces and forming a stable “gas–solid–soil” composite structure.

(4) A composition–structure–property relationship model is established and verified. The macroscopic properties of the material are shown to be direct manifestations of its microstructure, which in turn is governed by the geopolymer composition, CO₂ foam system and alkali activator through coupled reactions. The optimum mix essentially corresponds to a spatiotemporal matching point at which physical foaming, chemical carbonation and geopolymerization are synchronously coordinated. This work not only develops a high-performance, low-carbon foamed concrete, but also provides theoretical

and technical support for the high-value, low-cost resource utilization of fluid spoil in applications such as foundation backfilling and trench filling.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/buildings16071396/s1>, Figure S1: Preparation process; Figure S2: Test instruments.

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