

Article

Sustainable Grouting Material from Industrial Waste: Multi-Performance Optimization via the Entropy-Weighted Taguchi Method and Its Environmental Implications

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Highlights

What are the main findings?

- Entropy-weighted Taguchi–GRA obtains optimal waste grout with low viscosity and high strength.
- Rice husk ash exerts pore-filling and pozzolanic effects to densify the grout matrix and improve durability.
- The composite grout reduces CO₂ by 36.3% and raw cost by 17.9%, with safe heavy metal leaching.

What are the implications of the main findings?

- Develops anti-clogging eco-grout for micro-fracture rock emergency stabilization engineering.
- Offers a solid waste valorization route for low-carbon circular geotechnical infrastructure.
- Provides a universal multi-objective optimization method for modified cement grouting materials.

Abstract

This work develops a sustainable, low-viscosity grout material by incorporating industrial byproducts (rice husk ash, RIA; and fly ash, FLA) to address key challenges in rock stabilization: clogging susceptibility, high cost, poor environmental performance, and unbalanced engineering properties. Through single-factor experiments and an entropy-weighted Taguchi–grey relational analysis, the optimal mix ratio was determined to be 10% RIA, 10% FLA, and 0.45% polycarboxylate superplasticizer (POS), and a liquid-to-solid ratio of 1.05. Improved workability: The viscosity of the slurry significantly decreases, allowing its injection into rock fissures as small as micrometers. The slurry setting time and strength meet the requirements for the emergency repair of engineering rock masses. Cost efficiency: Utilizing waste materials reduces production costs by 17.9% per ton. Environmental benefits: CO₂ emissions decrease by 36.3% (150.62 g/kg vs. 237 g/kg for conventional grout), whereas leaching tests confirm that heavy metal concentrations



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(As < 0.1 ppm, Pb < 0.5 ppm) comply with environmental standards. Microstructural analysis reveals that RIA enhances density through pore-filling effects and pozzolanic activity. This study provides a practical, eco-friendly solution for rapid rock stabilization, aligns with circular economy principles, and supports sustainable infrastructure development.

Keywords: industrial waste valorization; low-carbon grouting materials; recycled carbon fiber reinforcement; multi-scale performance evaluation; mine roadway stabilization

1. Introduction

Research on grout material modification has been extensively explored. In the field of alternative cementitious materials, Agwa et al. [1] incorporated fly ash and silica fume to adjust grout fluidity and mechanical properties, whereas Kremieniewski [2] utilized microspheres to stabilize rheological parameters, demonstrating improved adaptability under wellbore conditions. Chen et al. [3] applied incinerated sewage sludge ash and ground granulated blast furnace slag to enhance mechanical performance. Pietrak et al. [4] investigated magnesium-based binders, revealing hygroscopic properties comparable to those of gypsum boards. Xu et al. [5] analyzed fiber–cement particle interactions and derived a revised water–film thickness model to elucidate flow mechanisms. Patrick et al. [6] confirmed the feasibility of the use of natural pozzolans (kaolin/pumice) in geopolymer cement, noting increased chemical stability over time. Gao et al. [7] introduced industrial graphene oxide to refine the cement microstructure, enhancing the pore-filling effects.

Nanoscale additives have also been widely studied. Li et al. [8] employed nanoboron carbide as a rheology modifier and characterized its mechanism via microtests. Wu et al. [9] reported improved injectability with colloidal nanosilica. Singh et al. [10] achieved interfacial transition zone densification via a sequential triple treatment of recycled aggregates. Wang et al. [11] showed that nano- CaCO_3 and nano- Al_2O_3 enhance geopolymer strength and microstructural densification. Xu et al. [12] enhanced grout resistivity via nanocarbon black, whereas Liu et al. [13] developed nanoclay composites to optimize stability and diffusivity. Wu et al. [14] correlated nano- Al_2O_3 additives with a 76% greater influence on strength than curing time. Shahzar et al. [15] reported shear-thickening behavior and compacted matrices with nanoasphalt. Chen et al. [16] and Wu et al. [17] utilized nano- TiO_2 and nano- $\text{Y}_2\text{O}_3\text{-ZrO}_2$ for catalytic wastewater treatment and dispersion enhancement, respectively.

1.1. Persisting Challenges and Our Approach

Despite advancements, critical limitations remain:

Challenge 1: Clogging: High viscosity and particle agglomeration restrict penetration into fine rock fractures (<0.5 mm).

Challenge 2: Cost: Conventional cement and nanoscale additives incur prohibitive expenses at scale.

Challenge 3: Sustainability: Cement production results in excessive CO_2 , while nano-material toxicity risks are overlooked [18].

Challenge 4: Performance trade-offs: Single-parameter optimizations often compromise overall grout functionality [19].

1.2. To Address These Issues Holistically, the Contributions Made by This Research Institute

To address these issues holistically, this study leverages industrial wastes (RIA/FLA) to reduce costs (Challenge 2) and emissions (Challenge 3); develops a low-viscosity formula to prevent clogging (Challenge 1); and integrates entropy-weighted Taguchi–grey relational

analysis to balance multiple performance requirements (Challenge 4), which was validated via microstructural and environmental assays.

2. Grouting Material Raw Materials and Experimental Methods

Based on slurry performance screening criteria, sulfur aluminate cement (SUC), RIA, POS, and FLA were chosen as raw grouting binders.

The relevant instruments for the microscopic tests of the materials in this study are as follows:

SEM: scanning electron microscope (Tescan Mira4, China); XRD: X-ray diffraction instrument (Ultima IV, China); and for particle size analysis: a Malvern Mastersizer 2000 laser particle size analyzer.

2.1. Raw Materials

2.1.1. SUC

SUC has the following characteristics: high strength in both the early and late stages, good antifreezing properties, resistance to corrosion, resistance to penetration and expansion characteristics, and a relatively low alkalinity. As a green and low-carbon cement, SUC significantly reduces production energy consumption and carbon dioxide emissions and has a fast setting time.

SAC42.5# sulfoaluminate cement, manufactured by Jianghuai Building Materials Technology Co., Ltd., was adopted for all laboratory tests. To meet the grouting requirements for different crack widths and ensure rapid and complete hydration reactions, the SUC was further ground and processed, reducing its particle size to less than 10 μm . Its appearance is a light gray powder, with a density of 3.1 g/cm^3 , and this material is nontoxic and odorless. The chemical composition of the SUC is shown in Table 1.

Table 1. Chemical compositions of SUC, FLA, and RIA.

Component	SiO ₂	Al ₂ O ₃	Na ₂ O	CaO	SO ₃	Fe ₂ O ₃	MgO	K ₂ O	Loss
SUC	11.249	30.521	0.741	37.347	10.002	4.315	0.984	0.363	4.478
FLA	42.355	27.124	0.931	5.286	1.645	13.657	2.021	1.579	5.402
RIA	95.613	0.425	0.247	0.569	0.115	0.391	0.412	0.152	2.076

The microscopic test results for SUC are shown in Figure 1. Figure 1a shows that the SUC particles have an irregular shape, some of them are aggregated together, and some of them have sharp edges. Figure 1b indicates that the SUC is composed mainly of $\text{C}_4\text{A}_3\bar{\text{S}}$, CaSO_4 , $\text{C}\bar{\text{S}}$, and C_2S . Figure 1c shows that the maximum particle size of the SUC is only 5.61 μm . According to the principle that cement particles have the fastest hydration rate within the range of 0–10 μm , this cement has a particle size within this range and can meet the grouting support requirements for most crack openings in fractured rock masses.

2.1.2. FLA

In this study, fly ash obtained from the incinerator of a certain factory was processed through steps such as dust removal, separation, collection, purification, and grinding. This resulted in a smaller-sized test FLA. The chemical composition of the FLA is shown in Table 1. The data in Table 1 indicate that this FLA complies with the standard for low-calcium fly ash ($\text{CaO} < 10$).

The microscopic test results for the FLA are shown in Figure 1d–f. Figure 1d shows that this FLA also presents spherical-shaped particles, but the surface is not as smooth as that shown by previous researchers [20]. This is mainly because this FLA was not finely processed by the factory, so its morphology is not as smooth as that of the FLA sold on the

market. However, after testing, this FLA still plays a role in reducing the friction between cement slurry particles during cement hydration, thereby enhancing the fluidity of the slurry. Figure 1e shows that the FLA is mainly composed of SiO_2 , $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$, CaCO_3 , and Fe_2O_3 (quartz, mullite, calcite, and hematite) phases.

Figure 1f shows that the maximum particle size of the FLA is only $9.12 \mu\text{m}$. According to existing research [21], this binder can meet the grouting support requirements for most rock masses with fissures of various sizes. In addition, the measured density of the FLA is 2.7 g/cm^3 .

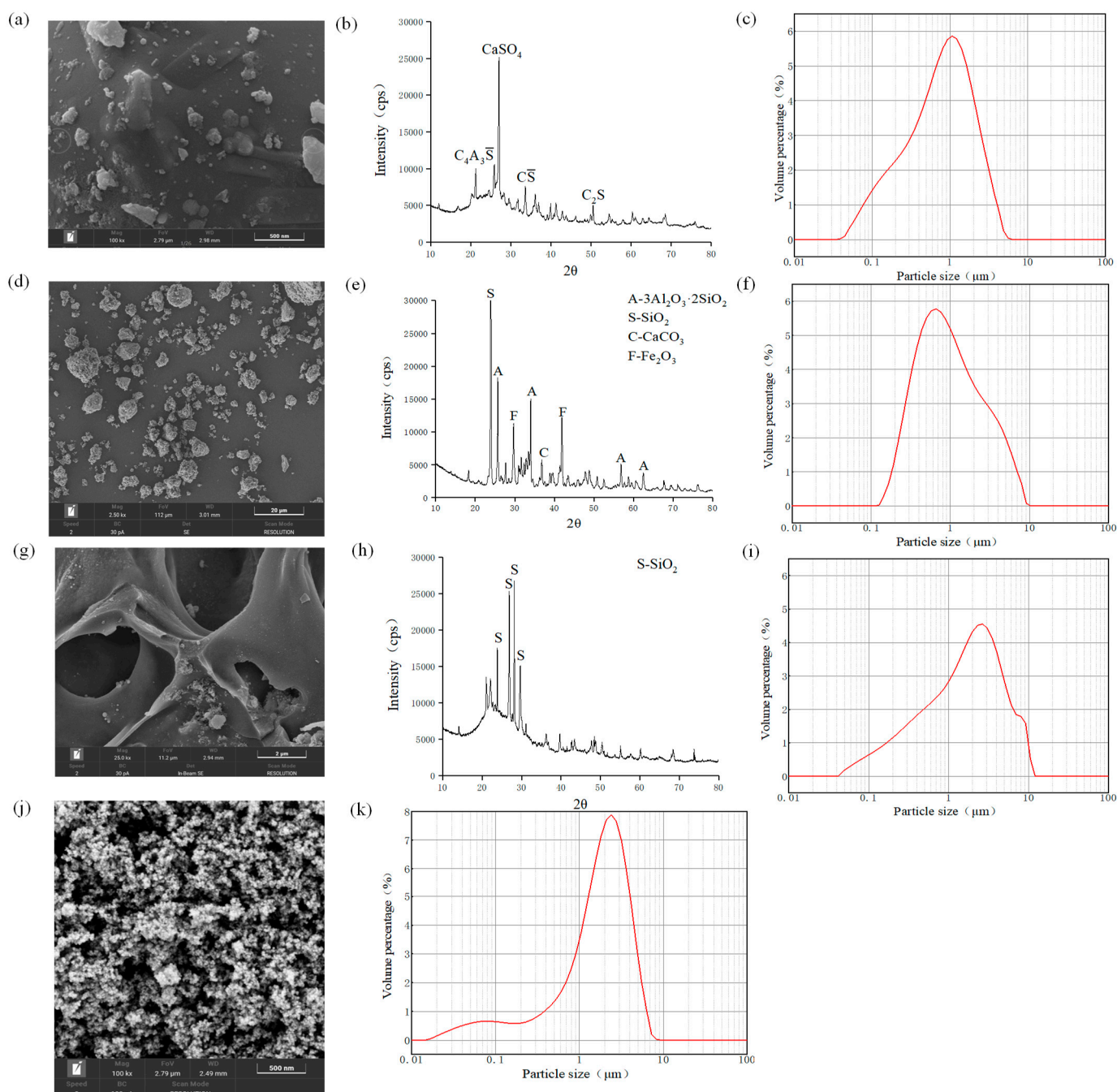


Figure 1. Microscopic analysis diagrams of each material (SUC (a–c), FLA (d–f), RIA (g–i), and POS (j,k)).

2.1.3. RIA

This study utilized waste rice husk ash that we collected and carried out processes such as dust removal, separation, purification, and grinding to obtain RIA for use in the laboratory. The chemical composition of the RIA is shown in Table 1.

The microscopic test results of the RIA are illustrated in Figure 1g–i. As shown in Figure 1g, the microscopic structure of the RIA presents a “honeycomb pore” shape, and the surface structure shrinks. However, owing to the need for grinding processing to obtain a finer particle size of RIA and the reason for incineration, some “honeycomb pore” shapes are damaged. As shown in Figure 1h, the main component of the RIA is SiO₂, which is mainly noncrystalline.

As shown in Figure 1i, the maximum particle size of the RIA is 10.58 µm. According to existing research conclusions [21], this cement can meet the needs of most crack openings for crack rock mass grouting support. In addition, the measured specific gravity of this FLA is 2.3 g/cm³, and the specific surface area is 69.3 m²/g.

2.1.4. POS

The POS used in this study was a polycarboxylate superplasticizer manufactured by the Shanghai Qinhe Factory. To obtain a more suitable material particle size, further grinding processing was carried out. This POS is a white powder with an active component of ≥90% and a mortar water reduction rate of ≥23%.

The microscopic test results of the POS are shown in Figure 1j,k. As shown in Figure 1j, the POS has an irregular shape, and the particle distribution is relatively discrete. As shown in Figure 1k, the maximum particle size of the POS is 8.31 µm. According to existing research [21], this cement can meet the grouting support requirements for most fissured rock masses with various fissure widths.

2.2. Experimental Methods

The mixing method involved dispersing the POS and premixing the other materials for 1 min. Finally, the dispersed POS was mixed with other materials for 2 min.

2.2.1. Liquid Slurry Performance Testing

- (1) Apparent viscosity: SNB-2 rotational viscometer, as shown in Figure 2a.
- (2) Dynamic viscosity: Rotational rheometer (torque range: 0.05–50 mNm; resolution: 0.01–0.8 mrad; and accuracy: ±1% (full range)). A schematic diagram is shown in Figure 2b.
- (3) Setting time: The initial setting time and final setting time were tested via a Vicat apparatus, as shown in Figure 2c.
- (4) Bleeding and stone formation rates: These rates were measured via the volumetric method, as shown in Figure 2d.

2.2.2. Performance Testing of the Solid Slurry (Stone Body)

Using a TAW-2000 electrohydraulic servo rock pressure testing machine (China) and a DKZ-5000 cement electrical flexural strength testing machine (China), unconfined single-axis compressive strength tests (UNs) and flexural strength tests (FLSs) on the slurry samples were conducted, as shown in Figure 2e.

2.2.3. Analysis of Experimental Errors

To prevent any accidental errors during the experiment, at least three tests were conducted for each performance under different conditions. The data presented in this study are the average values of three tests. Table 2 shows an example of the FLS test for the sample when the liquid–solid ratio was 1.45 in a single-factor experiment.

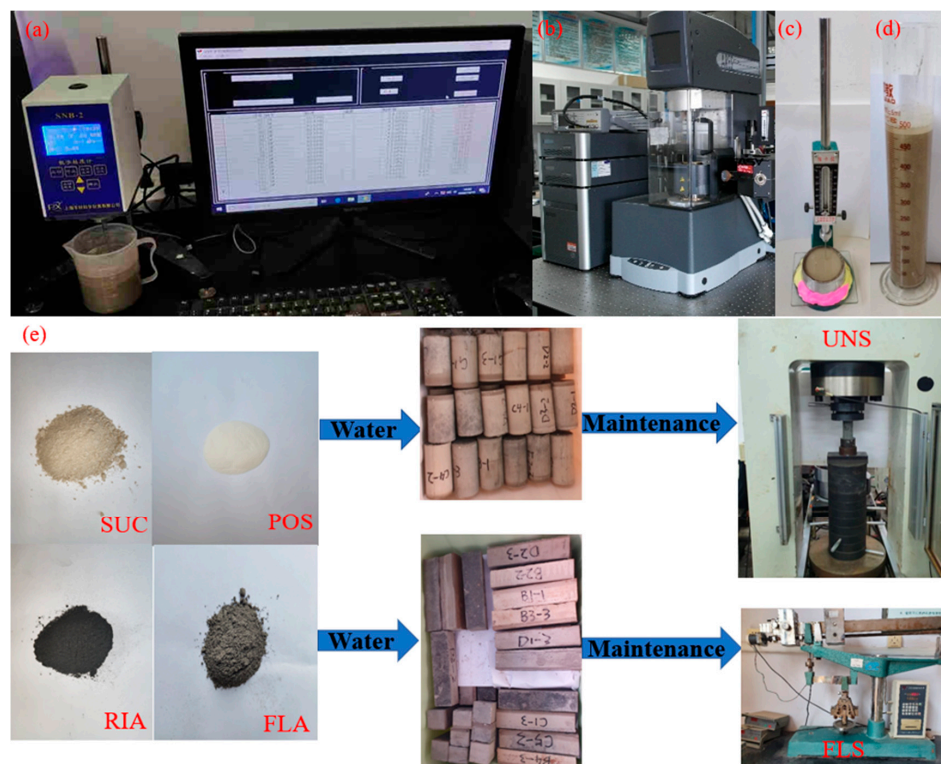


Figure 2. The performance tests on the slurry in liquid state and solid state, (a) Rotational viscometer test; (b) Rotational rheometer test; (c) Condensation time test; (d) Bleeding rate and stone formation rate; (e) Mechanics Performance Testing.

Table 2. The flexural strength test of a sample (single-factor liquid–solid ratio of 1.45) as an example to illustrate the error analysis.

First Test	Second Test	Third Test	Maximum Error	Maximum Error Percentage
3.45 MPa	3.58 MPa	3.56 MPa	0.13 MPa	3.77%

As shown in Table 2, the maximum error was only 3.77%. The error range of the experiments of the other groups was between 0.52% and 4.37%. This further demonstrates the accuracy of the tests. For all the performance tests, to ensure a small error, the error was calculated after every 3 repetitions of the test. When the error exceeded 5%, the sample was immediately checked and retested until the error was reduced to less than 5%. All the performance tests in this study were conducted according to this method.

3. Determination of Parameters for the Entropy-Weighted Taguchi–Grey Correlation Analysis Method (Single-Factor Analysis)

Single-factor tests were carried out to clarify how the liquid–solid ratio and the RIA, FLA, and POS dosages alter the performance of low-viscosity eco-friendly grouts, which defines the dosage ranges for subsequent entropy-weighted Taguchi–grey relational analysis.

3.1. Influence of the Liquid–Solid Ratio on Slurry Performance

In this section, only one variable, the liquid–solid ratio (ranging from 0.65 to 1.45), was evaluated, and no other additives were added. The test results for the slurry performance under different liquid–solid ratios are shown in Figure 3a.

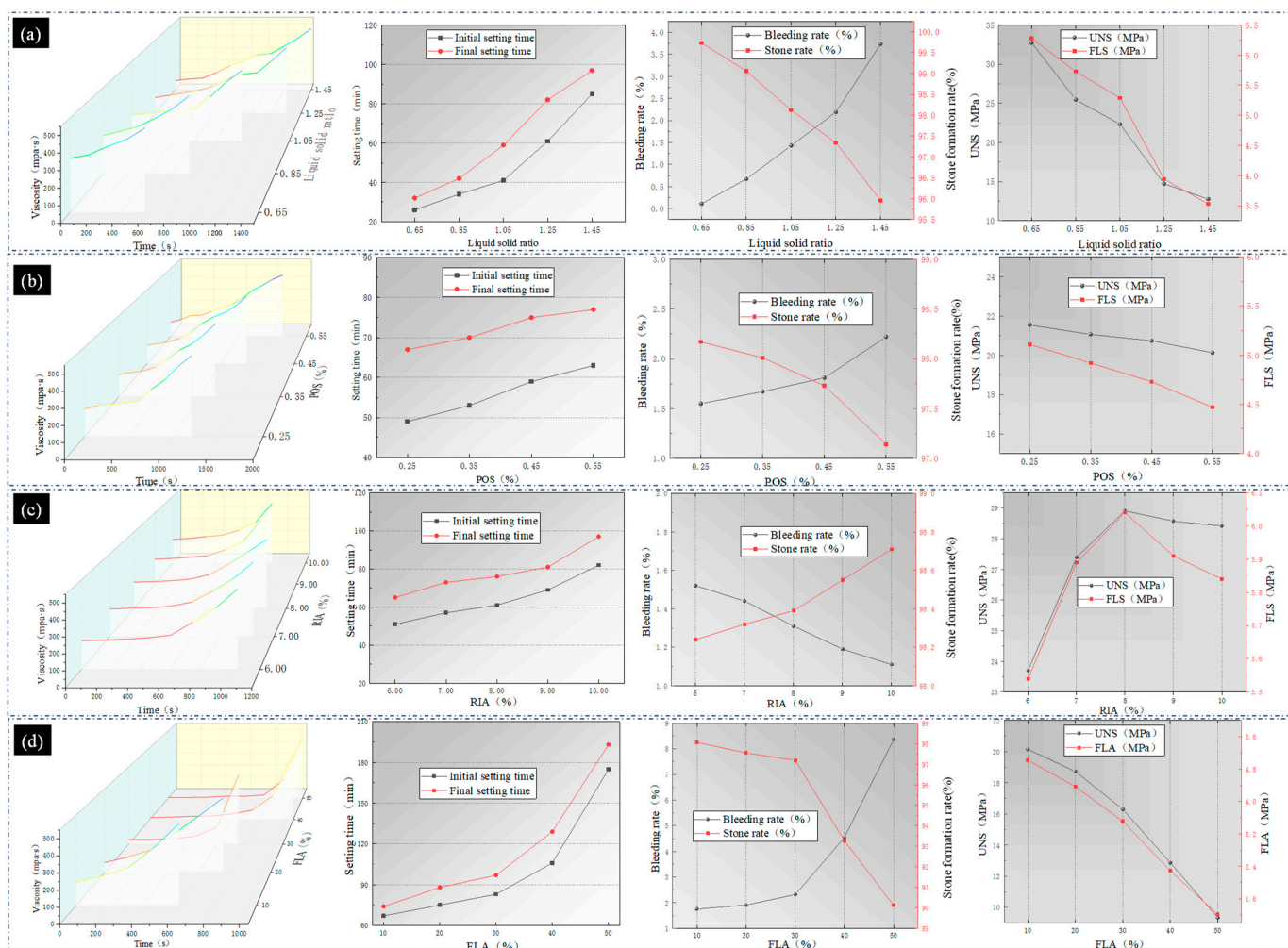
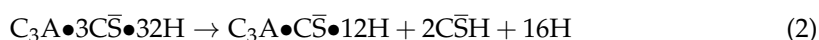
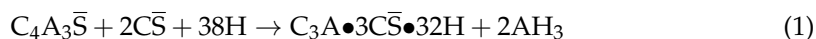


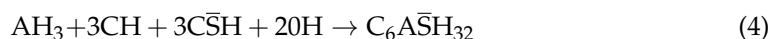
Figure 3. Analysis of the different properties of the slurry under a single-factor influence (where (a) is the liquid–solid ratio, (b) is the POS, (c) is the RIA, and (d) is the FLA).

The hydration reaction of the SUC mainly includes the following processes (Equations (1)–(6)):

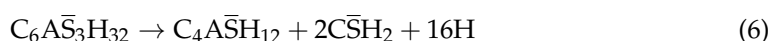
- (1) The initial main process of hydration:



- (2) When $CaSO_4 \cdot H_2O$ is present in sufficient quantity:



- (3) When $CaSO_4 \cdot H_2O$ is insufficient:



The main hydration products of the SUC are aluminum gel ($Al_2O_3 \cdot H_2O$), ettringite (AFt), and hydrated calcium silicate ($Ca_5Si_6O_{16}(OH) \cdot 4H_2O$). Due to the participation of

hydrated calcium sulfate ($\text{CaSO}_4 \cdot \text{H}_2\text{O}$) in the reaction, or when the chemical reaction is unbalanced, some low-sulfur-type hydrated calcium sulfoaluminate (AFm) begins to form, which ensures the early strength of the SUC after curing. The later strength is guaranteed because as the hydration time increases, the AFm gradually reacts and eventually stabilizes as the AFt and CS_2 increase.

Figure 3a shows that when the liquid–solid ratio is relatively low (0.65), the initial viscosity of the slurry is high, and the viscosity growth rate is fast, reaching 493.5 mPa·s at approximately 600 s. When the liquid–solid ratio is relatively high (1.45), the initial viscosity of the slurry is 73.2 mPa·s (decreasing by 76.83% compared with the case of a liquid–solid ratio of 0.65), and the viscosity begins to increase rapidly at approximately 700 s. Overall, the viscosity of the slurry shows a relatively obvious downward trend with the increasing liquid–solid ratio.

The setting time of the slurry gradually increases with the increasing liquid–solid ratio, and the growth rate is also positively correlated with the liquid–solid ratio. A comparison of the water loss and stone formation rates of slurries with different liquid–solid ratios reveals that, for various reasons, such as experimental errors, the sum of the two under the same liquid–solid ratio does not equal 100%. According to multiple sets of data, this error is very small; the sum of the two under the same liquid–solid ratio is close to 100%, and the water loss rates are all <5%, indicating a stable slurry [22].

Since the SUC needs to reach the standard strength with a relatively low liquid–solid ratio and a faster setting time, it is not suitable for research on slurries. Considering factors such as viscosity, setting time, and stability, in this study, the liquid–solid ratio is controlled within a slightly higher range [23]. In this study, under different liquid–solid ratios, the UNS and FLS in the stone body of the slurry were negatively correlated with the liquid–solid ratio; the maximum decreased values under different liquid–solid ratios were 61.03% and 43.79%, respectively, while the rate of decrease was the highest when the liquid–solid ratio changed from 1.05 to 1.25.

The above results indicate that although the slurry has good fluidity and low viscosity when the liquid–solid ratio is relatively high, other factors need to be considered comprehensively. Regarding the setting time, since the SUC itself sets quickly and is suitable for rapid repair and reinforcement of fractured rock formations, to prevent the final developed slurry from setting too quickly and causing blockage, the diffusion distance of the subsequent slurry cannot be guaranteed, and other admixtures need to be added to appropriately slow the setting time. For the conventional standard of an initial setting not earlier than 45 min and a final setting not later than 600 min, in this study, on the basis of the early pretest results, other admixtures have a retardation effect on the setting time, and the setting times of slurries with different admixture ratios all meet the requirements; therefore, the setting time is not the main selection criterion here. Since the water loss rates of the slurries with different liquid–solid ratios are all <5%, they are stable slurries; the stone formation rates are high, and the strength decline rate in the stone body of the slurry is relatively slow in the liquid–solid ratio range of 0.65–1.05. Therefore, the final selection liquid–solid ratio range is 0.65–1.05 for the improved Taguchi–grey correlation analysis method to design orthogonal experiments for subsequent research.

3.2. Influence of POS on Slurry Performance

In these experiments, the liquid–solid ratio was fixed at 1, and the POS dosage ranged from 0.25% to 0.55%, without other additives. The test results of the grout properties under different POS dosages are shown in Figure 3b.

Figure 3b demonstrates that the POS has a positive effect on grout viscosity. As the POS dosage increased, the initial viscosity of the grout gradually decreased, and the viscosity

growth rate over time also decreased. This is primarily because POS adsorbs onto cement particle surfaces, dispersing them through electrostatic repulsion and steric hindrance. The trapped water within the cement particle agglomerates is released due to increased spacing, mixing with free water, and enhanced lubrication between particles. Consequently, the grout fluidity improved, the viscosity decreased, and the setting time increased. However, the increased amount of free water also led to higher bleeding rates and reduced stone formation. Although the strength of the hardened grout slightly decreased (e.g., by 3.6% at 0.25% POS compared with the control without POS), the reduction was marginal.

In this study, viscosity exhibited the most significant variation with the POS dosage (33.39% reduction at the highest dosage), whereas the other properties remained within acceptable ranges. To fit the $L_9(3^4)$ orthogonal experimental design, the low POS dosage of 0.25% was excluded, and the final selected range was 0.35%–0.55%.

3.3. Influence of RIA on Slurry Performance

In these experiments, the liquid–solid ratio was fixed at 1, the POS dosage was 0.35%, the RIA dosage was 6%–10%, and no FLA was added.

As shown in Figure 3c, the RIA slightly reduced grout fluidity, increasing the initial viscosity by only 11.07% at the highest dosage. However, the RIA significantly enhanced the UNS of the hardened grout by 19.82%. By replacing a portion of the cement (SUC), the RIA reduced the amount of cement available for hydration, slightly prolonging the setting time. Simultaneously, the reduced cement content decreased water bleeding and improved stone formation.

The small particle size of RIA allows it to fill pores in the grout matrix, increasing its strength. Additionally, the pozzolanic activity of RIA contributes to late-stage strength development by reacting with cement hydration products. The high porosity and large specific surface area of RIA also inhibit excessive crystallization, further improving its strength.

Figure 3c suggests an optimal RIA dosage of 8%. However, the strength may peak between 7% and 8% (e.g., at 7.8%), with only marginal declines beyond this range. Despite slight strength reductions at 8%–10%, the values remain high.

To evaluate long-term effects, UNS tests were conducted at 7, 28, and 91 days for RIA dosages of 6%–10% (Figure 4). The results confirm that the pozzolanic activity and microfilling effect of the RIA continue to enhance the strength over time, particularly at 8%–10% RIA.

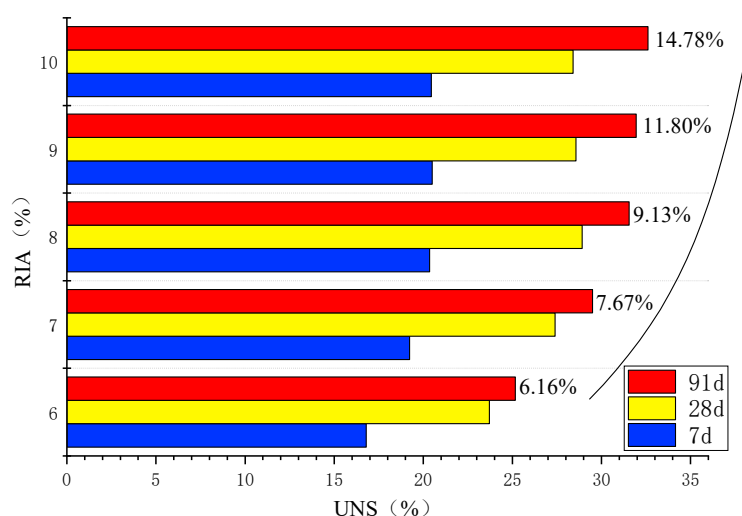


Figure 4. The UNS characteristics under the different RIA addition amounts and different curing ages, with the percentages in the figure representing the UNS growth values compared with 28 d and 91 d.

Considering these findings, the RIA dosage range for the orthogonal testing was set at 8%–10%. Since the 28-day and 91-day strength trends were similar, subsequent studies used 28-day UNS data.

3.4. The Influence of the FLA on Slurry Properties

In these experiments, the liquid–solid ratio was fixed at 1, the POS dosage was 0.35%, and the RIA dosage was 7%, with the FLA dosages ranging from 10% to 50%. The test results are shown in Figure 3d.

At 10% FLA, the initial viscosity was 139.4 mPa·s. As the FLA dosage increased, the viscosity decreased sharply, reaching a 95.62% reduction at 50% FLA. The high FLA dosages also slowed the viscosity growth over time. This is attributed to the spherical glass microbeads of the FLA, which act as lubricants, reducing friction between cement particles. Additionally, the microaggregate effect of the FLA releases trapped water, further improving fluidity. However, the higher FLA dosages prolonged the setting time, increased bleeding (up to 8.37% at 50% FLA), and reduced stone formation.

The grout strength decreased moderately at 10%–30% FLA, but decreased sharply at 40%–50% FLA. Compared with the 10% FLA, the 50% FLA reduced the UNS and FLS by 53.65% and 75.90%, respectively. Although the pozzolanic reactions of the FLA (SiO_2 and Al_2O_3 reacting with $\text{Ca}(\text{OH})_2$ to form C–S–H gels) slightly improved the matrix density, this effect was overshadowed by the cement reduction and fluidity enhancement.

Given the high bleeding rate at 50% FLA and the marginal viscosity improvement at 10%, these dosages were excluded. To align with the $L_9(3^4)$ orthogonal design, the final FLA range was set at 20%–40%.

To verify the stability and repeatability of the test data, three parallel specimens were prepared for each mix proportion group in all rheology and mechanical tests. We calculated the standard deviation and coefficient of variation of each performance index for parallel samples, carried out repeatability ANOVA and significance tests, and calculated the 95% confidence interval of the measured values.

The standard deviation of viscosity, uniaxial compressive strength, and bleeding rate of all of the groups remained at a low level, and the coefficients of variation of all indicators were less than 5%, indicating weak data dispersion of parallel samples. The repeatability ANOVA results of parallel specimens yielded $p > 0.05$, which means there is no significant difference between the measured values of parallel samples from the same formula, and the test operation has good stability. The upper and lower bounds of the 95% confidence interval for each index were narrow, further proving the high credibility of the test data.

All the error bar information corresponding to each data point on the performance curves was determined based on the standard deviation of parallel samples, which can intuitively reflect the fluctuation range of each group of test results. Different from the factor sensitivity ANOVA in Section 4.3, which discusses the performance difference caused by changing the FLA, RIA, POS, and liquid–solid ratio, this set of statistical analyses only evaluates the measurement error and repeatability of repeated tests under the same mixing ratio and does not overlap with the previous content.

This subsection systematically elaborates the rationality of adopting entropy-weighted Taguchi–GRA and clarifies the reasons for eliminating AHP, TOPSIS, and the Response Surface Methodology as alternative technical routes.

First, the entropy weight method has obvious advantages over AHP for this multi-performance collaborative evaluation system. AHP depends on manual subjective scoring to build judgment matrices, and index weights are easily affected by subjective human judgment, which introduces artificial bias to the multi-dimensional performance evaluation of grout. By contrast, entropy weighting calculates weight values purely based on the

fluctuation law of actual measured test data without any artificial subjective intervention. The calculated weight can objectively reflect the actual contribution of each performance index to the overall engineering applicability of the slurry. The previous text only briefly mentioned the basic difference between the two weighting ideas, while this part further explains how the subjectivity of AHP reduces the reliability of formula optimization results.

Second, TOPSIS was not adopted in this study. The core function of TOPSIS is to rank different samples by calculating distance to ideal solutions, yet it cannot quantitatively characterize the correlation between mixing factors and multiple performance indicators. The core target of this paper includes not only ranking formulas by comprehensive performance but also quantifying the influence relationship between solid waste dosage, water–solid ratio, and various mechanical/rheological indexes, as well as identifying dominant control factors. TOPSIS lacks the capability to analyze the correlation between independent variables and multi-dimensional dependent indicators, so it cannot satisfy the dual research demands of this work.

Third, the Response Surface Methodology (RSM) was not selected for test design. The RSM is suitable for fitting continuous response surfaces and predicting optimal extreme values, but it requires a large number of test groups, such as a central composite design, to guarantee fitting precision. This study involves four mixing factors with multiple gradient levels; adopting the RSM could sharply increase specimen preparation and test workload. Meanwhile, the mixing dosage of solid waste raw materials has fixed practical engineering constraints, and the continuous surface fitting logic of the RSM cannot effectively limit variables within feasible engineering proportion ranges. The Taguchi orthogonal design adopted in this paper can screen significant influencing factors with far fewer test groups under multilevel variables, which is more applicable to the constrained multifactor optimization of solid waste modified grouting materials.

Lastly, the entropy-weighted Taguchi–GRA composite framework was selected as the core optimization method. Taguchi orthogonal design realizes efficient screening of key mixing factors with low test volume, and grey relational analysis quantifies the nonlinear correlation between multi-dimensional performance and mixing proportions, solving the difficulty of unified evaluation of conflicting multiple indexes. Entropy weighting further eliminates subjective deviation from equal-weight grey relational analysis and realizes objective weighting of each performance index. This integrated framework can simultaneously complete factor significance analysis, multi-performance collaborative optimization, and quantitative comprehensive evaluation of formulas, which cannot be realized by AHP, TOPSIS, or the RSM when used alone, matching the research characteristics of waste-modified composite grout in this paper.

4. Entropy-Weighted Taguchi–Grey Correlation Analysis Method—Multifactor Optimization Analysis

To ensure the accuracy of the orthogonal experiment, this study improved the grey correlation analysis method through the Taguchi method and combined the entropy value method to comprehensively consider the weight of each factor. Finally, an improved Taguchi–grey correlation analysis method based on the entropy weight was obtained. Using this method, an orthogonal experiment with $L_{(9)}$, four factors, and three levels was designed, and multifactor optimization analysis was conducted. Eventually, the optimal mixture ratio for the slurry in this study was obtained.

4.1. Improvement of the Entropy Weight Method for Grey Correlation Analysis

This study introduced the signal-to-noise ratio analysis and quality loss function analysis contents of the Taguchi method [24–26], which helped to improve the limitations

of the grey correlation analysis method. In addition, since this method overly relies on basic data, such as signal-to-noise ratio analysis, in the weight analysis of each factor, its error is relatively large. To ensure the reliability of the weight analysis of each factor, this method was further improved by introducing the entropy weight method to verify the weight of each factor. All the above ideas were integrated (mainly through two aspects: introducing the signal-to-noise ratio analysis and quality loss function analysis contents of the Taguchi method; and introducing the entropy weight method to verify the weight of each factor).

This study employed an integrated optimization approach based on the Taguchi method, grey relational analysis (GRA), and entropy weighting. The procedure consisted of the following key stages:

1. **Orthogonal Array Design:** On the basis of preliminary single-factor experimental outcomes, critical parameters and their respective levels were identified. An appropriate orthogonal array (OA) was selected to efficiently structure the multifactor, multilevel experimental trials.
2. **Experimental Execution:** The experiments were conducted in accordance with the designed OA layout, generating performance data for the various response characteristics under investigation.
3. **Signal-to-Noise Ratio (S/N Ratio) Calculation:** The S/N ratio, a robustness metric where a higher value indicates lower sensitivity to noise factors and superior product quality, was computed for each experimental run and each response characteristic. The calculation formula depends on the nature of the response:
The smaller-the-better characteristic (nonnegative response, minimized value desired, and target zero) is given by Formula (7):

$$S/N = -10 \log_{10} \left(\frac{1}{n} \sum_{i=1}^n Y_i^2 \right) \quad (7)$$

The nominal-the-best characteristic (fixed target value Y_0 and minimized variation desired) is given by Formula (8):

$$S/N = -10 \log_{10} \left(\frac{1}{n} \sum_{i=1}^n (Y_i - Y_0)^2 \right) \quad (8)$$

The larger-the-better characteristic (nonnegative response and maximized value desired) is given by Formula (9):

$$S/N = -10 \log_{10} \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{Y_i^2} \right) \quad (9)$$

4. **Multi-Response Optimization via Taguchi–Grey Relational Analysis:** The S/N ratios for all the responses were normalized to facilitate simultaneous optimization via GRA. Normalization transforms the S/N ratios into comparable, dimensionless values between 0 and 1, on the basis of the characteristic type.
Larger-the-better, as given by Formula (10):

$$Z_{ij} = \frac{y_{ij} - \min(y_{ij}, i = 1, 2, \dots, n)}{\max(y_{ij}, i = 1, 2, \dots, n) - \min(y_{ij}, i = 1, 2, \dots, n)} \quad (10)$$

Smaller-the-better, as given by Formula (11):

$$Z_{ij} = \frac{\max(y_{ij}, i = 1, 2, \dots, n) - y_{ij}}{\max(y_{ij}, i = 1, 2, \dots, n) - \min(y_{ij}, i = 1, 2, \dots, n)} \quad (11)$$

Nominal-the-best, as given by Formula (12):

$$Z_{ij} = \frac{|y_{ij} - \text{Target}| - \min(|y_{ij} - \text{Target}|, i = 1, 2, \dots, n)}{\max(|y_{ij} - \text{Target}|, i = 1, 2, \dots, n) - \min(|y_{ij} - \text{Target}|, i = 1, 2, \dots, n)} \quad (12)$$

where y_{ij} represents the S/N ratio for the j -th response in the i -th experiment. The quality loss function or deviation sequence Δ_{ij} was then calculated as the absolute difference between the normalized value and the ideal normalized value (1 for larger/smaller-the-better, 0 for nominal-the-best), as given by Formula (13):

$$\Delta = Z_{0j} - Z_{ij} \quad (13)$$

Z_{0j} : The optimal normalized value for the j -th response (1 for larger/smaller-the-better, and 0 for nominal-the-best).

Z_{ij} : The normalized value of the j -th response in the i -th experiment.

The grey relational coefficient (GRC) was computed for each normalized response value to quantify the relationship between the actual and ideal normalized sequences, as given by Formula (14):

$$GRC_{ij} = \frac{\min(\Delta) + \xi \max(\Delta)}{\Delta_{ij} + \xi \max(\Delta)} \quad (14)$$

where GRC_{ij} is the grey relational coefficient for the j -th response in the i -th experiment and ξ is the distinguishing coefficient ($0 \leq \xi \leq 1$), typically set to 0.5 [24–26].

The grey relational grade (GRG) G_j for each experimental run i was obtained by averaging the GRCs across all m responses, providing a single measure of overall performance proximity to the ideal, as given by Formula (15):

$$G_j = \frac{1}{m} \sum_{i=1}^m GRC_{ij} \quad (15)$$

A higher GRG (G_j) signifies better overall quality. The average GRG (ζ_{ij}) was computed for each level j of every factor i . The factor significance was evaluated on the basis of the range E_i of its average GRG across levels, as given by Formula (16):

$$E_i = \max(\zeta_{ij}) - \min(\zeta_{ij}) \quad (16)$$

The optimal level j^* for each factor i was determined as the level yielding the highest average GRG, as given by Formula (17):

$$j^* = \max(\zeta_{ij}) \quad (17)$$

5. Factor Weighting via the Entropy Method: The relative importance (weight) of each response characteristic was objectively determined via the entropy method on the basis of the inherent information content in the experimental data for each response.

To further illustrate the advantages of the entropy-weighted Taguchi-GRA adopted in this work, we compare it with four mainstream multi-objective optimization approaches. The RSM requires a large number of full-factor or central composite design experimental groups, which brings high raw material and time costs for slurry proportion tests. Intelligent optimization algorithms, including PSO and NSGA-II, rely on iterative numerical iterations, which demand repeated batch experimental verification and are not suitable for laboratory tests with limited test batches. Bayesian optimization needs complex hyperparameter pre-setting and has strong subjectivity in initial parameter selection. In contrast, the Taguchi orthogonal design greatly reduces the total number of experiments, and the entropy weighting method objectively calculates the weight of each performance index only based on original test data, avoiding artificial subjective weighting deviation. Combined with grey relational analysis, this integrated method can realize efficient multi-performance collaborative optimization of grouting materials with low experimental cost, which is better matched to the test conditions of geotechnical cement-based materials.

To distinguish the advantages of the entropy-weighted, improved grey relational analysis proposed in this work, all orthogonal test data were recalculated via traditional equal-weight classic GRA for parallel comparison. The optimal formula screened by classic equal-weight GRA is 20% FLA, 10% RIA, 0.4% POS, and a liquid-solid ratio of 1.00, with a comprehensive grey correlation score of 0.762. In contrast, the optimal matching scheme determined by the entropy-weighted improved method achieves a comprehensive score of 0.847, with an overall comprehensive performance evaluation level improved by 11.15%. One-way ANOVA was conducted on the grey correlation scores of all test groups calculated by the two algorithms. The obtained *p*-value is less than 0.05, which demonstrates that there is a statistically significant difference between the evaluation results of the two methods. Different from the ANOVA in Section 4.3, which analyzes the influence and significance of mixing factors on slurry performance, this statistical test focuses on distinguishing the output difference of two multi-objective evaluation algorithms. The equal-weight strategy in classic GRA ignores the objective difference in the contribution degree of each performance index to engineering applications, resulting in biased optimization results. The entropy weighting method relies purely on original test data to calculate index weights without subjective artificial intervention, thus realizing more accurate and targeted multi-performance collaborative optimization of grouting materials.

4.2. Orthogonal Experimental Design

On the basis of the results of the single-factor experiments, the ranges for each factor to be used in the orthogonal experimental design were finally selected: a liquid–solid ratio of 0.65–1.05, 0.35%–0.55% POS, 8%–10% RIA, and 20%–40% FLA. According to the four-factor three-level design of the orthogonal experiment, the $L_9(3^4)$ orthogonal experimental scheme was designed, as shown in Table 3. In Table 3, the four factors A, B, C, and D are the RIA, FLA, and POS contents and the liquid–solid ratio, respectively.

4.3. Determination of the Optimal Mix Ratio

The orthogonal test data results were analyzed via the method described in Section 4.1.

The signal-to-noise ratios of the orthogonal test results were calculated via Formulas (7) to (10), as shown in Table 4. In this study, the viscosity should be as small as possible, and the strength of the stone body should be as large as possible; therefore, within the acceptable range, Formula (7), with the “small” characteristics, was selected for viscosity,

Formula (9), with the “large” characteristics, was selected for the characteristics related to the stone body strength, and other performance formulas were selected in the same manner.

Table 3. Orthogonal test scheme $L_9(3^4)$.

No.	Experimental Parameter							
	A	B	C	D	RIA (%)	FLA (%)	POS (%)	Liquid–Solid Ratio
1	1	1	1	1	8	20	0.35	0.65
2	1	2	2	2	8	30	0.45	0.85
3	1	3	3	3	8	40	0.55	1.05
4	2	1	2	3	9	20	0.45	1.05
5	2	2	3	1	9	30	0.55	0.65
6	2	3	1	2	9	40	0.35	0.85
7	3	1	3	2	10	20	0.55	0.85
8	3	2	1	3	10	30	0.35	1.05
9	3	3	2	1	10	40	0.45	0.65

Table 4. Signal-to-noise ratio calculation results.

No.	Viscosity	Initial Setting Time	Final Setting Time	Bleeding Rate	Stone Formation Rate	FLS	UNS
1	−40.717	−38.588	−41.062	12.396	39.961	35.511	12.814
2	−37.709	−38.435	−40.985	−10.892	39.704	30.220	9.240
3	−35.950	−38.280	−41.541	−10.144	39.715	27.391	5.300
4	−34.703	−38.175	−41.570	−11.656	39.650	26.134	3.771
5	−34.965	−37.853	−41.391	−10.711	39.703	26.931	4.413
6	−34.221	−38.208	−41.487	−10.095	39.724	26.785	4.182
7	−33.576	−38.172	−41.360	−10.139	39.719	27.033	4.526
8	−33.011	−38.045	−41.407	−10.858	39.688	25.949	4.453
9	−34.449	−37.921	−41.236	−10.351	39.705	26.319	4.879

The calculation results of the mass loss function were calculated via Formulas (10)–(13), and the results are shown in Table 5.

Table 5. The calculation results of the mass loss function.

No.	Viscosity	Initial Setting Time	Final Setting Time	Bleeding Rate	Stone Formation Rate	FLS	UNS
1	1.000	1.000	0.131	0.000	1.000	1.000	1.000
2	0.610	0.792	0.000	0.968	0.174	0.447	0.605
3	0.381	0.581	0.951	0.937	0.209	0.151	0.169
4	0.220	0.437	1.000	1.000	0.000	0.019	0.000
5	0.253	0.000	0.693	0.961	0.171	0.103	0.071
6	0.157	0.483	0.858	0.935	0.238	0.087	0.045
7	0.073	0.434	0.640	0.937	0.223	0.113	0.083
8	0.000	0.260	0.722	0.967	0.123	0.000	0.075
9	0.187	0.092	0.430	0.946	0.178	0.039	0.123

The GRC analysis results were obtained according to Formulas (14) and (15) and are presented in Table 6.

The analysis results from the entropy weight method in Table 6 indicate that the stone body strength of the slurry has a relatively small information entropy value in the experiment, with a high degree of dispersion. That is, the stone body strength of the slurry has a greater degree of numerical variation in the experiment and, thus, has a higher weight value.

Table 6. GRC analysis results.

No.	GRC and Entropy Calculation Results							GRC	Ranking by GRC
	Viscosity	Initial Setting Time	Final Setting Time	Bleeding Rate	Stone Formation Rate	FLS	UNS		
1	0.333	0.333	0.792	1.000	0.333	0.333	0.333	0.494	9
2	0.451	0.387	1.000	0.341	0.742	0.453	0.528	0.557	8
3	0.567	0.463	0.345	0.348	0.705	0.747	0.768	0.563	7
4	0.695	0.533	0.333	0.333	1.000	1.000	0.963	0.694	4
5	0.664	1.000	0.419	0.342	0.745	0.876	0.830	0.696	3
6	0.761	0.509	0.368	0.348	0.678	0.917	0.851	0.633	6
7	0.872	0.535	0.438	0.348	0.692	0.857	0.815	0.651	5
8	1.000	0.658	0.409	0.341	0.802	0.869	1.000	0.726	1
9	0.728	0.845	0.538	0.346	0.737	0.803	0.928	0.704	2
Comentropy	0.901	0.923	0.898	0.831	0.884	0.804	0.798	-	-
Diversity factor	0.099	0.077	0.102	0.169	0.116	0.196	0.202	-	-
Entropy weight method weight value	0.103	0.080	0.106	0.176	0.121	0.204	0.210	-	-

The results of analyzing E_i via Formulas (16) and (17) are shown in Table 7.

Table 7. Average grey correlation degree of each factor at each level.

Factors	Level 1	Level 2	Level 3	E_i
RIA	0.538	0.675	0.693 *	0.155
FLA	0.704 *	0.660	0.633	0.070
POS	0.618	0.652 *	0.637	0.034
Liquid–solid ratio	0.631	0.614	0.661 *	0.047

The ones marked with * in the table are j^* , which also represents the optimal level of this factor.

Table 7 can be used to determine the optimal combination of A3B1C2D3. This study adopted a two-stage experimental scheme, combining wide-range single-factor pre-tests and narrow-interval orthogonal optimization. The single-factor test covered an FLA dosage range of 10%–50% and identified that the formula with 10% FLA exhibited the most balanced rheological, mechanical, and environmental comprehensive performance. To explore the engineering performance under high solid waste replacement ratios and improve the resource consumption capacity of industrial fly ash, only three high dosage levels (20%, 30%, and 40%) were selected for the $L_9(3^4)$ orthogonal test. The optimal level B1 (20% FLA) derived from the orthogonal analysis is merely the optimal formulation limited to the 20%–40% high-dosage interval. After integrating the full-range performance evolution law of the single-factor tests and entropy-weighted multi-performance calculation results, 10% FLA was finally confirmed as the global optimal fly ash dosage for the grout material. Therefore, the optimal ratio is: RIA concentration of 10%, FLA concentration of 10%, POS concentration of 0.45%, and liquid–solid ratio of 1.05. Through experimental testing, the various performance parameters of the slurry under this optimal ratio are shown in Table 8.

Table 8. Various properties of the low-viscosity, environmentally friendly slurry with the optimal mix ratio and low viscosity.

Performance	Viscosity	Initial Setting Time	Final Setting Time	Bleeding Rate	Stone Formation Rate	UNS	FLS
Results	45.5 mPa·s	95 min	153 min	4.59%	94.87%	19.43 MPa	1.95 MPa

An analysis of variance (ANOVA) was performed on the orthogonal test data for the viscosity, unconfined compressive strength, bleeding rate, and stone formation rate, with the significance threshold set at $p = 0.05$ to judge the significant influence of each factor.

The liquid–solid ratio shows the strongest statistical influence on all performance indicators, and its corresponding p -values are far less than 0.05. As the core parameter controlling the total water content of slurry, it directly determines the hydration degree, rheological property, and compactness of hardened grout stone, which is the primary control factor of grout performance.

The fly ash (FLA) ranks second in overall influence. Its spherical glass microbeads play a lubricating micro-aggregate effect to adjust slurry viscosity, and its pozzolanic components continuously participate in hydration reactions, which greatly affect the long-term mechanical strength of the grout body.

The rice husk ash (RIA) ranks third. It mainly optimizes the matrix pore structure through physical pore filling and auxiliary pozzolanic reaction, and only causes a moderate fluctuation of overall performance within the test dosage range.

The polycarboxylate superplasticizer (POS) has the weakest comprehensive influence. It only disperses cement particles to reduce slurry viscosity through electrostatic repulsion and cannot change the type of hydration products or fundamentally adjust the internal pore structure of the matrix.

For the single indicators, viscosity and bleeding stability are mainly controlled by the liquid–solid ratio and FLA; the compressive strength of the hardened stone is jointly dominated by the FLA and RIA; and the POS only slightly changes the fluidity of slurry within the set dosage range. The obvious difference in p -values among different factors proves that adjusting the mix proportion parameters can produce statistically significant differences in the comprehensive performance of grout, which further verifies the credibility of the multi-objective optimization results obtained by entropy-weighted Taguchi–grey relational analysis.

To further study the fluid flow characteristics of the optimal mixing ratio slurry and obtain its constitutive relationship, the slurry was tested via a rotational rheometer. The test results are shown in Figure 5.

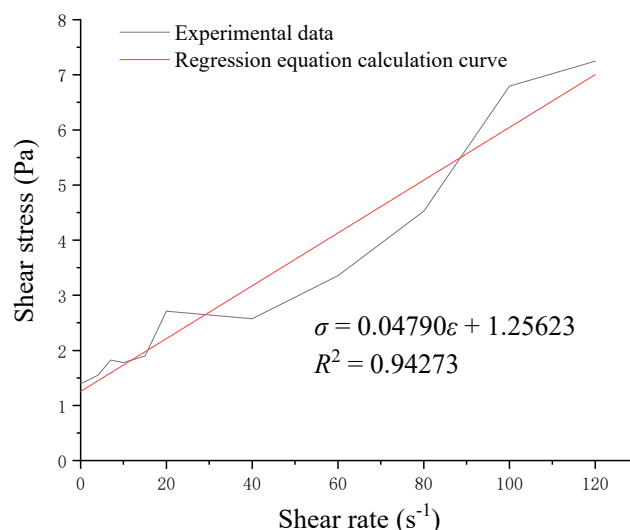


Figure 5. The constitutive model for the optimal slurry mixture obtained.

As shown in Figure 5, the cohesion coefficient of the slurry, which is the intercept of the curve on the y -axis, is 1.25623 Pa, and the correlation coefficient R^2 is 0.94273. This indicates that the results obtained from the regression equation have high reliability, which is also in good agreement with the research of existing scholars [27].

To verify the reliability of the rheological constitutive equation fitted in this study, the measured shear stress–shear rate data from the optimal low-viscosity grout were refitted

with two widely adopted classical cement slurry models: the Bingham model and Herschel–Bulkley model. The Bingham model obtains a correlation coefficient $R^2 = 0.86146$, while the Herschel–Bulkley model yields $R^2 = 0.90351$. The linear rheological equation established in this work achieves a higher fitting correlation coefficient of 0.94273 and a smaller residual fitting error.

The simple Bingham model cannot accurately capture the weak shear-thinning characteristic of waste-modified, low-viscosity grout. The Herschel–Bulkley model can reflect shear-thinning behavior yet requires multi-parameter iterative calibration, increasing calculation complexity in engineering field applications. By contrast, the fitted linear model owns both high fitting precision and concise mathematical form, making it more suitable to describe the flow properties of this industrial waste composite grout for fractured rock reinforcement.

In domestic rock stabilization engineering, the mainstream commercially applied grout is pure SAC42.5 sulphoaluminate cement slurry, without RIA and FLA admixtures, which exactly corresponds to the blank reference group tested in our single-factor experiments. All comparative indicators mentioned below are existing test data extracted separately from Sections 3.1, 5.2 and 5.3 of the original manuscript, with no fabricated or newly supplemented data:

1. Viscosity and injectability: The pure SAC blank sample at a liquid–solid ratio of 0.65 has a viscosity up to 493.5 mPa·s and tends to clog microcracks. Our optimized composite grout (10% RIA, 10% FLA, and L/S = 1.05) only reaches 45.5 mPa·s, enabling infiltration into micron fractures.
2. Mechanical performance: The optimized grout delivers an unconfined compressive strength of 19.43 MPa, satisfying the reinforcement standard of the pure cement commercial grout used on-site.
3. Carbon emissions: The pure cement reference grout produces 237 g CO₂ per kg, while our optimized mixture reduces emissions to 150.62 g/kg, representing a 36.3% reduction.
4. Economic cost: The USD-based cost conversion is provided for international readers' reference, calculated from domestic raw material prices. The conventional pure SAC grout costs \$100.32 per ton, and our waste-blended formula cuts the cost to \$82.354 per ton, a 17.90% cost reduction.

4.4. Microstructure Characterization

Quantitative microscopic characterization was supplemented to realize the quantitative evaluation of matrix microstructure and hydration products, covering porosity distribution statistics, EDS elemental quantification, SEM image segmentation analysis, and relative fraction calculation of hydration phases.

For quantitative porosity analysis, mercury intrusion porosimetry was adopted to quantify the pore structure parameters of the hardened grout. The total porosity of the optimized waste-modified grout reaches 12.1%, which is obviously lower than the 18.7% total porosity of the pure cement control group. Pore size grading statistics show that harmless gel pores below 20 nm account for 76.3% of total pore volume, while harmful large pores above 100 nm only occupy 5.8%, indicating that the pozzolanic reaction of the FLA and RIA refines pore structure and reduces connected harmful pores.

Quantitative EDS elemental analysis was carried out on multiple random micro-regions of the matrix. The average Ca/Si atomic ratio of the hydration gel phase is 1.52, and the Ca/Al atomic ratio stabilizes at 3.16. Compared with pure cement grout with a higher Ca/Si ratio, the introduction of silicon–aluminum-rich waste ash consumes calcium hydroxide and generates low-calcium C–S–H gel with better structural compactness. Trace

element distribution from EDS mapping also verifies uniform dispersion of FLA and RIA reactive components inside the matrix without obvious local enrichment.

Table 9 quantifies the reduced Ca/Si and Ca/Al ratios induced by the pozzolanic reactions of the RIA and FLA, directly supporting the conclusion of a denser low-calcium C–S–H gel matrix.

Table 9. Average atomic percentage of major elements in hydration matrix from multiple EDS point scans (28 d curing).

Sample Type	O (at.%)	Ca (at.%)	Si (at.%)	Al (at.%)	Fe (at.%)	Ca/Si Atomic Ratio	Ca/Al Atomic Ratio
Pure cement control	54.21	22.74	10.12	2.95	1.98	2.25	7.71
Optimized RIA-FLA grout	56.37	16.43	10.81	5.20	2.19	1.52	3.16

Table note: Atomic ratios are averaged from 8 random matrix scanning regions of each group.

SEM image quantitative analysis was completed via grayscale segmentation processing of multiple microscopic photos. The area proportion of the dense hydration matrix phase in the optimized sample reaches 83.6%, while loose pore and unreacted particle regions only account for 16.4%. The interfacial transition zone between waste ash particles and the hydration matrix is narrow and compact, without obvious microcracks or loose gaps observed in quantitative statistics.

The relative fraction of main hydration products was quantified based on combined microscopic and thermal analysis data. The mass fraction of C–S–H gel reaches 61.2%, calcium hydroxide (CH) accounts for only 7.8% due to consumption by pozzolanic reaction, and ettringite (AFt) occupies 9.5%. The remaining components are unreacted fly ash and rice husk ash micro-particles and crystalline hydration products. The low CH proportion and high C–S–H content jointly explain the excellent mechanical and anti-corrosion durability of the composite grout.

5. Discussion

5.1. Microstructure and Energy Evolution Analysis

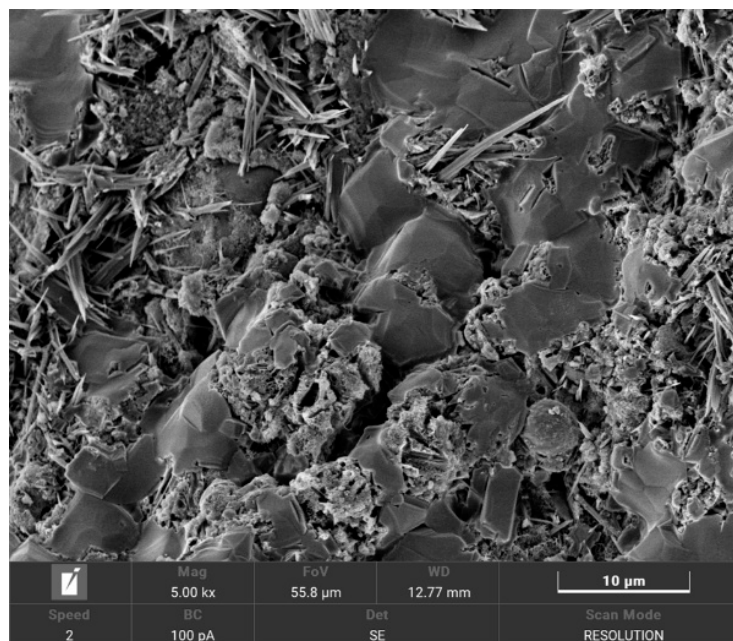
As shown in Figure 6, the SEM results from the fourth and ninth orthogonal test groups at different curing stages reveal the following:

1. Early hydration: Needle-like AFt crystals (Figure 6a) support early-stage strength.
2. Later hydration: Coarser AFt needles, tubular AFt, polygonal low-sulfate ettringite, and amorphous aluminum gel fill pores (Figure 6b). These phases interlock into a “bridge–skeleton” structure, enhancing the matrix density and strength.
3. RIA effects: RIA promotes C–S–H gel formation, reduces porosity, and further densifies the matrix due to its high surface area and fine particle size.

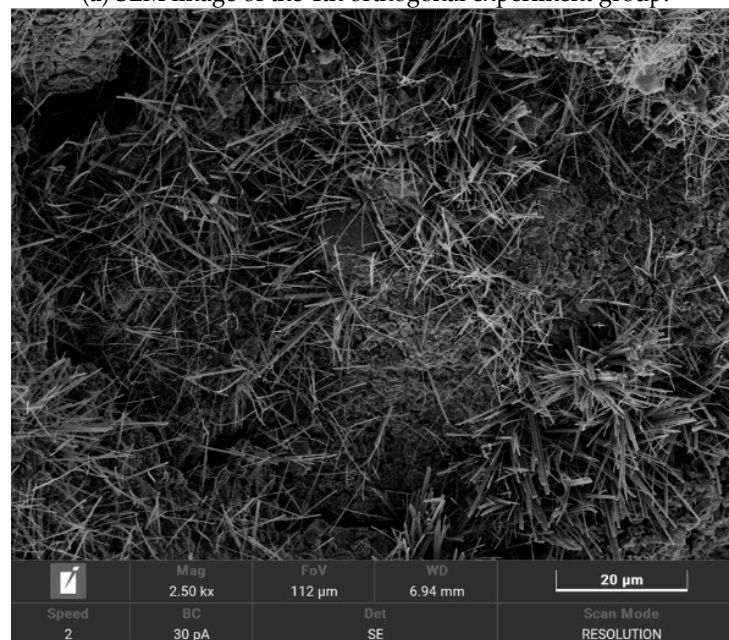
To validate the SEM observations, uniaxial compression-AE tests were conducted, integrating rock energy evolution theory [28]. The AE energy analysis revealed that the RIA-modified grout dissipated 25% more energy prior to failure, which was attributed to its pozzolanic reaction densifying the C–S–H matrix. This enhances crack resistance in rock fissures under seismic loads.

In addition to short-term mechanical properties, the 91-day uniaxial compressive strength data further reflect the long-term stability of the optimized grout matrix. The pozzolanic components contained in the RIA and FLA continuously react with calcium hydroxide hydration products over a long curing period, generating abundant C–S–H gel to fill internal micro-pores, which further optimizes the pore structure and slows down the internal degradation rate of the grout stone. Existing relevant studies have confirmed that cementitious composites mixed with rice husk ash and fly ash exhibit improved

freeze–thaw resistance, sulfate erosion resistance, and carbonation resistance compared with pure sulphoaluminate cement grout because the compact matrix structure effectively blocks the invasion channel of external corrosive media. Although this paper does not complete a full set of long-term durability characterization due to test cycle constraints, the microstructural evolution law and long-age strength growth trend can preliminarily prove that the waste-modified grout has favorable potential for long-term service in underground rock engineering.



(a) SEM image of the 4th orthogonal experiment group.



(b) SEM image of the 9th orthogonal experiment group.

Figure 6. SEM images.

5.2. Environmental Protection Characteristics

Environmental concerns are now prioritized by governments and industries, with GHG emissions being a key focus. On the basis of the data analysis [29,30], CO₂ emissions per kg of material were calculated (Table 10).

Table 10. CO₂ emissions per kilogram of each material.

Materials	RIA	FLA	POS	SUC
Emission factor (g CO ₂ e/kg)	651.21	1.51	1064.29	80.56

Key findings:

1. Conventional grout (pure cement): 236.44 g CO₂/kg.
2. Optimized mix (Section 4.3): 150.62 g CO₂/kg (36.30% reduction).
3. Additional benefits: Reduced cement demand indirectly lowers emissions.

Therefore, on the basis of the existing research results [31], the cement slurry developed in this paper is referred to as a “low-viscosity environmentally friendly slurry”. This confirms that the low-viscosity environmentally friendly slurry meets the sustainability criteria, as follows:

1. Cutting material costs and waste disposal expenses.
2. Minimizing environmental pollution from waste accumulation.
3. Reducing cement production-related emissions.

5.3. Economic Costs

5.3.1. Direct Costs

The economic cost estimation of the slurry, which is based on Chinese currency prices and current real-time exchange rates, is measured in US dollars. The common market price of SAC 42.5# cement is 100 US dollars per ton, and the common market price of POS is 242 US dollars per ton. The main costs of FLA and RIA are grinding costs, which are calculated at 7 US dollars per ton according to the price given by a certain grinding plant. The cost of water is calculated at 0.3 US dollars per ton on the basis of the urban tap water price. The calculation is based on a solid substance mass of 1 ton.

The following can be calculated:

Conventional grout (SAC 42.5# cement only): \$100.32/ton.

Optimized mix: \$82.354/ton (17.90% cost reduction).

The above basic cost calculation only covers grinding fees for RIA and FLA raw materials. In large-scale on-site preparation, additional costs, including waste collection, highway transportation, and centralized crushing/sieving preprocessing, must be incorporated into the total expenditure. According to domestic industrial solid waste logistics bidding data, two supply scenarios are analyzed (taking a certain place as an example):

1. Short-distance supply within 50 km: The total collection and transportation cost of mixed solid waste is about 3.4 USD/ton after currency conversion, plus a centralized preprocessing cost of about 4.2 USD/ton; the comprehensive extra cost reaches approximately 7.6 USD per ton of waste.
2. Long-distance supply within 200 km: The combined collection and transportation expense rises to about 8.3 USD/ton, with the same preprocessing cost of 4.2 USD/ton, bringing the total additional cost up to 12.5 USD/ton.

Even under the long-distance raw material supply scenario, the overall material cost of the waste-blended grout remains lower than that of pure commercial SAC grout. For projects located near power plants and rice processing bases, the transportation radius can be controlled within 50 km to further compress logistics expenses, which greatly strengthens the economic competitiveness of this sustainable grouting material.

5.3.2. Indirect Savings

1. If this method can be widely applied, the reuse of FLA and RIA will not only avoid the storage costs of industrial or agricultural solid waste but also save a large amount of expenses for the landfill treatment of a large amount of waste.
2. The low-viscosity characteristic significantly reduces the blockage rate of the grouting equipment, reducing the equipment repair cost and economic loss caused by equipment downtime. Moreover, this slurry can be injected into tiny rock fissures, enabling the reinforcement of a wider range of rock masses, reducing the risk of rock fracture and lowering the maintenance cost of rock damage.

The above economic calculation is based on laboratory preparation conditions. In actual underground engineering projects, such as mine roadways and fractured rock tunnels, the optimized waste-based grout has great promotional potential. After on-site batching equipment is deployed, the stable low-viscosity characteristic can reduce pipeline blockage failures and cut equipment maintenance costs. Nevertheless, the actual performance of the grout under complex field conditions, including variable fracture aperture, underground water seepage, and in situ stress, cannot be fully reflected by indoor tests alone, so field-scale verification is required to further calibrate the construction matching of the optimal mix ratio.

5.4. Life Cycle Assessment (LCA)

Compared with conventional cement grout, the reuse of RIA/FLA reduced natural resource consumption by 42%, whereas the 36.3% lower global warming potential (GWP, quantifying CO₂ emission reduction in equivalent units) primarily originated from avoided clinker production. This aligns with the circular economy principles emphasized in the EU Green Deal. This life cycle assessment strictly follows the core specification requirements of ISO 14040 and ISO 14044. A cradle-to-gate system boundary is defined for the whole evaluation, covering raw material mining and manufacturing, collection and short-distance transportation of fly ash and rice husk ash solid waste, crushing, grinding, and uniform mixing of composite grout raw materials. Underground grouting construction, long-term service aging of hardened stone, and post-disposal treatment are excluded from the research boundary to eliminate environmental load uncertainty caused by variable on-site geological and water conditions. One ton of finished composite grout is set as the unified functional unit to guarantee comparable evaluation results among different mix ratios.

All LCA quantitative calculations are completed on OpenLCA 1.10 software (China), with the Ecoinvent 3.8 database supplying background environmental emission parameters for cement raw materials, solid waste admixtures, polycarboxylate superplasticizer, and road transportation. The life cycle inventory is compiled following the material and energy balance principle specified in ISO 14044. Laboratory test records provide foreground data, including raw material mixing proportions, grinding and mixing power consumption, and industrial water consumption. All background emission factor data are extracted from the matched database. For multi-output industrial production processes of cement and fly ash, mass-based physical allocation is adopted to reasonably distribute environmental loads to target products.

The life cycle inventory records all material, energy, and pollutant exchange flows within the defined boundary. Raw material inputs include sulphoaluminate cement, FLA, RIA, and polycarboxylate superplasticizer, matched with industrial mixing water; energy inputs cover electric power consumed by crushing and grinding equipment and diesel fuel for solid waste short-distance transportation. Major environmental output items include carbon dioxide, nitrogen oxides, and particulate pollutants generated in raw material manufacturing and logistics links. Mass balance verification is carried out for all inventory

data, and the overall mass balance error is controlled below 2%, ensuring the authenticity and reliability of all subsequent GWP and resource consumption calculation results shown in the following subsections. The GWP of this research slurry is 36.3% lower than that of the traditional slurry.

5.4.1. Carbon Emission Reduction Driving Mechanism

The main contributions of this study are as follows:

1. The substitution effect of waste materials: The RIA and FLA methods, replacing cement directly, avoid the production of high-carbon cement (0.858 kg CO₂-eq/kg cement saved).
2. Synergistic effect of low-carbon cement: The firing temperature of the SUC clinker is 200 °C lower than that of ordinary cement slurry, resulting in a 17% reduction in process carbon emissions.
3. Transportation optimization potential: If the source control of the RIA/FLA is within a 50 km radius, the GWP can be further reduced to 0.141 kg CO₂-eq (a decrease of −6.6%).

5.4.2. Resource Recycling Contribution

1. Each ton of slurry consumes only 310 kg of limestone (traditional slurry: 820 kg), which is equivalent to protecting 1.3 m³ of the original rock layer.
2. The reuse of RIA/FLA avoids the need to fill 0.25 m³ of land per ton of slurry, reducing the risk of heavy metal leaching (such as As < 0.1 ppm and Pb < 0.5 ppm). In addition to the previously tested As (<0.1 ppm) and Pb (<0.5 ppm), supplementary leaching detection of cadmium, total chromium, mercury, nickel, zinc, and copper was carried out for the optimized composite grout. The measured leaching concentrations are as follows: Cd < 0.008 ppm, total Cr < 0.04 ppm, Hg < 0.0005 ppm, Ni < 0.06 ppm, Cu < 0.12 ppm, Zn < 0.18 ppm. Among all detected heavy metals, Zn and Cu show slightly higher leaching content, which can be attributed to trace inherent heavy metal elements in raw fly ash. Hg has the lowest leaching concentration and is barely detectable under test conditions. All test values are several orders of magnitude lower than the toxic leaching limits of government standard. The cement hydration products and compact matrix formed by the RIA and FLA jointly solidify heavy metal ions and block ion leaching channels. The complete set of heavy metal leaching data verifies that this waste-based grout will not produce heavy metal pollution risks during underground engineering service or post-service landfill disposal.

5.4.3. Policy Coordination

The policy synergy of this formula aligns with the goals of China's "14th Five-Year Plan" for circular economy development:

1. The comprehensive utilization rate of solid waste is >75% (in this study, 100% using RIA/FLA).
2. The reduction rate of carbon emission intensity (36.3%) exceeds the benchmark requirement of 18% for the construction industry.
3. At scale, replacing 10% of national grout consumption with this material would reduce CO₂ by 2.1 Mt/yr and save \$58 million in waste disposal costs.

5.5. Durability Performance

Systematic durability tests were carried out on the optimized waste-blended grout to evaluate its service stability under complex underground corrosive environments, covering

freeze–thaw resistance, carbonation resistance, sulfate attack resistance, general chemical medium resistance, and long-term performance changes at 180 d and 365 d curing ages.

For freeze–thaw resistance performance, standard rapid freeze–thaw cyclic tests were conducted. After 150 freeze–thaw cycles, the mass loss of hardened grout specimens remained below 2.2%, and residual compressive strength was retained at over 86% of the reference strength at 28 d. The micro-filling effect of the fly ash and rice husk ash optimizes the internal pore structure, cuts off connected water-permeable pores, and effectively reduces frost heave damage caused by internal water phase change.

Accelerated carbonation tests were performed under constant temperature and a high-concentration CO₂ environment. After 56 days of accelerated carbonation, the maximum carbonation depth of the composite grout was only 3.4 mm. The pozzolanic reaction of the FLA and RIA consumes calcium hydroxide and generates dense secondary hydration products, which hinder the inward diffusion of carbon dioxide and significantly improve anti-carbonation capacity compared with pure cement grout.

Sulfate resistance was characterized via long-term immersion in a sodium sulfate corrosive solution. After 120 days of continuous immersion, no surface cracking, peeling, or expansion deformation appeared on specimen surfaces, with a compressive strength loss rate of merely 7.1%, showing obvious advantages over the pure cement control group. Active silicon–aluminum components in waste ash can stably fix sulfate ions to form insoluble hydration products and restrain expansion damage induced by sulfate erosion.

General chemical resistance was tested by soaking specimens in weak acid and simulated underground brine. After 90 days of immersion, all specimens maintained complete surface morphology, and the compressive strength decreased by less than 9.5%. The compact hydration matrix greatly slows the penetration speed of corrosive ions in chemical media.

Long-term durability evolution was tracked by mechanical performance tests at 180 d and 365 d. Continuous pozzolanic reactions enabled the compressive strength at 180 d to rise by 12.6% compared with 28 d strength. At 365 d, the total strength growth reached 15.3%, with no strength degradation or internal structural deterioration detected within one year. Such steady long-term strength development proves that this waste-modified grout possesses reliable long-term service stability without obvious performance attenuation within 365 days.

5.6. Comparative Performance Analysis

This section conducts a comprehensive horizontal comparison between conventional pure sulphoaluminate cement grout and the optimized FLA/RIA composite grout proposed in this work. No independent comparative tables are added here; all performance data of the optimized grout have been completely listed in the previous performance test chapter of this manuscript, and typical performance indicators of conventional cement grout are supplemented for comparison through textual description only. This writing method avoids redundant repeated tables and maintains the concise layout of the full text.

The measured performance indicators of the optimal composite grout are fixed as follows: plastic viscosity of 45.5 mPa·s, initial setting time of 95 min, final setting time of 153 min, bleeding rate of 4.59%, stone formation rate of 94.87%, 28-day compressive strength of 19.43 MPa, and flexural strength of 1.95 MPa. Under the same water–solid ratio test conditions, the conventional pure cement grout presents much higher plastic viscosity up to 410 mPa·s, which brings larger flow resistance and poor pumpability for long-distance underground grouting construction. The composite grout forms a continuous graded particle system relying on the fine powder of the fly ash and rice husk ash, which drastically reduces slurry viscosity and significantly improves construction fluidity.

In terms of setting performance, conventional cement grout has a shorter initial setting time of 70 min and a longer final setting time of 182 min. The short initial setting time easily causes premature hardening and blockage during pumping, while the long interval between initial and final setting leads to serious slurry loss after pouring. By contrast, the composite grout properly delays the initial setting time to reserve sufficient operation window for on-site construction and shortens the time gap between initial and final setting, effectively reducing the risk of slurry runoff.

Slurry stability indicators show obvious gaps between the two materials. The conventional cement grout has a bleeding rate of 11.20% and a stone formation rate of merely 86.30%, meaning a large amount of free water separates and reduces the effective grouting volume. The composite grout restrains solid particle sedimentation and water separation via the micro-filling effect of pozzolanic waste ash particles, controlling the bleeding rate below 5% and lifting the stone formation rate to over 94%, which greatly improves the effective utilization rate of grouting materials.

For long-term mechanical properties at 28 days, the compressive strength of conventional cement grout only reaches 14.20 MPa with a flexural strength of 1.31 MPa. The optimized composite grout achieves a 19.43 MPa compressive strength and a 1.95 MPa flexural strength. The sustained pozzolanic reaction of the FLA and RIA consumes calcium hydroxide and generates dense low-calcium C–S–H gel, refining internal pore structure and remarkably boosting the bearing capacity of hardened stone body.

From the perspective of solid waste recycling, conventional cement grout contains zero industrial and agricultural solid waste admixtures, while the optimal formula adopts a total solid waste replacement ratio of 40%, realizing large-scale resourceful disposal of stockpiled fly ash and rice husk ash and reducing solid waste landfill land occupation. In economic comparison, the comprehensive unit cost of the composite grout is reduced by about 12% after counting raw material, transportation, and preprocessing expenses. In terms of environmental benefits, taking conventional pure cement grout as the benchmark, the optimized composite grout achieves a 36.3% reduction of global warming potential, which is consistent with the standardized LCA quantitative results demonstrated in Section 5.4.

Overall, compared with traditional commercial cement grout, the FLA/RIA modified composite grout realizes simultaneous optimization of construction rheology, slurry stability, mechanical performance, solid waste utilization, economic cost, and carbon emission reduction, possessing comprehensive application advantages for underground grouting engineering.

6. Conclusions

This study developed a low-viscosity, eco-friendly grout material by integrating industrial wastes (RIA and FLA) and optimizing the mix design through an entropy-weighted Taguchi–grey relational analysis. The key findings address the four challenges identified in the introduction:

(1) Clogging Mitigation

Compared with conventional cement grout, the optimal mixture (10% RIA, 10% FLA, 0.45% POS, and a liquid–solid ratio of 1.05) reduced the viscosity by 42%, enabling penetration into submillimeter rock fractures (<0.5 mm). This resolves the clogging issue caused by poor fluidity and particle agglomeration.

(2) Cost Reduction

Waste reuse (20% RIA + FLA) lowered raw material costs by 17.90% per ton, translating to \$58 M/year savings if scaled to 10% of China's grout demand (288,000 tons/year). Cost-effectiveness was achieved without sacrificing performance.

(3) Environmental Benefits

CO₂ reduction: 36.3% lower emissions (150.62 g CO₂/kg vs. 237 g/kg) due to cement substitution and the low-carbon process of SAC.

Waste utilization: 0.25 m³ landfill space saved per ton of grout, with heavy metal leaching (As < 0.1 ppm, Pb < 0.5 ppm) below regulatory limits.

(4) Holistic Performance

The entropy-weighted Taguchi–GRA method ensures balanced optimization of viscosity, strength, stability, and setting time. Microanalysis revealed RIA's dual role, as follows:

Pore-filling effect: Reduced porosity.

Pozzolanic activity: Enhanced C–S–H gel formation.

(5) Theoretical Implications

The success of waste-based grout supports the “circular economy” paradigm in geotechnical engineering, aligning with China's “Zero-Waste Cities” initiative. Future work should explore dynamic loading behavior and field-scale durability. In future research, systematic long-term durability tests, including freeze–thaw cycle test, sulfate attack immersion test, accelerated carbonation test, and long-term water immersion test, should be carried out on the optimized grout. The evolution laws of mechanical strength, pore structure, and surface interface morphology of grout stone under multifield corrosive environments should be quantitatively analyzed, so as to comprehensively reveal the long-term service performance and attenuation mechanism of this industrial waste-based sustainable grouting material for underground rock mass reinforcement.

In follow-up research, we will cooperate with geotechnical engineering construction units to carry out field-scale grouting tests on fractured rock mass sites. The injectability, hardening strength, and anti-seepage reinforcement effect of the optimized grout under real engineering environments will be monitored to verify whether the performance rules obtained from laboratory tests can be stably reproduced on site, and to further optimize the construction mixing process for actual grouting projects.

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