

Article

Experimental Study on Ratio Optimization and Nonlinear Response Characteristics of Grouting and Fire-Protecting Filling Material Coal Mining Area

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Abstract

In order to improve the fluidity, pumpability, and strength of separation-layer grouting fire-protecting filling material and reliability with multiple parameters and factors in traditional orthogonal tests, the coupling theory of the response surface-satisfaction function is applied to optimize the ratio of separation-layer grouting fire-protecting filling material. Cement content, the ash–gangue ratio, slurry concentration, and admixture were selected as the influencing factors for the ratio optimization of separation-layer grouting fire-protecting filling material and slump, with the bleeding rate and compressive strength selected as the evaluation indexes of material properties. The Box–Behnken experimental design method was applied to conduct 25 groups of experiments with different material ratios, and the response surface functions of various material performance evaluation indexes were constructed. The relationship between the influencing factors of fire protecting and filling material ratios and the target responsiveness was studied, as well as the optimal ratio of separation-layer grouting fire-protecting filling materials under multi-objective conditions. The results show that the influence of the slurry concentration and cement content on the degree of collapse is significant. The cement content and slurry concentration had significant influence on the compressive strength. The ash–gangue ratio has a significant impact on bleeding rate. Meanwhile, the interaction of the ash–gangue ratio, slurry concentration, and cement content also has a significant impact on the bleeding rate. For waste rock cementation abscission-layer grouting fire protecting and filling material, the optimal ratio is an ash and gangue ratio of 1:2, the cement content is 12.12%, the admixture is 1.49%, and the slurry concentration is 52%. The ratio of the corresponding response under the condition of prediction result is a slurry slump of 28.5 cm, bleeding rate of 2.36%, and filling body strength of 4.62 MPa, which basically coincide with the experimental results and verification and provide evidence for the abscission layer grouting field industrial test.

Keywords: fire protecting and filling material; response surface method; satisfaction function; matching; multi-objective optimization



Academic Editor: Thomas H. Fletcher

Received: 25 September 2025

Revised: 23 October 2025

Accepted: 28 October 2025

Published: 31 October 2025

Citation: Chen, Z.; Shi, J.; Zhang, Z.; Li, L. Experimental Study on Ratio Optimization and Nonlinear Response Characteristics of Grouting and Fire-Protecting Filling Material Coal Mining Area. *Fire* **2025**, *8*, 430.

<https://doi.org/10.3390/fire8110430>

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1. Introduction

With a large number of coal resources mined out, the shallow coal seam has been almost exhausted. Coal enterprises had to develop deep mining, and mining depth gradually increased [1]. With the increase in mining depth and intensity, the risk of mine

fires has become increasingly severe, with high temperature and fire hazards significantly aggravated, posing new challenges to the structural stability and safe operation of deep mines [2]. Affected by the mining of adjacent working faces and the variation in underground ventilation conditions, the roadway surrounding the rock and filling structures under high-temperature conditions are more prone to coal wall spalling, roof caving, and floor heave, which seriously threaten mine personnel, transportation systems, and ventilation safety and constitute a major hidden danger to the continuous and safe production of coal mines [3]. In order to effectively eliminate the adverse effects of floor heave, spalling, and structural deformation caused by fire and high temperatures, fireproof grouting reinforcement technology has gradually become one of the important means of structural safety protection in high-temperature mining environments. The slurry is injected into the fractures of broken coal and rock mass by grouting equipment. After curing, the slurry consolidates the originally loose and broken coal and rock structures into a complete whole, alters their original structural characteristics, repairs mechanical defects, and thereby improves the overall mechanical and thermal stability of surrounding rock mass under high-temperature conditions [4].

Layered grouting reinforcement technology is a grouting reinforcement method in which drilling is performed from the surface, and the grouting system is separated from the production system [5]. This technology does not require the modification of underground mining processes and is suitable for reinforcement and fire prevention operations in deep, high-temperature, and high-risk areas, contributing to efficient, safe, and green coal mining. In recent years, more and more coal mining enterprises have attempted to apply this technology to structural fireproof reinforcement in high-temperature zones. Since grouting and filling can effectively block heat transfer and control thermal damage, its application value in key roadways, goaf areas, and “three-under” mining regions has gradually emerged. At present, scholars’ research mainly focuses on thermal insulation mechanisms, grouting process parameters, and temperature rise control effects (e.g., fire resistance limit and heat transfer efficiency), and many valuable results have been achieved. Research methods mainly include similar material simulation, high-temperature numerical simulation, and on-site testing under fire conditions.

At present, domestic and foreign researchers have carried out extensive explorations on the applicability of layered grouting reinforcement technology in high-temperature mine fire environments. Research mainly focuses on the performance regulation of grouting materials under high-temperature conditions, the mechanical response mechanism of the grouting system, and engineering application verification under complex geological and disaster coupling conditions. In terms of material performance research, some scholars have conducted experimental studies on the coupling mechanism of temperature and humidity in mine fire environments, preparing grouting reinforcement samples by simulating typical fire scenarios such as high temperatures and high humidity and systematically analyzing the cementation performance, thermal deformation resistance, and structural stability of the slurry under different temperature gradients and moisture conditions [6]. In terms of grouting mechanisms, researchers have constructed mechanical models of layered grouting systems based on elastic thin plate theory, deeply analyzing the load response characteristics of grouting structures under thermo-mechanical coupling and their relationship with structural parameters, which provide a theoretical basis for grouting design and stability evaluation in high-temperature disaster scenarios [7]. To address the challenges of complex surrounding rock and fracture development conditions on grouting adaptability in actual fire environments, some studies have also proposed a composite grouting strategy combining layered and gap grouting so as to improve the compactness,

continuity, and thermal deformation resistance of grouting filling in high-temperature areas and effectively control settlement and thermal damage in the grouting zone [8].

Meanwhile, some engineering practices have verified the feasibility of this technology under complex overburden conditions in coal mines. For example, a continuous, integrated grouting mode has been proposed for the efficient filling of separation zones in goaf areas and has been successfully applied [9]. Other studies have focused on rock burst prevention and explored the use of separation grouting to restrict overburden movement induced by high stress, providing a new approach for disaster prevention in high-risk areas [10]. However, in meeting the complex requirements of high temperatures and high in situ stress in coal mines, the adaptability of existing grouting materials still needs further optimization and improvement. High-quality grouting materials should possess good thermal insulation, fluidity, cementation, and stability under high-temperature conditions, ensuring that the filling body can perform multiple functions such as cementing, supporting, compacting, buffering, and resisting thermal expansion, which is a key guarantee for mine heat hazard control and disaster prevention [11,12].

In some engineering practices, grouting materials for coal mines mainly include a slurry prepared by mixing fly ash and water in different proportions, as well as high-water fire protecting and filling materials composed of different components [13]. However, the application of high-water grouting materials in fire environments still has certain limitations, so fly ash and coal gangue are generally used as the main aggregates in engineering practice. Practical results show that these materials are effective in slowing down temperature rise and enhancing structural stability, but several problems also exist: (1) Fly ash is light and difficult to control during transportation and grouting; it easily spills or disperses as dust, causing environmental pollution; (2) the quality of fly ash in power plants fluctuates greatly, with high surface porosity and strong water adsorption, leading to increased slurry viscosity, poor fluidity, higher risk of pipeline blockage, and poor continuity of grouting [14]; (3) the cementation performance of the fly ash slurry is insufficient under high-temperature conditions, and large amounts of water may precipitate, affecting the compactness of filling structures, and in severe cases, even causing leakage or collapse risks [15]; and (4) although high-water fire protecting and filling materials have the advantages of good fluidity, a short setting time, and early strength, they suffer from low shrinkage, high brittleness, easy fracture, low long-term strength, and poor thermal stability [16–18].

Despite growing attention to the optimization of fireproof grouting materials, most existing studies have focused on single-factor effects or assumed simplified linear relationships between material components and performance indicators, which restrict their applicability under the complex thermal–mechanical coupling conditions of deep coal mines. Few works have quantitatively investigated the multi-parameter interactions among cement, fly ash, coal gangue, and admixtures, particularly under fire or high-temperature environments. Moreover, conventional optimization methods such as orthogonal or single-objective approaches are limited in their ability to simultaneously consider multiple responses—such as slump, bleeding rate, and compressive strength—thus hindering their effectiveness in practical design.

To bridge these gaps, this study proposes a fireproof grouting material system that utilizes coal gangue and fly ash as aggregates and cement as the binding material, aiming to achieve high fluidity, strong thermal resistance, and long-term structural stability for underground coal mine reinforcement. More importantly, a coupled response surface-satisfaction function (RSM–SF) framework is developed to perform multi-objective nonlinear optimization, enabling the simultaneous quantitative analysis of the coupled effects among cement content, the ash–gangue ratio, slurry concentration, and admixture. Through

the establishment of predictive response equations verified by 25 Box–Behnken experiments, the proposed method enhances both the scientific interpretability and engineering practicality of mixed proportion design. This research thus provides a systematic and generalizable approach for the optimization of fireproof grouting materials, filling a methodological and application gap in the study of grouting systems under high-temperature mining conditions.

2. Materials and Methods

2.1. Response Surface-Satisfaction Function Method Optimization Theory

2.1.1. Response Surface Theory

The response surface theory is to present the experimental data in the form of coordinate graphs by fitting equations under multi-objective conditions so as to obtain the influence of each element on the response value. The second-order response surface function is as follows:

$$y = g(x_1, x_2, \dots, x_n) = \sum_{i=1}^n \beta_{ii}x_i^2 + \sum_{i=1}^{n-1} \sum_{j=1}^n \beta_{ij}x_ix_j + \sum_{i=1}^n \beta_i x_i + \beta_0 \quad (1)$$

Among them, y is the response, x_i is the influence factor, and n is the number of influence factors. β_{ii} , β_{ij} , β_i , β_0 is an unknown parameter.

If m experiments are designed, Y and ϵ are the dependent variable and the error, respectively; then, the response surface function can be expressed as follows [19]:

$$Y = \beta X + \epsilon \quad (2)$$

This can be simplified by

$$\beta = (X^T X)^{-1} X^T Y \quad (3)$$

Bringing the data obtained from the experiment into Equation (3), we can find β and then substitute it into Equation (1) to obtain the desired function, which is the response surface function.

2.1.2. Satisfaction Function

Because there are multiple responses, when performing satisfaction optimization, the multi-objective optimization method is adopted, taking $d_i(y_i(x))$ as the response satisfaction, where i is the i th response and the satisfaction value is 0~1. When the response value is directly proportional to the satisfaction, it can be expressed by Formula (4a); when the response value is inversely proportional to the satisfaction, it can be expressed by Formula (4b); and Equation (4c) is applicable when the response volume has a target value, and the closer to the target value, the higher the satisfaction. In the formula, L_i , U_i , and T_i represent the lower limit, upper limit, and target response value of the range of the i th response volume, respectively. s and t represent the strictness of the response value based on the objective optimization value. The single response satisfaction function is as follows:

$$d_i(y_i(x)) = \begin{cases} 0, & y_i(x) < L_i \\ (\frac{y_i(x)-L_i}{U_i-L_i})^s, & L_i \leq y_i(x) \leq U_i \\ 1, & y_i(x) > U_i \end{cases} \quad (4a)$$

$$d_i(y_i(x)) = \begin{cases} 0, & y_i(x) < L_i \\ (\frac{y_i(x)-L_i}{U_i-L_i})^t, & L_i \leq y_i(x) \leq U_i \\ 1, & y_i(x) > U_i \end{cases} \quad (4b)$$

$$d_i(y_i(x)) = \begin{cases} 0, & y_i(x) < L_i \\ (\frac{y_i(x)-L_i}{T_i-L_i})^s, & L_i \leq y_i(x) < T_i \\ 1, & y_i(x) = T_i \\ (\frac{U_i-y_i(x)}{U_i-T_i})^t, & T_i < y_i(x) \leq U_i \\ 0, & y_i(x) > U_i \end{cases} \quad (4c)$$

Therefore, the overall satisfaction D can be calculated by Formula (5):

$$D = (\prod_{i=1}^k d_i^{r_i})^{\frac{1}{r_1+r_2+\dots+r_k}} \quad (5)$$

In the formula, r_i represents the weight of the response and k represents the number of responses.

2.2. Fire-Protecting Filling Material's Physical and Chemical Properties

The grouting materials selected in this experiment are cement, fly ash, coal gangue, and grouting admixture. The cement is selected from Lingyuan 42.5 R ordinary cement produced by Shandong Chongzheng Special Cement Co., Ltd. (Zibo, China), with a density of 3.21 g/m³, a specific surface area of 413 m²/kg, and a standard consistency of 23.7% water consumption. Chemical composition is SO₃ 2.0%, MgO 1.8%, 3CaO·SiO₂ 44.2%, and 3CaO·Al₂O₃ 0.8%; loss on ignition is 1.5%; and 28 days compressive strength is 53.9 MPa. The gangue is taken from the gangue after the third-level crushing of a mine from the Zi Mining Group, and the chemical composition analysis is carried out by X-ray fluorescence spectrometer (XRF), in which SiO₂ 51.76%, Al₂O₃ 40.45%, TiO₃ 1.19%, CaO 1.34%, MgO 0.29%, K₂O 0.47%, TiO₂ 1.03%, etc. For fly ash, power plant grade II fly ash is used, with a fineness of 11.7%, a loss on ignition of 2.96%, a water requirement ratio of 106%, SO₃ 4.15%, CaO 15.13%, and free CaO 4.96%. In order to improve the fluidity and micro-expansion of the slurry and ensure the durability of the filling body, the grouting admixture is used. The CGM high-strength, non-shrinking grouting material produced by Shandong Kaishun New Building Materials is composed of silicate, lignin, acrylamide epoxy resin, high water-reducing polycarboxylic acid, high-carbon alcohol fatty acid ester compound, etc.

The admixture was used in a dosage range of 1–5% by weight of the total cementitious material. Its typical formulation includes approximately 40–50% silicate minerals acting as the primary binder modifier, 15–20% lignin-based dispersant to improve slurry fluidity, 10–15% acrylamide epoxy resin to enhance micro-expansion and bonding, and 5–10% polycarboxylate superplasticizer for water reduction. Minor additives such as high-carbon alcohol fatty acid esters (<5%) were included to improve early-age stability and reduce segregation. These proportions represent the manufacturer's recommended ranges for the CGM system and ensure consistency across the experimental batches.

A total of 25 groups of tests were prepared. After the slurry was evenly mixed, the slump and bleeding rate were measured, and 3 test blocks were poured. The specifications were (150.0 × 150.0 × 150.0) mm³. The electro-hydraulic servo was controlled by MTS Microcomputer. The compressive strength was measured using a universal testing machine.

2.3. Experimental Design

The Box–Behnken design (BBD) was selected in this study because it provides a highly efficient response surface methodology for four independent factors at three levels, while requiring fewer experimental runs than the central composite design (CCD). The BBD arranges experimental points within a defined cube but avoids combinations where all factors simultaneously reach extreme levels, thereby reducing the likelihood of non-

physical or unstable mix proportions. This feature makes the BBD particularly suitable for material optimization studies in which extreme compositions may lead to segregation or rapid setting. In addition, 25 experimental runs were sufficient to establish second-order regression equations with a good predictive capability while maintaining experimental economy and repeatability.

Fire protecting and filling material performance parameters such as filling body strength, bleeding, stratification, pumpability, fluidity, and fire protecting and filling material cost all reflect the quality of the fire protecting and filling material. In engineering practice, the factors that affect the performance of the fire protecting and filling material are slurry concentration, cement content, density, gradation, temperature, grouting additive type, etc. In this regard, this paper focuses on the study of the impact of cement content, the ash–gangue ratio, slurry concentration, and grouting admixture on the three responses of the compressive strength, slump, and bleeding rate of the filling body and the optimal ratio. The design is carried out using four factors and three levels, as shown in Table 1. The code value is calculated as follows:

$$X_i = (x_i - x_0) / \Delta x_i \quad (6)$$

Table 1. Factors and levels in response surface analysis.

Influencing Factors	Coded Value	Coding Level		
		Low (−1)	Center (0)	High (1)
cement content	X_1	5.00	10.00	15.00
ash-gangue ratio	X_2	0.5	1.25	2.00
slurry concentration	X_3	52	55	58
grouting admixture	X_4	1.00	3.00	5.00

Among them, x_i , X_i , and x_0 respectively represent the increment value, coding value, and value at the center of each factor. A total of 25 sets of experiments were conducted. The experimental results are shown in Table 2. To quantify measurement variability, all experimental results in Table 2 are expressed as mean $\pm$ standard deviation (SD) based on three repeated tests. The ANOVA results in Table 3 are derived from these averaged values and are thus presented without SD to maintain statistical consistency.

In this study, the grouting admixture (X_4) was treated as a single factor representing the total dosage of a fixed-composition composite additive. The admixture was produced by the same manufacturer in a single batch to ensure consistent internal composition, while only its overall dosage (1–5 wt%) relative to the total binder mass was varied among experimental runs. Thus, the internal proportions of its constituents—silicate minerals, lignin-based dispersant, acrylamide epoxy resin, and polycarboxylate superplasticizer—remained constant throughout all experiments. This approach isolates the effect of dosage on material properties without introducing additional uncertainty from component variability.

Table 2. Test results of mixture ratio of fire protecting and filling material.

Serial Number	Cement Content (%)	Ash–Gangue Ratio	Slurry Concentration (%)	Grouting Admixture (%)	Slump (cm) $\pm$ SD	Bleeding Rate (%) $\pm$ SD	Compressive Strength (MPa) $\pm$ SD
1	15.00	1.25	55.00	1.00	23 $\pm$ 0.3	3.7 $\pm$ 0.1	5.3 $\pm$ 0.2
2	15.00	1.25	58.00	3.00	19 $\pm$ 0.2	3.3 $\pm$ 0.05	5.8 $\pm$ 0.1
3	5.00	1.25	55.00	5.00	23 $\pm$ 0.4	2.6 $\pm$ 0.1	3.9 $\pm$ 0.2
4	10.00	0.50	52.00	3.00	26 $\pm$ 0.3	1.9 $\pm$ 0.1	4.3 $\pm$ 0.15
5	6.00	1.25	58.00	3.00	22 $\pm$ 0.2	3.4 $\pm$ 0.05	5.1 $\pm$ 0.25
6	5.00	1.25	58.00	3.00	19 $\pm$ 0.3	3.4 $\pm$ 0.1	3.8 $\pm$ 0.2
7	15.00	2.00	52.00	3.00	21 $\pm$ 0.2	3.5 $\pm$ 0.1	5.1 $\pm$ 0.2
8	10.00	2.00	52.00	5.00	23 $\pm$ 0.3	3.0 $\pm$ 0.1	4.8 $\pm$ 0.2
9	5.00	0.50	58.00	3.00	24 $\pm$ 0.3	2.4 $\pm$ 0.1	4.6 $\pm$ 0.2
10	15.00	1.25	55.00	5.00	22 $\pm$ 0.3	3.8 $\pm$ 0.2	5.3 $\pm$ 0.2
11	5.00	1.25	52.00	3.00	23 $\pm$ 0.4	3.2 $\pm$ 0.1	4.5 $\pm$ 0.2
12	6.00	0.50	55.00	3.00	24 $\pm$ 0.3	3.0 $\pm$ 0.1	4.9 $\pm$ 0.2
13	10.00	1.25	55.00	3.00	25 $\pm$ 0.3	2.9 $\pm$ 0.1	5.1 $\pm$ 0.2
14	5.00	1.25	58.00	5.00	23 $\pm$ 0.2	3.2 $\pm$ 0.1	5.2 $\pm$ 0.2
15	10.00	1.25	55.00	3.00	24 $\pm$ 0.3	3.1 $\pm$ 0.2	5.0 $\pm$ 0.3
16	6.00	2.00	52.00	3.00	22 $\pm$ 0.3	3.5 $\pm$ 0.1	4.7 $\pm$ 0.2
17	15.00	2.00	58.00	3.00	21 $\pm$ 0.3	3.8 $\pm$ 0.1	5.2 $\pm$ 0.2
18	10.00	1.25	55.00	3.00	23 $\pm$ 0.4	3.6 $\pm$ 0.2	5.3 $\pm$ 0.3
19	6.00	1.25	52.00	3.00	22 $\pm$ 0.3	3.0 $\pm$ 0.1	4.8 $\pm$ 0.2
20	5.00	1.25	58.00	3.00	19 $\pm$ 0.3	3.5 $\pm$ 0.1	3.7 $\pm$ 0.1
21	15.00	1.25	55.00	1.00	23 $\pm$ 0.3	3.2 $\pm$ 0.2	5.1 $\pm$ 0.2
22	10.00	1.25	55.00	3.00	24 $\pm$ 0.3	3.7 $\pm$ 0.1	5.2 $\pm$ 0.2
23	15.00	2.00	58.00	3.00	22 $\pm$ 0.3	3.9 $\pm$ 0.2	5.3 $\pm$ 0.3
24	5.00	2.00	52.00	3.00	22 $\pm$ 0.3	3.8 $\pm$ 0.1	5.0 $\pm$ 0.2
25	10.00	2.00	55.00	1.00	22 $\pm$ 0.4	6.2 $\pm$ 0.2	3.8 $\pm$ 0.2

Table 3. Analysis of variance with the regression model for different response surfaces.

Source of Variation	Sum of Square			Mean Square			F Value			p Value		
	Y ₁	Y ₂	Y ₃	Y ₁	Y ₂	Y ₃	Y ₁	Y ₂	Y ₃	Y ₁	Y ₂	Y ₃
Model	116.95	45.19	19.19	8.35	3.23	1.37	100.54	78.89	96.20	<0.0001	<0.0001	<0.0001
X ₁	0.17	3.495×10^{-3}	0.26	0.17	3.495×10^{-3}	0.26	2.01	0.085	18.42	0.1864	0.7761	0.0016
X ₂	2.30	0.73	0.18	2.30	0.73	0.18	27.71	17.96	12.31	0.0004	<0.0017	0.0057
X ₃	4.76	4.686×10^{-4}	0.12	4.76	4.686×10^{-4}	0.12	57.31	0.011	8.59	<0.0001	0.9169	0.0150
X ₄	0.25	0.068	0.13	0.25	0.068	0.13	3.04	1.67	9.33	0.1119	0.2258	0.0122
X ₁ X ₂	2.500×10^{-3}	0.063	0.040	2.500×10^{-3}	0.063	0.040	0.030	1.53	2.81	0.8657	0.2447	0.1248
X ₁ X ₃	0.12	0.000	0.022	0.12	0.000	0.022	1.47	0.000	1.58	0.2525	1.0000	0.2375
X ₁ X ₄	2.500×10^{-3}	0.023	0.022	2.500×10^{-3}	0.023	0.022	0.030	0.55	1.58	0.8657	0.4754	0.2375
X ₂ X ₃	0.72	0.040	0.12	0.72	0.040	0.12	8.70	0.98	8.60	0.0146	0.3461	0.0150
X ₂ X ₄	0.25	0.30	1.000×10^{-2}	0.25	0.30	1.000×10^{-2}	3.01	7.39	0.70	0.1135	0.0216	0.4218
X ₃ X ₄	0.063	0.023	2.500×10^{-3}	0.063	0.023	2.500×10^{-3}	0.75	0.55	0.18	0.4061	0.4754	0.6842
X ₁ ²	0.050	0.013	7.059×10^{-3}	0.050	0.013	7.059×10^{-3}	0.60	0.31	0.50	0.4550	0.5919	0.4976
X ₂ ²	0.060	3.137×10^{-3}	4.412×10^{-4}	0.060	3.137×10^{-3}	4.412×10^{-4}	0.72	0.077	0.031	0.4151	0.7875	0.8638
X ₃ ²	2.20	1.225×10^{-3}	0.099	2.20	1.225×10^{-3}	0.099	26.52	0.030	6.97	0.0004	0.8661	0.0248
X ₄ ²	5.931×10^{-3}	0.031	0.028	5.931×10^{-3}	0.031	0.028	0.071	0.75	1.98	0.7948	0.4071	0.1896
Residual	0.83	0.41	0.14	0.083	0.041	0.014						

3. Results

3.1. Response Surface Function Fitting

According to the experimental results, the response surface function between the influencing factors and the evaluation index is obtained through the theoretical fitting of the response surface [20].

$$Y_1 = 0.7425x_1 + 10.07037x_2 + 10.28102x_3 + 1.23125x_4 - 6.66 \times 10^{-3}x_1x_2 - 0.011667x_1x_3 - 2.5 \times 10^{-3}x_1x_4 - 0.18889x_2x_3 - 0.16667x_2x_4 - 0.020833x_3x_4 - 5.33 \times 10^{-3}x_1^2 - 0.25926x_2^2 - 0.098x_3^2 - 0.011458x_4^2 - 244.79942;$$

$$Y_2 = 0.0525x_1 + 5.55741x_2 - 0.31157x_3 - 0.72292x_4 - 0.0333x_1x_2 - 3.7285x_1x_3 + 7.5 \times 10^{-3}x_1x_4 - 0.0444x_2x_3 - 0.18333x_2x_4 + 0.0125x_3x_4 - 2.66667 \times 10^{-3}x_1^2 + 0.059259x_2^2 + 2.3148 \times 10^{-3}x_3^2 - 0.026042x_4^2 + 10.61192;$$

$$Y_3 = 0.4475x_1 + 3.88889x_2 - 2.10972x_3 - 4.16667 \times 10^{-3}x_4 + 0.026667x_1x_2 - 5.00 \times 10^{-3}x_1x_3 - 7.5 \times 10^{-3}x_1x_4 - 0.07778x_2x_3 - 0.03333x_2x_4 + 4.1667 \times 10^{-3}x_3x_4 - 2.000 \times 10^{-3}x_1^2 + 0.02222x_2^2 + 0.020833x_3^2 - 0.02500x_4^2 + 55.03194.$$

The experiment uses the R^2 test, that is, the method of fitting test to evaluate the significance of the model. The coefficient of R^2 indicates the difference between the actual value and the predicted value. Taking 0~1 as the standard, when it is equal to 1, it means that there is no difference between the two. The R^2 fitted by the three response surface functions in this experiment are 0.9929, 0.9910, and 0.9926. At the same time, the p value represents the size of the selected factors in the entire model. When $p > 0.05$, it means that the influencing factors are not significant. When p is between 0.0001 and 0.05, it indicates that the influencing factors are significant. When $p < 0.0001$, it indicates that the influencing factor is very significant. Perform a corresponding analysis of variance on the response surface regression model, as shown in Table 3. The p value of each model is less than 0.001, indicating that the model regression effect is significant; the experimental results have high reliability.

At the same time, according to the experimental data, the predicted values of different responses are used as the values on the vertical axis, and the actual values are the values on the horizontal axis. Make an (X,Y) scatter plot, if the distribution of the scattered points approximates a straight line and they are distributed around the $Y = X$ straight line at the same time, then the model can be considered to have a good fit. From Figure 1, it can be seen that the fit of the response surface of each index is better and the reliability is higher.

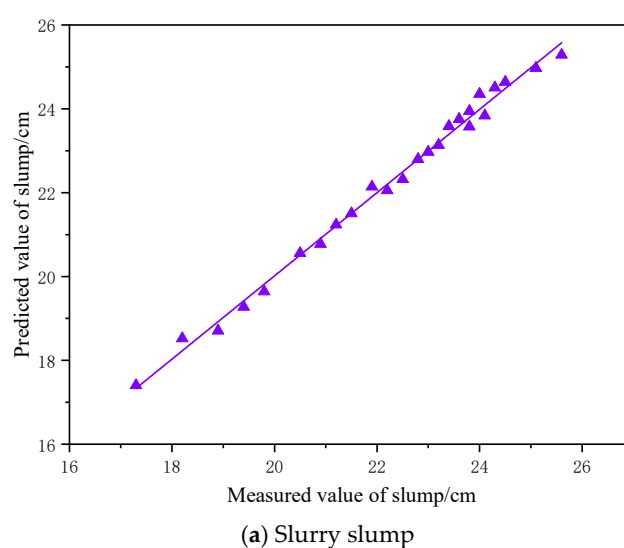


Figure 1. Cont.

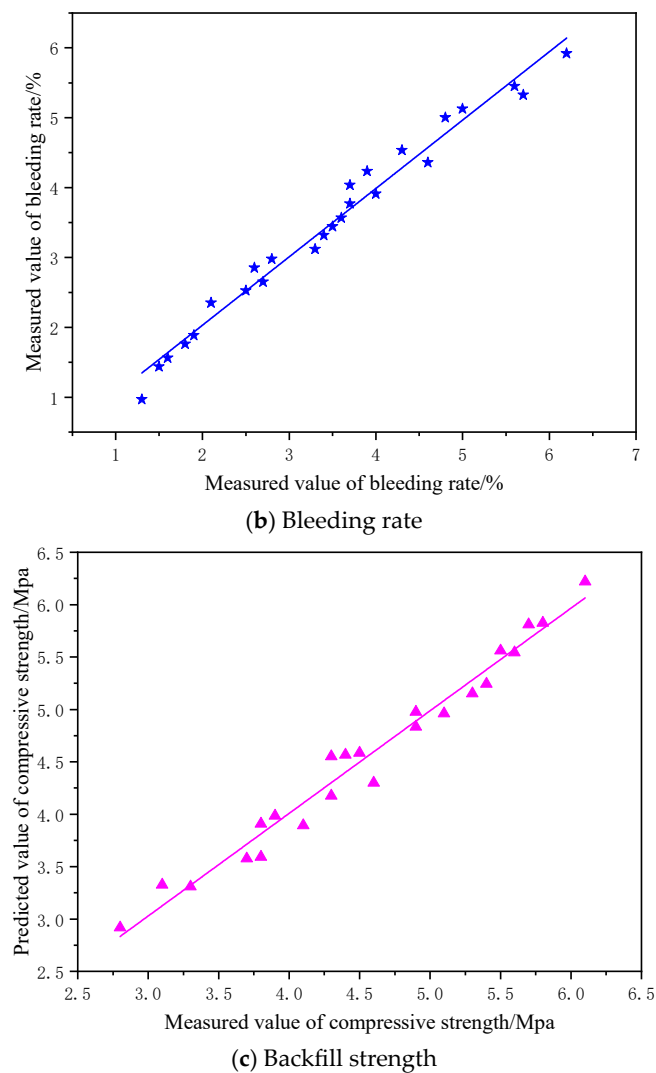
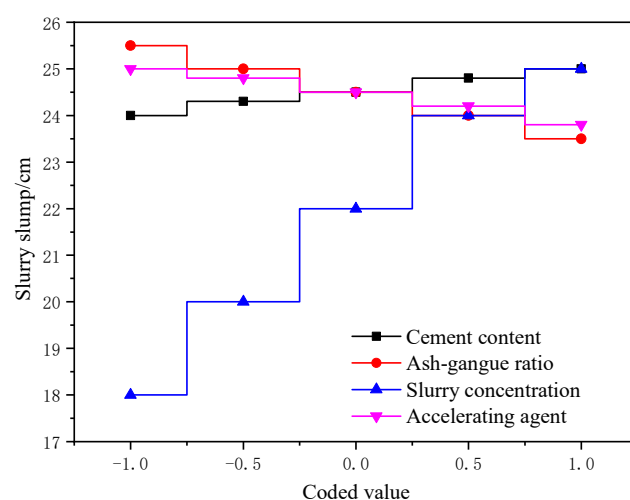


Figure 1. Comparison between the predicted value of different responses and the measured value.

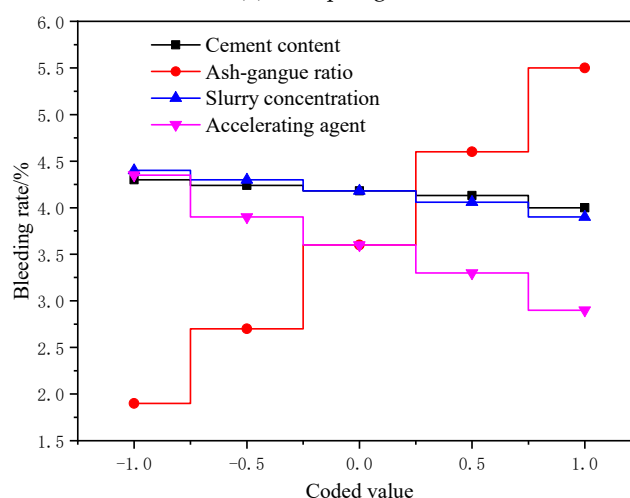
3.2. The Influence of Single-Factor Response Surface Parameters on the Performance of the Filling Body

In the slump response surface regression model, the interaction between various factors has little effect on it, and a single factor has a greater effect on it. Among them, the influence of the slurry concentration on slump is far greater than other factors; the effect of ash–gangue on the slump is more significant. However, the effects of cement content and grouting additives on the slump are not significant. As the slurry concentration increases, the slump significantly decreases. This is because as the slurry concentration increases, the viscosity increases, which in turn increases the friction and reduces slump [21]. Before the slurry concentration is 63%, the slump is close to 10 cm. After 63%, the slump decreased significantly. This shows that the slurry concentration should be appropriate. If the slurry concentration is too high, the fluidity of the slurry will be poor, and if it is too low, segregation will occur. As the ash ratio increases, the slump will slowly decrease. This is because the fly ash has a certain dependence on water. The more fly ash there is, the more water it absorbs, which reduces the fluidity, that is, the slump [22]. Increased cement content will cause a slight decrease in the slump. This is because cement will undergo a hydration reaction and generate hydrated crystals with better crystallinity, such as $\text{Aft}(\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O})$ and $\text{C}_3\text{AH}_6(3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 6\text{H}_2\text{O})$. They are needle-shaped, rod-shaped, and disordered. At the same time, the hydration reaction releases heat, which

further accelerates the hydration reaction and reduces the slump. The single factor of slump is shown in Figure 2a [23]. However, increasing the amount of cement used in coal mine filling and mining will significantly increase the cost of filling. Therefore, as long as the mechanical properties of the filling body meet the requirements, the amount of cement should be reduced as much as possible to reduce the material cost. The single factor of the bleeding rate is shown in Figure 2b. With the increase in the slurry concentration, the compressive strength has increased, and because the slurry concentration increases, the bleeding rate will decrease, which increases the compressive strength. As the ash ratio increases, the compressive strength will decrease. This is because the specific surface area of gangue is very large, the water absorption is enhanced, and the strength is reduced, making it difficult to form a stable system in the middle and late stages [24]. The grouting admixture also has an effect on the strength of the filling body. The single factor of compressive strength is shown in Figure 2c. With the increase in grouting admixture, it forms stable complexes with Ca^{2+} , Fe^{3+} , Al^{3+} , and other metal ions. These complexes form a soluble zone with the free water in the system, increase the diffusion rate of hydration products, shorten the incubation period of the hydration reaction of coal gangue and other cementing materials, and increase the compressive strength of the system at different ages.



(a) Slump degree



(b) Bleeding rate

Figure 2. Cont.

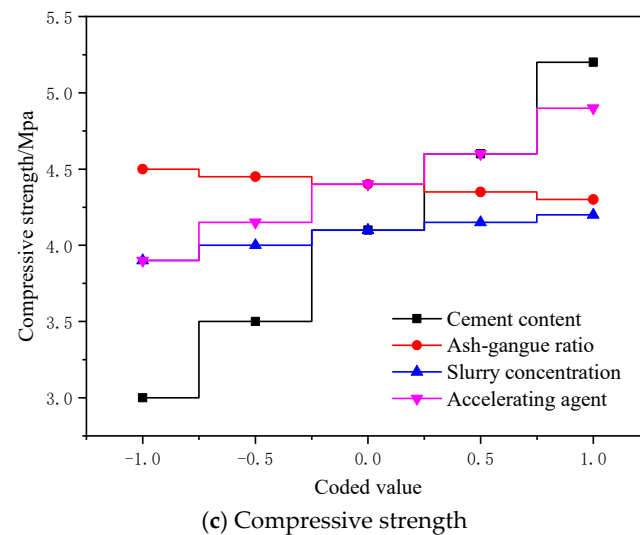


Figure 2. Single-factor influence analysis of different response quantities.

3.3. Influence of Interactions of Response Surface Parameters on Performance of Filling Body

It can be seen from Table 2 that the slump degree, bleeding rate, and compressive strength are not only affected by individual factors but also by the interaction of various factors. Among them, the slump degree is not significantly affected by the simultaneous action of multiple factors, and the bleeding rate has a significant effect on compressive strength.

Figure 3 shows the effect of the interaction between the cement content and ash ratio on the bleeding rate when the slurry concentration is 58% and the grouting admixture is 3%. When the ash is relatively low, the increase in cement content will cause a slight decrease in the bleeding rate. With the increase in the ash–gangue ratio, the bleeding rate increases significantly. When the gangue ratio increases from 0.5 to 2.0, the bleeding rate increases by approximately 35%. The analysis shows that when the cement content is 10% and the slurry concentration is 58%, an effect of the interaction between the ash–gangue ratio and the grouting admixture on the bleeding rate. When there are less additives in the grouting, as the ash ratio increases, the bleeding rate increases. When the grouting admixture is increased to a certain degree, the grouting admixture has a significant effect on the bleeding rate, but it reduces the sensitivity of the grouting admixture to the grouting admixture. When the cement content is 10.00% and the grouting admixture is 3.00%, in the case of relatively low ash–gangue, as the slurry concentration increases, the bleeding rate will increase slightly, and when the ash–gangue ratio increases, it will also increase the sensitivity of the bleeding rate to the slurry concentration, as shown. At this time, with the increase in the ash refuse ratio, the bleeding rate increases significantly.

This phenomenon can be explained by the differences in particle size distribution and surface morphology between fly ash and coal gangue. Fly ash particles are fine and spherical, which improve slurry fluidity but weaken the network structure required for water retention. In contrast, coal gangue particles are coarse and angular, forming a rigid skeleton that helps to restrain segregation and bleeding. When the ash–gangue ratio is excessively high, the dense packing of fine particles limits water migration, resulting in lower bleeding. However, when the ratio is too low, large interparticle voids lead to increased bleeding. Therefore, an intermediate ratio of approximately 1:2 yields an optimal packing structure that balances fluidity and stability.

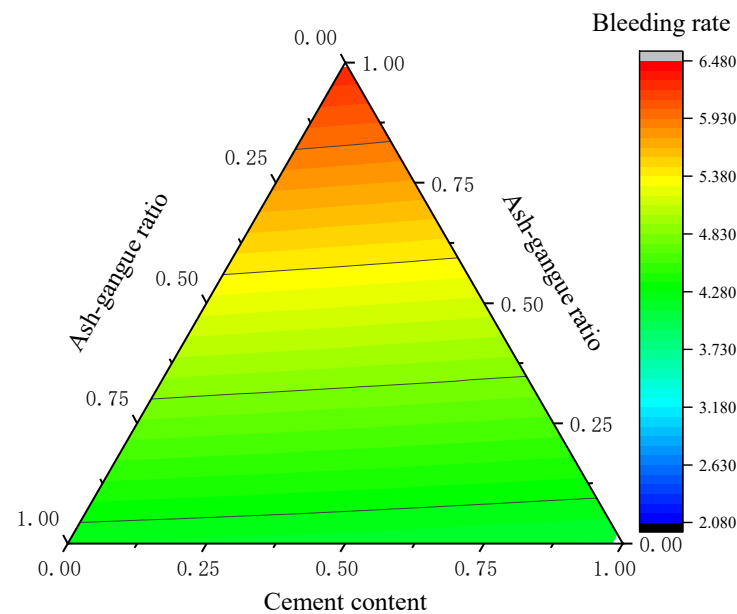


Figure 3. Interaction of cement and ash–gangue ratio on bleeding rate.

Figure 4 shows the influence of the slurry concentration of 54.19% and the grouting admixture of 3.00%, an interaction of the cement content and ash ratio on the compressive strength. It can be seen from Figure 4 that when the ash is relatively low, as the cement content increases, the strength of the filling body gradually increases, and the increase rate is larger and larger. When the cement content increases from 5% to 15%, the strength of the filling body increases by approximately 27%. When the cement content is low, as the ash–gangue ratio increases, the strength of the filling body increases, but the increase is not significant. This reminds us that, in engineering practice, the amount of cement can be increased at the same time, the use of fly ash can be reduced, and the cost of filling can be reduced. When the ash gangue ratio is 1.25 and the grouting admixture is 3%, the interaction between the slurry concentration and cement content is more significant. With the increase in both, the compressive strength continues to increase, but the increase is slower. In practical applications, the content of the two can be maintained at a certain level; it is unnecessary to increase the content too much, thereby increasing the filling cost. When the cement content is 10% and the slurry concentration is 58%, the interaction between the grouting admixture and the ash ratio is more significant. When the grouting admixture is low, the bleeding rate gradually increases with the increase in the gangue ratio, and when the grouting admixture reaches a certain amount, the sensitivity of the bleeding rate to fly ash and coal gangue will be reduced. The increase in the water rate is reduced. When the slurry concentration is 58% and the ash ratio is 1.25, the interaction between the cement content and the grouting admixture has a significant effect on the strength of the filling body. When there are fewer additives in the grouting, as the cement content increases, the compressive strength increases significantly. When the cement content is small, the compressive strength increases with the increase in the grouting admixture. The interaction between the remaining factors has no significant effect on the compressive strength.

Through the analysis of the influence of each single factor and the interaction of multiple factors, it is concluded that the slump is significantly affected by the single factor, and the interaction of multiple factors is not obvious. The order of single-factor influence is the slurry concentration > cement content > ash ratio > grouting admixture. The bleeding rate is significantly affected by the ash–gangue ratio, and the interaction of the ash–gangue ratio with the slurry concentration, cement content, and grouting admixture also has a

greater impact on the bleed rate. The order of each influencing factor is the ash–ganguer ratio > interaction between the ash–ganguer ratio and grouting admixture > interaction between the ash–ganguer ratio and slurry concentration > interaction between the cement content and ash–ganguer ratio; other factors are not significant. The compressive strength of the filling body is relatively significantly affected by the interaction between single factors and multiple factors. Mainly for the single factor, the most significant influencing factors are the cement content > ash–ganguer ratio > slurry concentration > grouting admixture > interaction between the ash–ganguer ratio and slurry concentration > interaction between the cement content and ash–ganguer ratio > cement content interaction with the slurry concentration.

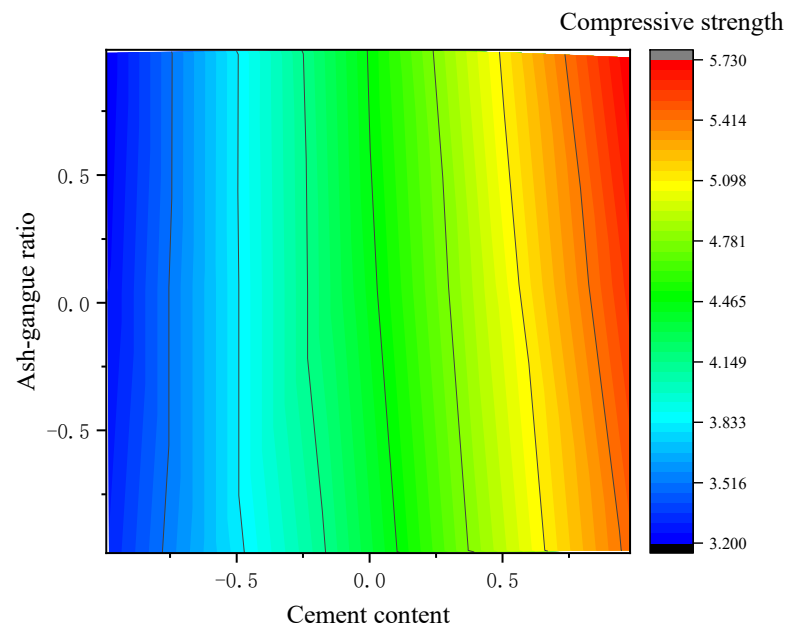


Figure 4. Interaction between gray–ganguer ratio and the compressive strength of cement content.

3.4. Optimization of Fire Protecting and Filling Material Ratio Satisfaction

When mixing the grouting and fire protecting and filling material, the following principles should be followed: “The compressive strength of the filling body and the conveying process should meet the requirements, the filling quality should reach a certain index, and the filling cost should be minimized [25].” Assuming that the weights of the three responses are equal, use Equations (4) and (5) to calculate the overall satisfaction. Figure 5 is an analysis of the impact of single factors on satisfaction. It can be seen from Figure 5 that the cement content has a significant effect on the overall satisfaction of the filling performance, the satisfaction is between 0.5 and 0.8, the trend is to increase first and then decrease, and the cement content is between 5% and 7%, which can achieve a greater satisfaction. When the pulp concentration is between 52% and 57%, it has little effect on satisfaction, but it has been maintained between 0.6 and 0.8 to maintain greater satisfaction. When the gangue ratio is in the range of 0.5–2, satisfaction decreases slightly, and when the gangue ratio is 0.5, the satisfaction can reach 0.8. The grouting admixture has a significant impact on overall satisfaction in the range of 1% to 5%, and satisfaction shows a trend of increasing first and then decreasing. When the grouting admixture is between 1.3 and 1.5, satisfaction can reach 0.9, which means that the grouting admixture needs an appropriate amount, and excessive admixture will not only increase the filling cost but also reduce the filling quality. The optimal fire protecting and filling material ratio fitted by Matlab data is a cement content of 12.12%, ash–ganguer ratio of 0.5, slurry concentration of 52%, and grout

admixture of 1.49%, and the corresponding response is a slump of 28.5 cm, bleeding rate of 2.36%, and the filling body strength is 4.62 MPa.

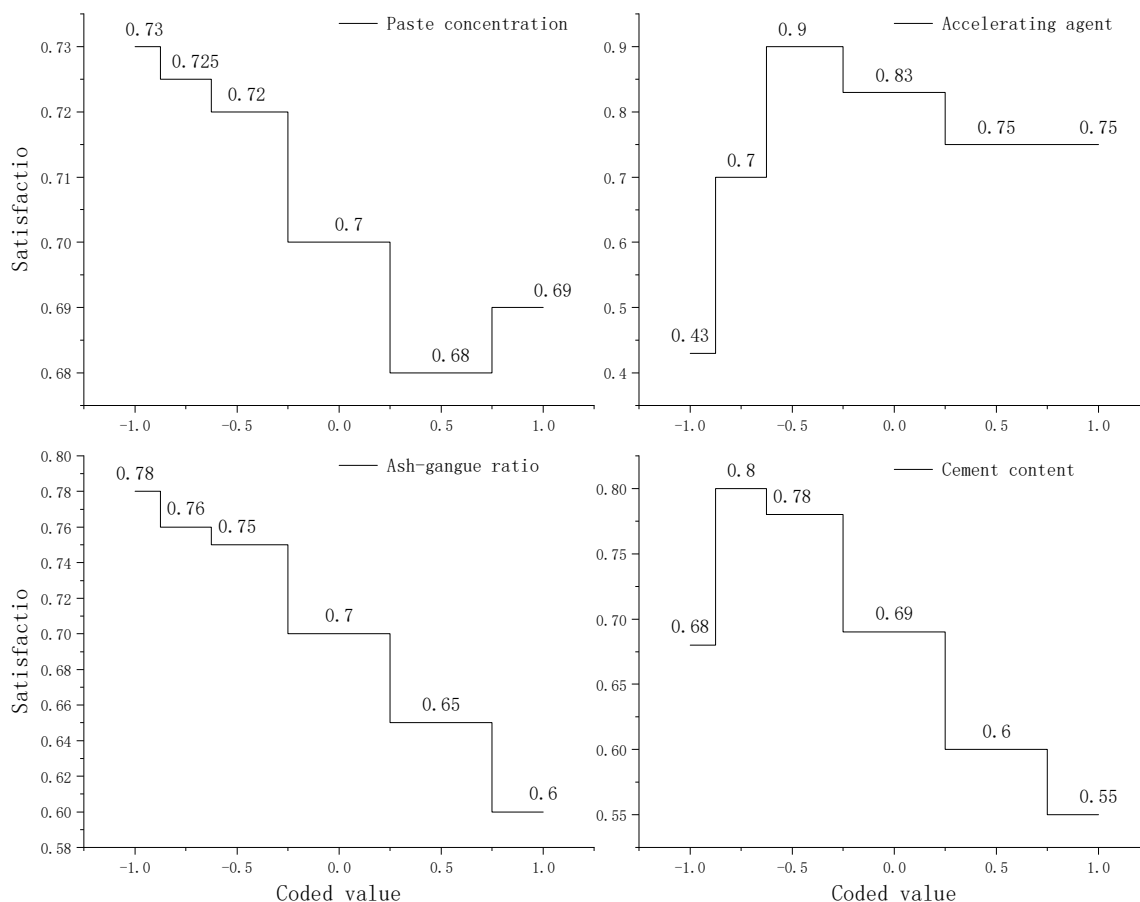


Figure 5. Single-factor influence on overall satisfaction of backfill performance.

According to relevant engineering guidelines for grouting materials in coal mining [26,27], a slump range of 25–30 cm ensures adequate pumpability, a bleeding rate below 3% indicates good stability, and a 28-day compressive strength exceeding 3.0–4.0 MPa satisfies the structural requirement for separation-layer filling. The optimized mix in this study (28.5 cm slump, 2.36% bleeding, and 4.62 MPa strength) fully meets and slightly surpasses these standards, demonstrating both high workability and mechanical reliability. These results confirm that the proposed RSM–SF-optimized formulation is suitable for practical fireproof grouting applications in coal mines.

3.5. Verification Test

In order to ensure the reliability of the experimental results, according to the optimized data obtained from the analysis, under the same experimental conditions, a verification test was conducted. The weight is a total of 5 kg of grouting material, of which 566.6 g is cement, 2355.6 g is materials, 1265.4 g is fly ash, 700 g is coal gangue, and 112.4 g is grouting admixture. The material experiment shows that the slump is 28.14 cm, which is basically consistent with the actual value. The bleeding rate is 2.42%, which is higher than the theoretical value, and the error is about 2.5%, which may be caused by external interference factors. The compressive strength of the filling body at 28 days of age is 4.47 MPa, and its value is shown in Figure 6, which is lower than the theoretical value, and the error is 3.23%. The cause of the error may be due to external factors such as the influence of the temperature during the experiment. The above shows that the ver-

ification test results are basically consistent with the optimization results; that is, it is feasible to use the response surface-function method to optimize the relevant ratio of the grouting material in the separation layer. However, the subsequent experiments should consider the influence of various external factors so that the experimental results reach a higher accuracy.

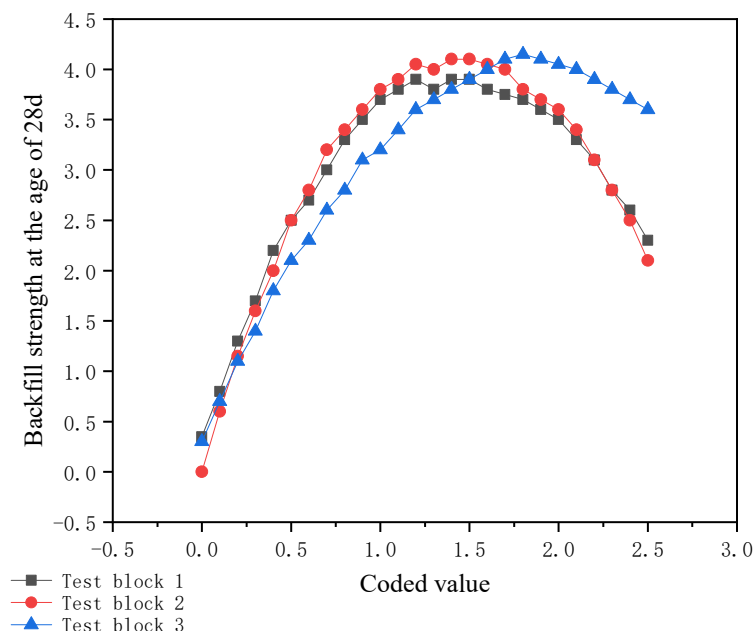


Figure 6. Backfill strength at the age of 28 d.

A small deviation was observed between the predicted and measured bleeding rates (2.36% vs. 2.42%, $\approx 2.5\%$). This discrepancy is mainly attributed to external factors during specimen preparation and testing: (i) minor fluctuations in ambient temperature and relative humidity, which affect hydration kinetics and early segregation; (ii) variability in manual mixing and batching (water dosing and shear history), leading to subtle differences in slurry rheology; (iii) time lag between mixing and testing, allowing partial settling or water migration; and (iv) non-uniform curing temperature among molds. To minimize such discrepancies in future tests and practical applications, several measures are recommended: (1) perform mixing and bleeding tests in a climate-controlled environment ($\pm 1^\circ\text{C}$, RH $50 \pm 5\%$) and use constant-temperature water baths or curing chambers; (2) implement automated batching and mixing protocols with fixed shear rate and duration, and timestamp all operations; (3) calibrate measuring devices (balances, slump cone, and cylinders) before each batch and standardize the bleeding test procedure; (4) increase the number of replicates ($n \geq 5$) with randomization and blocking, and report results as mean $\pm$ SD; and (5) precondition aggregates (e.g., pre-saturate coal gangue to stabilize water demand) and verify slurry rheology immediately before testing.

A moderate deviation was observed in the compressive strength (predicted 4.62 MPa vs. measured 4.47 MPa; $\approx 3.23\%$). Beyond curing-temperature effects, several factors may contribute to this reduction: (i) slight fluctuations in curing humidity and premature moisture loss from molds, increasing early shrinkage and microcracking; (ii) air entrainment due to non-uniform mixing energy or insufficient compaction, leading to higher void content; (iii) minor variations in the effective water-to-binder ratio (e.g., incomplete SSD conditioning of aggregates or evaporation during batching), altering hydration kinetics and final porosity; (iv) bleeding or segregation channels that locally weaken the matrix–gangue interfacial transition zone (ITZ); (v) variability in admixture dispersion at low dosages

affecting early rheology and packing; and (vi) testing artifacts such as end-surface planarity, capping quality, loading rate, or equipment calibration. To reduce this margin of error in future tests and practical applications, several measures are recommended: (1) apply humidity-controlled curing ($\geq 95\%$ RH) or lime-saturated water at 20 ± 1 °C with sealed molds to minimize moisture loss; (2) standardize mixing with a fixed-speed and fixed-time protocol, verify rheology before casting, and adopt vibration, compaction, or brief vacuum de-airing to minimize entrapped air; (3) control the effective water-to-binder ratio through the SSD preconditioning of gangue, closed-loop water dosing, and time-stamped batching to limit evaporation; (4) increase replicates ($n \geq 5$) with randomization and report mean $\pm$ SD to quantify uncertainty; and (5) improve mechanical testing fidelity by applying sulfur or neoprene capping, maintaining a standard loading rate, and periodically calibrating testing equipment. These measures aim to reduce the variability associated with porosity, ITZ integrity, and test accuracy, thereby improving the reproducibility of compressive strength measurements.

3.6. Industrial Feasibility and Field Implementation

From an engineering perspective, the optimized fireproof grouting material developed in this study not only improves rheological and mechanical performance but also exhibits clear economic and industrial feasibility. Based on the current market unit prices of cement, fly ash, and gangue, a comparative cost analysis was conducted. The results show that the optimized composition achieves an approximately 12–15% reduction in material cost compared with traditional cement-based formulations, primarily due to the efficient utilization of low-cost gangue and the optimized ash–gangue ratio. This demonstrates that the proposed formulation is both technically effective and economically sustainable, supporting its large-scale applicability in coal mine grouting operations.

In addition to cost advantages, the optimized mixture exhibits high fluidity, a low bleeding rate, and sufficient compressive strength under high-temperature conditions, confirming its suitability for industrial-scale abscission layer grouting. To ensure its reliable transition from laboratory validation to real-world applications, pilot-scale and on-site grouting tests will be conducted in the next phase. These experiments will evaluate key parameters such as pumping performance, setting time, and long-term durability under actual mining and thermal environments. The field data will also be used to refine the cost model and guide the formulation of engineering standards for practical fireproof grouting operations. Overall, the integration of technical reliability, cost efficiency, and application feasibility demonstrates that the optimized fireproof grouting material provides a practical, safe, and sustainable solution for underground fire prevention and water control in coal mines. This section bridges the gap between theoretical optimization and industrial implementation, highlighting the potential for future engineering-scale deployment.

4. Discussion

4.1. The Position of the Grouting Drilling Hole

The location of the separation grouting hole and the depth of the hole can be determined according to the separation location, the development degree of the separation, and the separation range. In order to further ensure the control of the overlying strata after mining in the north area of the minefield, a certain coal mine continued to implement separation grouting and filling projects at the working faces. The grouting pressures at different positions in the separation grouting system are also different, including the following: $P_{\text{separation}}$ is the pressure in the separation zone, P_{orifice} is the orifice pressure of the grouting hole, and P_{pump} is the outlet pressure of the grouting pump, as shown in Figure 7.

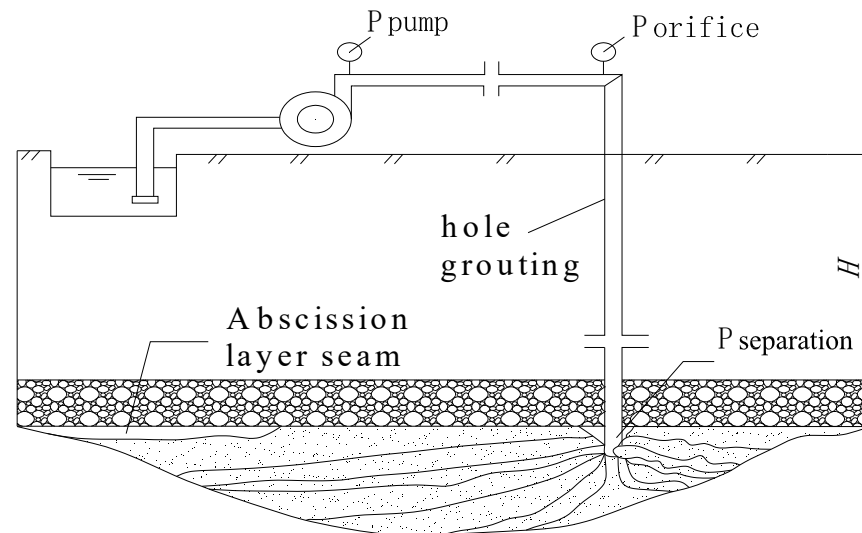


Figure 7. Diagram of grouting pressure.

It can be seen that, in the negative pressure grouting stage, the outlet pressure of the grouting pump is

$$P_{\text{pump}} = P_{\text{orifice}} + h_f \quad (7)$$

In the positive pressure grouting stage, the grouting pressure in the separation layer is

$$P_{\text{separation}} = P_{\text{pump}} - h_f + \gamma_{\text{slurry}} H \quad (8)$$

In this formula, h_f is the resistance of the slurry pipeline; γ_{slurry} is the bulk density of the slurry; and H is the separation depth. According to Formulas (7) and (8), the grouting pressure in the separation layer is related to the resistance of the slurry pipeline, the bulk density of the slurry, and the development degree of the separation layer. Take the 10-2# grouting hole in a certain coal mine as an example. When the working face is mined 50–80 m away from the orifice, the orifice pressure drops significantly, indicating that the separation layer is well developed. When the separation layer is fully developed and the orifice pressure drops to the lowest pore pressure of 1.2~2 MPa (usually for 150–180 days), it is a favorable opportunity for grouting, and the amount of grouting should be increased. When the grouting hole is about 300 m past the working face, the orifice pressure begins to rise significantly and gradually approaches the initial injection pressure of 3.8~4 MPa, indicating that the separation has become closed and stable.

4.2. The Process of Separate-Layer Grouting

(1) Pre-fracturing stage

Before being affected by mining, the advanced high-pressure water injection into the formation and hydraulic power will overcome the formation pressure so that the formation layer will undergo fracturing under the influence of strength and resistance. At this stage, the pumping pressure is higher, and the pumping volume is smaller.

(2) Stage of increasing injection volume

This stage is a critical stage. As the working face advances, the separation of the fracturing layer zone gradually develops, and the amount of slurry injected will increase accordingly. At this time, the pump pressure fluctuates significantly, and the amount of slurry should be increased to give full play to the filling effect of the grouting material.

(3) The injection volume reduction stage

As the coal mining face continues to advance, when it exceeds a certain distance from the grouting borehole, the pump pressure gradually increases, and the pump slurry volume decreases accordingly.

(4) End of grouting

The end of grouting is mainly based on three indicators: whether the total grouting volume exceeds the designed grouting volume; whether it has reached the allowable rock penetration volume; and whether it has reached the final grouting pressure. When the above indicators are reached, grouting can be ended.

During the grouting process, you should always observe and check the changes in the discharge volume and grouting pressure in time to avoid abnormal conditions that affect the filling effect. At the same time, you must adjust the pump volume and pump pressure at any time; stir grouting according to the design ratio in advance so as not to interrupt the grouting and affect the injection volume.

4.3. The Effect of Fireproof Grouting on a Certain Coal Mine

The results of fire monitoring and rock deformation observation in a certain coal mine under high-temperature conditions show that by grouting and filling the lower separation layer of the key thick strata, an active support measure, it effectively blocked the channel of heat transfer, reduced the large-scale fracture and collapse of the coal and rock mass, and lowered the risk of secondary disasters induced by fire. The results show that this technology not only reduces the possibility of structural instability and the secondary ignition of coal and rock mass under high-temperature conditions but also decreases the rate of temperature rise in high-temperature areas by about 50%. The overall process of the coal mine fire prevention and extinguishing grouting system is illustrated in Figure 8. Scholars have carried out related research on the mechanism of separation grouting in controlling the thermo-mechanical coupling effect of strata under fire conditions. They believe that the water and cementitious components in the slurry can fill the separation space, and the condensate of the slurry can form a thermal insulation support body supporting the overlying strata and roadway space, thereby delaying the spread of fire. At the same time, scholars pointed out that although a certain coal mine has adopted many measures to prevent and control fires, including advanced thermal insulation zones, microseismic and temperature monitoring, fireproof coal pillars, and insulation layers, the effect is not very good, and thermal hazards and fire risks still occur in the roadway and working face. The study found that the expansion of fractures and the thermal cracking of coal and rock masses are the main causes of mine fire development and disaster evolution. Through the regional fire prevention zoning method to divide the thermal fracture zones of the key strata, and by putting forward fireproof reinforcement measures combined with separation grouting, it can significantly improve the thermal stability and structural integrity of the surrounding rock under high-temperature conditions.

During the fire evolution process at the working face, a large separation space often forms in the lower part of the key thick strata so that the stress concentration occurs around the separation zone, which is superimposed with the original support pressure, forming a thermal impact stress field and, under the action of dynamic thermal disturbance, induces the spread of fire. By using separation grouting, the filling body can effectively fill the separation space, reduce the thermal stress accumulation of coal and rock mass, cut off the transmission path of the fire source, and fundamentally prevent the spread of fire. Secondly, separation grouting technology can effectively reduce the peak thermal energy in high-temperature areas. Through the comparison of temperature monitoring and microseismic energy changes in the No. 4 coal working face of a certain coal mine before and after the application of this technology, it can be seen that, before grouting, thermal energy

accumulation was obvious and the fire risk was very high, but after grouting, the rate of the temperature rise slowed significantly, the energy of microseismic events decreased markedly, and the peak value also declined. Practice also shows that, after the application of fireproof grouting technology in the No. 4 coal working face of a certain coal mine, fire hazards have been significantly controlled, and both the frequency and intensity of high-temperature fires have been obviously reduced.

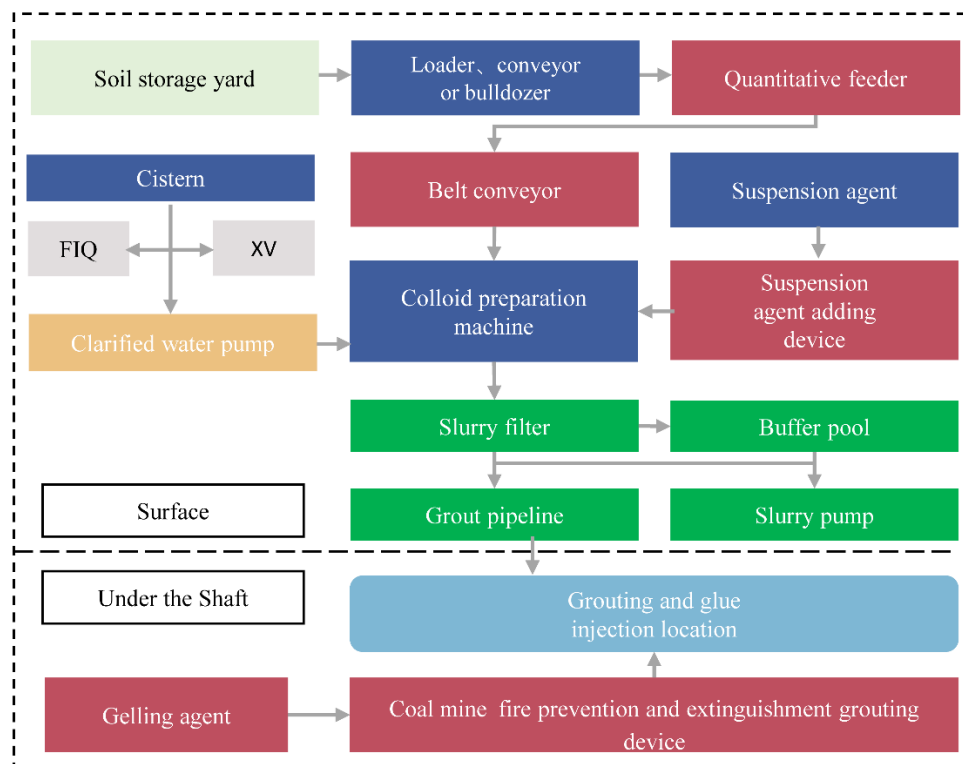


Figure 8. Fireproof grouting measures and their effect on a certain coal mine.

5. Conclusions

- (1) A method for optimizing the performance ratio of fireproof layered grouting material based on RSM-DF is proposed. Compared with the traditional orthogonal test method, this approach requires fewer tests when considering multiple parameters and multiple factors, allows for the continuous optimization of factor levels, and provides more accurate prediction and higher reliability, offering a new idea for material design under high-temperature fire conditions in coal mines.
- (2) The influence of cement content, the ash–gangue ratio, slurry concentration, and admixture on the slump, bleeding rate, and compressive strength was systematically studied, and a response surface regression equation was established. This provides a convenient approach for the multi-objective and multi-factor studies of fireproof filling materials under mine fire environments and has guiding significance for fire hazard prevention and control in coal mines.
- (3) The results indicate that slurry concentration and cement content have significant effects on the slump; cement content and slurry concentration have significant effects on the compressive strength; and the ash–gangue ratio has a very significant effect on the bleeding rate and shows notable interactions with the slurry concentration and cement content. The optimal ratio of the fireproof filling material is cement content of 12.12%, ash–gangue ratio of 0.5, slurry concentration of 52%, and admixture of 1.49%. The corresponding responses are a slump of 28.5 cm, bleeding rate of 2.36%, and filling body strength of 4.62 MPa. These results are basically consistent with the verification

experiments, providing a basis for the engineering application of fireproof grouting under high-temperature coal mine conditions.

- (4) The properties of layered fireproof grouting filling materials not only include the slump, bleeding rate, and compressive strength but also viscosity, diffusivity, creep, and rheological behavior. Influencing factors also involve pumping pressure, filling cost, and environmental temperature. Future research should explore more response indicators and influencing factors related to fire environments to make the conclusions more consistent with field practice and to further improve the basic research system of fireproof grouting and filling technology.
- (5) Industrial test results show that the layered fireproof grouting material exhibits good fluidity and pumpability under high-temperature conditions. Pocket assay grouting technology was proposed, which can be used for transverse extrusion and longitudinal reinforcement in the separation area, transforming traditional passive grouting into active fireproof reinforcement. This technology can effectively prevent slurry leakage or exudation at high temperatures, improve construction stability under fire conditions, reduce grouting cost, enhance fireproof efficiency, and serve as an important safeguard for safe, efficient, and green coal mining. In addition, a comparative economic evaluation indicates that the optimized mixture achieves approximately 12–15% lower material cost than conventional cement-based formulations, mainly due to the efficient utilization of low-cost gangue and the optimized ash–gangue ratio. This finding confirms that the proposed grouting material is not only technically reliable but also economically feasible for large-scale industrial application.

Future research should further expand the response indicators and influence factors of fireproof grouting materials under high-temperature and fire conditions. In addition to conventional parameters such as the slump, bleeding rate, and compressive strength, it is essential to consider the thermal conductivity, heat capacity, and gas permeability to comprehensively evaluate the coupled thermo-mechanical performance. The evolution of microstructure and pore connectivity under prolonged thermal exposure should also be examined through advanced techniques such as scanning electron microscopy (SEM) and X-ray diffraction (XRD), as these methods can effectively capture thermal degradation, microcracking, and phase transformation phenomena in cementitious composites [28]. Moreover, variations in curing time and admixture composition may critically influence fire resistance and post-fire residual strength, indicating the need for systematic experiments under different heating curves and exposure durations [29]. Future optimization frameworks could thus integrate multi-physics simulation and long-term high-temperature testing to improve the predictive accuracy and engineering applicability of fireproof grouting materials for deep coal mining operations.

Author Contributions: Z.C.: Methodology, Formal analysis, Validation, Investigation, Data curation, Visualization, Writing—original draft, Writing—review and editing; J.S.: Conceptualization, Methodology, Writing—review and editing, Supervision, Funding acquisition; Z.Z.: Data curation, Supervision, Formal analysis, Writing—review and editing; L.L.: Data curation, Supervision, Formal analysis, Writing—review and editing. All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by the Natural Science Foundation of Shandong Province [ZR2024MG039]: Research on Risk Sensing and Early Warning Decision-Making of Major Coal Mine Disasters Based on Multimodal Big Data.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

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

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analysis, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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