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Optimization of Mix Proportion for Special Cement-Based Materials Used in Coal Mines

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Abstract: Aiming at the problem of high cost of filling materials in the coal mining process, a cement-based high-water material is proposed, and cement is used as the basic cementing material for high-water materials in coal mines. The type of cement is determined by single-factor test, and the orthogonal test design is used as the dependent variable. According to the test results, the mathematical model of the water bleeding rate is obtained by using the response surface analysis method. Combined with the two-factor correlation analysis, the influence of different single factors and two factors on the water bleeding rate is determined. The factors that are more related to the water-cement ratio, fly ash content, and thickener ratio are determined. The mathematical model of strength is established by using the response surface test design with these three factors as independent variables.

Keywords: Response surface analysis; analytic hierarchy process; ratio optimization.

1. Introduction

With the continuous innovation of coal mining technology, the application of various new materials in coal mining has gradually increased[1]. However, for coal enterprises, the high cost of finished materials and technical limitations still exist, and self-research of materials under different needs has become an important task in the application of new materials in coal mines[1]. In practical applications, common materials include support materials along empty roadways and goaf filling materials. Due to the special operating environment of coal mines, choosing materials with advantages such as easy operation and convenient transportation can not only improve construction efficiency, but also effectively reduce labor and transportation costs[4, 5].

The purpose of this paper is to study a kind of cement-based material for coal mine that can achieve higher water-cement ratio in order to achieve similar properties to high-water materials. By improving the water-cement ratio, the optimal utilization of raw materials can be achieved under the premise of ensuring engineering performance, and the transportation volume of coal mine resources can be effectively reduced. To achieve this goal, this paper uses cement as a cementitious material and cooperates with other common materials to study its use effect under higher water-cement ratio. Considering the wide variety of materials, this paper selects several common materials as raw materials in the experiment, and studies its impact on performance through the combination of different materials.

In this paper, the bleeding rate is used as a dependent variable, and different raw materials and corresponding content are used as independent variables. Through the corresponding experimental design and test results, the model constructed by the corresponding relationship between the bleeding rate and the raw materials is finally obtained. The coal mine can choose the raw materials corresponding to the model according to the performance requirements of different scenarios. At the same time, the raw materials can be changed according to the method of this paper to obtain new models for use.

2. Test Raw Materials

2.1. Cement Selection

Cement, as a commonly used cementing material, plays an important role in the cementing process. The hardening of cement after cementation has a certain strength because the minerals in cement undergo hydrolysis or hydration reaction in contact with water, and the hydration products form a cement stone structure in a certain way to produce strength. In general, the water-to-cement ratio for high water materials during use is 1.3:1 to 3:1[6]. In this article, the preliminary tests conducted when selecting the type of cement controlled the water-to-cement mass ratio at 1:1, 2:1, 3:1, 4:1, and 5:1, comparing the volume of bleeding rate of different types of cement under different water-to-cement ratios, thereby determining a type of cement with a lower bleeding rate as the raw material. The commonly used cements are two types: Portland cement and sulfoaluminate cement. In this article, three types of cement are selected for preliminary experiments with water. The identification and types of the three cements are as follows: 1 cement is ordinary Portland cement (cement grade designation 425), 2 cement is special sulfoaluminate cement (cement grade designation 425), and 3 cement is fast-setting sulfoaluminate cement (cement grade designation 425).

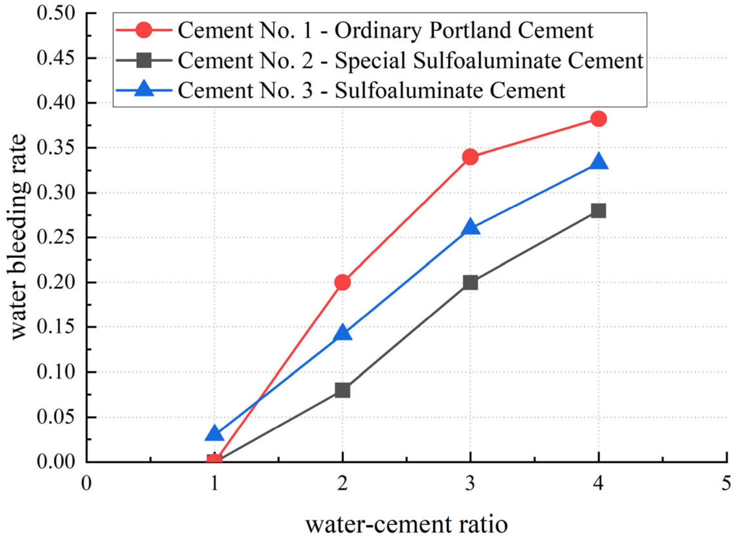


Figure 1. Bleeding Rate Results of A Single Type Of Cement at Different Water-Cement Ratios

As shown in Figure 1, comparing the water bleeding rate of the three types of cement with the same water-cement ratio and then standing for the same time, the water bleeding rate of 2# cement was lower than that of the other two cements under the same water-cement ratio, so special sulfoaluminate cement was determined as the raw material.

At the same time, in order to ensure the effect of the combination of different materials and cement, different admixtures and admixtures are added to change the properties of the materials, and the effects of the proportion of different raw materials in the mixed materials on the properties of the finished materials are further compared, and the optimal mix ratio suitable for engineering applications can be determined.

2.2. Determination of Supplementary Cementitious Materials Types

Fly ash refers to the fine ash and bottom slag obtained from the flue gas of coal-fired power plants and the comprehensive utilization of coal gangue and coal slurry resources after being collected by dust collectors[2]. Fly ash is a waste generated from the combustion of coal, and if not controlled and discharged into the air indiscriminately, it can cause certain pollution. Typically, fly ash is used as a raw material for cement production. However, considering the large amount of energy consumed annually in our country, with coal accounting for more than half of the energy consumption, there is a significant gap between the fly ash produced from coal combustion and the consumption amount. A considerable portion of fly ash lacks effective utilization pathways for treatment. Therefore, reusing fly ash in the coal mining process through various methods is a good energy-saving and emission-reduction measure. To maximize the environmental benefits of material usage, fly ash is selected as a cement admixture.

2.3. Determination of Admixture Types

The initially selected types of additives include thickeners, cellulose ethers, concrete accelerators, concrete water reducers, guar gum, gypsum, and mineral powder. During the experiments, the mass of the solid materials added is kept constant. When comparing results with the same water-cement ratio while varying the water-cement ratio, the test groups with less bleeding are recorded. Based on this method, a large number of experiments were conducted, which preliminarily excluded cellulose ethers, guar gum, and mineral powder, as their impact on the bleeding of cement when used in combination is minimal and does not significantly reduce the bleeding rate. Ultimately, thickeners, concrete accelerators, and gypsum were selected as cement additives.

3. Experimental Design and Result Analysis

3.1. Experimental Design and Results

Before conducting the water release test, the raw materials in the experiment are first identified as independent variables, and a horizontal division is made based on the proportional relationships between the independent variables. The BBD (Box-Behnken Design) experimental design method is employed to construct the experimental table, in which the water bleeding rate is treated as the dependent variable. The raw materials involved in the experiment are regarded as five independent variables, and after division into different levels, the corresponding experimental table is generated. The levels of each parameter of the independent variables are as follows, with the actual values of each parameter during the experiment indicated in parentheses:

A, water-cement ratio, mass ratio of water to preparation materials: 1 (1); 2 (2); 3 (3); 4 (4); 5 (5);

B, the amount of fly ash accounts for the sum of the weight of cement and fly ash, %: 1 (50); 2 (60); 3 (70); 4 (80); 5 (90);

C, the percentage of thickener in the sum weight of cement and fly ash, %: 1 (0.5);2 (1);3 (1.5);4 (2);5 (2.5);

D, the percentage of quick-setting agent in the sum weight of cement and fly ash, %: 1 (0.5);2 (1);3 (1.5);4 (2);5 (2.5);

E, gypsum to cement and fly ash by weight percentage, %: 1 (0.5);2 (1);3 (1.5);4 (2);5 (2.5);

The experimental design was conducted using the L25(56) orthogonal array, with the level factor table and the selected orthogonal test table as shown in Tables 1 and 2. The experiments were carried out according to the design of the orthogonal tests, and the results were recorded. Prior to preparing the experiments, it was necessary to determine the weights of the various materials involved for ease of operation. As the primary binding materials, the weights of cement and fly ash were fixed at 100 parts, with each part weighing 2g. The weights of the thickening agent, accelerator, and gypsum were calculated as a percentage based on the total weight of cement and fly ash.

Table 1. Level Factor Table

Factor	Unit	Level 1	Level 2	Level 3	Level 4	Level 5
A(W/C ratio)		1	2	3	4	5
B(Fly ash)	%	50	60	70	80	90
C(Thickening agent)	%	0.5	1	1.5	2	2.5
D(Quick-setting agent)	%	0.5	1	1.5	2	2.5
E(Gypsum)	%	0.5	1	1.5	2	2.5

Table 2. Orthogonal Design Table

Serial number	A	B	C	D	E	24-hour bleeding rate
1	1	1	1	1	1	0
2	1	2	3	4	5	0
3	1	3	5	2	4	0
4	1	4	2	5	3	0
5	1	5	4	3	2	0
6	2	1	5	4	3	0
7	2	2	2	2	2	0.1875
8	2	3	4	5	1	0
9	2	4	1	3	5	0.2291
10	2	5	3	1	4	0
11	3	1	4	2	5	0.1176
12	3	2	1	5	4	0.4705
13	3	3	3	3	3	0.4545
14	3	4	5	1	2	0.0588
15	3	5	2	4	1	0.3676
16	4	1	3	5	2	0.5333
17	4	2	5	3	1	0
18	4	3	2	1	5	0.7159
19	4	4	4	4	4	0
20	4	5	1	2	3	0.6629
21	5	1	2	3	4	0.7088
22	5	2	4	1	3	0.6835
23	5	3	1	4	2	0.7088
24	5	4	3	2	1	0.6923
25	5	5	5	5	5	0.0384

In this study, the software Design-Expert 13.0 was used to fit the experimental data of each group in order to obtain the corresponding mathematical model. During the model selection process, significance tests were conducted for the linear model, two-factor interaction model (2FI), second-order model, and third-order model. Through a comparative analysis of the significance of the models, detection of lack of fit, and correlation tests, the optimal model was ultimately determined. After evaluation, the second-order model was selected as the final mathematical model, and variance analysis and significance tests were performed on the obtained model. The mathematical model regarding the bleeding rate (Rb) and the five independent variables obtained after fitting the experimental data is:

$$\begin{aligned} R_b = & -1.40233A+0.050657B-0.294060C+0.25998D-0.350341E+0.056910AB \\ & -0.048637AC+0.594569AD-0.010676BD+0.091440CD+0.108945CE \\ & +0.100672DE-0.000260B^2-0.019515ABD-0.031338CDE-0.000431AB^2 \\ & +0.000052B^2D+0.000145AB^2D-1.65796 \end{aligned} \quad (1)$$

3.2. Analysis of Test Results

The analysisThe of significance variance of (theAN constantOVA term), was linear conducted term to, test quadratic the term significance (ofinteraction the term constant), term and, square linear term term (,cur quadraticvature term effect ()interaction in term the), quadratic and equation square model term was (testedcur usingvature analysis effect of) variance in. the The quadratic results equation of model the. model The's AN analysisOVA of results variance of are the shown model in are Table shown in3 Table. The3 Prob, > where F the value model of's the Prob model > is F less value than is less than 0.01, indicating that the constructed model is highly significant0. The model fit. statistics01 are, presented indicating in that Table the constructed model is highly4 significant. The model, fit with an R statistics are² shown in value Table of 04..974 The R2² and value an of Ade thequ predictionate model Precision is value of0 .9.9742,3539 and in the the signal-to prediction-no modelise fit ratio statistics (,Ade whichqu isate much Precision greater) than is 94.. This353 suggests9 that, which the model is much has greater good than fitting accuracy4 and. can This be result used indicates for that subsequent the optimization fit design is good using the, and response the surface response approximation surface model model. can be used for subsequent optimization design.

Tbale 3. Analysis of Variance Table

source	Sum of Squares	df	Mean Square	F Value	P-value Prob＞F	
Mean vs total	1.76	1	1.76			
Linear vs mean	1.61	5	0.3219	12.50	＜0.0001	Suggested
2FI vs linear	0.2861	10	0.0286	1.27	0.3665	
Quadratic vs 2FI	0.0006	1	0.0006	0.0243	0.8800	aliased
residual	0.2027	8	0.0253			
total	3.86	25	0.1543			

Table 4. Goodness-of-Fit Statistic

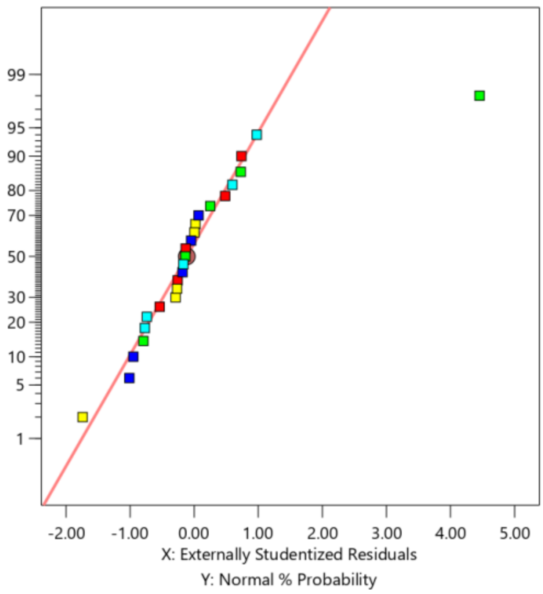
Std.dev.	Mean	C.V.%	R^2	Adjust R^2	Predicted R^2	Adeq precision
0.0950	0.2652	35.84	0.9742	0.8967	0.6964	9.3539

The actual values and predicted values with an error of less than 5% are classified as high-precision predictions, those less than 10% are considered excellent predictions, and those greater than 10% but less than 15% are regarded as successful and effective predictions. As shown in Table 5, by comparing the actual values with the predicted values obtained from the model, it can be observed that for the 25 groups of materials mixed with water in the actual experiment, the water bleeding rate after 24 hours and the predicted values calculated by the model, except for the 8 experimental groups where the actual water bleeding rate after 24 hours is 0, the maximum difference between the actual values and predicted values for this type of data is 0.09, which can be classified as high-precision data. Additionally, there are 11 groups of data that are defined as high-precision predictions, while the remaining 5 groups have larger errors. By comparing the normal probability distribution of the model's residuals, the distribution of residual values against predicted values, and the distribution of actual values against predicted values, it can be seen that there are points that are far from the overall model. The occurrence of this phenomenon may be due to systematic errors in the experiment, which is a normal occurrence and has a minimal impact on the overall accuracy of the model, thus demonstrating that the accuracy of the model can support the corresponding optimization design.

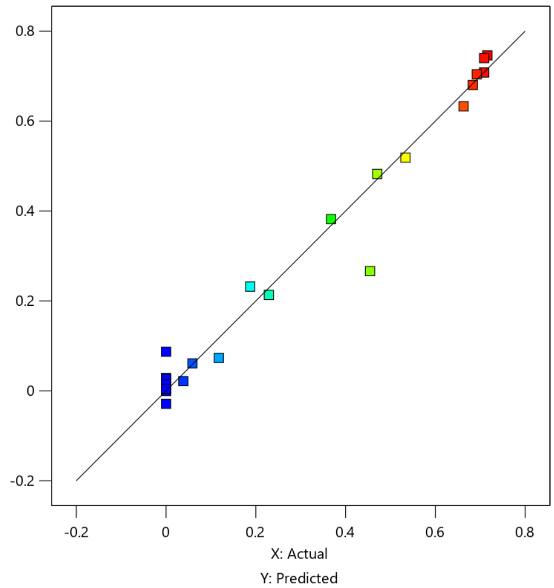
Table 5. Table of Differences Between Observed and Theoretical Values

Serial number	A	B	C	D	E	24-hour bleeding rate	Predicted value	Error
1	1	50	1	1	1	0	0.00	0
2	1	60	3	4	5	0	0.00	0
3	1	70	5	2	4	0	0.03	—
4	1	80	2	5	3	0	0.02	—
5	1	90	4	3	2	0	-0.03	—
6	2	50	5	4	3	0	0.01	—
7	2	60	2	2	2	0.1875	0.23	19.50%
8	2	70	4	5	1	0	0.01	—
9	2	80	1	3	5	0.2291	0.22	-6.51%
10	2	90	3	1	4	0	0.03	—
11	3	50	4	2	5	0.1176	0.07	-58.45%
12	3	60	1	5	4	0.4705	0.48	2.78%
13	3	70	3	3	3	0.4545	0.27	-69.30%
14	3	80	5	1	2	0.0588	0.06	7.70%
15	3	90	2	4	1	0.3676	0.39	4.70%
16	4	50	3	5	2	0.5333	0.52	-2.51%
17	4	60	5	3	1	0	0.01	—
18	4	70	2	1	5	0.7159	0.75	4.40%
19	4	80	4	4	4	0	0.09	—
20	4	90	1	2	3	0.6629	0.64	-3.97%
21	5	50	2	3	4	0.7088	0.74	4.46%
22	5	60	4	1	3	0.6835	0.68	-0.10%
23	5	70	1	4	2	0.7088	0.71	0.45%
24	5	80	3	2	1	0.6923	0.71	2.32%
25	5	90	5	5	5	0.0384	0.03	-34.83%

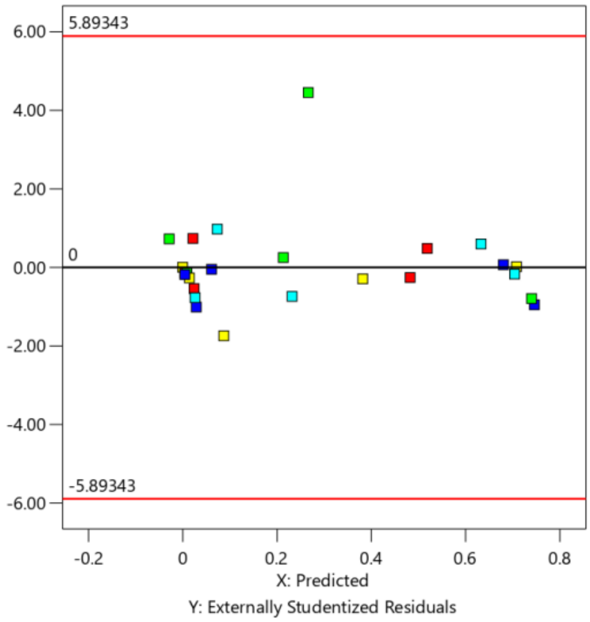
The adaptability of the model is assessed based on the normal probability distribution of the residuals, as well as the distribution of residual values and predicted values. Generally, if the model is well-adapted, the points in the normal probability distribution of the residuals should lie on a straight line, and the distribution of residuals and predicted values should show no regular pattern. As shown in Figure 2(a), it can be observed from the normal probability distribution graph that the points in the residual and predicted value distribution are essentially aligned on a straight line. In Figure 2(b), the distribution of residuals and predicted values shows no regular pattern, leading to the conclusion that the model has good adaptability. In Figure 2(c), it can be seen from the distribution of actual values and predicted values that the predicted values of the model and the actual values are essentially aligned on a straight line, indicating a strong consistency between the predicted values of the model and the data obtained from actual experiments.



(a) Normal Plot of Residuals



(b) Predicted vs.Actual



(c) Residuals vs.Predicted

Figure 2. Residual Analysis Plots

The three-dimensional graph and contour distribution illustrating the impact of the interaction of different factors on the water bleeding rate, as shown in Figures 3 to 9, indicate that the interaction of factors AB, AC, and AD has a greater effect on the dependent variable water bleeding rate compared to the interactions of CD, CE, and DE. Additionally, the color variations suggest that the interactions of AC and AD have the most significant impact on the dependent variable. This demonstrates that the influence of the interactions of AC and AD on the dependent variable is more pronounced than that of other dual-factor interactions. Therefore, it can be preliminarily concluded that the influence of factor A on the water bleeding rate, whether as a single factor or through interaction effects, is quite significant, establishing factor A as an important independent variable for analysis in this study.

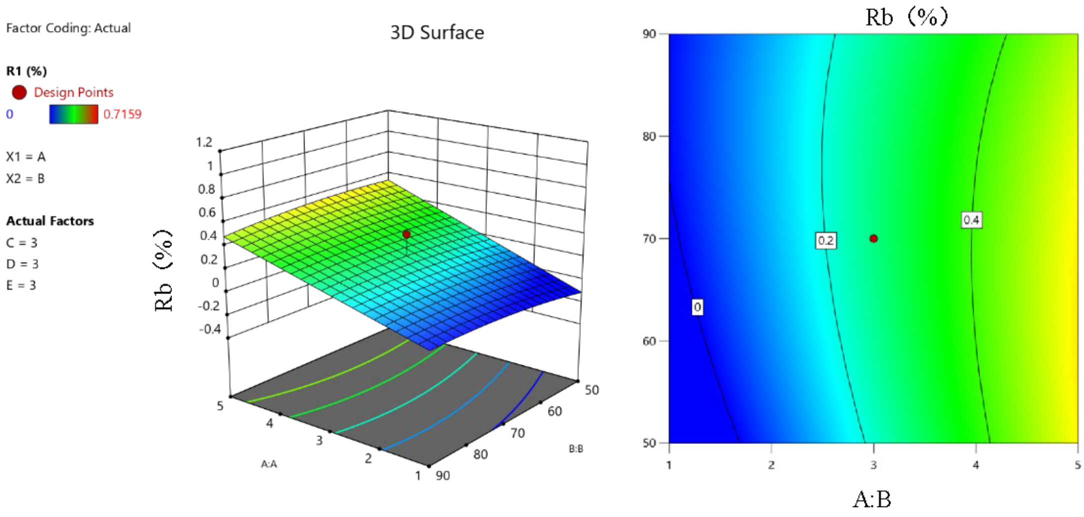


Figure 3. The Influence of AB Interaction on Water Bleeding Rate

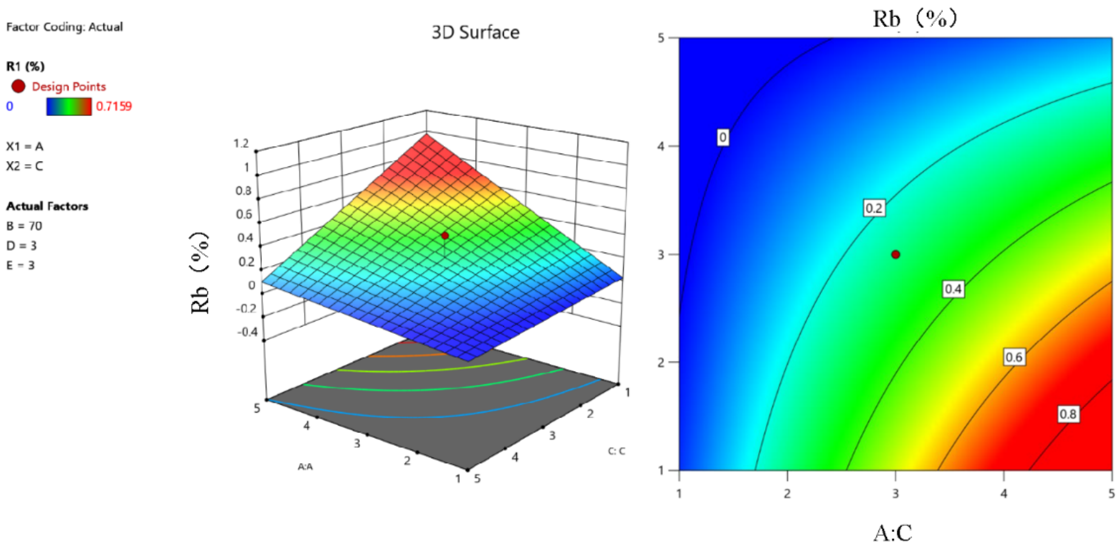


Figure 4. The Influence of AC Interaction on Water Bleeding Rate

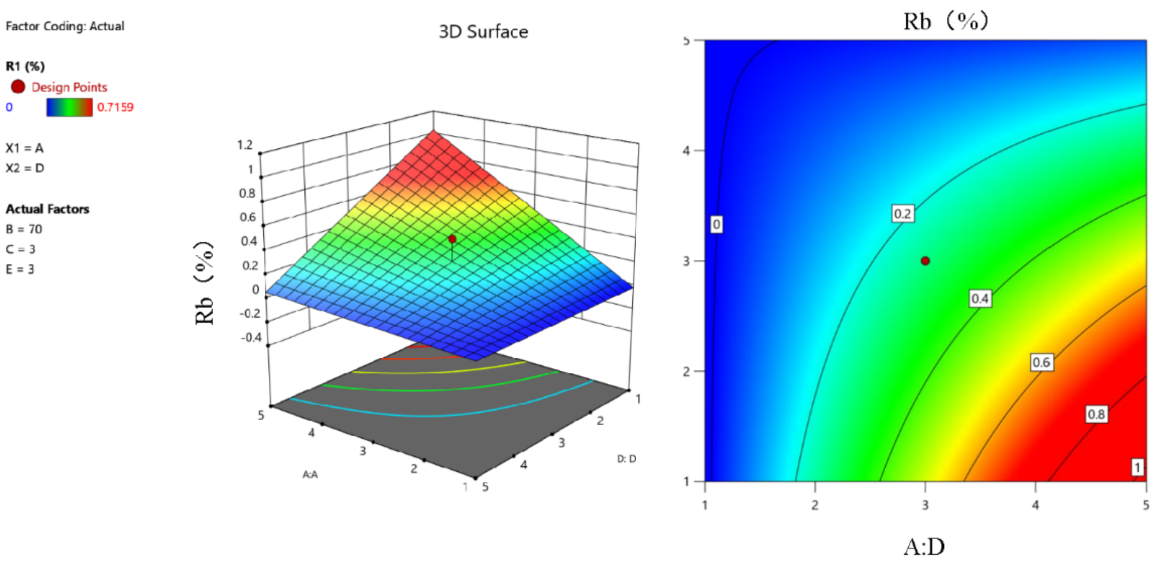


Figure 5. The Influence of AD Interaction on Water Bleeding Rate

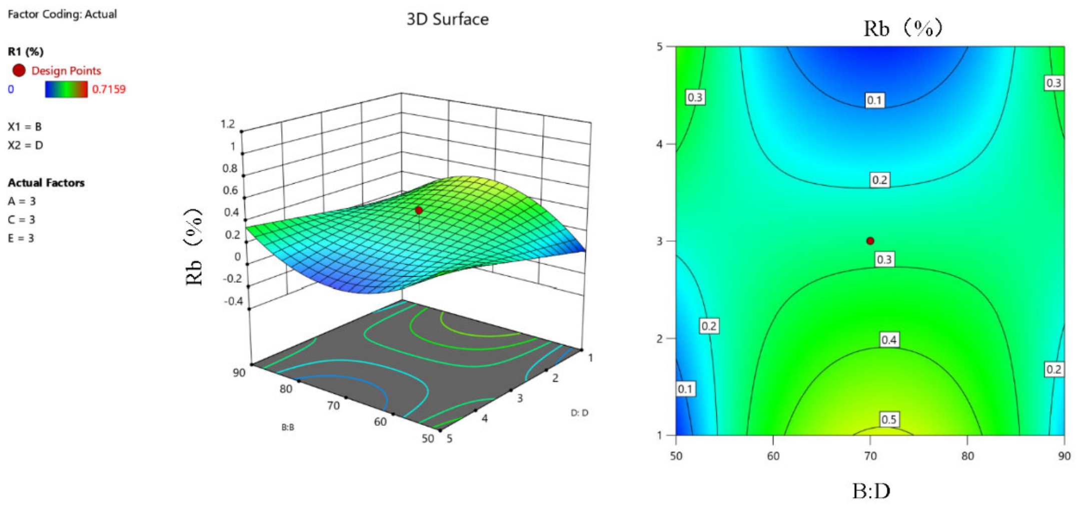


Figure 6. The Influence of BD Interaction on Water Bleeding Rate

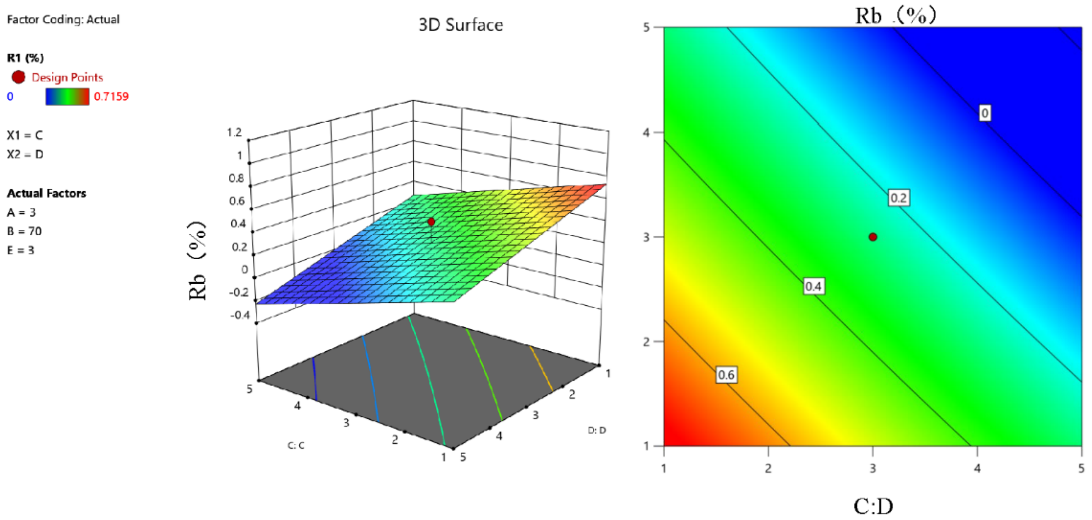


Figure 7. The Influence of CD Interaction on Water Bleeding Rate

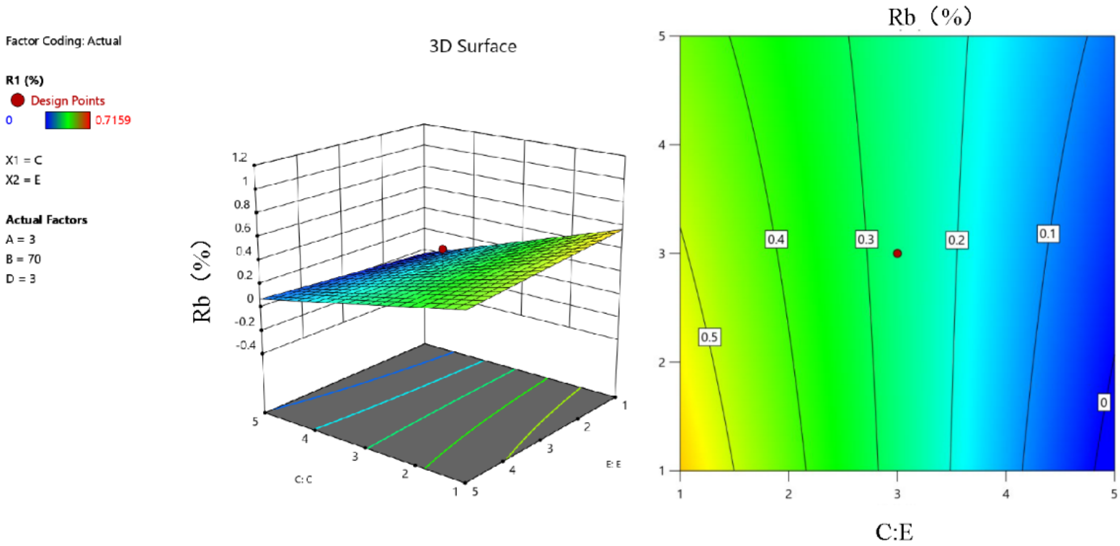


Figure 8. The Influence of CE Interaction on Water Bleeding Rate

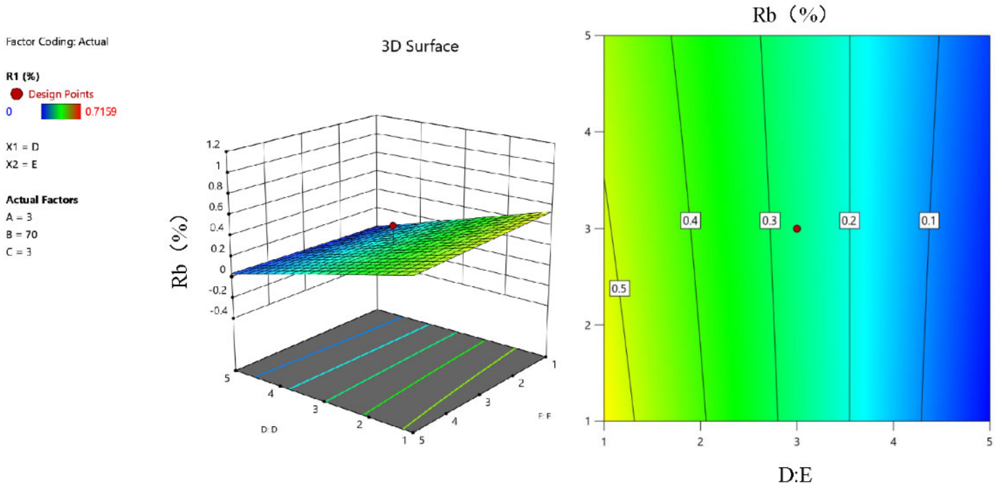


Figure 9. The Influence of DE Interaction on Water Bleeding Rate

The optimal mix ratio obtained from the model based on five factors, corresponds to the values A=3, B=3, C=3, D=3, E=3. However, substituting these results into the derived mathematical model yields a corresponding dependent variable of water bleeding rate Rb=0.266281, which is too high to meet normal usage requirements. To ensure that the water bleeding rate meets on-site demands, a two-factor correlation analysis is employed to further reduce the number of independent variables, thereby ensuring that the optimal solution obtained can satisfy on-site requirements.

4. Model Optimization

4.1. Two-Factor Correlation Analysis

Based on the analysis of response surface methodology, the influence of the interaction of two factors on the dependent variable of water bleeding rate was analyzed, determining the optimal values corresponding to five factors. However, considering the applicability of the optimal solution to practical situations, after substituting the obtained optimal solution into the model and performing calculations, it was found that the water bleeding rate was excessively high, making it unusable on-site. This section employs SPSS to conduct a two-factor correlation analysis between each factor and the dependent variable of water bleeding rate, further determining the optimal solution by excluding factors with minimal impact. To ensure that the experimental data can undergo two-factor correlation analysis, results demonstrated that the water bleeding rate, in addition to having an approximately linear relationship with the water-cement ratio, exhibited an irregular distribution in the scatter plots corresponding to the other four factors. To confirm the prerequisites for satisfying two-factor correlation analysis, a normality test was conducted on the experimental data, with the test results shown in Table 6. After conducting the normality test calculations using SPSS, it was found that both the Kolmogorov-Smirnov and Shapiro-Wilk significance values were less than 0.05. Additionally, it can be observed that the actual values and expected normal values in the normality plot of the water secretion rate are linearly correlated, as shown in Figure 10. Therefore, it can be concluded that the experimental data meet the criteria for normal distribution, allowing for the implementation of a two-factor correlation analysis.

Table 6. Normality Test

	Kolmogorov-Smirnov test			Shapiro—Wilk test		
	Statistic	Degrees of Freedom	Significance	Statistic	Degrees of Freedom	Significance
bleeding rate	.237	25	.001	.782	25	.000

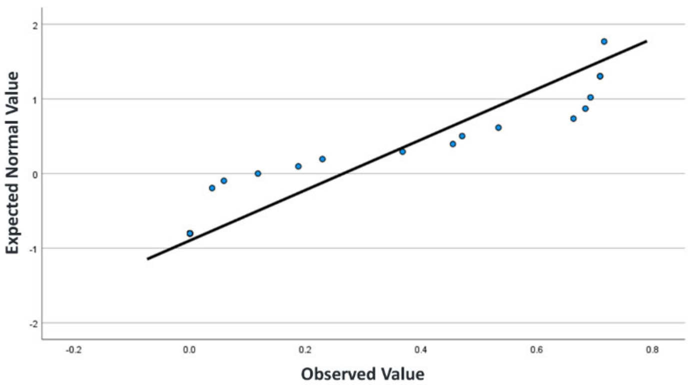


Figure 10. Normal Q-Q plot of water bleeding rate

The results of the analysis of the dual-factor correlation indicate that among the five selected independent variables and the dependent variable, the water-cement ratio and the bleeding rate show a significant correlation at the 0.01 level, while the thickening agent shows a significant correlation at the 0.05 level. Taking into account the cost and environmental benefits during application, the water-cement ratio, thickening agent, and fly ash are selected as the variables related to strength for the correlation experiment design. Additionally, considering the optimization of the mathematical model established for bleeding, the accelerator and gypsum are chosen as fixed values, with a dosage of 1.5%.

Table 7. Two-Factor Correlation Results

		W/C ratio	Fly ash	Thickening agent	Quick-setting agent	Gypsum	bleeding rate
W/C ratio	Pearson correlation	1	.000	.000	.000	.000	.699**
	Significance (Two-tailed)		1.000	1.000	1.000	1.000	.000
	Case count	25	25	25	25	25	25
Fly ash	Pearson correlation	.000	1	.000	.000	.000	-.092
	Significance (Two-tailed)	1.000		1.000	1.000	1.000	.662
	Case count	25	25	25	25	25	25
Thickening agent	Pearson correlation	.000	.000	1	.000	.000	-.500*
	Significance (Two-tailed)	1.000	1.000		1.000	1.000	.011
	Case count	25	25	25	25	25	25
Quick-setting agent	Pearson correlation	.000	.000	.000	1	.000	-.138
	Significance (Two-tailed)	1.000	1.000	1.000		1.000	.510
	Case count	25	25	25	25	25	25
Gypsum	Pearson correlation	.000	.000	.000	.000	1	-.022
	Significance (Two-tailed)	1.000	1.000	1.000	1.000		.916
	Case count	25	25	25	25	25	25
bleeding rate	Pearson correlation	.699**	-.092	-.500*	-.138	-.022	1
	Significance (Two-tailed)	.000	.662	.011	.510	.916	
	Case count	25	25	25	25	25	25

**. The correlation is significant at the 0.01 level (two-tailed).
*. The correlation is significant at the 0.05 level (two-tailed).

4.2. Model construction

The experimental group obtained through orthogonal experimental design was tested using the software Design Expert 13.0 and SPSS, ultimately resulting in a mathematical model of the water bleeding rate Rb in relation to the selected five factors.

$$\begin{aligned} R_b =& -1.40233A+0.050657B-0.294060C+0.25998D-0.350341E+0.056910AB \\ & -0.048637AC+0.594569AD-0.010676BD+0.091440CD+0.108945CE \\ & +0.100672DE-0.000260B^2-0.019515ABD-0.031338CDE-0.000431AB^2 \\ & +0.000052B^2D+0.000145AB^2D-1.65796 \end{aligned} \quad (2)$$

The excessive water bleeding rate cannot be applied on-site. A two-factor correlation analysis of various factors and the dependent variable of water bleeding rate was conducted using SPSS. By substituting the optimal solutions corresponding to D and E from the five factors into the derived formula, the mathematical model of water bleeding rate in relation to the water-cement ratio, fly ash, and thickening agent can be expressed as:

$$\begin{aligned} R_b =& 0.381377A-0.47629B+0.025053C \\ & +0.001635AB-0.048637AC-0.000104B^2 \\ & +0.000004AB^2-1.022971 \end{aligned} \quad (3)$$

5. Summary

In order to ensure that the material can achieve effective bonding after mixing with water, and that the water release meets the corresponding requirements during use, five types of raw materials were selected, with cement as the main bonding material. At the same time, to reduce costs, fly ash, accelerators, and other materials were chosen for use in combination. To obtain a reasonable proportion, a BBD experimental design was established to create an experimental table. Through the experiments, a mathematical model was developed for the dependent variable of water bleeding rate and the independent variables of raw materials and their corresponding contents, and variance analysis was conducted to assess the accuracy of the model. Additionally, to ensure that the model meets on-site requirements during practical application, a two-factor correlation analysis was employed to optimize the number of independent variables, further enhancing the model's practicality on site.

References

[1] Frías-Rojas, Moisés, et al. "New trends for nonconventional cement-based materials: Industrial and agricultural waste." Sustainable and nonconventional construction materials using inorganic bonded fiber composites. Woodhead Publishing, 2017. 165-183.

[2] Xia Junwu, Su Qiong, Liu Dandan. Optimal gypsum-lime content of high water material[J]. Materials Letters, 2018,215: 284-287.

[3] Zhang Yaohui, Wang Chun, Ren Yumeng, et al. Effects of mixing water and environmental pH value on the properties of sulfoaluminate cement-based ultra-high water materials[J]. Scientific Reports, 2024,14(1): 15939.

[4] Xi Xian, Sun Lulu, Shi Quanlin, et al. Effects of mineral admixture on properties of cement-based foam material

developed for preventing coal spontaneous combustion[J]. Fuel, 2023,342: 127785.

[5] Yao Suwan, Liu Songhui, Zou Dinghua, et al. Development of an Aerated Sulfoaluminate Cement–Based Material for Coal Mine Filling[J]. Journal of Materials in Civil Engineering, 2023,35(2): 4022436.

[6] BAI Jianbiao, ZHANG Zizheng, WANG Xiangyu, et al. Stress control and surrounding rock strengthening mechanism of gob-side entry retaining with high-water content material filling and its application[J]. Coal Science and Technology, 2022,50(6): 16-28.