Strength Analysis and Ratio Optimization of New Sealing Materials With Response Surface Method

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Research

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Abstract

The influence of the interaction among water-cement ratio, content of expansion agent, water reducing agent, and retarder on the compressive strength of new sealing material was studied. The Design-Expert software was used to design experiments, establish a quadratic polynomial regression model, draw response surface, and optimize parameters. The microstructure morphology of the sample is explored by scanning electron microscope (Hereinafter referred to as SEM). The research results show that the interaction between water-cement ratio and expansion agent content is the most crucial factor affecting the compressive strength of the new sealing material. Under the optimal condition of 0.4% water reducing agent, 0.04% retarder, 0.8 water-cement ratio, and 8% expansion agent, the compressive strength of the sealing material cured for 3 d and 7 d is 39.247 MPa and 41.044 MPa, with the maximum absolute error of 1.71% and 2.81%, which proves the high accuracy of the model. The main hydration products of the new sealing material are ettringite and C-S-H gel, interlacing each other to form a dense structure, which contributes to the high strength of the new sealing material.

1 Introduction

China is currently faced with the most severe gas disaster in coal mines in the world (Yuan 2016; Zhang et al. 2019a; Xue and Yuan 2017). Gas pre-drainage is effective for coal mine gas control, and the the performance of sealing material is the key to ensuring drainage efficiency.

The coal mine gas drilling sealing material can be divided into inorganic grouting material and organic grouting material. Inorganic sealing materials are mainly cement mortar low-cost but easy to shrink and crack (Guo et al. 2019; Zhu et al. 2019). Organic sealing materials, including polyurethane, modified resin (Gao et al. 2017; Niu et al. 2018; Yang et al. 2016) have a better sealing effect but are expensive, flammable, and toxic (Guan et al. 2020; Sui et al. 2015). In recent years, scholars have studied composite sealing materials for coal mines. Some researchers have developed a CF expansion sealing material with cement, the base material, and metal Al powder, gypsum, and other inorganic additives with good expansibility (Li et al. 2018a; Zhang et al. 2019b). Glass fiber would reduce the fluidity of pure cement paste, and the higher the content of glass fiber, the more pronounced the effect (Wang et al. 2018). Carbon nanofibers can effectively increase the shrinkage and crack resistance of ultra-fine cement while keeping the hardness unchanged (Hogancamp et al. 2017). Fly ash were using as a base material and adding water-retaining agent, expansion agent. This material, with a certain degree of expansion, is not easy to shrink and adheres tightly to coal, and could significantly improve the mechanical properties of grouting materials (Zhai et al. 2013; Zhang et al. 2019c; Zhai et al. 2016). The strength of the sulfoaluminate cement sealing material decreases rapidly with the increase of the water-cement ratio (Chen et al. 2019). Portland cement have used to modify the polyurethane material and developed a new grouting material, which not only keeps good mechanical properties of polyurethane material but also overcomes the shortcoming of flammability (Feng et al. 2013).

The optimization of sealing materials has not been extensively studied using multiple nonlinear regression analysis. This paper investigates the performance of composite sealing materials from the perspective of macromechanics and microstructure with the quadratic polynomial regression model. The results reveal that the interaction between the water-cement ratio, retarder, expansion agent, and water reducing agent in the composite sealing material system significantly affects the compressive strength of the sealing material cured for 3d and 7d. The optimal ratio is determined by the verification test, and the mechanism of action of the new sealing material is revealed by SEM analysis.

2 Materials And Method

2.1 Materials

The base material used in the experiment is ultrafine Portland cement. The measured value of D90 and D50 are 12.6 μm and 5 μm , respectively, which can be effectively injected into micro-cracks below 0.1 mm. The seaweed powder with a white appearance, which is easily soluble in water, is used as the retarder. The solution is a colorless and transparent viscous liquid, which can advance the setting time and improve the strength of cement materials (DU et al. 2019). After being added, PCE (Polycarboxylic acid water-reducing agent), a white powder, which is easily soluble in water, can disperse cement particles with the water-reduction rate of 20–35% (Shui et al. 2016; Zhang and Kong 2015). The compound expansive agent of calcium oxide and calcium sulfate (HCSA), with a gray-white appearance, promotes a good spatial structure of cement hydration products (Feng et al. 2012; Pan et al. 2020). The composition of ultrafine cement and expansion agent used in the experiment is shown in Table 1.

Table 1
Chemical composition of raw materials

Chemical composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Loss	Total
HCSA expanding agent	4.96	8.52	0.99	64.18	2.67	16.97	1.19	99.48
Superfine Portland cement	20.57	9.89	3.08	57.65	2	2.7	2.6	98.49

2.2 Method

A total of 29 sets of experiments with four factors and three levels was designed by Design-Expert 8.0.5 software (Table 2). The four factors are water-cement ratio, retarder content, water reducing agent content, expansion agent content, as shown in Table 2. The materials were mixed, then water was added, and the mixture was poured into the triple mold (7.07 cm × 7.07 cm × 7.07 cm). The mold surface was scraped flat and put into the curing box. After curing the mixture for one day, the mold was removed, and then the stone body was cured for 3d and 7d.

The compressive strength of stones of different ages was tested by the RMT uniaxial press (Fig. 2). The microstructure of the new sealing material and ordinary cement materials was then observed by the FEI Quanta 200 FEG environmental scanning electron microscope. After the experiment, the response surface regression analysis was carried out by Design-Expert 8.0.5 software. By taking compressive strength as the response value, a quantitative regression model of the relationship between the compound dosing amount of admixtures was obtained (Lan et al. 2019), which provides a basis for optimizing the sealing material.

Table 2
Range of factors in the experiment

Test group	Value range of each factor/%			
	Water–cement ratio	Retarder	Water–reducing agent	Expanding agent
ZJ1-29	0.8 ~ 1.0	0.03 ~ 0.05	0.3 ~ 0.5	8–10

3 Experimental Results

It can be seen from Table 3 that after 3d curing (temperature 20 ± 2°C, relative humidity above 95%), the minimum compressive strength of the new sealing material is 23.997 MPa, and the maximum is 39.918 Mpa; After 7d curing (the curing conditions are the same), the minimum compressive strength is 27.012 MPa and the maximum is 42.197 MPa. These results show that the new sealing material has good mechanical properties.

Table 3
Response surface test design and test results

NO	Parameter value				R _{3d} /MPa	R _{7d} /MPa
	Water–cement ratio	Retarder	Water–reducing agent	Expanding agent		
1	0.80	0.04	0.40	8.00	39.918	42.197
2	0.80	0.04	0.40	10.00	30.986	32.991
3	0.80	0.04	0.50	9.00	37.697	38.477
4	0.80	0.05	0.40	9.00	31.652	34.133
5	0.80	0.04	0.30	9.00	33.997	35.010
6	0.80	0.03	0.40	9.00	29.899	32.069
7	0.90	0.03	0.40	10.00	26.943	28.511
8	0.90	0.03	0.30	9.00	27.696	31.233
9	0.90	0.03	0.40	8.00	30.931	32.871
10	0.90	0.03	0.50	9.00	28.479	32.141
11	0.90	0.04	0.40	9.00	29.601	30.381
12	0.90	0.04	0.30	10.00	31.239	32.011
13	0.90	0.04	0.40	9.00	29.601	30.381
14	0.90	0.04	0.40	9.00	29.601	30.381
15	0.90	0.04	0.30	8.00	32.986	36.021
16	0.90	0.04	0.40	9.00	29.601	30.381
17	0.90	0.04	0.50	8.00	35.112	38.021
18	0.90	0.04	0.50	10.00	31.213	32.997
19	0.90	0.04	0.40	9.00	29.601	30.381
20	0.90	0.05	0.50	9.00	27.162	29.133
21	0.90	0.05	0.40	8.00	28.922	30.410
22	0.90	0.05	0.40	10.00	26.993	27.012
23	0.90	0.05	0.30	9.00	25.896	28.033
24	1.00	0.04	0.40	8.00	28.911	31.036
25	1.00	0.04	0.50	9.00	26.976	29.989
26	1.00	0.04	0.40	10.00	26.513	27.978
27	1.00	0.03	0.40	9.00	26.295	28.091
28	1.00	0.05	0.40	9.00	23.997	27.843
29	1.00	0.04	0.30	9.00	27.664	29.008

4 Response Surface Optimization Analysis

According to the experimental results in Table 3, the water-cement ratio, retarder, water reducing agent, and expansion agent were set as independent variables A, B, C, D, and the compressive strength of the samples cured for 3d and 7d were used as the objective function for multiple nonlinear regression fitting.

4.1 Model establishment

The quadratic equation model is the best fitting model, according to Design-Expert 8.0.5 software. The multinomial regression equations of 3d compressive strength (Y3d) and 7d compressive strength (Y7d) related to independent variables A, B, C, and D were:

$$Y_{3d} = +306.38742 - 250.81417 \times A + 2642.85833 \times B + 64.7475 \times C - 45.55025 \times D - 1012.75 \times A \times B - 109.7 \times A \times C + 16.335 \times A \times D + 120.75 \times B \times C + 51.475 \times B \times D - 5.38 \times C \times D + 84.275 \times A^2 - 28622.5 \times B^2 + 104.425 \times C^2 + 1.613 \times D^2 \quad (1)$$

$$Y_{7d} = +429.00175 - 384.545 \times A + 1793.21667 \times B - 63.415 \times C - 49.48733 \times D - 578 \times A \times B - 62.15 \times A \times C + 15.37 \times A \times D + 48 \times B \times C + 24.05 \times B \times D - 2.535 \times C \times D + 144.49167 \times A^2 - 19728.33333 \times B^2 + 185.14167 \times C^2 + 1.84917 \times D^2 \quad (2)$$

The positive and negative coefficients indicate the increase and decrease of the response value caused by the change of the independent variable.

4.2 Model variance analysis and significance test

As shown in Table 4, the P values of the two models are both less than 0.0001, indicating a small experimental error (Bao 2019). It can be seen from the significance test that the significance order of each factor in the regression equation of the sample cured for 3d is as follows: A (water-cement ratio) > D (expansion agent) > C (water reducing agent) > B (retarder); Interaction of different factors: AD (water-cement ratio, expansion agent) > AC (water-cement ratio, water-reducing agent) > AB (water-cement ratio, retarder) > CD (water-cement ratio, expansion agent) > BD (retarder, expansion agent) > BC (retarder, water reducing agent). The P value of A and D is less than 0.0001, indicating extremely significant influence. The P value of B and C is greater than 0.05, showing the insignificant impact; The P value of AD and AC is less than 0.05, suggesting a significant effect. The P value of AB, BC, BD, and CD is greater than 0.05, indicating insignificant influence. The P value of the BC is 0.8102, meaning the least significant impact.

The order of the significance of each factor in the regression equation of specimen cured for 7 days is as follows: A (water-cement ratio) > D (expansion agent) > C (water reducing agent) > B (retarder); Interaction of different factors: AD (water-cement ratio, expansion agent) > AC (water-cement ratio, water-reducing agent) > AB (water-cement ratio, retarder) > CD (water-cement ratio, expansion agent) > BD (retarder, expansion agent) > BC (retarder, water reducing agent). The P value of A and D is less than 0.0001, indicating extremely significant influence. The P value of B and C is greater than 0.05, indicating an insignificant impact. The P value of AD is less than 0.05, indicating significant interaction. The P value of the AC changes from 0.0432 to 0.3482, suggesting that the interaction between A and C has a more significant impact on the sample cured for three days, and the longer the sample is cured, the less significant the effect. The P value of BC is 0.9413, indicating the least significant interaction effect.

Table 4
Response Surface Secondary Model and ANOVA Results

Curing age/d	Source	Sum of Squares	df	Mean Square	F value	P value	Significance
3	Model	332.63	14	23.76	24.40	< 0.0001	***
	A	159.82	1	159.82	164.12	< 0.0001	***
	B	2.63	1	2.63	2.70	0.1224	*
	C	4.27	1	4.27	4.39	0.0549	*
	D	43.67	1	43.67	44.85	< 0.0001	***
	AB	4.10	1	4.10	4.21	0.0593	*
	AC	4.81	1	4.81	4.94	0.0432	**
	AD	10.67	1	10.67	10.96	0.0052	**
	BC	0.058	1	0.058	0.060	0.8102	*
	BD	1.06	1	1.06	1.09	0.3145	*
	CD	1.16	1	1.16	1.19	0.2940	*
	A ²	4.61	1	4.61	4.73	0.0473	**
	B ²	53.14	1	53.14	54.57	< 0.0001	***
	C ²	7.07	1	7.07	7.26	0.0174	**
	D ²	16.88	1	16.88	17.33	0.0010	**
	Residual	13.63	14	0.97			
	Lack of Fit	13.63	10	1.36			
	Pure Error	0.000	4	0.000			
	Cor Total	346.26	28				
R ² = 0.9606	AdjR ² = 0.9213	***—P value [−∞,0.0001]; **—P value [0.0001,0.05]; *—P value [0.05,+∞]					
7	Model	328.94	14	23.50	14.33	< 0.0001	***
	A	139.62	1	139.62	85.15	< 0.0001	***
	B	5.81	1	5.81	3.55	0.0807	*
	C	7.43	1	7.43	4.53	0.0515	*
	D	70.35	1	70.35	42.91	< 0.0001	***
	AB	1.34	1	1.34	0.81	0.3819	*
	AC	1.55	1	1.55	0.94	0.3482	*
	AD	9.45	1	9.45	5.76	0.0308	**
	BC	9.216E−003	1	9.216E−003	5.621E−003	0.9413	*
	BD	0.23	1	0.23	0.14	0.7128	*
	CD	0.26	1	0.26	0.16	0.6981	*
	A ²	13.54	1	13.54	8.26	0.0123	**
	B ²	25.25	1	25.25	15.40	0.0015	**
	C ²	22.23	1	22.23	13.56	0.0025	**

Curing age/d	Source	Sum of Squares	df	Mean Square	F value	P value	Significance
	D ²	22.18	1	22.18	13.53	0.0025	**
	Residual	22.96	14	1.64			
	Lack of Fit	22.96	10	2.30			
	Pure Error	0.000	4	0.000			
	Cor Total	351.90	28				
R ² = 0.9348	Adj R ² = 0.8695	***—P value [−∞,0.0001]; **—P value [0.0001,0.05]; *—P value [0.05,+∞]					

As the items (F value, R²) of conformity degree comparison were relatively abstract, it was necessary to verify the selected model in other ways. The studentized residual was used to compare the predicted residual value and the actual value (Fig. 3). The points were mostly concentrated in the center of the map in terms of the abscissas and was distributed approximately on a straight line, indicating that the model was reliable.

4.3 Response surface analysis

Figure 4 is the three-dimensional surface obtained according to the response value under the interaction of each two independent variables, where the X-axis and the Y-axis show the factor level, and Z-axis shows the compressive strength. The interaction and law of the other two factors under the condition that any two of the four factors A (water-cement ratio), B (retarder content), C (water-reducing agent content) and D (expansion agent content) remain unchanged were determined.

Figure 4 (a) and (b) show the influence of the interaction between water-cement ratio and expansion agent on the compressive strength of the sample. The compressive strength of the sample varies greatly with the water-cement ratio or the value of the expansion agent, At this time, the response surface is steep, which indicates that the interaction of water-cement ratio and expansion agent has a significant influence on the response value. It is consistent with the results of the previous analysis of variance. When the water-cement ratio is low, the compressive strength of the sample increases rapidly with the increase of the expansion agent content. When the water-cement ratio increases to above 0.9, the compressive strength of the sample increases slowly with the increase of the expansion agent content. When the content of expansion agent is low, the compressive strength of the sample increases rapidly as the water-cement ratio decreases. When the content of expansion agent increases to more than 9%, the compressive strength of the sample increases slowly with the decrease of water-cement ratio.

Figure 4 (c) and (d) show the influence of the interaction between water-cement ratio and water reducing agent on the compressive strength of the sample. The compressive strength of the sample has a steeper change trend, indicating that the interaction between the water-cement ratio and the water reducing agent has a significant impact on the response value.

The smoother response surface in Fig. 4 (e) and (f) indicates less significant influence of the interaction of the retarder and water-cement ratio on the compressive strength. When the water-cement ratio remains unchanged, the compressive strength of the sample increases first and then decreases with the increase of retarder content. When the content of the retarder is constant, the compressive strength of the sample increases rapidly with the decrease of the water-cement ratio, and the steep response surface shows that the retarder has little effect on the compressive strength.

As shown in Fig. 4 (g) and (h), when the content of the expansion agent is constant, the compressive strength increases with the increase of the water reducing agent, indicating that the expansion agent has less influence on the fluidity. When the value of the water reducing agent reaches the upper limit, and the value of the expansion agent reaches the lower limit, the response surface is steep, indicating that the interaction of the two factors has the greatest impact on the compressive strength.

Figure 4 (i) and (j) show the influence of the interaction between retarder and expansive agent on the compressive strength of the sample. The change rule of the graph is consistent with the influence rule of the two-factor ANOVA on the response value in the Table 4. When the content of the expansion agent remains constant, the compressive strength increases and then decreases with the increase of the content of the retarder. When the content of the retarder remains unchanged, the compressive strength of the sample decreases rapidly with the increase of the content of the expansion agent, indicating that the effect of the water-cement ratio on the response value is more significant than that of the retarder. The response surface is relatively flat on the whole, indicating that the influence of the interaction between the retarder and the expansive agent is small.

As shown in Fig. 4 (k) and (l), when the retarder interacts with the expansion agent, the response surface is relatively smooth, indicating that the influence of the interaction on the compressive strength is the least significant, which is consistent with the results of the variance analysis.

4.4 Response surface optimization prediction and verification

The experimental results were further analyzed by taking the compressive strength of the sample as the optimization index. The optimized experimental scheme was obtained by the Design-Expert software where the content of water reducing agent is 0.4%, the content of retarder is 0.04%, the water-cement ratio is 0.8, and the content of expansion agent is 8%. The scheme was then verified. Due to limited space, the results were shown in Table 5 without being described in detail. It can be seen that the compressive strength of the specimen cured for 3 d and 7 d under the optimal condition are 39.247 MPa and 41.044 MPa, with the maximum absolute error between the predicted value and actual value of compressive strength as 1.71% and 2.81%, respectively, indicating that the model is relatively reliable.

Table 5
Optimum Scheme and Results

NO	3d Compressive strength(actual)/MPa	3d Compressive strength(prediction)/MPa	Error/%	7d Compressive strength(actual)/MPa	7d Compressive strength(prediction)/MPa	Error/%
1	39.918	39.247	1.71	42.197	41.044	2.81
2	39.554	39.247	0.78	42.031	41.044	2.40
3	39.487	39.247	0.61	41.567	41.044	1.27
4	39.593	39.247	0.88	41.214	41.044	0.41
5	39.229	39.247	-0.046	41.299	41.044	0.62

3 Microscopic Characterization Of Sealing Material

It can be seen from Fig. 5 (a), (b) that the connection between the hydration products of the ordinary cement material during curing is loose, resulting in a lower compressive strength. When cured for three days, the cement clinker is encapsulated by C-S-H gel with obvious pore structure and large pore volume; After 7-day curing, the surface area of the C-S-H gel gradually increases, the volume gradually expands, and the pore becomes smaller. With the hydration reaction, the number of C-S-H gels increases, and some needle-shaped AFt crystals appear.

It can be seen from Fig. 5 (c), (d) that the gel particles of the new sealing material are interconnected compactly, which increases the strength of the material. After 3d curing, C-S-H gel with dense structure and fewer pores were formed, which were wrapped with cement clinker; After 7d curing, a large number of ettringite crystals were formed, indicating that the admixture can promote cement hydration reaction, and AFt crystals were generated with expansion effect. The C-S-H gel and AFt were densely and uniformly cross-bonded together, thereby increasing the strength of the material (Li 2018b; Termkhajornkit et al. 2015; Sekkal et al. 2016).

5 Conclusions

The results obtained in this study made it possible to draw the following conclusions.

- (1) An orthogonal experiment, taking the compressive strength as the response value, was designed using Design-Expert software. The quadratic model about the compressive strength and test factors (water-cement ratio, water reducing agent, retarder, expansion agent) was established and optimized.
- (2) Through variance analysis and the significance test, the significance order of the influence of each component on the compressive strength were obtained as follows: Water cement ratio $\gg$ expansion agent $\gg$ water reducing agent $\gg$ retarder. According to the response surface drawn by the regression equation, the interaction between water-cement ratio and expansion agent has the most significant effect on the compressive strength, and the interaction between retarder and water reducing agent has the least significant effect on the compressive strength.

(3) The optimal experimental condition was obtained by response surface analysis: water reducing agent content of 0.4%, retarder content of 0.04%, the water-cement ratio of 0.8, and expansion agent content of 8%. The compressive strength of the new sealing material cured for 3d and 7d were 39.247 MPa and 41.044 MPa, with the maximum absolute error of 1.71% and 2.81%, respectively. The predicted value was highly consistent with the measured value.

(4) Compared with ordinary cement materials, the new sealing material has a denser overall structure and fewer pores. The hydration products C-S-H gel, Aft, and other cementing materials, which are uniformly cross-bonded together, increase the uniformity and compactness of the material, thus improving the strength of the material.

Declarations

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All authors contributed to this paper. Xin Guo prepared and edited the manuscript. Sheng Xue made a substantial contribution to the data analysis and revised the article. Chunshan Zheng and Yaobin Li reviewed the manuscript and processed the results of numerical simulation during the research process.

Data Availability

The data used for conducting classifications are available from the corresponding author authors upon request.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and / or publication of this article.

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Figures

(a) Ultrafine portland cement

(b) Expansion agent

(c) Water-reducing agent

(d) Retarder

Figure 1

Raw materials of the sealing material

Figure 2

Compressive strength test

Figure 3

Studentized Residual Distribution

Figure 4

Three-dimensional response curve of compressive strength

(a) 3d curing of ordinary cement

(b) 7d curing of ordinary cement

(c) 3d curing of new materials

(d) 7d curing of new materials

Figure 5

SEM micro-morphology of hydration products of samples at different ages