Ratio Optimization of Fly Ash-Based All-Solid Waste Paste Filling Materials

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Abstract. To address the high cost and energy consumption issues associated with traditional cement-based materials, and in response to the national policies of carbon peak and carbon neutrality. This study utilizes bulk solid waste to prepare a fully solid waste paste filling materials. Materials such as fly ash, slag, steel slag, and desulfurized gypsum were used. Single-factor experiments and orthogonal experiments were conducted to optimize the mix ratio. The results indicated that after mix ratio optimization, the filling materials samples achieved compressive strengths of 0.866,2.489 and 5.612 MPa at 3days,7days and 28days, respectively, with a fluidity of 220 mm and a bleeding rate of 2%, meeting the specification requirements for strength and fluidity in mine paste filling. The results of the study provide a reference for field operations and cost reduction and low carbon reduction for field operations and cost reduction and low carbon emission reduction.

Keywords: paste filling; orthogonal design; fluid performance; compressive strength

1 Introduction

With the development and utilization of mineral resources, a large amount of solid waste is generated, occupying land and causing significant environmental damage. Mines in China must undergo backfilling and restoration after mining [1]. The rapid development of mining engineering has led to the widespread application of the filling mining method [2]. Paste backfilling technology, characterized by its non-segregation and non-sedimentation of the slurry, has developed rapidly worldwide [3-4].Utilizing solid waste to replace a portion of cement in the preparation of high-performance mine backfill materials has become a trend [5-6]. The use of fly ash, slag, steel slag, and desulfurized gypsum in the production of cementitious materials has a broad range of applications. These materials leverage the pozzolanic reaction of fly ash to generate silicates gel, which provides strength to the system [7-8]. Lin et al. [9] prepared green and low-carbon cementitious materials using fly ash, steel slag, and granulated blast-furnace slag, which exhibited good strength and toughness. Liu et al. [10] used the Box-Behnken response surface method, with the amount of cement and lime as independent variables

W. Nie et al. (eds.), *Proceedings of the 2024 6th International Conference on Hydraulic, Civil and Construction Engineering (HCCE 2024)*, Atlantis Highlights in Engineering 37,
https://doi.org/10.2991/978-94-6463-726-7_53

and the compressive strength of the cemented materials as the response target value for mix optimization, allowing for a direct observation of the interactive effects of different factors.

This study aims to prepare all-solid-waste paste backfill materials, using both single-factor and orthogonal designs. To assess the impact of various factors on the filling performance, provides a reference for the green mining and low-carbon development in China.

2 Experiment

2.1 Experimental Material

The raw materials used in this experiment include fly ash(FA), slag(BFS), steel slag(SS), desulfurized gypsum(FGD), and water. The tailings used are iron tailings(IT) The moisture replacement of the IT is less than 5%, the density is 2.48 g/cm³. The coefficient of uniformity (Cu) is 18.06, and the coefficient of curvature (Cc) is 0.82.

The chemical composition of the aforementioned materials was analyzed by XRF as shown in Table 1. The specific surface area of the FA is 3126 cm²/g, the alkali coefficient M_0=0.19, the activity coefficient M_a=0.69, indicating that it is an alkaline material with good pozzolanic activity. The BFS alkali coefficient M_0=1.02, the activity coefficient M_a=0.56, classifying it as highly active alkaline slag. The SS alkali coefficient M_0=3.70, an activity coefficient M_a=4.13, indicating that it is highly active alkaline steel slag. FGD, used as an activator.

Table 1. Chemical composition of raw materials (%)

SMPL	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	SO_3	K_2O	TiO_2	MnO
FA	43.88	30.47	8.07	6.25	5.84	1.13	1.02	2.43	0.24
BFS	29.94	16.90	38.14	0.48	9.82	1.63	0.38	0.62	0.57
SS	10.56	3.74	43.63	30.47	4.67	0.29	0.08	0.64	3.74
FGD	0.56	1.32	37.98	0.45	4.06	51.27	0.06	—	—
IT	60.29	7.76	5.71	14.79	5.30	—	4.17	0.39	0.33

2.2 Experimental Flow

The samples are made by mixing cementitious materials with IT. Based on extensive literature review and preliminary experiments, the cement-sand ratio is determined to be 1:4, and the mortar mass concentration is set at 80%. The initial mix ratio for the cementitious materials is BFS: SS: FGD = 6:3:1. While keeping the ratio of the first three cementitious materials constant, a portion of the cementitious materials is replaced with FA. Single factor test and orthogonal test were used to optimize the ratio. The single-factor tests use the range with different FA replacements (0~60%) denoted as AF1~AF7, different mass concentrations (74%~86%) denoted as BM1~BM7, and different cement-sand ratios (1:4~1:10) denoted as CB1~CB7. The orthogonal tests employ a three-factor, four-level experimental method, with the factors and levels detailed in Table 2.

Table 2. Orthogonal experimental factors and level design

Level	Factor		
	(A) Fly ash replacement	(B) Mass concentration	(C) Cement-sand ratio
1	10	76	1:4
2	20	78	1:6
3	30	80	1:8
4	40	82	1:10

The materials are accurately weighed and placed into a slurry mixer for thorough stirring. After stirring, the fluidity and bleeding rate of the mortar are tested. Then, the well-mixed slurry is poured into cylindrical molds（$\phi\times h$=50mm×100mm）. The samples are then subjected to standard curing for 3 days,7 days and 28 days to test for uniaxial compressive strength. The fluidity test was conducted in accordance with the national standard GB/T2419-2016"Method for Determining the Fluidity of Cement Mortar"; the bleeding rate test was completed according to the industry standard JC/T2478-2018 "Tailings Concrete for Backfilling in Mining Voids"; the compressive strength of the hardened filling materials was tested in compliance with the national standard GB/T17671—2011"Method for Testing the Strength of Cement Mortar".

3 Experimental Results and Discussion

3.1 Analysis of Single Factor Test Results

Fig 1 group a illustrates the uniaxial compressive strength of the filling material with different FA replacements at various ages. Under the same mass concentration and cement-sand ratio, the initial sample without FA had strengths of 4.19 MPa, 6.25 MPa, and 11.17 MPa at 3 , 7 , and 28 days, respectively. Upon the addition of FA, the compressive strength of the samples gradually decreased. When the FA replacement was 10%, the compressive strengths of the samples were 3.09 MPa, 5.24 MPa, and 10.23 MPa, representing decreases of 26.3%, 16.2%, and 8.4%, respectively. When the FA replacement was increased to 60%, compared to the 10% FA replacement, the compressive strengths respectively decreased by 84.9%, 71.3%, and 67.7%. As the amount of FA increases, the calcium-silica ratio of the system decreases, weakening the pozzolanic reaction. The strength generated by the pozzolanic reaction of FA is far from sufficient to compensate for the loss in strength due to the unhydrated reactions in the system, leading to a gradual decrease in compressive strength with increasing FA replacement. It can also be observed that as the FA replacement increases, the fluidity of the filling slurry shows an overall upward trend. It is recommended to select a FA replacement between 10% and 40%.

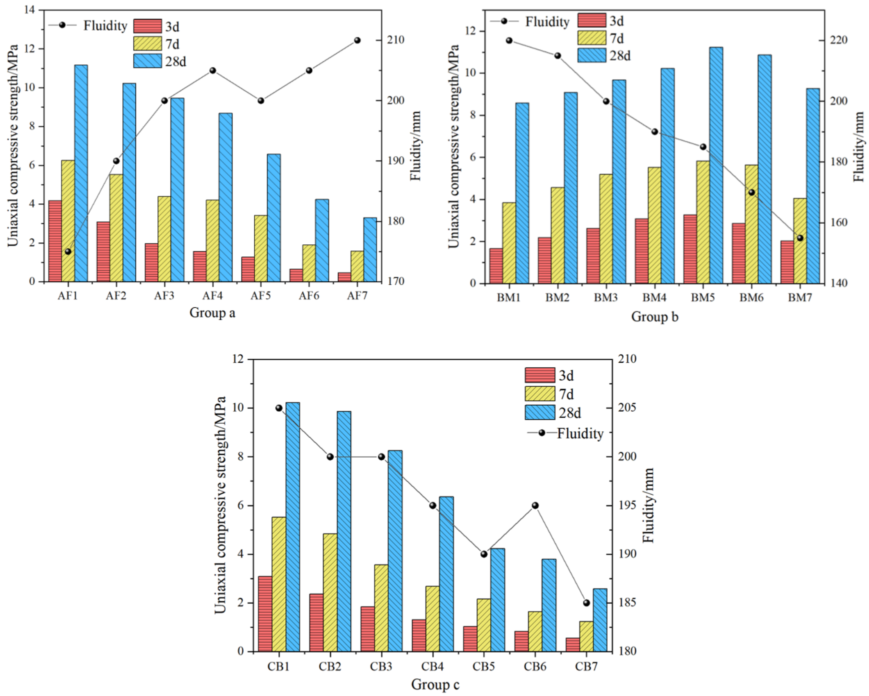

Fig. 1. Single factor design test results

Group b illustrates the uniaxial compressive strength of the filling material at different ages under various mass concentrations. With the same FA replacement and cement-sand ratio, the filling materials achieves its maximum compressive strength at a mass concentration of 82%, with 3day, 7day, and 28day compressive strengths of 3.07 MPa, 5.83 MPa, and 11.23 MPa, respectively. As the mass concentration increases, the compressive strength of the filling materials first rises and then declines, indicating that there is a critical value for mass concentration. Group b also shows the significant impact of mass concentration on fluidity. Mass concentration reflects the mass of mixing water; as mass concentration increases, the reduction in free water increases the cohesion of the filling slurry and the frictional resistance of particle settlement, leading to a decrease in fluidity and bleeding rate. To meet the requirements of paste backfilling, the mass concentration is chosen to be between 76% and 82%.

Group c illustrates the uniaxial compressive strength of the filling material at different ages under various cement-sand ratios. With the same fly ash replacement and mass concentration, the compressive strength of the filling materials decreases as the cement-sand ratio decreases. The filling materials with a cement-sand ratio of 1:10 has compressive strengths at different ages that are reduced by 81.7%, 76.5%, and 74.6% compared to those with a cement-sand ratio of 1:4, respectively. This indicates that the cement-sand ratio significantly affects the compressive strength. The fluidity also generally decreases as the cement-sand ratio decreases, due to the increased replacement of coarser tail sand particles and decreased replacement of finer cementitious materials in the filling slurry. The increased particle friction leads to reduced fluidity and poorer workability of the slurry. The IT tend to flocculate and settle, causing segregation in the mortar, which can lead to construction issues such as pipe blockages and blowouts. Moreover, the segregation phenomenon becomes more pronounced with increased amounts of sand and water used. Therefore, it is vital to carefully control the amount of water and sand used to avoid segregation, ensuring the stability and workability of the filling material during construction.

3.2 Analysis of Orthogonal Test Results

Orthogonal experiments are an efficient method for considering the interactive effects of multiple factors rapidly. By selecting the range derived from single-factor test results for mix optimization, we can identify paste backfill mortars that can be applied to various actual situations in mine backfilling. The results of the 16 groups of experiments are shown in Table 3.

Table 3. Orthogonal experimental design results

Number	Factor			Mechanical performance index			Conveying performance index	
	(A)Fly ash replacement /%	(B)Mass concentration /%	(C)Cement-sand ratio	Compressive strength/MPa 3d	7d	28d	Fluidity /mm	Bleeding rate /%
1	10	76	1:4	2.17	5.25	10.18	215	2
2	10	78	1:6	1.34	3.37	8.43	200	5
3	10	80	1:8	0.85	2.12	5.50	190	6
4	10	82	1:10	0.53	1.48	3.54	175	2
5	20	76	1:6	0.87	2.50	5.61	220	2
6	20	78	1:8	0.65	1.27	3.86	205	6
7	20	80	1:10	0.34	0.78	1.81	200	6
8	20	82	1:4	2.99	6.08	10.64	180	0
9	30	76	1:8	0.40	0.89	2.83	225	10
10	30	78	1:10	0.26	0.54	0.87	210	9
11	30	80	1:4	1.79	3.59	7.84	195	3
12	30	82	1:6	1.37	2.98	6.96	180	0
13	40	76	1:10	0.16	0.33	0.63	230	12
14	40	78	1:4	0.64	1.93	4.45	220	4
15	40	80	1:6	0.38	1.25	2.52	210	5
16	40	82	1:8	0.28	0.65	1.34	195	3

The range analysis is the difference between the maximum and minimum average values of the results, and it is an indicator of how significantly a factor affects the outcome being measured. According to Table 4, the factor that most significantly affects the fluidity is the mass concentration of the filling slurry, with the order of influence on fluidity being mass concentration > FA replacement > cement-sand ratio. The cement-sand ratio is the most important factor affecting the compressive strength of the filling materials, with the order of influence on the compressive strengths being cement-sand ratio > FA replacement > mass concentration. For bleeding rate, the most significant factor is also the mass concentration of the slurry, with the order of influence on bleeding rate being mass concentration > cement-sand ratio > FA replacement. Based on the analysis of the 3day, 7day, and 28day compressive strength results, the optimal mix ratio for compressive strength is $A_2B_4C_1$. However, due to its poor fluidity, it does not meet the requirements for pipeline transportation in mine filling. Considering compressive strength, fluidity, and bleeding rate, the practical applications are $A_1B_1C_1$ and $A_2B_1C_2$. To reduce filling costs and increase the use of tailings, the final selection is $A_2B_1C_2$. This mix ratio maximizes the use of solid waste, meets the requirements for mine filling, and achieves energy saving, emission reduction, low carbon, and cost-effectiveness.

Table 4. Range analysis results

Influence factor	Compressive strength/MPa			Fluidity	Bleeding rate
	3d	7d	28d	/mm	/%
Fly ash replacement(A)	3.44	8.05	18.72	75	15
Mass concentration(B)	2.28	4.07	7.33	160	22
Cement-sand ratio(C)	6.30	13.74	26.26	15	20

4 Conclusions

This study utilizes all-solid-waste paste backfill materials for tailings. Through single-factor experiments and orthogonal tests, the mix ratio is optimized to meet the control and application requirements of mine backfilling. The specific conclusions are as follows:

(1) By single-factor experiments, the trends of different factors' influences were confirmed. As the replacement of fly ash increased, the compressive strength of the filling materials gradually decreased, while the fluidity gradually increased. With the increase in mass concentration, the compressive strength of the filling materrial first increased and then decreased, and the fluidity gradually decreased. As the cement-sand ratio decreased, both the compressive strength and fluidity of the filling materrial gradually decreased. At the same time, the incorporation of fly ash is not conducive to the development of early strength.

(2) By orthogonal experiments, the optimal mix ratio was determined. With a fly ash replacement of 20%, and proportions of 60% for slag, 30% for steel slag, and 10% for desulfurized gypsum, the filling materials samples achieved compressive strengths of 0.866 MPa, 2.489 MPa, and 5.612 MPa at 3, 7, and 28 days, respectively. The fluidity

was 220 mm, and the bleeding rate was 2%, meeting the performance requirements for paste backfilling in mines.

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