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Experimental Study on Strength Development and Engineering Performance of Coal-Based Solid Waste Paste Filling Material

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Abstract: To explore the strength development characteristics and engineering performance of different coal-based solid waste filling materials cemented into filling body, coal gangue was used as coarse material, fly ash, desulfurization gypsum, gasification slag, and furnace bottom slag as fine material, and cement as a gelling agent. The uniaxial compressive strength (UCS) and bleeding rate of coal-based solid waste cemented backfill (CBSWCB) were tested by an orthogonal experiment, and the influencing factors of mechanical properties and strength development were analyzed. The multiple generalized linear model of strength and bleeding rate was established, and the optimal filling material ratio was determined. The engineering performance index of CBSWCB with the optimal ratio was tested. The results show the following points: (1) the concentration and content of desulfurization gypsum had a great influence on the early compressive strength of CBSWCB, while fly ash, gasification slag, and furnace bottom slag had little influence on the early compressive strength. (2) High concentration, high content of fly ash and furnace bottom slag, low content of desulfurization gypsum, and gasification slag can significantly improve the early strength. High concentration and high content of fly ash, low content of gasification slag, furnace bottom slag, and desulfurization gypsum are beneficial to the later strength increase. (3) Under the optimal ratio scheme, the bleeding rate of CBSWCB was 1.6%, the slump was 16.6 cm, the cohesion was general, the segregation resistance was good, the initial setting time was 5.42 h, the final setting time was 7 h, and the early strength after curing for 8 h reached 0.24 MPa.

Keywords: filling mining; coal-based solid waste; orthogonal experiment; strength development; regression analysis; engineering performance

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1. Introduction

With the rapid development of China's economy, the demand for coal resources has increased sharply, and ecological and environmental problems, such as surface subsidence, groundwater pollution, and gangue discharge caused by large-scale mining, have become increasingly prominent [1] (Figure 1). In recent years, filling coal mining technology has become one of the effective technical ways to solve the above-mentioned problems. Among them, paste filling makes solid waste into a paste in proportion and transports it to the goaf. It has developed rapidly in black, non-ferrous, gold, coal, and other systems [2], which effectively solves the coal-based solid waste generated during the deep processing of coal (Figure 2). It avoids environmental pollution, resource waste, surface subsidence, and other problems caused by mining, and achieves good social and economic benefits [3]. However, the comprehensive application of coal-based solid waste in China started late, and the comprehensive utilization path is relatively simple, and large-scale industrial utilization has not yet been achieved. At this stage, the method of land occupation and stacking is mostly adopted, occupying a large number of land resources, and the heavy metals contained in it will pollute the environment, and the added value of deep-processing products is generally low, which will increase the production cost of enterprises [4,5]. Taking desulfurized gypsum as an example, it is mainly used as a cement retarder, paper gypsum board, gypsum block, and other new wall materials. By 2019, the output will reach 71.5 Mt, and the utilization rate has not yet reached 80%, but the utilization rate of Germany, Japan, and other countries has already reached 100%, which is still a large gap compared with developed countries. Therefore, the reduction, harmlessness, and resource utilization of coal-based solid waste are imperative.

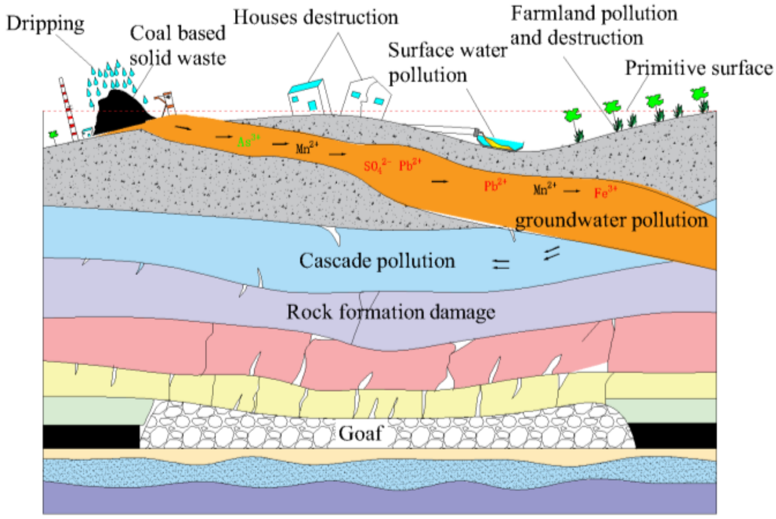


Figure 1. Ecological and environmental problems caused by large-scale mining of coal resources.

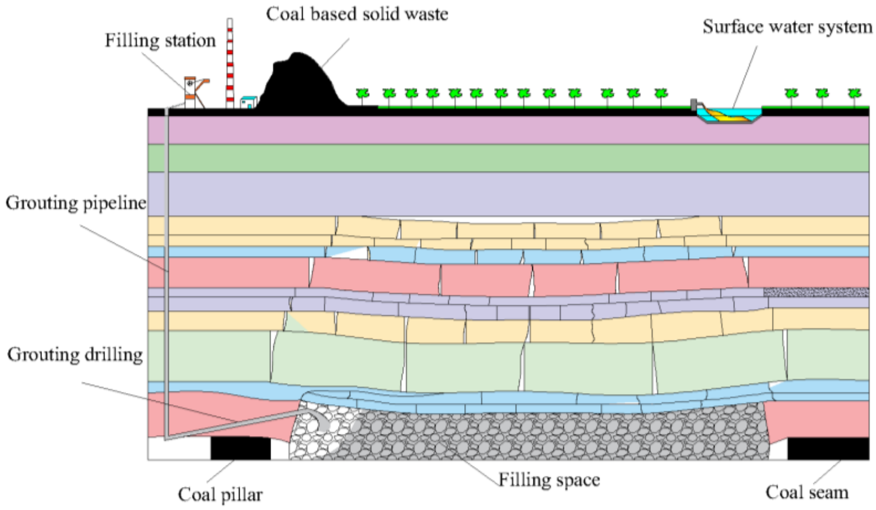


Figure 2. Application of coal-based solid waste in paste filling.

CBSWCB is formed from coal-based solid waste filling materials after a reasonable proportion design to support the rock mass around the stope. Therefore, exploring the strength characteristics, composition and mechanical properties of CBSWCB is the research foundation of backfill mining, and the ultimate goal of backfill mining is to choose low-cost, high-strength, low-pollution, and fluid-filling materials. In recent years, coal-green-filling mining has been widely used. Many scholars at home and abroad have carried out multi-dimensional research on filling materials, mainly focusing on the selection of filling materials and optimization of the ratio [6–9], the influence of mechanical properties [10–12], backfill mechanics constitutive models [13,14] and failure mechanisms [15,16].

At present, certain experimental studies have been carried out on coal-based solid waste filling materials. The filling materials are concentrated in coal gangue, gypsum, fly ash, slag, tailings, etc. [17–20], and the single factor analysis is mainly conducted through orthogonal experiments, regression analysis, visual modeling, neural network learning, response surface methodology, and other methods to analyze and optimize the factors affecting the strength development of the proportioning materials. Ref. [21] used fly ash, coal gangue, ordinary silicate cement, and water as the main filling materials, and found that the quality ratio of filling materials was 10%:20%:50%:20% without additives, which achieved a good filling effect. Ref. [22] tried to replace coal gangue with waste concrete to prepare filling paste, studied its initial flow properties, rheological properties, flow properties after standing, bleeding rate, and compressive strength, and determined the reasonable range of coarse aggregate replacing gangue.

Ref. [23] explored the evolution process and reaction mechanism of geopolymers synthesized by circulating fluidized bed fly ash, and investigated the influence of process parameters such as activator modulus, solid-liquid ratio, curing temperature, and time on the mechanical properties of geopolymers, and optimized the preparation conditions through a neural network. Ref. [24] explored the sensitive influence factors of the slump, compressive strength, and elastic modulus of the gangue-fly ash-tailings cementation through an orthogonal experiment, and they carried out a mix proportion design and determined the optimal mix proportion on this basis. Ref. [25] studied the influence of admixture content and tailings content, and slurry concentration on the fluidity and strength of semi-hydrated phosphogypsum fillings through orthogonal experiments. It was found that slurry concentration had an obvious impact on the strength and fluidity in the early stage, and admixture content had a great impact on the later stage. The water content of cemented paste filling (CPB) technology can ensure that the paste filling material does not ooze out, separate, or solidify when the water content reaches the lowest level during pipeline transportation [26]. The simultaneous unconfined compressive strength (UCS) test is a simple and the most commonly used method to assess the strength of CBSWB, but the information provided by the UCS test is limited. Based on the UCS test, understanding its hydraulic properties and exploring the hydraulic properties of CBSWB can provide a reasonable theoretical basis for the safe and economical structural design of CBSWB [27]. Xinguo Zhang et al. [28] used coal gangue to prepare cementitious paste filling material (CPB), added fly ash to partially replace ordinary Portland cement (OPC), and conducted a UCS test, slump test, bleeding test, and segregation through an orthogonal test, A series of hydraulic-mechanical tests were carried out. On this basis, the selected filling material was applied to the mine, and its filling performance was monitored. Dan Ma et al. [29] studied the evolution of the hydraulic and mechanical properties of coal-based solid waste CPB under different particle size distributions and cementing materials, and analyzed the deterioration mechanism of the mechanical properties. Although the above research has carried out extensive and detailed research, the material selection is generally single, and the research on the interaction between gasification slag and furnace bottom slag as paste filling materials is not in depth enough. At the same time, there are few studies on filling coal mining in the Ningdong coal power base, and the influencing factors of CBSWCB on the performance and strength development of paste filling engineering need to be further studied.

The Coal-Thermal Power-Chemical Industry Base in East Ningxia is a large-scale coal base of 100 million tons and a coal power base of tens of millions of kilowatts in China. In 2018, the coal output was about 91.55 million tons, the coal chemical production capacity was 22.25 million tons, and the thermal power capacity was 14.95 million kilowatts. In recent years, it has produced more than 90 million tons of coal-based solid wastes, such as coal gangue, fly ash, desulfurization gypsum, gasification slag, and furnace bottom slag [30], which is increasing year by year. Renjiazhuang Coal Mine is located on the edge of the Mu Us Desert 20 km northeast of Lingwu City in Ningxia, 30 km west of the Yellow River and facing Yinchuan City, and adjacent to the Hongshiwan Coal Mine in the north. Meihuajing Coal Mine is located in the Yuanyang Lake mining area of the Coal-Thermal Power-Chemical Industry Base in East Ningxia. Yuanyang Lake Power Plant is adjacent to Meihuajing Coal Mine, and the straight line distance between Yuanyang Lake Power Plant and Renjiazhuang Coal Mine is 25.4 km. The straight line distance between Meihuajing Coal Mine and Yuanyang Lake Power Plant is 2.5 km. Convenient transportation, geographical conditions, and sufficient sources of coal-based solid waste materials are conducive to the implementation of coal mine filling and mining. Taking the filling mine of Renjiazhuang Coal Mine in the Coal-Thermal Power-Chemical Industry Base in East Ningxia as the engineering background, this paper selected coal gangue as aggregate, fly ash, gasification slag, furnace bottom slag, and desulfurization gypsum as fine material and cement as cementitious material, established the multiple regression model of CBSWCB through an orthogonal experiment, explores the influence of coal-based solid waste filling material on the mechanical properties of CBSWCB, and analyzes its strength development characteristics. On this basis, the proportion optimization was carried out, and the engineering performance index of the optimization results was tested, to provide a reliable reference for guiding the paste filling mining of coal mine.

2. Test Materials and Schemes

2.1. Material Composition

The chemical composition of the dried coal-based solid waste filling material was determined by X-ray fluorescence (XRF), and the mineralogical composition was determined by X-ray diffraction (XRD). As shown in Figures 3 and 4, the main component of coal gangue is SiO_2, accompanied by some silica-based compounds. A large amount of SiO_2 ensures the high hardness and deformation resistance of gangue. Fly ash is the dry discharged ash of the Yuanyanghu Power Plant. The main components are SiO_2, Fe_2O_3, and CaO. The content of CaO and SiO_2 reaches 92.27%, which reduces the polymerization degree of glass in fly ash and increases its activity. The gasification slag is mainly composed of SiO_2, the bottom slag is mainly composed of SiO_2, $Al_2O_3 \bullet SiO_2$, and Fe_2O_3, and the desulfurization gypsum is mainly composed of $CaSO_4$ and $CaSO_4 \bullet 2H_2O$.

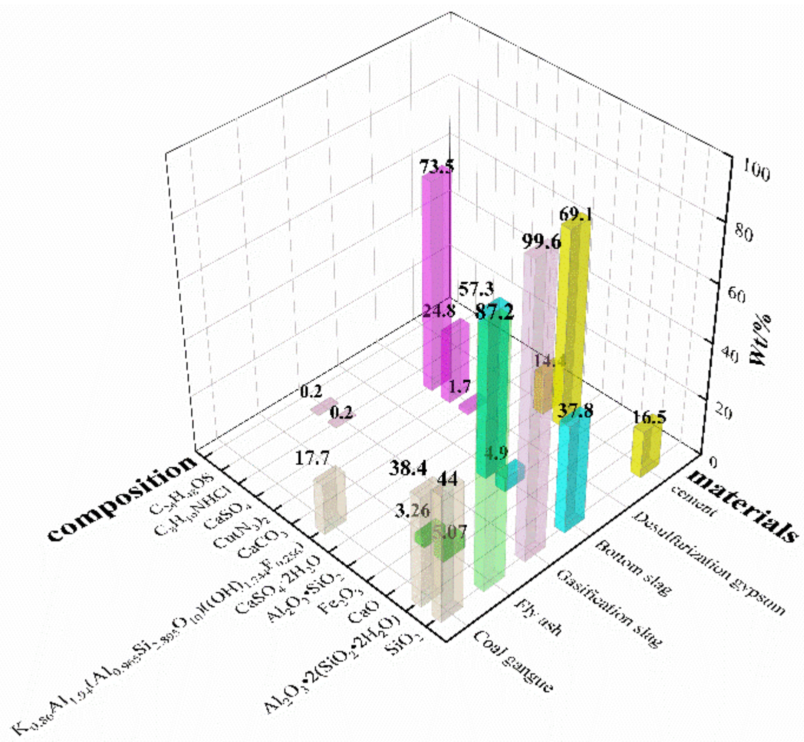


Figure 3. Analysis of main components of coal-based solid waste filling materials.

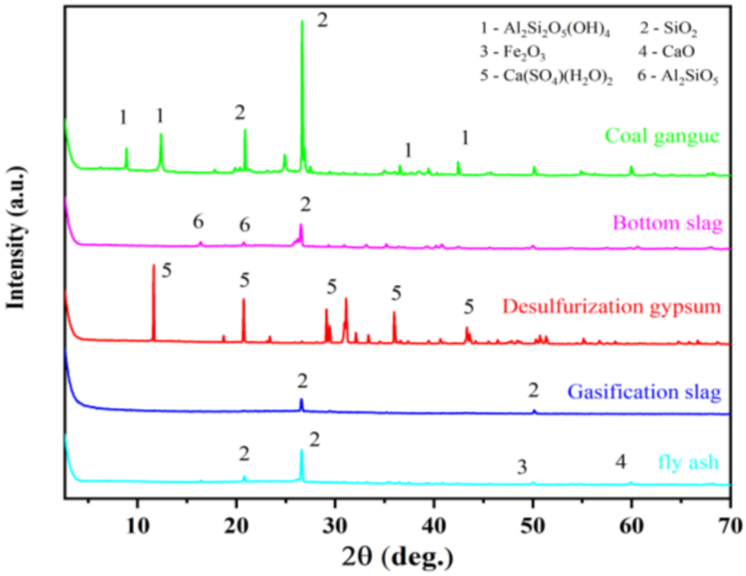


Figure 4. X-ray diffraction (XRD) of coal-based solid waste filling material.

Through the scanning electron microscope (SEM) on six kinds of experimental material microstructure tests, as shown in Figure 5, the gangue microscopic form is mainly a block, flake and other irregular forms. This irregular block form resting on the plane does not easily flow, the gangue large particle structure formed for the maintenance of the initial specimen provides a certain strength, and in the late maintenance stage, the generation of crystalline and gel material attached to the gangue and other large particles occurs. The pores are formed by the particles. The large particles of the morphological structure mean that it is easier for it to play its structural role, increasing the compressive strength of the filled specimen. The fly ash microscopic form is more regular, showing spherical particles and these spherical particles are conducive to their own transport and flow; it is easier for them to penetrate the pores between the large particles, increasing the opportunity to react with the cement hydration products, in addition to desulfurization. The microscopic form of gypsum is irregular in size and prismatic block form, and its particle size is moderate and uniform, which makes the particle composition of the mixture more reasonable; the microscopic form of gasification slag is not uniform, and the surface is rough and irregular, and the rough surface increases its specific surface area, which is more conducive to the adhesion of hydration gel and other substances on its surface, thus making the bonding between the particles of the mixture more tight. The microscopic morphology of furnace slag is irregularly lumpy, with obvious sintering traces on the surface, and the microscopic surface is smooth with fine pores, which are conducive to the entry of free water, and the outward migration of free water in the middle and later stages of maintenance is conducive to the continuation of hydration reaction inside the specimen, thus increasing the strength of the specimen. The microscopic morphology of No. 425 ordinary silicate cement is irregularly lumpy, and the particle size is on the high side. The microscopic form of No. 425 ordinary silicate cement is irregularly lumpy, the particle size is small, and the surface is rough, so the tiny particles will enter the pores between the large particles faster and mix with other materials fully in the mixing process so that the hydration product $Ca(OH)_2$ can react with other components more easily and generate calcium alumina and other crystalline substances, which will increase the strength of the specimen.

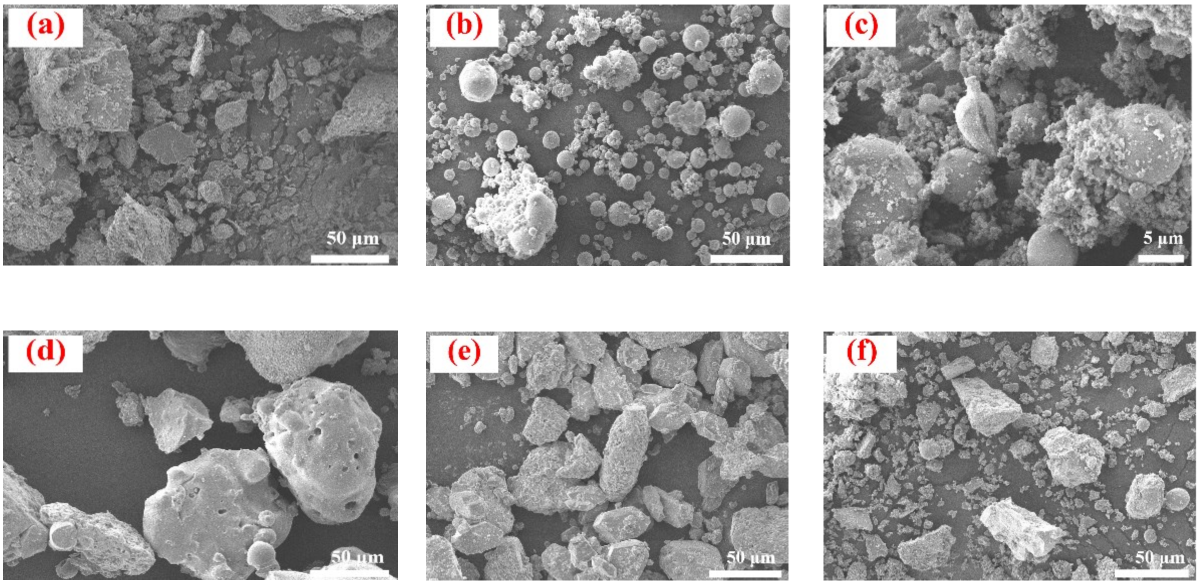


Figure 5. SEM microstructure of coal-based solid waste filling material, with (**a**) coal gangue, (**b**) fly ash, (**c**) gasification slag, (**d**) furnace bottom slag, (**e**) desulfurization gypsum, and (**f**) cement in the figure.

2.2. Test Scheme

An L16(4^5) orthogonal test is adopted. Without special instructions, the ratios involved in this paper are all mass ratios. Considering mainly exploring the filling performance of coal-based solid waste, we took the quality of coal gangue and cement as invariants, the cement mixture was 5% of the total mass of coal-based solid waste, and we set five research factors, namely A (concentration) and B (ash gangue ratio), C (gasification slag: coal gangue), D (bottom slag: coal gangue); E (desulfurized gypsum: coal gangue). Studies have shown that 75% to 80% of paste filler slurry concentration is beneficial to the stability of the roof and surrounding rock in the goaf [3]. Fly ash is coal-based solid waste. Due to its porous structure and spherical particle size, it has good permeability in a loose state and is a good cementitious material. Desulfurization gypsum has a small particle size, stable composition, and less harmful impurities. Fly ash and desulfurized gypsum account for a large proportion, which can meet the requirements of large-scale green economic filling. Gasification slag and bottom slag have pozzolanic properties, and blast slag has been gradually used at home and abroad to replace some cement. Considering the specific filling requirements and output, a gradient design is made for these two kinds of coal-based solid waste. The mixing amount of the two is close to but less than the above three kinds of coal-based solid waste. After comprehensive consideration of how to save the consumption of coal-based solid waste filling materials and test cost, four horizontal gradients were specifically set, as shown in Table 1.

Table 1. L16(4^5) orthogonal experimental factors and levels.

Level	Factor				
	A (Concentration/%)	**B (Ash Gangue Ratio)**	**C (Gasification Slag: Coal Gangue)**	**D (Bottom Slag: Coal Gangue)**	**E (Desulfurized Gypsum: Coal Gangue)**
L1	74	0.3	0.2	0.1	0.2
L2	76	0.4	0.25	0.15	0.3
L3	78	0.5	0.3	0.2	0.4
L4	80	0.6	0.35	0.25	0.5

2.3. Sources of Experiment Materials and Their Preparation

The experiment uses coal gangue as the coarse material, fly ash, gasification slag, furnace bottom slag, desulfurized gypsum as the fine material, and 425 ordinary Portland cement as a cementitious material. Among them, coal gangue comes from Renjiazhuang and Meihuajing coal mines, and fly ash, desulfurization gypsum, gasification slag and furnace bottom slag come from Yuanyang Lake Power Plant. The geographical location and material sources of the mining area are shown in Figure 6. At the same time, the particle size of coal-based solid waste filling materials has an important influence on the strength of coal-based solid waste cemented backfill (CBSWCB). In order to obtain the best strength effect of CBSWCB, the gradation of five kinds of coal-based solid waste took place. The gangue rock blocks were crushed and sieved into three particle size intervals, including (0.0–1.25) mm, (1.25–2.5) mm (2.5–5.0) mm of rock particles, and fly ash, gasification slag, furnace bottom slag. The large blocks in the desulfurized gypsum were crushed and sieved into particles with a particle size of less than 2.5 mm. The particle size composition of gangue is shown in Table 2.

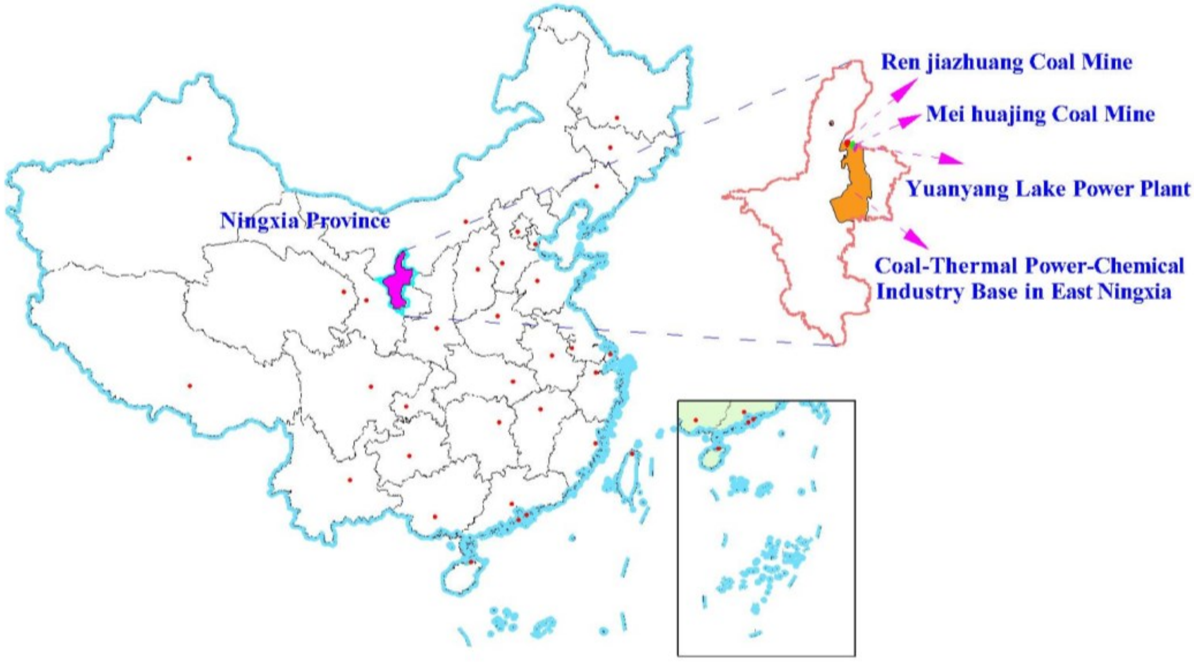
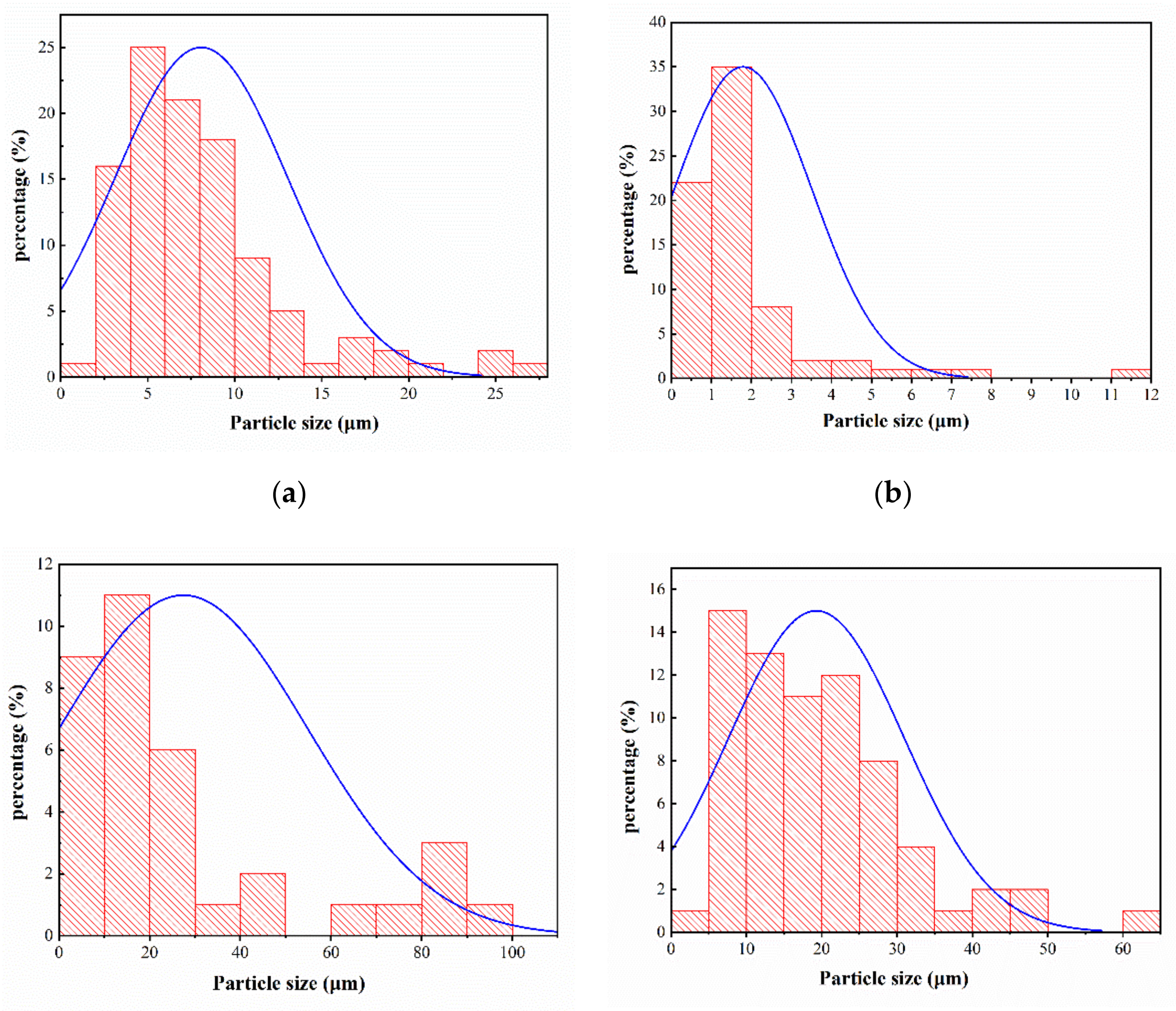


Figure 6. The geographical location of the mining area and sources of coal-based solid waste materials.

Table 2. Particle size composition of gangue.

Particle Size (mm)	Mass Percentage (%)
0–1.25	29.8
1.25–2.5	42.5
2.5–5.0	27.7

Particle size distribution, or particle gradation, is an important parameter for the properties of powder materials, such as fly ash, gasification slag, furnace bottom slag, and desulfurized gypsum. According to the SEM image, the particle size distribution of the four kinds of coal-based solid waste materials is counted. As shown in the Figure 7, the particle size of fly ash is mainly distributed in 5–10 μm, the particle size of gasification slag is mainly distributed in 0–3 μm, and the particle size of furnace bottom slag is mainly distributed in 0–3 μm. The particle size is mainly distributed in 10–30 μm, and the particle size of desulfurized gypsum is mainly distributed in 5–25 μm.



(**a**) (**b**)

(**c**) (**d**)

Figure 7. Particle size distribution diagram of coal-based solid waste materials, including (**a**) fly ash, (**b**) gasification slag, (**c**) bottom slag, (**d**) desulfurized gypsum.

According to the GB\T50080-2016 national standard [31], the quality of coal gangue is fixed, and according to the design plan of various coal-based solid waste materials, the test water and cement were weighed in turn, we used a cement paste mixer to fully stir the mixture, and casted the mixed cementitious material into a standard cube mold of 70.7 × 70.7 × 70.7 mm. We used the vibrating table to reduce the air and gap in the mold, then used the scraper to level the mold surface, and placed the leveled mold indoors, as shown in Figure 8. At the same time, part of the mixed cementitious material was added into the plexiglass container with a height of about 120 mm to ensure that the height of the measured filling surface was consistent and then it was sealed with fresh-keeping film. After standing indoors for 24 h, the bleeding rate was measured and recorded. The 48 sets of experimental data obtained were recorded. Three standard cube molds of 70.7 × 70.7 × 70.7 mm were made in each group of experiments, and a total of 144 standard cube molds were made in the experiment. After indoor curing for 12 h, we took the sample out of the mold and placed it in a constant temperature and humidity curing box at 20 °C and humidity of 90 ± 5% until it was taken out during the test, as shown in Figure 9. It completed the uniaxial compressive strength test on an electro-hydraulic servo rock pressure tester (loading rate is 0.05 KN/s) according to the national standard [32] of GB\T23561.12-2010.

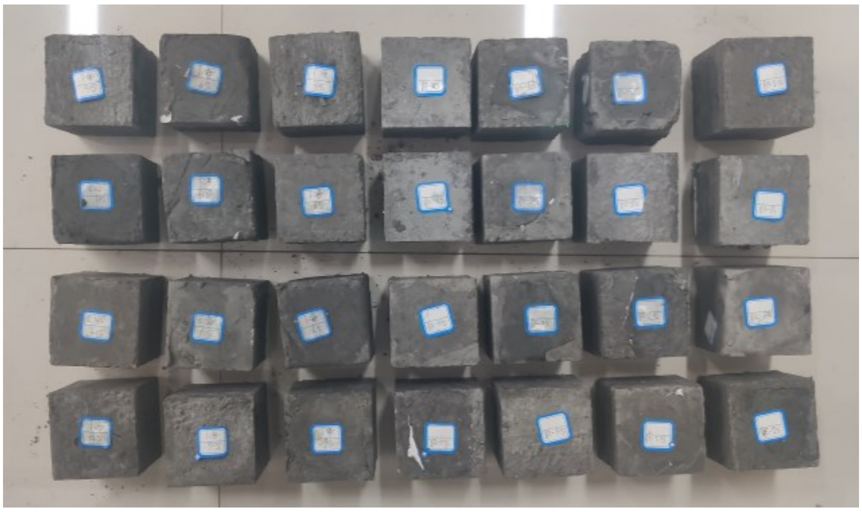


Figure 8. Partial specimen of CBSWCB.

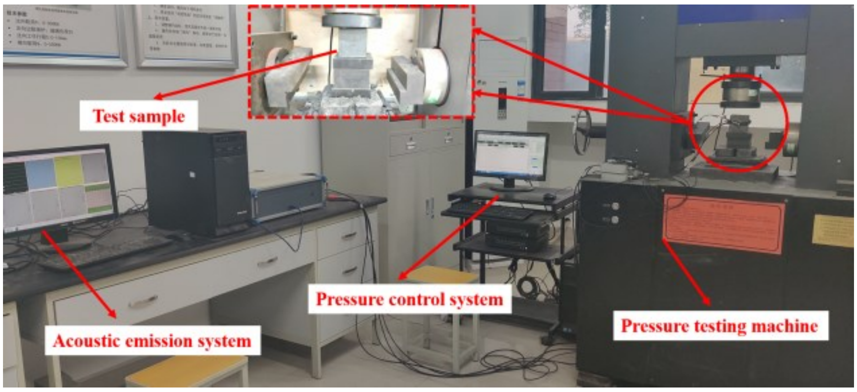


Figure 9. Test system.

While considering the coal-based solid waste filling material dosage and test cost, the optimization model was predicted through the development and regression analysis of coal-based solid waste cemented filling strength, and the required coal gangue, fly ash, gasification slag, furnace bottom slag, and desulfurization gypsum were re-screened according to the optimized proportioning scheme of filling materials obtained, and the secondary experiments were conducted according to GB\T50080-2016. Slump, expansion, initial and final setting time, and strength tests were conducted for the optimized proportioning test to verify the engineering performance and strength development characteristics, which will be described in detail in the later part of this paper.

3. Results

3.1. The Development of Backfill Strength

3.1.1. Sensitivity Analysis of Filling Body Strength

The bleeding rate and uniaxial compressive strength (3 d, 7 d, 28 d) of the coal-based solid waste material backfill were tested by the L16(4^5) orthogonal experiment (3 d, 7 d, 28 d) and the three datasets of bleeding rate are averaged, and the experimental results are shown in Table 3. The arithmetic average of the uniaxial peak compressive strength under the same value of a single factor was taken to obtain the influence of a single factor on the strength development of CBSWCB under the synergistic influence of multiple factors, as shown in Figures 10 and 11.

Table 3. Results of L16 (45) orthogonal experiment of CBSWCB.

Test No.	Factor					UCS (MPa)			Bleeding Rate (%)
	A	B	C	D	E	3 d	7 d	28 d	
S1	74	0.3	0.2	0.1	0.2	0.184	0.272	0.254	1.98
S2	74	0.4	0.25	0.15	0.3	0.161	0.27	0.31	2.39
S3	74	0.5	0.3	0.2	0.4	0.188	0.361	0.35	2.42
S4	74	0.6	0.35	0.25	0.5	0.175	0.245	0.261	4.76
S5	76	0.3	0.25	0.2	0.5	0.147	0.369	0.448	2.78
S6	76	0.4	0.2	0.25	0.4	0.173	0.396	0.604	3.16
S7	76	0.5	0.35	0.1	0.3	0.193	0.345	0.411	1.22
S8	76	0.6	0.3	0.15	0.2	0.343	0.621	0.796	1.15
S9	78	0.3	0.3	0.25	0.3	0.368	0.764	0.626	1.23
S10	78	0.4	0.2	0.2	0.2	0.324	0.583	0.704	2.01
S11	78	0.5	0.35	0.15	0.5	0.276	0.571	0.433	1.49
S12	78	0.6	0.25	0.1	0.4	0.365	0.971	1.723	0.5
S13	80	0.3	0.35	0.15	0.4	0.348	0.601	0.616	4.89
S14	80	0.4	0.3	0.1	0.5	0.317	0.643	0.697	0.55
S15	80	0.5	0.25	0.25	0.2	0.454	0.809	0.839	1.08
S16	80	0.6	0.2	0.2	0.3	0.406	0.582	0.574	0.53

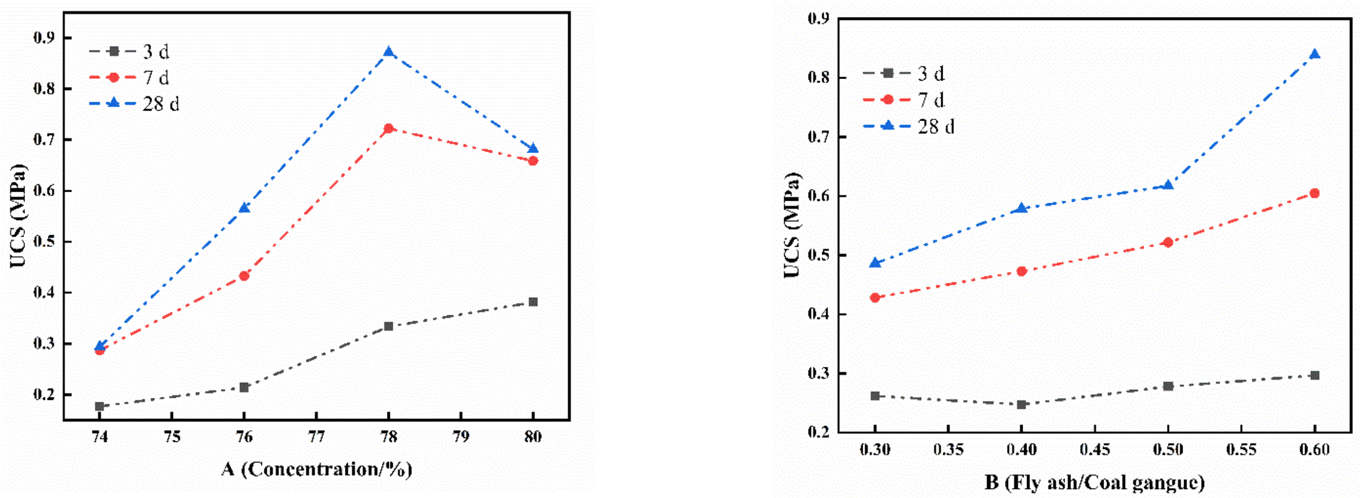


Figure 10. Influence of concentration and ash/gangue ratio on strength development.

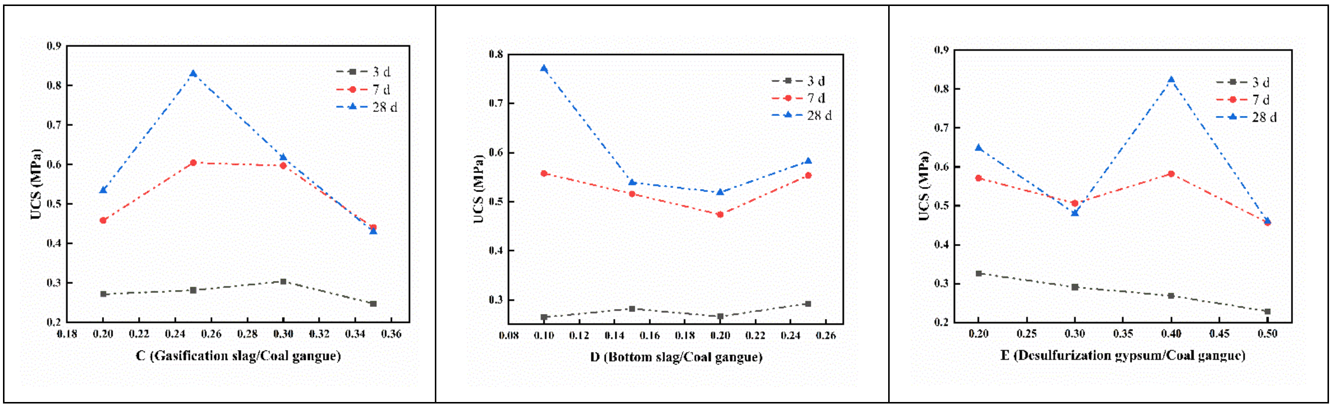


Figure 11. Influence of gasification slag, bottom slag and desulfurized gypsum on strength development.

In Figure 10, the compressive strength of CBSWCB shows a trend of first increasing and then decreasing with the increase in concentration. When the concentration is lower than 78%, the compressive strength increases rapidly and decreases when it is higher than 78%. At the same time, the compressive strength increases rapidly before 7 days, increases slowly in the later stage, and finally tends to ease. It can be observed from the 3 d strength that fly ash has little effect on the early strength. With the increase in fly ash content in different curing ages, the compressive strength gradually increases, and there is large room for the rise in late strength.

In Figure 11, with the increase in the content of gasified slag, the compressive strength of CBSWCB increases first and then decreases. When the factor C is 0.25, it reaches the maximum, then decreases rapidly, and finally tends to be stable. At the same time, with the increase in curing age, the compressive strength gradually decreases and the change rate gradually increases. It can be observed that the content of gasified slag has an important negative impact on the later strength. With the increase in the content of the furnace bottom slag, the compressive strength decreases first and then increases, and the change in 3 d strength is small. It can be observed that the content of the furnace bottom slag has little effect on the early strength. The content of desulfurized gypsum is negatively correlated with the 3 d compressive strength. With the increase in curing age, the compressive strength decreases first, then increases, and then decreases with the increase in the content of desulfurized gypsum, but on the whole, the content of desulfurized gypsum is negatively correlated with the compressive strength.

3.1.2. Analysis of Filling Body Strength Range

The range analysis of compressive strength after different curing ages (3 d, 7 d, and 28 d) at different levels of each factor of CBSWCB is carried out. As shown in Table 4, the order of influencing factors on 3 d compressive strength is concentration > desulfurization gypsum content > fly ash content > gasification slag content > furnace bottom slag content. The order of influencing factors on 7 d compressive strength is concentration > gasification slag content > fly ash content > desulfurization gypsum content > furnace bottom slag content. The order of influencing factors on 28 d compressive strength is concentration > gasification slag content > separated gypsum content > fly ash content > furnace bottom slag content.

AiBiCiDiEi is used to characterize the optimal test scheme combination, where A, B, C, D, and E represent the five factors, respectively, and i (for example, 1, 2, 3, 4) represents the different level gradients of the corresponding factors. The optimal test scheme combination of 3 d compressive strength in this test is $A_4B_4C_3D_4E_1$. The best test scheme combination of 7 d compressive strength is $A_3B_4C_2D_1E_3$. The optimal test scheme combination of 28 d compressive strength is $A_3B_4C_2D_1E_3$. According to the analysis in Table 4, on the whole, the concentration and the content of desulfurized gypsum have a great impact on the early compressive strength, while fly ash, gasification slag, and furnace bottom slag have little impact on the early compressive strength, and the influence degree of the three is close. High concentration and high content of fly ash and furnace bottom slag, low content of desulfurization gypsum, and gasification slag can significantly improve the early strength. High concentration and high content of fly ash, low content of gasification slag, furnace bottom slag, and desulfurization gypsum is conducive to the increase in later strength. At the same time, the compressive strength of 3 d to 7 d increases rapidly. With the increase in curing age, the influence on the compressive strength gradually decreases and finally tends to be stable. Comprehensive consideration of the optimal test scheme combination of this test is $A_3B_4C_2D_1E_3$, that is, the concentration is 78%, the ash: gangue ratio is 0.6, the gasification slag: coal gangue ratio is 0.25, the furnace bottom slag: coal gangue ratio is 0.1, and the desulfurization gypsum: coal gangue ratio is 0.4.

Table 4. Range analysis of compressive strength of coal based solid waste backfill at different curing ages.

Factor	Level	3 d UCS (MPa)	7 d UCS (MPa)	28 d UCS (MPa)
A	74	0.177	0.287	0.29375
	76	0.214	0.43275	0.56475
	78	0.33325	0.72225	0.8715
	80	0.38125	0.65875	0.6815
B	0.3	0.26175	0.427958333	0.486
	0.4	0.24735	0.473	0.57875
	0.5	0.27775	0.5215	0.61725
	0.6	0.29655	0.60475	0.8385
C	0.2	0.27175	0.45825	0.534
	0.25	0.28175	0.60475	0.83
	0.3	0.304	0.59725	0.61725
	0.35	0.248	0.4405	0.43025
D	0.1	0.26475	0.55775	0.77125
	0.15	0.282	0.51575	0.53875
	0.2	0.26625	0.47375	0.519
	0.25	0.2925	0.5535	0.5825
E	0.2	0.32625	0.57125	0.64825
	0.3	0.29085	0.50645	0.48025
	0.4	0.2685	0.58225	0.82325
	0.5	0.22875	0.457	0.45975
Significance		A > E > B > C > D	A > C > B > E > D	A > C > E > B > D
Optimal level		$A_4B_4C_3D_4E_1$	$A_3B_4C_2D_1E_3$	$A_3B_4C_2D_1E_3$

3.2. Regression Analysis of Filling Body

Intuitive analysis can not analyze the influence the degree of various factors on the test results in the test process. To make up for this defect, the regression analysis method is used to perform regression analyses of the bleeding rate and uniaxial compressive strength of 3 d, 7 d, and 28 d.

3.2.1. Regression Analysis of the Bleeding Rate

To facilitate the regression analysis and simplify the regression model, multiple linear regression analysis was carried out on the test data of the bleeding rate measured in Table 3 through IBM SPSS statistics software. It was found that the determination coefficient R^2 of the regression model was 0.49 and the significance coefficient was 0.177, indicating that there was collinearity independence between the independent variables and the fitting of the regression model was poor. By using MATLAB 2020 to perform multiple generalized linear analyses, we can obtain the following CBSWCB generalized linear model as shown in Formula (1):

$$\begin{gathered} Y_B = 207.3x_1 + 118x_2 + 62.2x_3 - 330.4x_4 + 351.7x_5 - 1.3{x_1}^2 - 264.5{x_2}^2 - 393.5{x_3}^2 \\ -389.3{x_4}^2 - 429.3{x_5}^2 + 10.4x_1x_2x_3x_4x_5 + 41.1x_1x_2x_3x_4 - 9.1x_1x_2x_3x_5 - 3.6x_1x_2x_4x_5 - 806 \\ R^2 = 0.97 \end{gathered} \tag{1}$$

where Y_B is the bleeding rate, x_1 is A (concentration), x_2 is B (ash gangue ratio), x_3 is C (gasification slag/coal gangue), x_4 is D (furnace bottom slag/coal gangue), x_5 is e (desulfurized gypsum/coal gangue). The bleeding rate data in Table 3 are interpolated with scattered nodes through MATLAB software, and the five factors are taken as independent variables and the bleeding rate as dependent variables. As shown in Figure 12, a visual model is constructed to intuitively analyze the effect of various factors on the bleeding rate.

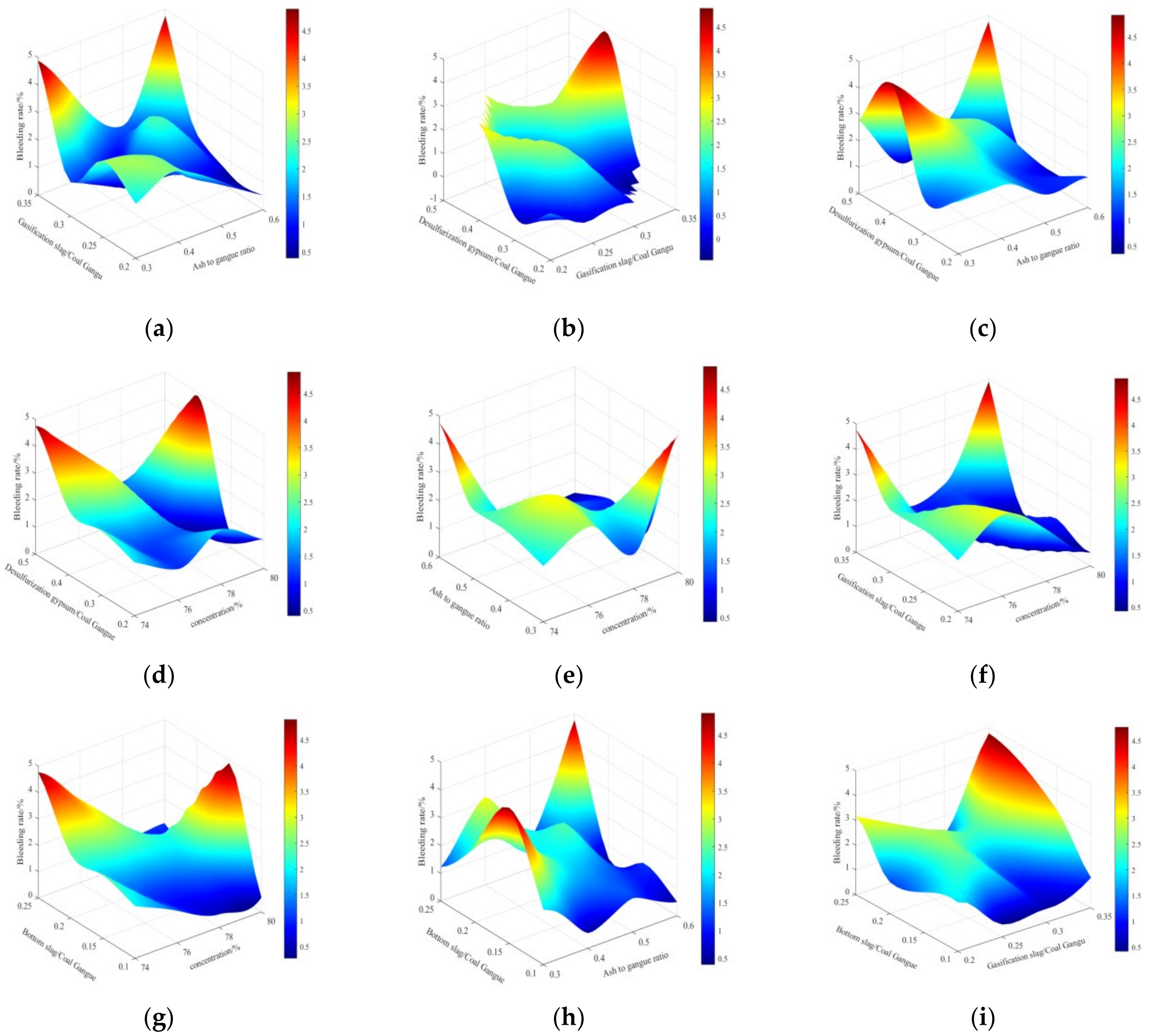


Figure 12. Effect of coal-based solid waste filling material on bleeding rate. (**a**) Factors of B and C; (**b**) factors of C and E; (**c**) factors of B and E; (**d**) factors of A and E; (**e**) factors of A and B; (**f**) factors of A and C; (**g**) factors of A and D; (**h**) factors of B and D; (**i**) factors of C and D.

Factor C in Figure 12a significantly affects the bleeding rate. When B = 0.3 and C = 0.35, the bleeding rate reaches the maximum, indicating that appropriately increasing the content of gasification slag can promote the increase in bleeding rate. In Figure 12b, with the increase in factors C and E, the bleeding rate shows an increasing trend as a whole, reaching the maximum when E is 0.4–0.45 and C = 0.35, both of which positively affected the bleeding rate. In Figure 12c, when the ash gangue ratio is less than 0.5, with the decrease in factor B and the increase in E, the bleeding rate gradually increases, and there is a certain negative correlation between them. In Figure 12d, when the concentration is constant, the bleeding rate shows an upward trend with the increase in factor E, indicating that the desulfurization gypsum significantly affects the bleeding rate. In Figure 12e, with the increase in factors A and B, the bleeding rate gradually decreases, indicating that A and B simultaneously negatively affect the bleeding rate. In Figure 12f, when the concentration is less than 76%, the negative changes in factors A and C will promote the filling body bleeding. When the concentration is greater than 76%, the two changes in the same direction will increase the bleeding body rate. In Figure 12g, it can be observed that factors A and D have a significant negative correlation and affect the bleeding rate, and their negative changes can promote an increase in the bleeding rate. In Figure 12h, when factors B and D change in the same direction, the bleeding rate increases significantly, reaching the maximum when B = 0.6 and D = 0.25. In Figure 12i, with the increase in factors C and D, the bleeding rate gradually increases, reaching the maximum when C = 0.35 and D are 0.2–0.25, indicating that factors C and D positively affect the bleeding rate.

Combined with the comprehensive comparative analysis of the established regression model, it is found that the amount of furnace bottom slag has a greater impact on the bleeding rate. Appropriately reducing the amount of furnace bottom slag can promote an increase in bleeding rate, which is closely related to the loose and porous structure of furnace bottom slag, more carbon particles, and high loss of ignition, resulting in strong water absorption. The content of desulfurized gypsum has little impact on the bleeding rate, and the concentration and content of fly ash negatively affect the bleeding rate, while gasification slag, furnace bottom slag, and desulfurized gypsum positively affect the bleeding rate, but the effects of the three are not very different.

3.2.2. Regression Analysis of Compressive Strength

The uniaxial compressive strength obtained from the L16(4^5) orthogonal test is analyzed by multiple regression. Through linear regression, it is found that the determination coefficient R^2 of the 3d compressive strength linear regression model is 0.9007, 7d is 0.6356 and 28 d is 0.4448. With the increase in curing age, the regression model coefficient gradually decreases and the significance of the model decreases. Therefore, MATLAB is used for generalized linear fitting. Due to many design factors, the generalized linear model is simplified, the significance level is set as 0.05, and the multiple linear regression model of CBSWCB uniaxial compressive strength is proposed, as shown in Formula (2). Formulas (3)–(5) are multiple generalized linear models of 3 d, 7 d, and 28 d UCS, respectively.

$$\begin{aligned} Y_i = {} & b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_5x_5 + b_6{x_1}^2 + b_7{x_2}^2 + b_8{x_3}^2 + b_9{x_4}^2 + b_{10}{x_5}^2 \\ & + b_{11}x_1x_2x_3x_4x_5 + b_{12}x_1x_2x_3x_4 + b_{13}x_1x_2x_3x_5 + b_{14}x_1x_2x_4x_5 + b_{15} \end{aligned} \quad (2)$$

In the above equation, Y_i is the uniaxial compressive strength, where i = 3, 7, 28, respectively, representing the uniaxial compressive strength of coal-based solid waste filling at 3 d, 7 d, and 28 d. x_1 is A (concentration), x_2 is B (ash gangue ratio), x_3 is C (gasification slag/coal gangue), x_4 is D (furnace bottom slag/coal gangue), x_5 is e (desulfurized gypsum/coal gangue). b_k is the multiple regression coefficients (k = 1, 2, $\cdots$, 15).

$$\begin{aligned} Y_3 = {} & 2.65x_1 - 1.84x_2 + 11.36x_3 - 2.95x_4 + 2.2x_5 - 0.02{x_1}^2 - 0.49{x_2}^2 - 24.19{x_3}^2 - 7.64{x_4}^2 \\ & -4.92{x_5}^2 - 104.58 \\ & R^2 = 0.99 \end{aligned} \quad (3)$$

$$\begin{aligned} Y_7 = {} & 10.87x_1 - 3.68x_2 + 12.3x_3 - 14.66x_4 + 18.45x_5 - 0.07{x_1}^2 - 3.36{x_2}^2 - 43.82{x_3}^2 \\ & -9.98{x_4}^2 - 24.45{x_5}^2 - 0.8x_1x_2x_3x_4x_5 + 2.27x_1x_2x_3x_4 + 0.014x_1x_2x_3x_5 - 0.07x_1x_2x_4x_5 \\ & -422.85 \\ & R^2 = 0.97 \end{aligned} \quad (4)$$

$$\begin{aligned} Y_{28} = {} & 33.2x_1 + 10.8x_2 - 6.3x_3 - 56.461x_4 + 68.1x_5 - 0.2{x_1}^2 - 27.5{x_2}^2 - 53.4{x_3}^2 \\ & -12{x_4}^2 + 2.8x_1x_2x_3x_4x_5 + 6.8x_1x_2x_3x_4 - x_1x_2x_3x_5 - 1.3x_1x_2x_4x_5 - 1290.5 \\ & R^2 = 0.94 \end{aligned} \quad (5)$$

3.3. Engineering Performance Test of Filling Body

The engineering performance of CBSWCB includes transportation performance and mechanical performance. Transportation performance is the ability to transport coal-based solid waste materials to the goaf of the working face, to avoid problems such as slurry segregation and blocking the pipeline. Mechanical performance is the ability to control the deformation of overlying strata after filling, that is, compressive strength and deformation resistance [33].

Slump can well reflect the cohesion and friction resistance of cementitious materials. Bleeding rate and water retention can reflect the stability of CBSWCB and the segregation capacity of slurry. A large bleeding rate and poor water retention will loosen the surface of the filling body and reduce its durability. Cohesion can reflect the uniformity of the filling body and the stratification and segregation of pumped slurry. The initial and final setting time is an important engineering performance index. If the initial setting time is too short, it will cause problems, such as the blockage of slurry transmission pipeline, the tension of filling operation, and difficulty of filling body self-reliance. If the final setting time is too long, it will seriously affect the normal production and operation of the coal mine.

In this test, indicators such as slump, bleeding rate, initial setting time, final setting time, cohesion, water retention, and early strength are selected to evaluate the engineering performance of new coal-based solid waste filling materials. Combined with the field experience of filling mining at home and abroad, the slump is between 18 and 23 cm, the bleeding rate between 1.5% and 5%, which are considered to have “good” and “excellent” transportability, respectively. The concentration of paste filling slurry is 76–80%, which can ensure that no bleeding in the stope occurs and effectively avoids the problem of cementation of ore and rock [3]. Early strength refers to the strength required by the filling material to maintain its structural stability. The high productivity of general coal mines requires that the early strength is the comprehensive strength within 8 h after filling, which is mainly estimated by empirical Formula (6) [34].

$$h^2 = a\sigma^3 \tag{6}$$

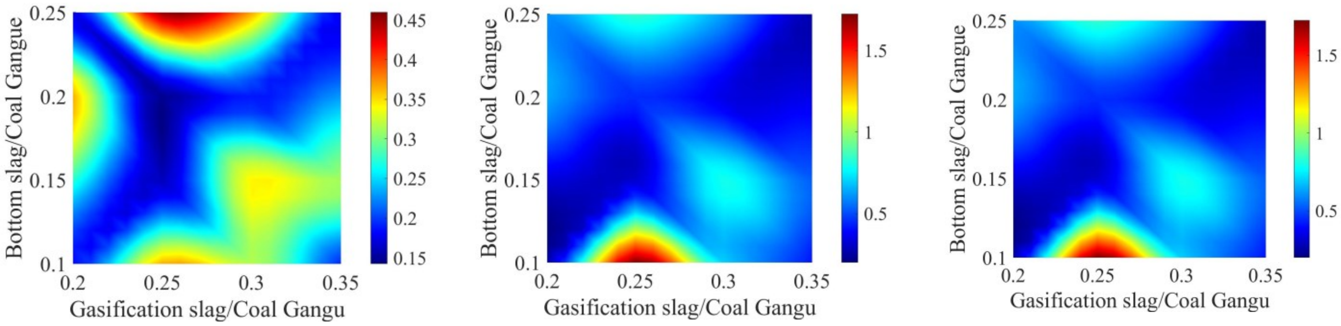
where “h” is the mining height, “a” is the empirical coefficient, the filling body of the coal mine is generally taken as 600, and “σ” is the early strength. The mining height of the filling face in Renjiazhuang Coal Mine is 3.8 m. To ensure the filling effect and normal operation of the working face, the early strength of CBSWCB needs to be higher than 0.29 Mpa.

4. Discussion

4.1. Analysis of Factors Influencing the Development of Backfill Strength

During different curing ages, the compressive strength of CBSWCB first increased and then decreased with the increase in concentration, and gradually increased with the increase in fly ash content (Figure 10), which may be the spherical surface of fly ash. Smaller spherical particles adhere unevenly, and there are pores between the small spherical particles and the matrix. The compressive strength gradually increases with the adsorption and compaction of the pores in the later stage, which reduces the ability of the slurry to produce plastic deformation, resulting in glue. The cementing effect is better in the coagulating material, and the compressive strength is increased. The compressive strength of CBSWCB first increases and then decreases with the increase in gasification slag content. With the increase in furnace bottom slag content, it first decreases and then increases. As the content of desulfurized gypsum increases, it first decreases. The ups and downs of rising and falling are shown in Figure 11.

After the CBSWCB strength sensitivity analysis in Section 3.1.2, it was found that gasification slag and furnace bottom slag have different effects on the early strength and late strength of CBSWCB, and the current research on gasification slag and bottom slag as paste filling materials is not enough. In order to further study the influence of the interaction of different material factors on CBSWCB, the control variable method was used to analyze the influence mechanism of the interaction between gasification slag and bottom slag on the strength of CBSWCB. The factors remain unchanged, and the griddata function in MATLAB software was used to plot to explore the effect of gasification slag and furnace bottom slag content at different curing ages on the compressive strength of coal-based solid waste backfill, as shown in Figure 13. The high-strength areas of 3 d and 7 d are concentrated in factor C of 0.22–0.27, D of 0.1–0.13, and 0.22–0.25 in the area, with the increase in the curing age, the high-intensity area shifts to the area with a factor C of 0.22–0.27 and D of 0.1–0.12, and finally reaches the maximum when C is 0.25 and D is 0.1. It can be observed that the influence of gasification slag content on the strength of CBSWCB is lower than that of the furnace bottom slag. Low content of gasification slag and high content of furnace bottom slag is beneficial to increase the compressive strength. This may be due to the high residual carbon content of the gasification slag. The high content of the gasification slag will hinder the gelation reaction between it and cement or lime. At the same time, under the co-stimulation of the hydration reaction of other materials, for the furnace bottom slag, the Al_2O_3, Fe_2O_3, and active SiO_2 react with calcium silicate and calcium aluminate gel, resulting in the formation of a dense structure with a certain strength of CBSWCB. As the curing age increases, the pores left by the surface of the furnace bottom slag burnt are packed tightly, and the influence of the content of gasification slag and furnace bottom slag on the compressive strength gradually decreases.



(**a**) (**b**) (**c**)

Figure 13. Influence of C and D factors on compressive strength of different curing ages. (**a**) 3 d UCS; (**b**) 7 d UCS; (**c**) 28 d UCS.

4.2. Effects of Different Factors on Bleeding Rate

Combined with the established regression model (Formula (1)), a comprehensive comparative analysis of the influence of different factors on bleeding rate (Figure 12) found that the amount of furnace bottom slag has a greater impact on the bleeding rate. Appropriately reducing the amount of furnace bottom slag can promote bleeding. The increase in water rate is related to the loose and porous structure of furnace bottom slag, many carbon particles, and the high loss of ignition, which leads to strong water absorption. The amount of desulfurized gypsum has a small effect on the bleeding rate, the concentration and fly ash mixing. The amount negatively affects the bleeding rate, while the gasification slag, furnace bottom slag, and desulfurized gypsum positively affect the bleeding rate, but the effects of the three are not much different.

4.3. Engineering Performance Index Test Analysis

According to the GB/T1346-2011 national standard [35], the test piece was tested and analyzed (Figure 14). The CBSWCB slump under the optimal ratio was 16.6 cm, the bleeding rate was 1.6%, the cohesion was average, and there was no segregation. Water sedimentation problems, good water retention, no water condensate precipitation in the central stack or edge and good segregation resistance show that CBSWCB has good transportation performance. The initial setting time is 5.42 h, and the final setting time is 7 h.

The uniaxial compression test was performed on three specimens after 8 h of curing, and the average value of the three combined test data was taken. The compressive strength reached 0.24 MPa. Although it did not reach 0.29 MPa, the strength of the filling body determined by the empirical formula was relatively large. Emphasis is placed on safety considerations and there is a certain amount of overflow. It can be considered that the CBSWCB under the optimal ratio meets the early filling strength requirements of the target mine, and provides a reference for its subsequent filling and mining, which has certain guiding significance.

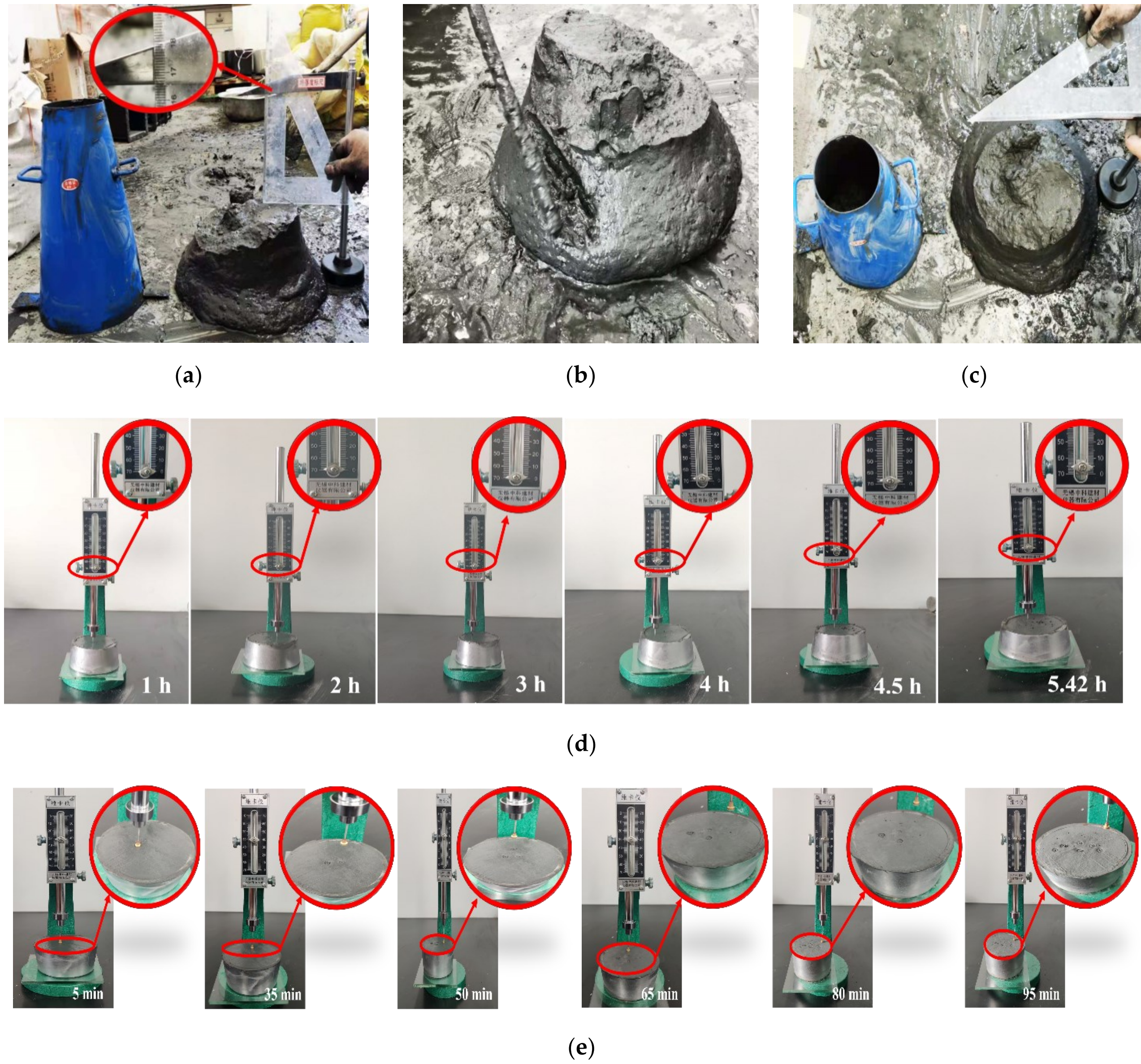


Figure 14. Engineering performance inspection of coal-based solid waste filling body. (**a**) Slump test; (**b**) cohesion test; (**c**) water retention test; (**d**) initial setting time test; (**e**) final setting time test.

5. Conclusions

In this paper, the UCS and bleeding rate of CBSWCB were tested by orthogonal experiment, and the influencing factors of mechanical properties and strength development were analyzed. The multiple generalized linear model of strength and bleeding rate was established, and the optimal filling material ratio was determined. The engineering performance index of CBSWCB with the optimal ratio was tested, and the following conclusions are drawn.

(1) Based on the development characteristics of the industrialized structure of the Coal-Thermal Power-Chemical Industry Base in East Ningxia, a multi-source coal-based solid waste filling material was preliminarily prepared, the strength development law of CBSWCB was analyzed, and the optimal ratio scheme of this test was determined, that is, the concentration was 78%, the ash: gangue ratio was 0.6, the gasification slag: coal gangue was 0.25, the furnace bottom slag: coal gangue was 0.1, and the desulfurization gypsum: coal gangue was 0.4.

(2) Concentration and desulfurized gypsum content had a great influence on the early compressive strength of CBSWCB, while fly ash, gasification slag, and furnace bottom slag had little influence on the early compressive strength, and the influence degree of the three was close. High concentration and high content of fly ash and furnace bottom slag, low content of desulfurization gypsum, and gasification slag can significantly improve the early strength. High concentration and high content of fly ash, low content of gasification slag, furnace bottom slag, and desulfurization gypsum were conducive to the increase in the later strength. At the same time, the compressive strength increases rapidly in the early stage. With the increase in curing age, the impact on the compressive strength gradually decreases and finally tends to be stable.

(3) The content of furnace bottom slag had a greater impact on bleeding rate, while the content of desulfurized gypsum had a small effect on bleeding rate. Concentration and fly ash content were negatively correlated to the bleeding rate, while gasification slag, furnace bottom slag, and desulfurized gypsum had a positive impact on the bleeding rate, and the impact of the three was close.

(4) The engineering performance test of CBSWCB under the optimal proportioning scheme shows that the bleeding rate of the filling body was 1.6%, the slump was 16.6 cm, the cohesion was general, the segregation resistance was good, and the initial setting time was 5.42 h. The final setting time was 7 h, and the early strength after curing for 8 h reached 0.24 MPa, which meets the requirements of the target mine paste filling. The consumption of coal-based solid waste filling materials exceeds 95%, which ensures the resource utilization of coal-based solid waste, reduces the cost of mine filling, and has significant economic and social benefits.

Author Contributions: J.Z. and K.Y. conceived and designed the theoretical framework; J.Z. and X.H. performed the experiments and wrote the manuscript; Z.W., X.Z. and J.F. corrected the tables and figures. All authors participated in the finalization of the written manuscript. J.Z. acted as the supervisor of the project and acquired all necessary funding. All authors have read and agreed to the published version of the manuscript.

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