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Study on the proportion of coal gasification slag paste filling material

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Abstract: Aiming at the utilization of coal gasification slag, it is proposed to prepare coal gasification slag as mine filling material. The optimum ratio of cement: coal gasification slag: gangue is 1:5:1, the mass fraction is 70%, and the dosage of quicklime is 4%. The strength of backfill reaches 4.23mpa, which can meet the requirements of mine backfill strength.

1. Introduction

Coal gasification technology has brought new impetus to China's industrial development, but the emissions of coal gasification slag have increased significantly, and are increasing at the rate of 4.8 ~ 5.2 million tons per year, and are expected to reach 10.3 million tons in 2023. If not handled properly, great environmental harm will be caused [1,2]. At present, the research on coal gasification slag has the disadvantages of low utilization rate and poor large-scale utilization [3]. The application of coal gasification slag in concrete field provides us with experience and ideas. According to Liu Kaiping [4] et al., the compressive strength of concrete mixed with grinding coal gasification coarse slag is much higher than that of benchmark concrete, and the compressive strength of concrete mixed with grinding coal gasification fine slag is lower than that of benchmark concrete. Coal gasification fine slag with relatively high carbon content is not good for the compressive strength of concrete, so it is not recommended to be used in concrete. Yoshitaka[5] conducted concrete test and aggregate test on coal gasification slag and concluded that coal gasification slag may be used in concrete. Gao Peng, Li Qinghong et al. [7] studied the long-term mechanical properties, frost resistance and construction performance of coal gasification slag pavement base material, and demonstrated the technical feasibility of coal gasification slag pavement base material in highway engineering. Lei Tong [8] found that coal gasification coarse slag can be applied. The amount of coal gasification coarse slag was determined by experiment. The author made paste filling material with coal gasification slag from a shaanxi mine as the main raw material, and met the requirements of the minimum compressive strength of filling body of the coal mine for 28d 3.8mpa.

2. Study on physicochemical properties of coal gasification slag

Table 1 Chemical composition test results of coal gasification slag

composition	SiO_2	Al_2O_3	CaO	Fe_2O_3	SO_3	Na_2O	K_2O	MgO	TiO_2	P_2O_5	MnO
content%	41.79	21.96	12.69	11.99	4.92	2.13	1.80	1.18	0.04	0.22	0.21

The chemical composition of coal gasification slag (Hereinafter referred to as CGS) is shown in Table 1. The chemical composition of coal gasification slag is mainly composed of SiO_2, Al_2O_3, CaO, Fe_2O_3 and other four oxides, the sum of which is as high as 70%, similar to low calcium fly ash standard, it has a certain pozzolanic activity. The content of four oxides in coal gasification residue is more than 85%, the content of acid oxides (SiO_2 + Al_2O_3) in coal gasification residue is more than 60%, while the content of basic MgO and TiO_2 oxides is less than 30%.The ash content of CaO, MgO and Fe_2O_3 in coal is higher than that in common coal. The above characteristics are the important material basis of CGS resource utilization technology.

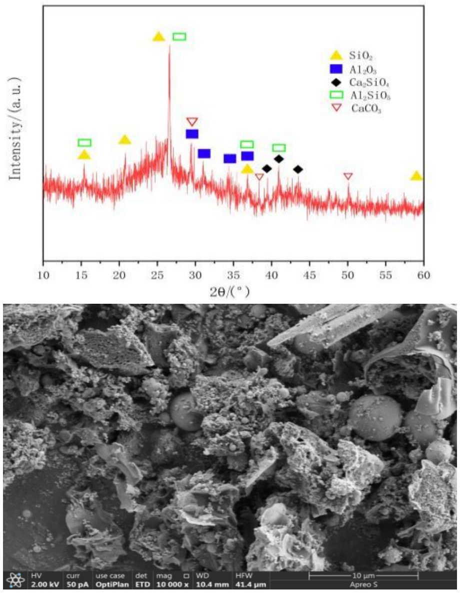

Fig.1 XRD and SEM patterns of coal gasification slag

Figure 1 shows the XRD pattern of CGS. In the XRD pattern of CGS, "steamed bread peak" appears in the region of 12° ~ 50°, and there are few sharp and intense diffraction peaks, with a small amount of magnesium aluminite and part of calcium aluminite.The mineral composition mainly consists of quartz (SiO_2), andalusite (Al_2SiO_5), Ca_2SiO_4, active Al_2O_3, and a small amount of $CaCO_3$.SEM shows that CGS is mainly composed of some irregular particles with different sizes, loose intergranular, porous surface and complex morphology. The spheroidal melt on the surface is rich in content and concentrated in distribution, and the shape of unburned carbon particles is different. The surface pores of CGS are well developed, and it can be clearly seen that mineral ash and unburned carbon are closely bonded as polyhedron.

Relevant literature [8] pointed out that when CGS is used in concrete field, the carbon content of CGS decreases with the increase of particle size, and the burning loss of CGS can be controlled below 6% by screening and sorting CGS by 1.16mm. It will not have a negative effect on cement hydration. The CGS with particle size ≥1.18mm was poured into the ball mill and ground at a speed of 180 RPM /min to take it out for later use. The CGS was ground in the mill for 30min to reach the limit particle size, and the specific surface area measured after ball milling was 351m^2/kg.

3. The experiment part

3.1 Orthogonal test

Table 2 Orthogonal test factor level table

level	factorA Mass ratio of cement to gasification slag	factor B Mass ratio of cement and gangue	factorC Mass concentration(wt.%)	factorD Quick lime(wt.%)	Water reducing agent
1	1:4	1:1	69%	2%	1%
2	1:5	1:2	70%	3%	1%
3	1:6	1:3	71%	4%	1%

Four factors were used in this orthogonal test: mass ratio of cement to CGS A, mass ratio of cement to gangue B, mass concentration of slurry C, and amount of quicklime D. The mass ratio of cement to CGS was 1:4~1:6, the mass ratio of cement to gangue was 1:1~1:3 and the mass concentration was 69wt. %~ 71wt. %, and the addition of quicklime was 2%~4%.Three levels were taken for each factors. This time, L_9 (3^4) was selected in the orthogonal test of 4 factors and 3 levels, and a total of 9 tests were required. The main indexes are slump, bleeding rate, setting time and uniaxial compressive strength at different ages, as shown in Table 2.

3.2 The experimental process

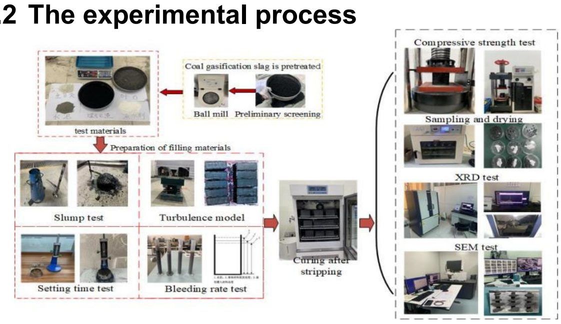

Fig. 2 Schematic diagram of test flow

3.3 The test results

The orthogonal test results are shown in Table 3.

Table 3 Orthogonal test results table

Test no.	Exudation rate(%)	Setting time(min) Initial	Setting time(min) Final	slump(mm)	Uniaxial compressive strength at different ages(MPa) 8h	3d	7d	28d
1#	3.1	5.53	8.55	197.17	0.24	0.68	1.72	3.48
2#	2.71	5.2	8.04	192.34	0.23	0.71	1.85	3.66
3#	2.31	4.88	7.73	181.05	0.23	0.66	1.92	3.77
4#	2.44	4.81	7.41	186.33	0.22	0.59	2.12	4.23
5#	2.23	4.48	7.17	176.18	0.23	0.69	1.89	4.08
6#	2.77	5.12	7.91	188.15	0.20	0.48	1.81	3.80
7#	2.08	4.08	6.45	169.37	0.19	0.59	2.37	4.28
8#	2.65	4.72	7.61	177.27	0.14	0.36	2.25	4.45
9#	2.26	4.37	6.89	180.11	0.18	0.54	1.92	4.01

3.4 Range analysis

Range analysis can be used to determine the influence weight of various factors on various performance indicators of backfill, and the trend diagram of the relationship between various factors and performance is more conducive to intuitive analysis of the trend of change, with four factors A, B, C and D as abscissa, and the average value of each performance indicator of K_1, K_2 and K3 as ordinate. By drawing the trend chart of factors and indicators, we can see the change trend of each indicator with factors more intuitively.

(1)Extreme difference analysis of conveying performance

Table 4 Transmission performance range analysis table

K_i and R value	slump(mm)				Exudation rate(%)			
	A	B	C	D	A	B	C	D
k_1	190.19	184.29	186.87	184.49	2.71	2.54	2.84	2.53
k_2	182.89	181.93	186.06	182.62	2.48	2.53	2.47	2.52
k_3	175.58	181.44	175.53	181.55	2.33	2.45	2.21	2.47
Poor R	14.60	2.85	11.33	2.94	0.38	0.09	0.63	0.06
Factors affecting	A-C-D-B				C-A-B-D			

To slump poor R calculation results show that the primary and secondary order of factors affecting slump A > C > D > B (mass ratio of cement to CGS > mass concentration of slurry > content of quicklime > mass ratio of cement to gangue), The main influencing factor is the mass concentration of slurry, the second is the quality of slag cement and coal gasification ratio, and then the cement 26 ratio and quick lime content and waste rock mass. The calculation results of range R of bleeding rate showed that the primary and secondary factors affecting bleeding rate were C>A>B>D (mass concentration of slurry > mass ratio of cement to CGS > mass ratio of cement to gangue > dosage of quick lime), and the main influencing factor was mass concentration of slurry, which had an influence of 0.63% on bleeding rate.The second is the mass ratio of cement to CGS, then the content of quicklime and the mass ratio of cement to gangue.

(2)Analysis of condensation time range

Table 5 Paste setting time range analysis table

K_i and R value	Initial setting time(min)				Final setting time(min)			
	A	B	C	D	A	B	C	D
k_1	5.20	4.81	5.12	4.79	8.11	7.47	8.02	7.53
k_2	4.80	4.8	4.79	4.80	7.50	7.61	7.45	7.47
k_3	4.39	4.79	4.48	4.80	6.98	7.51	7.12	7.57
Poor R	0.81	0.02	0.64	0.01	1.13	0.14	0.91	0.10
Factors affecting	A-C-B-D				A-C-B-D			

The calculation results of range R of initial setting time show that the factors affecting initial setting time are in the order of A>C>B>D (mass ratio of cement to CGS > mass concentration of slurry > mass ratio of cement to gangue > content of quicklime). The proportion of cementing material is the main influencing factor, followed by concentration. Among them, the increase of CGS as auxiliary cementing material shorteneth the initial setting time of paste material by 0.81h, and the influence of slurry mass concentration from low to high on initial setting time can be shortened by 0.64h, the effects of mass ratio of cement to gangue and dosage of quicklime were 0.1h and 0.2h, respectively, it has little effect on initial setting time. Range R of calculation for the final setting time, according to the results of the primary and secondary order of factors influencing the final setting time of A > B > C > D(Mass ratio of cement to CGS > mass concentration of slurry > mass ratio of cement to gangue > content of quicklime), factors affecting rules similar to the effects of initial setting time, gelled material proportion and concentration is the main factor affecting the final setting time.

Comprehensive the above analysis, the quality of slurry concentration and the quality of the CGS and cement ratio is the main factor affecting the transmission performance, the nine groups of experiments, 5 #, 7 #, 8 # does not meet the material requirements, the characteristic of slump of paste material of the slump is less than 180 mm, 1 # does not satisfy the paste material to let stand exudation rate lower than 3%.Therefore, 1#, 5#, 7# and 8# do not meet the requirements of paste material for conveying performance. The more CGS and cement in the paste filling material, the shorter the setting time. According to the characteristic requirements of the filling material, the 9 groups of tests all met the setting time requirements.

(3) Strength range analysis

Table 6 Analysis of the uniaxial compressive strength range of different ages of paste

K_i and R value	The intensity of 8h(MPa)				The intensity of 3d(MPa)			
	A	B	C	D	A	B	C	D
k_1	0.23	0.21	0.19	0.22	0.68	0.62	0.51	0.64
k_2	0.22	0.20	0.21	0.21	0.59	0.59	0.61	0.59
k_3	0.17	0.20	0.22	0.2	0.49	0.56	0.65	0.54
Poor R	0.06	0.01	0.03	0.02	0.19	0.06	0.14	0.10
Factors affecting	A-C-D-B				A-D-B-C			

The calculation results of range R of 8h uniaxial compressive strength show that the primary and secondary factors affecting 8h uniaxial compressive strength of paste filling materials are A > C > B > D (mass ratio of CGS to cement > mass concentration of slurry > quantity of quicklime > mass ratio of cement to gangue).The main influencing factor is the mass ratio of CGS to cement, that is, the higher the proportion of CGS, the lower the 8h strength. With the increase of the ratio of CGS to cement from 1:4 to 1:6, the influence reaches 0.06mpa.The influence of slurry mass concentration on strength at 8h also reached 0.03mpa, while the negative influence of quick lime on strength at 1d reached 0.02mpa, which is because the excessive addition of quick lime will affect the hydration reaction of cement in the early stage to a certain extent. The calculation results of 3d uniaxial compressive strength range R show that the primary and secondary factors affecting the uniaxial compressive strength of paste filling materials are A > C > B > D (mass ratio of CGS to cement > mass concentration of slurry > quantity of quicklime > mass ratio of cement to gangue).As an important stage to judge early strength, the main influencing factors of early strength were similar to those of 8h childbearing age. CGS and quicklime still have a negative effect on the strength of filling materials.

Table 7 Analysis of the uniaxial compressive strength range of different ages of paste

K_i and R value	The intensity of 7d(MPa)				The intensity of 28d(MPa)			
	A	B	C	D	A	B	C	D
k_1	1.83	2.07	1.93	1.84	3.64	4.00	3.91	3.86
k_2	1.94	2.00	1.96	2.01	4.04	3.99	3.97	3.91
k_3	2.18	1.88	2.06	2.09	4.25	3.86	4.04	4.15
Poor R	0.35	0.19	0.13	0.25	0.61	0.14	0.13	0.29
Factors affecting	A-D-B-C				A-D-B-C			

The calculation results of the 7d uniaxial compressive strength range R show that the primary and secondary order of influencing paste filling strength is A > D > B > C (mass ratio of gasification slag to cement > amount of quicklime > mass ratio of cement to gangue > mass concentration of slurry).At 7d, CGS and quicklime began to show a positive correlation on strength, and the influence of high level to low level reached 0.35mpa and 0.25mpa, respectively. The calculation results of the range R of uniaxial compressive strength at 28d showed that the primary and secondary order of influencing the paste strength at the later stage was A > D > B > C (mass ratio of gasification slag to cement > quantity of quicklime > mass ratio of cement to gangue > mass concentration of slurry).The main influencing factors of 28d strength are the mass ratio of CGS cement and the dosage of quicklime, and the influence level of the two factors on the strength continues to increase.

The main factors affecting the strength are the mass ratio of CGS to cement and the addition of quicklime, which have a significant impact on the later strength. Although it has a negative impact on the early strength, it still meets the early strength requirements of filling materials. The 8h strength of 9 groups of tests 7#, 8# and 9# is less than 0.2mpa.The strength of 1#, 2# and 3# tests was lower than 3.8mpa on 28d, which did not meet the requirements of strength.

3.5 Result analysis and discussion

Based on the above analysis, when the mass ratio of cement to gasification slag is 1:5, the mass ratio of cement to gangue is 1:1, the mass concentration of slurry is 70%, and the dosage of quicklime is 4%, the experimental group is 4#, which achieves good results in terms of conveying performance and strength and is recommended for this test.

4. Conclusion

coal gasification slag, coal gangue, cement and admixture are used as the main raw materials to prepare paste filling materials. Solid waste is used as the raw material of mine filling materials, which can save production cost.

The minimum strength of backfill in goaf of Shaanxi coal mine is 3.8mpa and the mass concentration of backfill slurry is 70%. Under the condition of satisfying the slurry mass concentration, the filling body was prepared when the mass ratio of coal gasification slag, coal gangue and cement was 5:1:1, the content of quicklime was 4% and the water reducing agent was 1%. The strength of the filling body reached 4.23MPa after 28 days. Meet the requirements.

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