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Research Article

Effect of Different Activators on Rheological and Strength Properties of Fly Ash-Based Filling Cementitious Materials

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In this paper, lime, gypsum, NaOH, and Na_2SO_4 are mainly used to study the activation degree and activation mechanism of fly ash, and L9 (3^4) orthogonal table is used for the orthogonal test. The influence of different activators on the rheological and strength properties of slurry was analyzed. The microstructure and hydration products of fly ash cement cementitious body were studied by SEM and XRD. The results show that the bleeding rate of slurry containing activator fly ash system is between 2.24% and 3.37%, which is much higher than that of pure cement slurry (9.25%). Therefore, the addition of this system can improve the fluidity of slurry, and the optimal scheme is $a_3b_2c_2d_2$. The results show that the compressive strength of cement with activator fly ash system is much lower than that of pure cement, but the increase of strength is between 31% and 85%, which is much greater than that of pure cement (35%–46%). The optimal scheme is $A_2B_2C_3D_3$ or $A_3B_2C_3D_3$ at 3 days and $A_1B_3C_2D_3$ at 28 days. According to the scanning results of SEM and XRD, the addition of activator can significantly improve the hydration degree of fly ash and form a more complex network structure without obvious gap.

1. Introduction

tWith the development of mining technology, filling method is considered a future development direction for an increasing number of large, medium, and small mines, and the reduction of filling cost has always been the research emphasis of scholars all over the world [1, 2]. Fly ash mainly refers to fine particles generated from coal combustion, the particle size of which is 1–100 m and the main chemical compositions are SiO_2 and Al_2O_3 [3]. By the end of 2020, the annual production of fly ash in China has reached 580 million tons. If the fly ash is released into the air, it will seriously affect the air quality, but if it is stacked on the ground, it will not only pollute the local environment but also inevitably cause a waste of resources. Since fly ash is enriched with Si, Al, Ca, and other chemical substances [4], it can be used to substitute some types of cement in the fields of concrete [5, 6] or mine filling [7–9]. This is a generally accepted treatment method. However, as an inert material, fly ash can substitute cement as much as possible only when its internal potential activity is fully stimulated. For this purpose, it is affirmatively necessary to study the excitation principle and mode of fly ash.

In the physical excitation mode, the particle size of fly ash is changed by mechanical milling technology, so its particle fineness is improved, and specific surface area is increased, which facilitates the dissolution of elements such as SiO_2 and Al_2O_3 and the penetration of Ca^{2+}, thereby improving the activity of fly ash [10, 11]. As for the chemical excitation mode, different fly ash excitants are added to stimulate the internal potential activity of fly ash [12]. Fu et al. [13] developed a compound additive and its ratio to effectively activate fly ash by using the methods of orthogonal design and uniform design and working out a computer program. Sun et al. [14] studied the mechanism and persistence of different chemical excitants stimulating the activity of fly ash by using XRD, SEM, EDS, and other devices and determined the optimal combination of excitants by single mixing and double mixing. Hoang et al. [15] investigated the compressive strength of fly ash cement gelatin at 5°C and 20°C by using three different additives, that is, sodium thiocyanate, diethanolamine, and glycerin. The results showed that cement hydration is promoted after these three additives are added, and the content of calcium carboaluminate in hydration products is increased, and the compressive strength of gelatin is also improved to a certain extent.

Al_2O_3 and SiO_2 stored in fly ash bind with Ca^{2+} in slurry to form calcium silicate and calcium aluminate, so that the internal structure of gelatin becomes denser, and its long-term strength is improved [16]. The reaction mechanism is as follows.

CaO in cement reacts with water to generate $Ca(OH)_2$:

$$CaO + H_2O \longrightarrow Ca(OH)_2 \quad (1)$$

Al_2O_3 and SiO_2 in fly ash continue to react chemically with $Ca(OH)_2$ to generate $CaO{\cdot}SiO_2$ and $CaO{\cdot}A1_2O_3$:

$$m_1 \cdot Ca(OH)_2 + SiO_2 + nH_2O \longrightarrow m_1 \cdot CaO \cdot SiO_2 \cdot nH_2O \quad (2)$$

$$m_1 \cdot Ca(OH)_2 + Al_2O_3 + nH_2O \longrightarrow m_1 \cdot CaO \cdot Al_2O_3 \cdot nH_2O \quad (3)$$

However, Ca^{2+} in slurry is not enough for Al_2O_3 and SiO_2. Hence, excitant is added to the slurry to supplement Ca^{2+} needed by Al_2O_3 and SiO_2 and also to crack the internal structure of fly ash. Then, the excitant reacts chemically with Al_2O_3 dissolved from fly ash in the solution containing CaO to generate ettringite (Aft). The reaction mechanism is as follows:

$$AL_2O_3 + Ca(OH)_2 + 12H_2O \longrightarrow C_3AH_{12} \quad (4)$$

$$C_3AH_{12} + 3C\overline{S}H_2 + 14H_2O \longrightarrow C_3A \cdot 2C\overline{S}H_{12} + Ca(OH)_2 \quad (5)$$

Excitant is added to also increase the concentration of OH- in the slurry [17], so that the reaction speed between fly ash and $Ca(OH)_2$ is improved. In addition, it also has a certain promoting effect on the early strength of gelatin. The reaction mechanism is as follows:

$$-O-S-OH + NaOH \longrightarrow -O-Si-ONa + H_2O \quad (6)$$

$$-O-Si-O-Si + 2NaOH \longrightarrow 2(-O-Si-ONa) + H_2O \quad (7)$$

According to the above analysis, excitant can be added to densify the gelatin, generate more stable hydration products, and improve the compressive strength of gelatin.

2. Composition of Test Materials

The test materials used include fly ash produced by China Huadian Group Kunming Power Plant; 32.5# Portland slag cement produced by Beikong Cement Plant; lime powder with calcium content ≥85% and fineness ≥180 meshes produced by Kunming Maoshan Qiangda Lime Building Materials Factory; and gypsum powder produced by Yunnan Hongshi Mining Co., Ltd., as well as NaoH, Na_2SO_4, and other chemical additives.

3. Characteristics of Fly Ash

The fly ash has a density of 1.8~2.3 g/cm^3, bulk density of 0.6~0.9 g/cm^3, measured bulk density of 0.79 g/cm^3, and compacted bulk density of 1.26 g/cm^3. Its chemical composition and technical indexes are shown in Tables 1 and 2, respectively.

4. Characteristics of Cement

The cement has a density of 3.01~3.15 g/cm^3, bulk density of 1.3~1.8 g/cm^3, measured bulk density of 1.35 g/cm^3, and compacted bulk density of 1.95 g/cm^3. Its chemical composition is shown in Table 3.

4.1. Test Program. In this paper, four different excitants are used to perform compound activation test on fly ash. Because the activation effect on fly ash varies among different excitants, the test program is designed by the orthogonal test in this paper. According to a great number of preliminary studies, the four excitants used in this test are determined: lime, gypsum, NaoH, and Na_2SO_4. $L9(3^4)$ orthogonal table is used for orthogonal test. The specific test programs are shown in Tables 4 and 5, respectively.

According to the test method by Huang et al. [18], the evenly mixed slurry is poured into a standard triple test mold (7.07 cm ∗ 7.07 cm ∗ 7.07 cm) and then cured for 24 h and demoulded after tamping. The specimen is watered every 3 d and covered with water-retaining material, and cured for 3 d, 7 d, and 28 d at room temperature.

The evenly mixed slurry is poured into a measuring cylinder with a measuring range of 200 mL until reaching the 100 mL scale. The bleeding amount of the slurry is recorded at 2 h, and the bleeding rate is calculated according to formula (8) [19], where V is the total volume of slurry; ΔV is the secreted water.

The slurry prepared is poured into a slump barrel with both top and bottom openings. The slump barrel is placed vertically upward and then lifted at a constant speed. The maximum diffusion diameter of the slurry, that is, the fluidity of slurry, is measured by a straight rule.

4.2. Test Equipment. The microstructure of fly ash cement gelatin and the morphology of its hydration products are observed by Philips XL30 ESEM-TMP SEM (Figure 1(a)) and D/Max 2200 XRD (Figure 1(b)) provided by Research Center for Analysis and Measurement, Kunming University of Science and Technology.

Table 1: Chemical composition of fly ash.

Composition	Fe_2O_3	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	Loss on ignition
Content (%)	11.26	51.79	22.95	2.89	1.6	0.46	0.9	0.06	7.69

Table 2: Technical indexes of fly ash.

Item	Unit of measurement	Technical index	Test value
Fineness	%	≤8 (0.08 screen residue)	1.1
Loss on ignition	%	≤5.0	7.69
Water demand	%	≤95	90
Water content	%	≤1.0	0.03
SO3	%	≤3.0	0.06
Free CaO	%	≤4.0	Trace amount

Table 3: Chemical composition of P.S 32.5 cement.

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3	Total
Content (%)	19.97	6.34	3.74	65.74	1.11	1.1	0.12	98.12

Table 4: Orthogonal table design of compound activation test.

Level	A	B	C	D
	Lime dosage (%)	Gypsum dosage (%)	NaOH dosage (%)	Na_2SO_4 dosage (%)
1	8	4	1	2
2	12	5	2	3
3	16	6	3	4

Table 5: Orthogonal test scheme of compound activation.

Test no.	A Lime dosage (%)	B Gypsum dosage (%)	C NaOH dosage (%)	D Na_2SO_4 dosage (%)
FH-1	8	4	1	2
FH-2	8	5	2	3
FH-3	8	6	3	4
FH-4	12	4	2	4
FH-5	12	5	3	2
FH-6	12	6	1	3
FH-7	16	4	3	3
FH-8	16	5	1	4
FH-9	16	6	2	2

Notes: the ratio of cement to fly ash is 1 : 1; the water-cement ratio is 0.5.

5. Analysis of Test Results

5.1. Fluidity Analysis of Slurry. According to Table 6, slurry containing the system of excitant and fly ash has a bleeding rate of 2.24%~3.73% and a fluidity of 110 mm~155 mm. The bleeding rate and fluidity of unmixed cement mortar are 9.25% and 150 mm, respectively. According to field filling experience, the slurry has a paste property if its bleeding rate is below 5% [20]. Therefore, it is believed that the system of excitant and fly ash can be added to improve the fluidity of slurry and ensure that no segregation or pipe blockage occurs during the slurry transportation. The primary cause is that although the lime used in the test is hydrated lime ($Ca(OH)_2$), it still contains a small amount of quicklime (CaO). The digestion reaction between CaO and water continues to generate $Ca(OH)_2$, and $Ca(OH)_2$ particles are in the form of colloidal dispersion. The diameter of these particles is about 1 m, and their surface is covered by a thick water film. As indicated by a large number of particles and large total surface area, lime has a good water-retaining property, so that the slurry has a certain stability without excess water secretion. There is no big difference in fluidity between unmixed cement mortar and slurry containing the system of excitant and fly ash, so no detailed analysis is conducted, and only the bleeding rate of slurry is analyzed by multiple linear regression and variance analysis, as shown in the following formula:

$$y = 4.32 - 13.67x_1 - 2.5x_2 + 6.5x_3 + 9x_4, \tag{8}$$

where y is the bleeding rate of slurry; x_1 is the lime dosage, %; x_2 is the gypsum dosage, %; x_3 is the NaoH dosage, %; x_4 is the Na_2SO_4 dosage.

The negative correlation coefficient R^2 of curve fitting is 0.848, indicating that the regression of this equation is significant and the curve fitting is highly accurate.

(a)

(b)

Figure 1: Morphology and phase test. (a) SEM. (b) XRD.

Table 6: Rheological properties of slurry.

Test No.	A Lime dosage (%)	B Gypsum dosage (%)	C NaOH dosage (%)	D Na_2SO_4 dosage (%)	Syneresis rate (%)	Fluidity (mm)
FH-1	1 (8)	1 (4)	1 (1)	1 (2)	3.47	131
FH-2	1	2 (5)	2 (2)	2 (3)	3.17	140
FH-3	1	3 (6)	3 (3)	3 (4)	3.73	155
FH-4	2 (12)	1	2	3	3.14	145
FH-5	2	2	3	1	3.02	138
FH-6	2	3	1	2	2.94	115
FH-7	3 (16)	1	3	2	2.45	136
FH-8	3	2	1	3	2.4	112
FH-9	3	3	2	1	2.24	110
KD-12	0	0	0	0	9.25	150

Notes: KD-12 is unmixed cement mortar; KD-11 is fly ash containing 50 additives, without excitant.

Variance analysis is carried out on the bleeding rate of slurry. According to Table 6, the influence of each factor on the bleeding rate of slurry from big to small is as follows: lime dosage > NaOH dosage > Na_2SO_4 dosage > gypsum dosage; the optimal scheme is $A_3B_2C_2D_2$, that is, 16% lime, 5% gypsum, 2% NaOH, and 3% Na_2SO_4 (due to limited space, variance analysis is not described).

5.2. Strength Analysis of Fly Ash Cement Gelatin. For some specimens that have reached the curing period, the compressive strength test is carried out by 200-C-1 compression-testing machine manufactured by Wuxi Building Materials Instrument Factory, and the test results are shown in Figure 2. Other specimens are divided by a cutting machine, and a small part in the middle is retained and placed in industrial alcohol to stop its hydration reaction. When necessary, the specimen is taken out and carbon sprayed on its surface, and then it can be scanned by SEM [21].

According to Figure 2, when the curing period is only 3 d, the addition of excitant can improve the compressive strength of fly ash cement gelatin, but its maximum strength value is far less than that of ordinary Portland cement. According to Figure 3, with the increase of curing period and when it reaches 7 d, the strength value of the gelatin containing the system of excitant and fly ash is 68%~85%, and that of the gelatin without excitant is 46%~60%. When the curing period reaches 28 d, the strength value of the gelatin containing the system of excitant and fly ash is 37%~78%, and that of the gelatin without excitant is 35%~68%. Therefore, excitant can be added to increase the hydration speed of fly ash and enhance the compressive strength of fly ash cement gelatin. When the curing period reaches 90 d or 180 d, the compressive strength of gelatin containing the system of excitant and fly ash is greater than that of ordinary Portland cement.

Multivariate linear regression analysis and variance analysis are carried out on the compressive strength of gelatin containing the system of excitant and fly ash at 3 d, 7 d, and 28 d, as shown in formulas (9)–(11):

$$y = 0.45 + 0.02x_1 - 0.06x_2 + 0.28x_3 + 0.04x_4. \quad (9)$$

The F-value test method is adopted, and the critical value of F-test is $F_{0.95}$ $(4.5) = 5.19 < F = 61.95$. The regression equation is significant and the negative correlation coefficient $R_2 = 0.9644$, where y is the compressive strength of gelatin; x_1 is the lime dosage, %; x_2 is the gypsum dosage, %; x_3 is the NaoH dosage, %; x_4 is the Na_2SO_4 dosage.

$$y = 4.62 + 0.001x_1 - 0.41x_2 + 1.63x_3 - 0.23x_4. \quad (10)$$

The F-value test method is adopted, and the critical value of F-test is $F_{0.95}$ $(4.5) = 5.19 < F = 71.12$. The

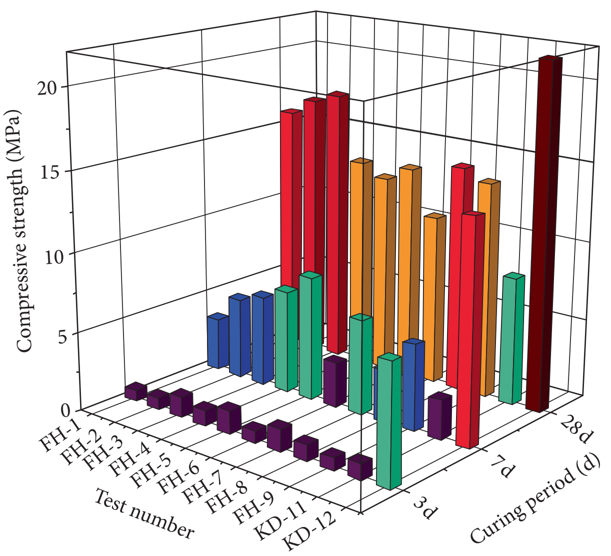


Figure 2: Variation of compressive strength of cementitious materials with different ratios.

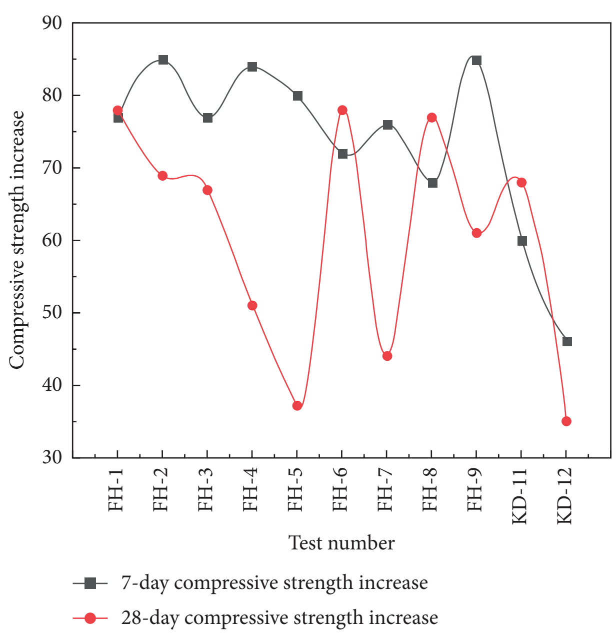


Figure 3: Increase of compressive strength with curing age.

regression equation is significant and the negative correlation coefficient $R_2 = 0.9689$, where y is the compressive strength of gelatin; x_1 is the lime dosage, %; x_2 is the gypsum dosage, %; x_3 is the NaoH dosage, %; x_4 is the Na_2SO_4 dosage.

$$y = 16.24 - 0.49x_1 + 0.72x_2 - 0.50x_3 + 0.46x_4. \quad (11)$$

The F-value test method is adopted, and the critical value of F-test is F0.95 (4.5) = 5.19 < F = 302.44. The regression equation is significant and the negative correlation coefficient $R_2 = 0.99259$, where y is the compressive strength of gelatin; x_1 is the lime dosage, %; x_2 is the gypsum dosage, %; x_3 is the NaoH dosage, %; x_4 is the Na_2SO_4 dosage.

Variance analysis is carried out on the compressive strength of gelatin containing the system of excitant and fly ash at 3 d, 7 d, and 28 d, and the analysis results are shown in Table 7. According to Table 7, the fluence of each factor on the early strength of gelatin from big to small is as follows: NaOH dosage > lime dosage > Na_2SO_4 dosage > gypsum dosage. The optimal scheme is $A_2B_2C_3D_3$ or $A_3B_2C_3D_3$ (the influence and optimal scheme for gelatin with a curing period of 7 d and 3 d are the same, so they are not listed). The influence on long-term strength from big to small is as follows: lime dosage > gypsum dosage > Na_2SO_4 dosage > NaOH dosage, and the optimal scheme is $A_1B_3C_2D_3$.

6. Analysis of Gelatin Morphology and Phase Test Results

6.1. SEM Analysis of Hydration Products. According to Figure 4(a), the solid particles in the slurry are uniformly dispersed in the fly ash cement gelatin without the addition of excitant upon the curing period of 3 d. There are few hydration products, and only a small amount of C-S-H flocs and tabular $Ca(OH)_2$ are produced, leading to loose gelatin structure and no stable structure. According to Figure 4(b), compared with gelatin at 3 d, the gelatin at 28 d has a large amount of acicular AFt, which extend and cross each other to form a denser network structure.

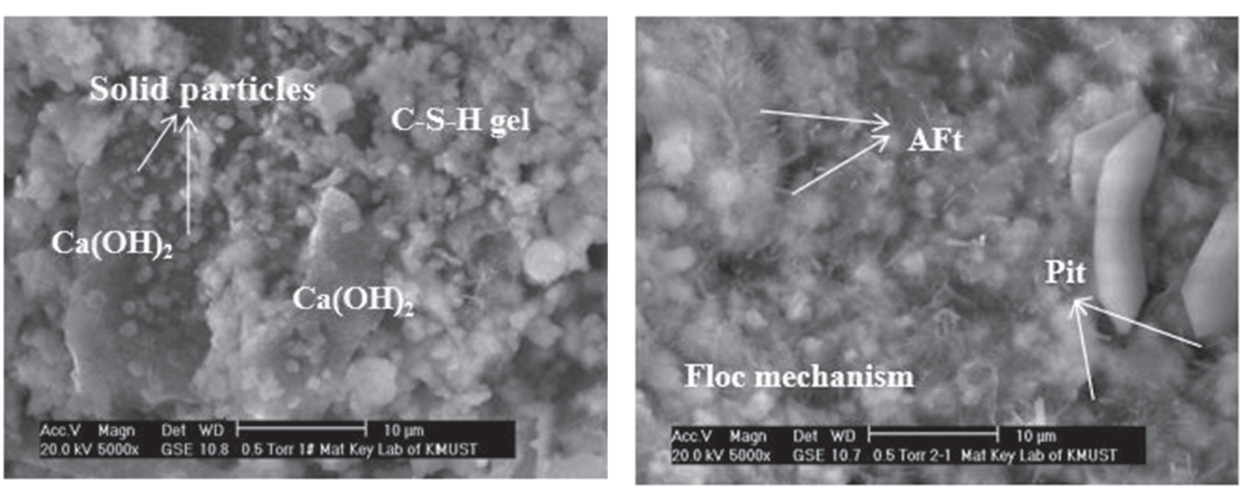
According to Figure 5(a), although the curing period of gelatin containing excitant is only 3 d, the solid particles have been wrapped by hydration products, and the particles are connected through AFt to form a dense network structure. Compared with Figure 4(a), the hydration of fly ash is obviously improved, the hydration products are increased, the network structure formed is more complex, and the gelatin is more dense. According to Figure 5(b) in which excitant is added, compared with Figure 4(b), the hydration of the gelatin is more significant, the internal structure is denser, and the hydration products are more complex, mainly including C–S–H gelatin, AFt, $Ca(OH)_2$, and $CaCO_3$. In addition, AFt becomes thicker and denser, so that all particles are tightly connected into one piece, and no obvious gap can be seen.

6.2. XRD Analysis of Hydration Products. As can be seen from Figure 6(a), the hydration reaction of the specimen after curing for 3 d generates substances such as $Ca(OH)_2$(C), AFt(E), and C-S-H gelatin. With the increase of hydration cycle, the diffraction peak of $Ca(OH)_2$ decreases obviously, which is mainly due to the chemical reaction between $Ca(OH)_2$ and quartz (Q) or mullite in fly ash, resulting in new hydration products. However, the diffraction peak of AFt basically has no change, which indicates that the reaction speed of fly ash is slow without excitant, resulting in a low growth in the strength of gelatin.

As can be seen from Figure 7(a), since the excitant contains $Ca(OH)_2$, the diffraction peak of $Ca(OH)_2$ in Figure 7(a) is higher than that of $Ca(OH)_2$ in Figure 6(a). Therefore, gelatin with t excitant has a greater early strength. The diffraction peak of $Ca(OH)_2$ in Figure 7(b) is obviously lower than that of $Ca(OH)_2$ in Figure 7(a), and the

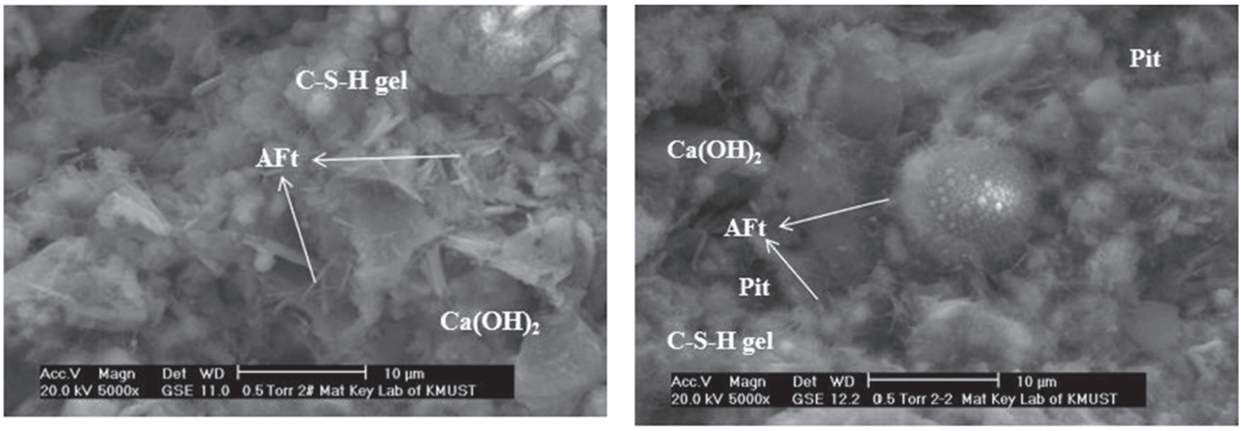
Table 7: Analysis results of orthogonal test for compressive strength of slurry.

Test No.		A Lime dosage	B Gypsum dosage	C NaOH dosage	D Na_2SO_4 dosage	Compressive strength (MPa) 3 d	7 d	28 d
FH-1		1 (8)	1 (4)	1 (1)	1 (2)	0.73	3.43	15.87
FH-2		1	2 (5)	2 (2)	2 (3)	0.75	5.17	16.93
FH-3		1	3 (6)	3 (3)	3 (4)	1.32	5.77	17.53
FH-4		2 (12)	1	2	3	1	6.53	13.4
FH-5		2	2	3	1	1.51	7.87	12.67
FH-6		2	3	1	2	0.78	2.87	13.6
FH-7		3 (16)	1	3	2	1.43	6	10.8
FH-8		3	2	1	3	1.05	3.33	14.33
FH-9		3	3	2	1	0.81	5.4	13.67
	K_1	2.8	3.16	2.56	3.05			
	K_2	3.29	3.31	2.56	2.96			
	K_3	3.29	2.91	4.26	3.37			
Comprehensive strength at 3 d	K1 = K1/3	0.933	1.053	0.853	1.017		C > A > D > B	
	K2 = K2/3	1.097	1.103	0.853	0.987			
	K3 = K3/3	1.097	0.97	1.42	1.123			
	Range	0.164	0.133	0.567	0.136			
Optimal scheme		A_2 or A_3	B_2	C_3	D_3		$A_2B_2C_3D_3$ or $A_3B_2C_3D_3$	
	K_1	50.33	40.07	43.8	42.2		A > B > D > C	
	K_2	39.67	43.93	44	41.33			
	K_3	38.8	44.8	41	45.27			
Compressive strength at 28 d	K1 = K1/3	16.78	13.36	14.6	14.07			
	K2 = K2/3	13.22	14.64	14.67	13.78			
	K3 = K3/3	12.93	14.93	13.67	15.09			
	Range	3.84	1.58	1	1.31			
Optimal scheme		A_1	B_3	C_2	D_3		$A_1B_3C_2D_3$	



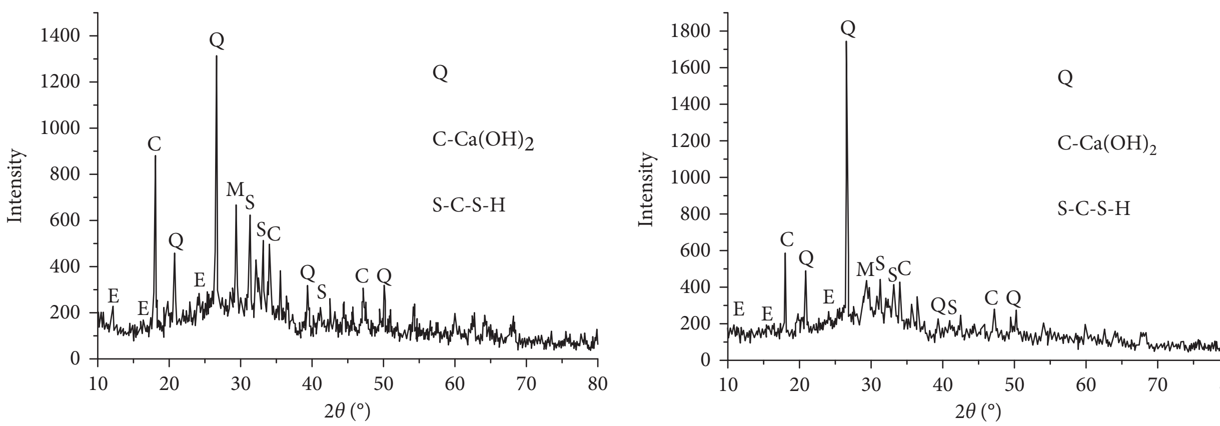
(a) (b)

Figure 4: Microstructure of cementite after 3 days and 28 days when fly ash content is 50% without activator. (a) 3 d curing period; (b) 28 d curing period.



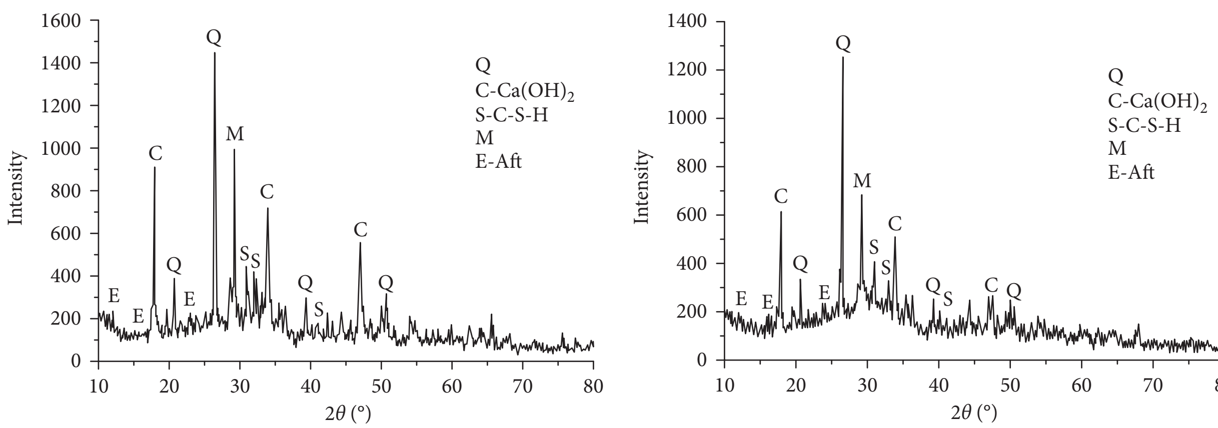
(a) (b)

Figure 5: Microstructure of cementitious body after 3 days and 28 days with 50% fly ash and activator. (a) 3 d curing period; (b) 28 d curing period.



(a) (b)

Figure 6: XRD patterns of hydration products at different ages with 50% fly ash without activator. (a) 3-day hydration; (b) 28-day hydration.



(a) (b)

Figure 7: XRD patterns of hydration products with 50% fly ash and activator at different ages. (a) 3-day hydration; (b) 28-day hydration.

diffraction peaks of mullite, quartz, and other substances in Figure 7(b) are all decreased. This is mainly because as the excitant is added, the active SiO_2 released from quartz reacts with $Ca(OH)_2$ in the slurry to form complex hydration products and increase the compressive strength of gelatin. The diffraction peak of $Ca(OH)_2$ in Figure 7(b) is significantly lower than that of $Ca(OH)_2$ in Figure 7(a), and the diffraction peaks of mullite, Shi Ying, and other substances in Figure 7(b) have all decreased. This is mainly due to the pozzolanic reaction between the active SiO_2 released from and $Ca(OH)_2$ in the slurry due to the addition of activator, which forms more complex hydration products and increases the compressive strength of the gel.

7. Conclusions

(1) The bleeding rate of slurry containing the system of excitant and fly ash is 2.24%~3.73%, which is far less than that of unmixed cement mortar (9.25%). Its fluidity is 110 mm~155 mm, which is not much different from that of unmixed cement mortar (150 mm). The influence of four excitants on the bleeding rate of slurry from big to small is lime dosage > NaOH > Na_2SO_4 > gypsum dosage, and the optimal scheme is $A_3B_2C_2D_2$.

(2) The influence of each factor on the early strength of gelatin from big to small is NaOH dosage > lime dosage > Na_2SO_4 dosage > gypsum dosage, and the optimal scheme is $A_2B_2C_3D_3$ or $A_3B_2C_3D_3$. The influence on long-term strength from big to small is lime dosage > gypsum dosage > Na_2SO_4 dosage > NaOH dosage, and the optimal scheme is $A1B_3C_2D_3$. The amount of excitant can be determined according to the strength requirements of the filling body.

(3) From the microstructure of gelatin, it can be seen that the addition of excitant makes the hydration products of fly ash more complex, mainly including C-S-H gelatin, AFt, $Ca(OH)_2$, and $CaCO_3$. In addition, with the increase of curing period, AFt becomes denser and coarser, so that the particles are tightly connected into one piece without obvious gap, and the internal structure is denser. The results of XRD analysis show that excitant can be added to stimulate the activity of fly ash, generate more complex hydration products, and increase the compressive strength of fly ash cement gelatin.

(4) According to test results, it is considered that the addition of excitant fly ash system can improve the fluidity of slurry, increase the long-term strength of filling body, and reduce its early strength. The added amount of compound excitant can be appropriately adjusted according to the needs of the mines, thereby achieving the purpose of controlling the strength of filling body.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Acknowledgments

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