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

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1. Introduction

With 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 μm and the main chemical compositions are SiO2 and Al2O3 [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 SiO2 and Al2O3 and the penetration of Ca2+, 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. 

$\text{Al}_2\text{O}_3$ and $\text{SiO}_2$ stored in fly ash bind with $\text{Ca}^{2+}$ 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.

$$m_1 \cdot \text{Ca(OH)}_2 + \text{SiO}_2 + n\text{H}_2\text{O} \longrightarrow m_1 \cdot \text{CaO} \cdot \text{SiO}_2 \cdot n\text{H}_2\text{O}$$ (2)

$$m_1 \cdot \text{Ca(OH)}_2 + \text{Al}_2\text{O}_3 + n\text{H}_2\text{O} \longrightarrow m_1 \cdot \text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$$ (3)

However, $\text{Ca}^{2+}$ in slurry is not enough for $\text{Al}_2\text{O}_3$ and $\text{SiO}_2$. Hence, excitant is added to the slurry to supplement $\text{Ca}^{2+}$ needed by $\text{Al}_2\text{O}_3$ and $\text{SiO}_2$ and also to crack the internal structure of fly ash. Then, the excitant reacts chemically with $\text{Al}_2\text{O}_3$ dissolved from fly ash in the solution containing $\text{CaO}$ to generate ettringite ($\text{Aft}$). The reaction mechanism is as follows.

$$\text{CaO} + \text{H}_2\text{O} \longrightarrow \text{Ca(OH)}_2$$ (1)

$$\text{Al}_2\text{O}_3 + \text{SiO}_2 + 3\text{C}_3\text{S}_1\text{H}_2 + 12\text{H}_2\text{O} \longrightarrow \text{C}_3\text{AH}_12$$ (4)

$$\text{C}_3\text{AH}_12 + 3\text{C}_3\text{S}_1\text{H}_2 + 14\text{H}_2\text{O} \longrightarrow \text{C}_3\text{A} \cdot 2\text{C}_3\text{S}_1\text{H}_12 + \text{Ca(OH)}_2$$ (5)

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

$$-\text{O} - \text{S} - \text{OH} + \text{NaOH} \longrightarrow -\text{O} - \text{Si} - \text{ONa} + \text{H}_2\text{O}$$ (6)

$$-\text{O} - \text{Si} - \text{O} - \text{Si} + 2\text{NaOH} \longrightarrow 2(-\text{O} - \text{Si} - \text{ONa}) + \text{H}_2\text{O}$$ (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 $\geq$85% and fineness $\geq$180 meshes produced by Kunming Maoshan Qiangda Lime Building Materials Factory; and gypsum powder produced by Yunnan Hongshu Mining Co., Ltd., as well as NaoH, NaSO$_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 NaSO$_4$. L$^9(3^4)$ 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; $\Delta V$ 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.
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 110mm∼155mm. The bleeding rate and fluidity of unmixed cement mortar are 9.25% and 150mm, 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 1mm, 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:

$$y = 4.32 - 13.67x_1 - 2.5x_2 + 6.5x_3 + 9x_4, \quad (8)$$

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$_2$SO$_4$ 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.
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 > Na2SO4 dosage; the optimal scheme is A3B2C2D2, that is, 16% lime, 5% gypsum, 2% NaOH, and 3% Na2SO4 (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 3d, 7d, and 28d, as shown in formulas (9)–(11):

\[ y = 0.45 + 0.02x_1 - 0.06x_2 + 0.28x_3 + 0.04x_4. \]  

\[ y = 4.62 + 0.001x_1 - 0.41x_2 + 1.63x_3 - 0.23x_4. \]

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 Na2SO4 dosage.

![Figure 1: Morphology and phase test. (a) SEM. (b) XRD.](image)

**Table 6: Rheological properties of slurry.**

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

Notes: KD-12 is unmixed cement mortar; KD-11 is fly ash containing 50 additives, without excitant.
Figure 2: Variation of compressive strength of cementitious materials with different ratios.

Figure 3: Increase of compressive strength with curing age.

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 influence of each factor on the early strength of gelatin from big to small is as follows: NaOH dosage > lime dosage > Na2SO4 dosage > gypsum dosage. The optimal scheme is A2B2C3D3 or A3B2C3D3 (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 > Na2SO4 dosage > NaOH dosage, and the optimal scheme is A1B3C2D3.

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 CaCO3. 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 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 Na2SO4 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 $F > 0.95$ ($4.5 = 5.19 < F = 302.44$). 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 Na2SO4 dosage.
Table 7: Analysis results of orthogonal test for compressive strength of slurry.

| Test No. | A Lime dosage | B Gypsum dosage | C NaOH dosage | D Na2SO4 dosage | Compressive strength (MPa) |
|----------|---------------|-----------------|---------------|-----------------|---------------------------|
| 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           |

Comprehensive strength at 3 d:
- K1 = K1/3 = 0.933
- K2 = K2/3 = 1.097
- K3 = K3/3 = 1.097

Range: 0.164 0.133 0.567 0.136

Optimal scheme: A2 or A3 B2 C3 D3

Compressive strength at 28 d:
- K1 = K1/3 = 16.78
- K2 = K2/3 = 13.22
- K3 = K3/3 = 12.93

Range: 3.84 1.58 1.0 1.31

Optimal scheme: A1 B3 C2 D3

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.
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.

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

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 SiO₂ 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 SiO₂ 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 > Na₂SO₄ > gypsum dosage, and the optimal scheme is A₁B₂C₃D₃.

(2) The influence of each factor on the early strength of gelatin from big to small is NaOH dosage > lime dosage > Na₂SO₄ dosage > gypsum dosage, and the optimal scheme is A₁B₂C₃D₃ or A₂B₂C₃D₃. The influence on long-term strength from big to small is lime dosage > gypsum dosage > Na₂SO₄ dosage > NaOH dosage, and the optimal scheme is A₁B₁C₂D₃. 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 CaCO₃. 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.

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