Research and Application of Fast-Strengthening Environment-Friendly Sulfoaluminate Cement Slurry on Taguchi Method

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Abstract: Most of the existing research on cement slurry materials are not environmentally friendly and environmental pollution is significant. Most researchers only test its performance, but do not conduct engineering feasibility verification. In this study, the materials extracted from various wastes were used to replace part of the sulfoaluminate cement, and orthogonal experiments were designed to analyze the reaction mechanism between different materials. Finally, the optimal mixing ratio was obtained. Then, through the regression equation analysis method, digital photograph restoration technology, the finite element method and various practical engineering conditions, the feasibility of the slurry under different applicable engineering conditions was compared and analyzed. The comparison between the experimental and numerical simulation results shows that the cement slurry obtained in this study has good reliability and feasibility. It can carry out rapid grouting reinforcement. The results of this study not only provide a feasible and environmentally friendly cement slurry for a wide range of construction projects, but also provide an effective method for the treatment of various wastes.

Keywords: sulfoaluminate cement; Taguchi grey relational analysis; strengthened by rapid grouting; waste recycling; feasibility verification

1. Introduction

In recent years, with the increasing mining intensity of coal resources, external resources are decreasing daily. The construction of deep wells has become an inevitable trend and the mining conditions are increasingly complex [1–5]. Grouting and plugging water in micro-fractures in deep wells is a bottleneck restricting the rapid advance of the wellbore and the safe production of deep wells. Grouting is one of the most effective methods to prevent water seepage in vertical shaft walls and strengthen the surrounding rock of the shaft wall [6–11]. The wellbore is accompanied by a large amount of water seepage during the excavation process. The ordinary cement that was adopted for post-wall grouting can effectively seal large cracks, but the sealing effect in micro-cracks was poor and the excellent wall still produced water in the form of large-area uniform sweat seepage. The chemical slurry is limited due to its high price, complex grouting process and environmental pollution. The research and application of grouting materials have turned to composite slurries [12]. Many scholars have studied the influence and mechanism of external admixtures (nano-SiO2, nano-CaCO3, limestone powder, etc.) on the performance of cement slurry to improve the performance of cement slurry and apply it to micro-crack grouting and water blocking.
In the research on the properties of fly ash–cement composite slurry, Wang et al. [13] studied the morphological characteristics of the interface formed between the fly ash mixed with cement and cement slurry through SEM and EDC. Li et al. [14] measured the fractal dimension of the pore volume of the fly ash–cement slurry at different ages through experiments and discussed the relationship between the fractal dimension of the pore volume and the porosity of the pore surface area, average pore size, pore distribution and macroscopic mechanical properties. Shi et al. [15] studied the effect of different amounts of fly ash on the early hydration and pore structure of hardened cement paste. Liu et al. [16] studied the influence of slurry concentration, fly ash content, cement content, coal gangue content and other factors on slurry fluidity through the flow test of a high-dosage fly ash slurry.

In the research on the mechanical properties and hydration of nano-CaCO_3 cement composites, Li et al. [17] found through experimental research that with the increase of the nano-CaCO_3 content, the slump first increased and then decreased and the initial setting time gradually increased. Although shortened, the final setting time did not change significantly, the strength at each age showed an increasing trend and the resistance to chloride ion penetration gradually improved. Kawashima et al. [18] studied the dispersion of calcium carbonate nanoparticles based on their hydration rate, solidification rate and resistance [19]; the study focused on the effect of nano-CaCO_3 particles with an average diameter of 60 nm on the properties of cement materials through experiments. The results showed that nano-CaCO_3 particles could improve the early cement hydration and interface properties to a certain extent. J. Camiletti et al. [20] found through experiments that nano-CaCO_3 can promote the process of cement hydration through the nucleation effect and is an effective filler. The dense microstructure produced by filling can improve the early mechanical properties of cement-based materials. Tian et al. [21] studied the mechanism of the effect of nano-CaCO_3 on concrete properties through the heat of hydration test method. They found that nano-CaCO_3 can promote the hydration reaction of cement and improve the microstructure of cement paste.

However, the cement materials used in these previous studies were not environmentally friendly. Moreover, researchers who choose waste materials for cement slurries usually neglect feasibility verification.

Therefore, combined with the Taguchi grey relational analysis method, the effects of different fine-grained sulfoaluminate cement (FISC), xCaCO_3·yH_2O (colloid, abbreviated as CAH) content, the water–cement ratio (WCR), fine particle size incineration ash (FPSA, not fly ash) and superplasticizer (SUPL) content on the performance of the cement slurry were studied and their mechanism of action was analyzed. Their application in the cement slurry field provides a theoretical basis. Finally, the optimal mixing ratio of the cement slurry was obtained. Through the regression equation analysis method, digital photographic restoration technology and the finite element method, combined with a variety of actual engineering conditions, the feasibility of the slurry under various actual engineering conditions was compared and analyzed. The feasibility verification shows that the cement slurry obtained in this study has good reliability and feasibility. It can carry out rapid grouting reinforcement. The results of this study not only provide a feasible and environmentally friendly cement slurry material for a wide range of construction projects, but also provide an efficient method for the treatment of various wastes.

2. Materials and Methods

2.1. Materials

2.1.1. FISC

FISC: To make the cement hydration reaction more fully, ultrafine cement with a smaller particle size was used (the particle size is mainly distributed: 0.51~15.36 μm and the strength grade is 42.5). In this study, sulfoaluminate cement with a faster setting time was used as the cement to ensure grouting reinforcement of the engineering rock mass. After testing, the chemical composition of fine-grained cement has been shown in Table 1.
Table 1. FISC chemical composition.

| Component | SiO$_2$ | Al$_2$O$_3$ | Na$_2$O | CaO | SO$_3$ | Fe$_2$O$_3$ | MgO | K$_2$O | Loss |
|-----------|---------|-------------|---------|-----|--------|------------|------|-------|------|
| Content (%) | 10.358  | 27.483      | 0.972   | 42.653 | 9.574   | 5.619      | 1.213 | 0.257 | 1.871 |

2.1.2. FPSA

FPSA is obtained from factory waste dumps. To have a sufficient reaction in the experiment and apply it to the tiny cracks in the rock, it was ground into a smaller particle size (particle size processed to 5 $\mu$m). The chemical composition of FPSA is shown in Table 2. This method avoids the high cost that researchers need to purchase FPSA in the past and provides a way to process FPSA, which better protects the environment. FPSA is not fly ash.

Table 2. FPSA chemical composition.

| Component | CaO | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | SO$_3$ | MgO | Na$_2$O | K$_2$O | TiO$_2$ | LOI |
|-----------|-----|---------|-------------|------------|--------|------|--------|-------|--------|-----|
| Content (%) | 4.12 | 40.57   | 17.78       | 28.95      | 1.27   | 1.43 | 1.02   | 1.35  | 0.95   | 2.56 |

A note on the environment and volcanic ash effects of FPSA:

Several technical conditions of FPSA are: Loss on ignition $\leq$ 3%, Arsenic $\leq$ 0.0003%, Heavy metal $\leq$ 0.0009%, Chloride $\leq$ 0.001%, Ammonia precipitates $\leq$ 0.0001%. This indicates that FPSA meets the requirements of the conventional experimental environment.

Usually, the fly ash pozzolanic effect is due to the chemical reaction of SiO$_2$, Al$_2$O$_3$ and other substances with the alkaline activator Ca(OH)$_2$. The FPSA in this study contains a lot of SiO$_2$, Al$_2$O$_3$ and other substances. Meanwhile, for FPSA, a single factor test was performed before the orthogonal test. That is, the experiment in which only the dosage of FPSA was changed. The experimental results show that when the dosage of FPSA is appropriate, the pozzolanic effect is stimulated due to the action of the activator Ca(OH)$_2$, gels such as calcium silicate hydrate are formed and the strength of the slurry consolidation increases slightly. When the amount of FPSA added is large, the relative content of the activator decreases and the FPSA that has not been activated by the pozzolanic activity acts as an inert material in the slurry consolidation. In addition, the carbon particles in the FPSA absorb the water in the slurry system to make the volume increase, resulting in a decrease in the stone rate of the slurry. This is in line with the conclusion of existing research [4] to a certain extent. Therefore, the authors believe that FPSA has pozzolanic properties and has the same effect as ordinary fly ash.

2.1.3. CAH

There is a large amount of abandoned limestone in nature. It was obtained by the chemical extraction method, grinding processing method and sol-gel method to obtain xCaCO$_3$·yH$_2$O (colloid):

1. Chemical methods are used to extract calcium carbonate from limestone.
2. The obtained CaCO$_3$ was processed into nano-scale powder by a grinder.
3. A sol-gel method was employed to obtain xCaCO$_3$·yH$_2$O (colloid), the CAH referred to in this paper. CAH is a sol and its particle size is nanoscale.

This is different from the calcium carbonate that researchers purchased directly from factories in the past. This method is not only cost-effective, but also environmentally friendly and can effectively support experiments. The obtained CAH particle size is nanoscale, which can well meet the experimental needs. Technical indicators of CAH are shown in Table 3. Figure 1 is the XRD ray diffraction pattern of CAH; it is visible that its crystal phase material is mainly CaCO$_3$. This method can not only obtain CAH to improve the performance of cement slurry, but also provides a way for waste disposal, which better protects the environment.
Table 3. Basic properties of CAH.

| Exterior   | Odor      | Specific Gravity | PH  | Average Particle Size | Quality Score |
|------------|-----------|------------------|-----|-----------------------|---------------|
| White powder | Odorless | 2.7 g/cm³        | 9.5 | 30 nm                 | ≥99.9%        |

Figure 1. XRD pattern of CAH.

2.1.4. SUPL

The SUPL used in this study is produced by a chemical company in Shanghai. The appearance is a white powder, the specific gravity is 1.08 g/cm³, the active ingredient is ≥99.9% and it has the effect of air entrainment and retardation. A certain amount of air bubbles can improve the fluidity of the slurry, but too many air bubbles will adversely affect the strength of the stone body [22]. Therefore, the air-entraining effect of the water-reducing agent should also be considered when applying the water-reducing agent. Figure 2 is a Scanning Electron Microscopy (SEM) image of SUPL. From Figure 2, SUPL is in a better dispersion state, is without flocculation and can be well used in experiments.

Figure 2. SEM image of SUPL.

2.2. Methods

Four main influencing factors were designed in this orthogonal experiment, namely CAH content, WCR, FPSA content and SUPL content (denoted as A, B, C, D, respectively). Three levels were set for each influencing factor. The experiment was arranged in an orthogonal table, with a total of 9 groups of experiments, and each group of experiments
was repeated at least 3 times to avoid accidental errors. Table 4 shows the factor levels of orthogonal experiments [4].

**Table 4. Experiment factor level table.**

| Level | Factor |
|-------|--------|
|       | A (%)  | B        | C (%) | D (%) |
| 1     | 0.5    | 1        | 20    | 0.3   |
| 2     | 1      | 1.2      | 30    | 0.4   |
| 3     | 2      | 1.5      | 40    | 0.5   |

The equipment and instruments required in the test process mainly include cement mortar mixer, Martens funnel, truncated circular cone die, measuring cylinder and DKZ-5000 cement electric anti-folding machine.

Viscosity (VIS): the apparent viscosity (unit is s) of the slurry was determined using a Marsh funnel, which was characterized by measuring the time required for the slurry (946 mL) to flow freely from the Marsh funnel under the action of gravity.

The viscosity (unit is mPa·s) was measured and recorded by an SNB-2 digital viscometer; the test time was 140 min.

Fluidity (FLU): the fluidity of the slurry was measured by using a truncated cone die with an upper diameter of 36 mm, a lower diameter of 60 mm, a height of 60 mm and a glass plate of \( 800 \times 800 \times 5 \) mm. The unit is cm.

Unconfined uniaxial compressive strength (UNS) and different confining pressure (1 MPa and 2 MPa) triaxial compression strength (1TS and 2TS): \( \phi 50 \times 100 \) mm samples were tested by JAW-2000 Electro-hydraulic servo triaxial instrument.

Stone rate (STR): 100 mL of the slurry was obtained with a graduated cylinder and placed in a standard curing box. The curing temperature was \((20 \pm 2) ^\circ \text{C}\), the humidity was 95% and the curing was for 24 h. Subsequently, the stone rate was measured. An STR greater than 95% is generally considered to be a stable slurry.

Flexural strength (FLS): the DKZ-5000 cement electric flexural strength machine was used to measure the flexural strength of the stone body. Samples to the instrument dimensions were made and cured for 24 h. Subsequently, the samples were placed in a standard curing box for 28 days.

### 3. Taguchi Orthogonal Experiment Results and Flow Performance Analysis

#### 3.1. Taguchi Orthogonal Experiment Design and Results

The orthogonal experimental schemes and experimental results are listed in Table 5.

**Table 5. Results and schemes of orthogonal experiments.**

| No. | Scheme | A | B | C | D | VIS (s) | FLU (cm) | FLS (MPa) | STR (%) | UNS (MPa) | 1TS (MPa) | 2TS (MPa) |
|-----|--------|---|---|---|---|--------|----------|-----------|---------|-----------|-----------|-----------|
| 1#  | A\(_3\)B\(_2\)C\(_2\)D\(_3\) | 2 | 1 | 30 | 0.5 | 32.26  | 45.57    | 4.56      | 96.7    | 31.52     | 35.54     | 38.21     |
| 2#  | A\(_3\)B\(_2\)C\(_3\)D\(_1\) | 2 | 1.2 | 40 | 0.3 | 28.31  | 53.74    | 3.66      | 96.4    | 26.16     | 30.03     | 33.56     |
| 3#  | A\(_3\)B\(_2\)C\(_1\)D\(_2\) | 2 | 1.5 | 20 | 0.4 | 27.84  | 55.36    | 3.27      | 90.5    | 20.34     | 24.55     | 26.84     |
| 4#  | A\(_2\)B\(_2\)C\(_2\)D\(_1\) | 1 | 1.5 | 30 | 0.3 | 28.06  | 54.98    | 3.01      | 92.3    | 16.82     | 20.97     | 23.25     |
| 5#  | A\(_3\)B\(_2\)C\(_3\)D\(_2\) | 1 | 1.2 | 40 | 0.5 | 28.22  | 53.99    | 4.05      | 94.0    | 27.01     | 31.15     | 35.72     |
| 6#  | A\(_2\)B\(_2\)C\(_1\)D\(_1\) | 1 | 1 | 20 | 0.4 | 31.21  | 45.43    | 4.11      | 96.6    | 27.55     | 32.02     | 36.01     |
| 7#  | A\(_1\)B\(_1\)C\(_1\)D\(_1\) | 0.5 | 1 | 20 | 0.3 | 32.39  | 42.35    | 4.91      | 97.1    | 32.97     | 35.99     | 38.84     |
| 8#  | A\(_1\)B\(_2\)C\(_2\)D\(_2\) | 0.5 | 1.2 | 30 | 0.4 | 28.52  | 51.73    | 3.95      | 95.1    | 25.31     | 29.74     | 32.23     |
| 9#  | A\(_1\)B\(_2\)C\(_3\)D\(_3\) | 0.5 | 1.5 | 40 | 0.5 | 27.11  | 62.31    | 3.02      | 90.9    | 17.06     | 21.52     | 25.03     |

#### 3.2. Flow Performance (VIS) Analysis

VIS is the main index to evaluate the flow performance of cement grout. The greater the VIS, the worse the flow performance of the cement grout and the worse the injection. The range analysis results of influencing factors of the slurry VIS are shown in Table 6.
3.2. Flow Performance (VIS) Analysis

VIS is the main index to evaluate the flow performance of cement grouts and injection materials. The smaller the VIS, the better the flow performance of the cement grout and the better the injection. As the amount of water added increases, the fluidity of the slurry increases, thereby reducing the VIS [23]. 

When the addition of SUPL was 0.5%, the VIS of the slurry was 29.34 s. When the dosage of CAH was increased to 0.5%, the VIS of the slurry was 29.19 s. When the dosage of CAH was increased to 1.5%, the VIS of the slurry was 27.67 s, respectively. The change rates were 11.27% and 2.40%, respectively. It is due to the fact that the water is added, then, as its fluidity is higher, it increases the fluidity of the slurry, hence reducing the VIS [23]. 

The analysis of Figure 3 and Table 6 indicates that increasing the amount of CAH added had little effect on the VIS of the cement-based slurry. When the amount of CAH added was 0.5%, the VIS of the slurry was 29.34 s. When the dosage of CAH was increased to 2%, the VIS of the slurry was 29.47 s, the growth rate was 1.4% and its influence could be ignored. The impact of WCR on the VIS of the ultrafine cement-based slurry was significant, the VIS gradually decreased with the increase of WCR. When the WCR was 1.0, 1.2 and 1.5, the VIS of the slurry was 31.95 s, 28.35 s and 27.67 s, respectively. The change rates were 11.27% and 2.40%, respectively. It is due to the fact that the water is added, then, as its fluidity is higher, it increases the fluidity of the slurry, hence reducing the VIS [23]. The VIS of an ultrafine cement-based slurry can be slightly reduced by increasing the amount of CAH added. When the amount of CAH added was 0.5%, the VIS of the slurry was 29.34 s. When the dosage of CAH was increased to 2%, the VIS of the slurry was 29.47 s, the growth rate was 1.4% and its influence could be ignored. The impact of WCR on the VIS of the ultrafine cement-based slurry was significant, the VIS gradually decreased with the increase of WCR. When the WCR was 1.0, 1.2 and 1.5, the VIS of the slurry was 31.95 s, 28.35 s and 27.67 s, respectively. The change rates were 11.27% and 2.40%, respectively. It is due to the fact that the water is added, then, as its fluidity is higher, it increases the fluidity of the slurry, hence reducing the VIS [23].

The VIS of an ultrafine cement-based slurry can be slightly reduced by increasing the amount of SUPL added. When the content of SUPL rose from 0.3% to 0.5%, the slurry VIS decreased by 0.40 s. It can be seen from the literature [24] that SUPL can change the surface characteristics of cementitious material particles. On the one hand, it can disperse solid particles; on the other hand, it can destroy the flocculation-like structure in the slurry so that the encapsulated flocculation water is released and the slurry is free. The VIS of the slurry decreased with increasing water. Due to the addition of FPSA, the VIS of the cement slurry was reduced. The larger the amount of FPSA added, the smaller the VIS of the cement slurry. When the content of FPSA increased to 40%, the VIS of the slurry decreased by 2.60 s.

Table 6. Range analysis table of influencing factors of VIS. (Note: Ki is the i-th level average value of the factor, the same below, i = 1, 2, 3).

| Level | A   | B   | C   | D   |
|-------|-----|-----|-----|-----|
| K1    | 29.34 | 31.95 | 30.48 | 29.59 |
| K2    | 29.16 | 28.35 | 29.61 | 29.19 |
| K3    | 29.47 | 27.67 | 27.88 | 29.19 |
| Range | 0.31  | 4.28  | 2.60  | 0.40  |

From Table 6, the influence degree of each factor on the slurry viscosity is WCR > FPSA > SUPL > CAH. The influence trend of each factor was shown in Figure 3.
3.3. Flow Performance (VIS Time-Variant) Analysis

The slurry denatures in the presence of its VIS before setting. As one of the foremost parameters to evaluate the rheological properties of grout, the time-variation of VIS must be considered in the research of grouting theory and numerical calculation. The VIS of 9 groups of samples in the orthogonal test was tested and recorded. The VIS time-varying curves were drawn (Figure 4) for visual analysis.

![VIS time-varying curve](image)

**Figure 4. VIS time-varying curve.**

From Figure 4, the initial VIS value of each group of slurries is between 5 and 15 mPa·s. Compared with the pure ultrafine cement slurry, its initial VIS is lower. The change of the VIS with time can be divided into two stages: the sound stage and the rising stage. The inflection point of the VIS time-varying curve mostly appears around 70 min and the VIS time-varying curve is relatively flat during the stable period. The change rate of the VIS does not exceed 15.5% and the slurry is in a relatively stable state.

With the progress of the hydration reaction, the time-varying curve of VIS appears at an inflection point and enters the rising period, and so the VIS of the slurry rises sharply. In the stable period, the VIS of the slurry is low and the diffusivity is strong, but it is easily eroded by dispersion. Therefore, it should be avoided that the slurry is scoured by the water, which can be used as a period of slurry migration and diffusion. During the rising period, the VIS rises rapidly in a relatively short period, which is a critical period for determining the grouting quality. In actual grouting construction, the grouting scheme can be adjusted according to the time-varying characteristics of the VIS.

3.4. Flow Performance (FLU) Analysis

The FLU reflects the diffusion performance of the grouting material and is also the leading indicator of the plasticity of the cement slurry. On-site construction requires the grouting material to have good FLU. The greater the FLU, the less likely the slurry tends to segregate and stratify. The results of factor range analysis are shown in Table 7.

| Level | A   | B   | C   | D   |
|-------|-----|-----|-----|-----|
| K1    | 52.13 | 44.45 | 47.71 | 50.36 |
| K2    | 51.47 | 53.15 | 50.76 | 50.84 |
| K3    | 51.56 | 57.55 | 56.68 | 53.96 |
| Range | 0.66 | 13.1 | 8.97 | 3.6 |

From Table 7, the influence degree of each factor on the FLU is WCR > FPSA > SUPL > CAH. The influence trend of each factor is shown in Figure 5.
The analysis of Figure 5 and Table 7 indicates that:

1. With the increase of the CAH content, the FLU of the cement-based slurry gradually decreased. The reasons are: On the one hand, the particle size of CAH is small. Due to the filling effect of fine particles, the gap-filling water of the cement clinker is replaced, which increases the free water molecules. On the other hand, CAH has a larger specific surface area. Its contact area with water molecules is also larger, which can adsorb a large number of water molecules, thereby reducing the FLU. In this experiment, the dispersion of CAH particles in the cement-based slurry was better, the adsorption effect was more obvious, so the FLU generally showed a decreasing trend.

2. FLU is greatly affected by WCR. When WCR is less than 1.2, the FLU of the cement-based slurry increases significantly. When the WCR is more significant than 1.2, simply growing the WCR will slow down the increase in the FLU of the cement-based slurry. The analysis shows that the dispersing effect of the rise of the relative water content on the cement is the main factor affecting the FLU. When the water content increases, the interaction between the attractive force between particles and the intermolecular force in the slurry is destroyed and so the FLU of the slurry increases. When the WCR is higher than 1.2, the water content is sufficient and the adsorption of particles becomes a secondary factor, leading to a slowdown in FLU variation.

3. The content of FLU and FPSA is approximately linear. The FLU continued to grow with FPSA incorporation. The analysis is that FPSA is composed of many spherical glass microbeads. Compared with FISC, its surface is smoother. At the same time, its particle size is finer, the particle shape is round, the specific surface area is small and the adsorption force for water molecules is small. These morphological characteristics reduce the water demand of the slurry and increase the fluidity and activity of the slurry.

4. Adding SUPL can effectively improve FLU. When the dosage of SUPL is 0.4% and 0.5%, the growth rate of fluidity is 0.9% and 6.14%, respectively. This is because SUPL can be adsorbed on the surface of cement particles, and the lubricating effect and dispersing effect of SUPL avoid the accumulation of the slurry. VIS is reduced and, therefore, FLU is improved.
4. Analysis of Solidification Characteristics of Cement Slurry

4.1. Analysis of Solidification Characteristics (STR)

STR is the ratio of the volume of the solidified slurry to the volume of the slurry and is an important index to evaluate the mechanical properties and stability of the slurry stone body. The results of the range analysis of the influencing factors of STR are shown in Table 8. The degree of influence of each factor on the STR of the slurry is: WCR > SUPL > FPSA > CAH. The influence trend of each factor is shown in Figure 6.

Table 8. Range analysis table of influencing factors of STR.

| Level | A   | B   | C   | D   |
|-------|-----|-----|-----|-----|
| K1    | 94.37 | 96.80 | 94.73 | 95.27 |
| K2    | 94.30 | 95.17 | 94.70 | 94.07 |
| K3    | 94.53 | 91.23 | 93.77 | 93.87 |
| Range | 0.23 | 5.57 | 0.97 | 1.4 |

The analysis of Figure 6 and Table 8 indicates that the STR of the slurry is more than 90%, indicating that the ultrafine cement grouting material mixed with FPSA and CAH has good stability. When the grout strength index and grouting meet the requirements, a reasonable grouting filling rate can be guaranteed.

WCR is the main factor affecting the rate of serious stones. With the increase of WCR, the rate of serious stones decreases continuously. The reason is analyzed as follows: the larger the WCR, the higher the free water content that acts as a dispersion effect, which leads to the decrease of STR. With the increase of CAH content, the STR first decreased and then increased. When the content of CAH was 1%, the STR was 94.30%, but its change range was small and the change rate did not exceed 0.24%.

The STR decreases with the increase of FPSA content and the reason is analyzed as follows: the pozzolanic activity of fly ash can only be manifested under the action of an activator; the hydration product of cement, Ca(OH)$_2$, is the most commonly used activator [25]. With the increase of the content of FPSA, the relative content of the activator decreases, the FPSA that has not been activated by the pozzolanic activity acts as inert material in the stone body. The carbon particles in FPSA absorb the water in the slurry system and increase its volume. This results in a decrease in the STR. The effect of SUPL on the STR is similar to that of FPSA. The STR decreases continuously with the increase of the
SUPL content and the magnitude of the change also decreases. When the content of SUPL was 0.4% and 0.5%, the change rate of the calculus rate of the serous fluid was 1.26% and 2.13%, respectively.

4.2. Analysis of Solidification Characteristics (FLS)

As can be seen from the experimental results, the intensity-related parameters show the same trend of change. Therefore, only FLS is taken as an example to analyze.

The results of the range analysis of the influencing factors of F are shown in Table 9. The influence degree of each factor on FLS is: WCR > FPSA > CAH > SUPL. The influence trend of each factor is shown in Figure 7.

**Table 9. Range analysis table of influencing factors of FLS.**

| Level | A   | B   | C   | D   |
|-------|-----|-----|-----|-----|
| K1    | 3.96| 4.53| 4.10| 3.86|
| K2    | 3.72| 3.89| 3.84| 3.78|
| K3    | 3.83| 3.1 | 3.58| 3.88|
| Range | 0.24| 1.47| 0.52| 0.10|

**Figure 7. Trend chart of the influence of various factors on FLS.**

The analysis of Figure 7 and Table 9 indicates that the CAH has a more significant impact on FLS. With the increase of CAH content, the FLS first increased and then decreased. After incorporating CAH, due to the surface effect of nanoparticles, calcium silicate hydrate gel (CSH gel), the main hydration product of cement, will form a spatial network structure with CAH as the core, making the slurry structure with an interior which is more compact [26]. In addition, when CAH and FPSA coexist, CAH will destroy the internal structure of fly-ash particles and the formed fibrous hydrate will grow in the fine cracks of the FPSA particles, which will have a certain impact on the FLS.

The FLS decreases approximately linearly with the increase of WCR. When the WCR increases, the more complete the hydration reaction of cement slurry is and the more CSH is produced. Due to its unidirectional fibrous distribution, it cannot effectively fill the pores [27], resulting in too high a porosity and thus a lower FLS.

With the increase in the SUPL dosage, the FLS showed a slight increase. When the content of SUPL was 0.3%, the FLS was 3.86 MPa. When the dosage of SUPL was increased to 0.5%, the FLS was 3.88 MPa, an increase of 0.02 MPa, with a growth rate of 0.5%. It can be seen that the amount of SUPL has little effect on the FLS.
With the increase of the FPSA dosage, the FLS showed a decreasing trend and its decreasing rate was also decreasing. The mechanism is the same as the effect of FPSA on STR: when the content of FPSA is too high, the FPSA that has not stimulated pozzolanic activity acts as an inert material and the volume expansion of carbon particles after absorbing water can easily cause the stone body to crack, resulting in a decrease in FLS.

After analysis, UNS, 1TS and 2TS all show similar characteristics to FLU.

5. Taguchi Grey Correlation Analysis of Optimal Mix Ratio and Slurry Constitutive Relation

To more accurately analyze and obtain the optimal mix ratio, in this article, due to limited space, Taguchi grey relational analysis methods [4,11,28] and commonly used equations are briefly introduced:

Step 1: According to the results of the aforementioned single factor experiment, select the appropriate parameters and orthogonal array and design the orthogonal experiment table.

Step 2: Perform multifactor and multilevel experiments according to the designed orthogonal test table.

Step 3: Calculate the signal-to-noise ratio of the test results, that is, the signal-to-noise ratio (S/N). The larger the ratio is, the smaller the interference and the better the product quality characteristics.

The signal-to-noise ratio is usually divided into three categories:

(1) The lower, the better (LB): The test index value should not be negative. The smaller the required \( Y \) value, the better, because the ideal value is zero. During the calculation process, the smaller the fluctuation of the data, the better. At this time, the calculation uses Equation (1).

\[
S/N = -10 \times \log_{10} \left( \frac{1}{n} \sum_{i=1}^{n} Y_i^2 \right)
\]

(2) The more nominal, the better (NB): This refers to the existence of a fixed target value for the test index value and requires that \( Y \) will fluctuate around the target value. At this time, Equation (2) is selected as the calculation equation.

\[
S/N = -10 \times \log_{10} \left( \frac{1}{n} \sum_{i=1}^{n} (Y_i - Y_0)^2 \right)
\]

(3) The higher, the better (HB): The test index value does not take a negative value. We require that the larger \( Y \) is, the better as the ideal value is positive infinity. During the calculation process, the greater the fluctuation of the data, the better. At this time, Equation (3) is selected as the calculation equation.

\[
S/N = -10 \times \log_{10} \left( \frac{1}{n} \sum_{i=1}^{n} \frac{1}{Y_i^2} \right)
\]

Step 4: The Taguchi grey relational method is used to analyze and optimize multiple performances and determine the optimal combination.

\[
Z_{ij} = \frac{y_{ij} - \min(y_{ij}, i = 1, 2, \ldots, n)}{\max(y_{ij}, i = 1, 2, \ldots, n) - \min(y_{ij}, i = 1, 2, \ldots, n)}
\]

In the case of S/N in HB, choose Equation (4).

\[
Z_{ij} = \frac{\max(y_{ij}, i = 1, 2, \ldots, n) - y_{ij}}{\max(y_{ij}, i = 1, 2, \ldots, n) - \min(y_{ij}, i = 1, 2, \ldots, n)}
\]
In the case of S/N in LB, Equation (5) is selected.

\[
Z_{ij} = \frac{|y_{ij} - \text{Target}| - \min(|y_{ij} - \text{Target}|, i = 1, 2, \ldots, n)}{\max(|y_{ij} - \text{Target}|, i = 1, 2, \ldots, n) - \min(|y_{ij} - \text{Target}|, i = 1, 2, \ldots, n)}
\]  

(6)

In the case of S/N in NB, Equation (6) is selected.

Equation (7) is adopted to calculate the mass loss function.

\[
\Delta = Z_{0j} - Z_{ij}
\]  

(7)

The calculation of the grey relational coefficients is performed by Equation (8).

\[
GRC_{ij} = \frac{\min(\Delta) + \xi \max(\Delta)}{\Delta + \xi \max(\Delta)}
\]  

(8)

The meaning of the values above are:

- \(GRC_{ij}\) is the grey relational coefficient of the \(j\)-th characteristic in the \(i\)-th test [11].
- \(Z_{0j}\) is the optimal value of the \(j\)-th characteristic.
- \(Z_{ij}\) is the standard value of the \(j\)-th characteristic.
- \(\xi\) is the discrimination coefficient and its value is generally between 0 and 1, usually 0.5.

The grey relational grade is calculated using Equation (9).

\[
G_j = \frac{1}{m} \sum_{i=1}^{m} GRC_{ij}
\]  

(9)

\(G_j\) represents the optimal level. The higher the \(G_j\), the better the quality of the product. For each factor \(i\), there is a corresponding level \(j\). By calculating its mean of grade values (\(\zeta_{ij}\)), the influence of \(E_i\) can be further calculated:

\[
E_i = \max(\zeta_{ij}) - \min(\zeta_{ij})
\]  

(10)

For different factors \(i\), the optimal level \(j^*\) is obtained by the following method:

\[
j^* = \max(\zeta_{ij})
\]  

(11)

Step 5: In addition, to obtain important statistically significant process parameters and the percentage contribution of these parameters to the performance of samples containing UFA and SH materials, it is also necessary to evaluate and analyze the data through the statistical technique of variance analysis.

The Taguchi grey relational analysis method was adopted, combined with the above analysis experimental data (among them, LB is used for VSI and coagulation time, HB is used for the rest) and the optimal mix ratio \((A_2B_3C_3D_3)\) of the orthogonal experiment was obtained. After the experiments, its performances were: 27.12 s (VSI), 51.36 cm (FLU), 96.21% (STR), 4.08 MPa (FLS), 30.58 MPa (UNS), 35.26 MPa (1TS) and 39.09 MPa (2TS).

After obtaining the optimal mixing ratio, to confirm whether the slurry can quickly perform grouting and water plugging reinforcement in the project, the Vicat instrument was used to test the setting time of the slurry. The initial setting time (79.4 min) and final setting time (115.2 min) were obtained. Compared with previous studies, the slurry has a shorter setting time and can be quickly grouted for reinforcement.

Cement slurry is more concerned about its fluidity in the grouting, therefore, the follow-up research of this study will verify the flow law and injectability of the cement slurry through numerical simulation and other means.

In this study, the constitutive model of the CAH new cement slurry (Figure 8) was obtained by analyzing the experimental data and using the regression equation method. According to the ideas in the research of previous researchers [4,11], the constitutive
In this study, the constitutive model of the CAH new cement slurry (Figure 8) was established. The constitutive equation can be input into the numerical simulation software through MATLAB in the future so that the numerical simulation calculation is more accurate and credible.

![Figure 8. CAH new cement slurry constitutive model (shear stress–shear rate curve).](image)

The correlation coefficient is 0.97854, indicating that the fitting effect is good. The cohesive force of the cement slurry, which is the intercept of the curve on the y-axis, has a value of 1.51326 Pa, which is also in good agreement with the research of Wu [4,11].

6. Feasibility Verification

To verify the feasibility of the cement slurry in this study, a rock with cracks (RTC) was obtained at a project site and cut into $50 \times 100$ mm specimens and $100 \times 100 \times 200$ mm specimens. The cement slurry obtained in the previous study was injected into the RTC through a pressure pump and then cured under standard conditions. Then, a comparative study was carried out on the RTC before grouting (BG) and after grouting (AG), under different conditions.

6.1. Numerical Simulation Comparative Study

Uniaxial compression tests and triaxial compression tests under different confining pressure conditions (5 MPa, 10 MPa) were carried out. The data analysis of the triaxial compression experiment showed that the Poisson’s ratio of the RTC (AG) was 0.271, the elastic modulus was 18.72 GPa, the internal friction angle was 29.7° and the cohesion was 10.23 MPa. At the same time, the strength of the cement slurry stone body was tested and calculated, the Poisson’s ratio of the cement slurry stone body was 0.542, the elastic modulus was 3.76 GPa, the internal friction angle was 13.2° and the cohesion was 4.29 MPa. These data are used in numerical simulation calculations to make the results more credible.

DPRT (digital photographic restoration technology, a method to restore blurred images using the improved Wiener filter image restoration principle in digital image processing) analyzes the obtained RTC (BG) and the major cracks it contains (the tiny cracks have little effect and are no longer shown) are restored to the computer. Through the FEM (finite element method), combined with ABAQUS software, the schematic diagram of the RTC (BG) model is established. The constitutive model of Figure 8 was imported into ABAQUS by the MATLAB algorithm. Slurry stone parameters are assigned to the slurry in RTC (BG) to obtain RTC (AG).

The results of the uniaxial compression experiment are taken as an example to illustrate the research effect. The mesh is divided by four-node linear tetrahedral elements. The constraints are consistent with the conventional uniaxial compression experiments (the bottom of the sample is fixed, the top of the sample is loaded downward and the other directions are unconstrained).

The calculation results of the uniaxial compression experiment are shown in Figure 9.
The unconfined uniaxial compressive strength of RTC (BG) is 86.71 MPa and the peak strain is $10.15 \times 10^{-3}$. The unconfined uniaxial compressive strength of RTC (AG) is 89.93 MPa (increased by 3.71%) and the peak strain is $11.24 \times 10^{-3}$ (increased by 10.74%). The unconfined uniaxial compressive strength of NS (BG) is 83.69 MPa and the peak strain is $9.83 \times 10^{-3}$. The unconfined uniaxial compressive strength of NS (AG) is 85.41 MPa (increased by 3.71%) and the peak strain is $10.59 \times 10^{-3}$ (increased by 10.74%).

The surrounding rock in actual grouting engineering is usually affected by a high confining pressure from in-situ stress and high grouting pressure during grouting. In this study, three variables (1 MPa, 6 MPa, 11 MPa) of the grouting pressure ($\sigma_g$) were set. The three variables (2 MPa, 7 MPa, 15 MPa) of the confining pressure ($\sigma_c$) were also set. The rock parameters adopted the parameters in Section 6.1. The equation in Figure 8 and the performances of the best ratio of the slurry were inputted into ABAQUS to set the cement slurry through the MATLAB algorithm. The FEM method and ABAQUS software were used to set the model of the fractured rock mass (Figure 10 is the model of JRC = 2 MPa, as the $\sigma_g$ increases from 1 MPa to 6 MPa and finally increases to 11 MPa, the corresponding $Q$ are $3.79 \times 10^{-6}$ m$^3$/s, $4.35 \times 10^{-6}$ m$^3$/s and $6.09 \times 10^{-6}$ m$^3$/s, respectively. When JRC = 3 and the $\sigma_g$ is 11 MPa, as the $\sigma_c$ increases from 2 MPa to 7 MPa and finally increases to 15 MPa, the corresponding $Q$ are $6.09 \times 10^{-6}$ m$^3$/s, $3.75 \times 10^{-6}$ m$^3$/s and $1.53 \times 10^{-6}$ m$^3$/s, respectively.

Figure 9. Comparison of uniaxial compression stress–strain curve experiment and numerical simulation (NS is the numerical simulation result).

From Figure 9, the unconfined uniaxial compressive strength of RTC (BG) is 86.71 MPa and the peak strain is $10.15 \times 10^{-3}$. The unconfined uniaxial compressive strength of RTC (AG) is 89.93 MPa (increased by 3.71%) and the peak strain is $11.24 \times 10^{-3}$ (increased by 10.74%). The unconfined uniaxial compressive strength of NS (BG) is 83.69 MPa and the peak strain is $9.83 \times 10^{-3}$. The unconfined uniaxial compressive strength of NS (AG) is 85.41 MPa (increased by 3.71%) and the peak strain is $10.59 \times 10^{-3}$ (increased by 10.74%).

The trend of RTC (BG) and RTC (AG) is similar, but the intensity of RTC (AG) is slightly higher. It shows that the cement slurry has a certain degree of reinforcement. Comparing the numerical simulation and experimental results in Figure 9, it can be found that there is a specific error between the two, but the error is small. The same characteristics are obtained in triaxial compression experiments with different confining pressures. This shows that the slurry obtained in this study has a better reinforcement effect on a rock under different conditions. That is, the slurry of this study can be applied to practical engineering.

6.2. Liquidity Rules under Different Geological Conditions

The joint roughness coefficient (JRC) in the existing study [4] is used to set the rock fracture joint surface. Two variables were set, JRC = 3 and JRC = 7, using the usual JRC [4]. The surrounding rock in actual grouting engineering is usually affected by a high confining pressure from in-situ stress and high grouting pressure during grouting. In this study, three variables (1 MPa, 6 MPa, 11 MPa) of the grouting pressure ($\sigma_g$) were set. The three variables (2 MPa, 7 MPa, 15 MPa) of the confining pressure ($\sigma_c$) were also set. The rock parameters adopted the parameters in Section 6.1. The equation in Figure 8 and the performances of the best ratio of the slurry were inputted into ABAQUS to set the cement slurry through the MATLAB algorithm. The FEM method and ABAQUS software were used to set the model of the fractured rock mass (Figure 10 is the model of JRC = 3). The volume flow rate is denoted as $Q$ ($10^{-6}$ m$^3$/s).

From Figure 11, when JRC = 3 and the $\sigma_c$ = 2 MPa, as the $\sigma_g$ increases from 1 MPa to 6 MPa and finally increases to 11 MPa, the corresponding $Q$ are $3.79 \times 10^{-6}$ m$^3$/s, $4.35 \times 10^{-6}$ m$^3$/s and $6.09 \times 10^{-6}$ m$^3$/s, respectively. When JRC = 3 and the $\sigma_g$ is 11 MPa, as the $\sigma_c$ increases from 2 MPa to 7 MPa and finally increases to 15 MPa, the corresponding $Q$ are $6.09 \times 10^{-6}$ m$^3$/s, $3.75 \times 10^{-6}$ m$^3$/s and $1.53 \times 10^{-6}$ m$^3$/s, respectively.
The geological conditions in underground engineering are complex. With the increase of the underground depth, the geological conditions of high ground temperature are usually encountered. The 100 × 100 × 200 mm sample obtained from the aforementioned engineering site was subjected to grouting experiments (using the cement slurry obtained in this study). After standard curing, direct shear experiments under different temperature conditions were carried out to compare the mechanical strength properties before and after grouting. Sample heating was performed by a box resistance meter. The temperature setting variables are 20 °C (normal temperature condition), 270 °C, 470 °C and 770 °C. The time setting variables are 1 h, 2 h and 3 h.
From Figures 12 and 13, with the increase in temperature, the shear strength gradually decreased and the rate of decrease was more significant when the temperature was higher. With the rise of heating time, the shear strength showed a negative correlation trend and the decline rate also showed a positive correlation trend with time. Compared with Figures 12 and 13, the shear strength after grouting was slightly improved, but with the increase in temperature and heating time, the shear strength decreased rapidly. The durability of the cement slurry stone body was lower on rocks. The changing trend of this conclusion is in good agreement with the research of existing scholars [11]. The accuracy and feasibility of this study are illustrated.

Figure 12. Shear strength of RTC (BG) under different geothermal conditions.

Figure 13. Shear strength of RTC (AG) under different geothermal conditions.

In summary, the results of this research can achieve good applicability in a variety of different engineering conditions.
7. Conclusions

The sulfoaluminate cement slurry analyzed by the Taguchi grey correlation analysis method has a good performance. The specific conclusions are as follows:

1. The ultrafine cement-based composite grouting material has a lower VIS. WCR was the main factor affecting the VIS, followed by the content of FPSA, and the change in the content of SUPL and CAH did not affect the VIS.

2. For the 9 groups of slurries designed in the orthogonal experiment, the time-varying VIS can be divided into two stages: the stable period and the rising period, and the VIS change rate in the stable period did not exceed 15.5%. The duration of the stable period of most of the slurry was about 70 min, which can be used as the period of slurry migration and diffusion. The rising period was shorter in duration and the VIS increased substantially.

3. The addition of CAH lead to a decrease in the FLU of the ultrafine cement-based slurry, the FLU increased with the increase of FPSA, SUPL content and WCR. STR decreased with the rise of the content of WCR, FPSA and SUPL. The WCR had the most excellent effect, while CAH had no significant impact on it.

4. The most significant effect on the FLS was WCR, followed by CAH. When the content of CAH was increased from 0.5% to 1%, the growth rate of the FLS was 22%. The content of SUPL and FPSA had little effect on the flexural strength.

5. The optimal mixing ratio (A_{2}B_{3}C_{3}D_{3}) of the cement slurry was obtained by the Taguchi grey relational orthogonal analysis method. Its optimal performance was: 27.12 s (VSI), 51.36 cm (FLU), 96.21% (STR), 4.08 MPa (FLS), 30.58 MPa (UNS), 35.26 MPa (1TS) and 39.09 MPa (2TS). In addition, the initial setting time was 79.4 min and final setting time was 115.2 min. The grout enabled rapid grouting reinforcement.

6. The constitutive model equation of the slurry was derived. Through rock sampling at the engineering site, combined with DPRT, FEM, ABAQUS and several indoor experimental types of equipment, the experimental data and numerical simulations were compared. The conclusions obtained were compared with those of previous studies. It was found that the slurry obtained in this study can adapt to a variety of engineering conditions and has good accuracy and feasibility.

7. The conclusions of this study have certain limitations; for example, the rock fracture opening is not discussed and the seepage characteristics are only discussed in terms of the flow rate. In the future, more in-depth research should be carried out to simulate grouting with multiple openings and micro-cracks and to further analyze and simulate the fluid–solid coupling effect in the grouting process. In addition, the test and field engineering verification should be combined to study the injectability of ultrafine cement grouting materials to micro-cracks in a real rock mass.

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