Development of Cementing Material of Fine Full-Tailings Filling by Slag Powder

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Abstract: The cementing material of fine full-tailings is developed with the fine tailings of Anshan Iron and Steel Company as aggregates, the water quenching slag of blast furnace as the main active material, and the desulphurized ash residue as one of the main activator materials. This paper explores the effects of composite activator with different content on the compressive strength of the filling body of the cementing material of fine full-tailings filling. After orthogonal test and analysis with different statistical methods, the optimum formula is obtained and verified by experiment. The test results indicate that the compressive strength of the new cementing material at 28 day is 2.01 times of that of cement under the same proportion. The X-ray diffraction and scanning electron microscopy are used to analyze the composition of hydration products and micro-morphology of the new cementing material. The results show that the hydration products of the cementing material are mainly flocculent C-S-H gel, and the more the hydration products are, the more compact the filling body structure will be, which form a network structure with higher strength. The new cementing material could replace cement and most of the filling materials are the waste, which could significantly reduce the cost of filling mining and is environmental-friendly. If the scientific filling technology is combined, the mine tight-filling ratio can be significantly improved, which is conducive to the safety of mining production as well as the mine construction cost control.

1. Introduction

Tailings are one of the products of the sorting operations. Due to the limitation of the current technology, it is no longer suitable for further separation, so the vast majority of tailings are stacked in the tailings, which not only takes up a lot of land resources, but also brings some negative impact on the environment. Nowadays, there are huge economic and environmental benefits for the recovery and reuse of tailings[1-2].

The water quenching slag of blast furnace is a valuable secondary resource and has been widely used in cement admixture and high-grade cement development. In order to develop the core filling cementing material of full tailings in Anshan Iron and Steel Company, the water quenching slag of blast furnace is used as the main active material, and the desulphurized ash slag is used as one of the excitant materials to study and develop the compound activator. Besides, the slag powder grained by the blast furnace water quenching slag is used to conduct stimulation, thus obtaining the new cementing material. Desulphurization ash slag has an effect on the mechanics and stability of cementing materials, whether as cement admixture or as agglomerating agent. In particular, different composition of flue gas in metallurgical industry as well as the different composition and content of desulfurization ash residue bring some difficulties and problems for the resource utilization of desulphurization ash slag[3-5]. Therefore, the current utilization of desulphurized ash is low, and most of the ashes are stacked, which lead to the
resource waste and pollute the environment. Thus, to make use of the desulfurization ash residue is an important way to protect the environment and sustainable development. Aiming at the mining method in Anshan Iron and Steel Mine, use the sintered desulfurization slag discharged from Anshan Coking Plant as the composite excitant material of slag powder and study the optimized formula of the core filling cementing material. The waste resource is fully utilized in filling mining technology, which could not only protect the environment but also minimize the cost of filling materials, thereby improving the economic benefits of mining resources, achieving the development and utilization of waste resources and greatly reducing the cost of filling mining methods\(^\text{[6-8]}\).

The physical and mechanical properties of the new cementing material are directly related to the physical and chemical properties of tailings, the physicochemical properties of slag powders and the ratio of activators\(^\text{[9]}\). The experimental study of developing the new cementing material of fine full-tailings filling using slag powder is described below.

2. A comprehensive analysis of the characteristics of filling material of full tailings

2.1. Analysis of the physical and chemical properties of the full tailings

The cementing material of full tailings filling is to use the activator to simulate the blast furnace water quenching slag with potential activity, thus resulting in hydration reaction and producing cementation. The strength of the new cementing filling is determined by the physical and chemical properties and the grain size gradation of the full tailings filling aggregate. Through the test analysis, the chemical composition analysis of the full tailings of Anshan Iron and Steel Mine and the grain size gradation results are as shown in Table 1.

| Mineral composition | TFe | FeO | SiO2 | CaO | MgO | Al2O3 | MnO | S | P | Ig |
|---------------------|-----|-----|------|-----|-----|-------|-----|---|---|----|
| Content /%          | 8.92| 6.26| 67.75| 3.44| 4.78| 1.48  | 0.31| 0.25| 0.074| 1.12 |

2.2. Analysis of grain size gradation of full tailings

According to the results of the granulated size analysis of the full tailings of Anshan Iron and Steel Company, the grain size distribution curve of full tailings in Anshan Iron and Steel Company is shown in Fig.1.

![Fig.1 Grain size gradation curve of full tailings in Anshan Iron and Steel Company](image-url)
2.3. Summary
Through the chemical analysis of the full tailings of Anshan Iron and Steel Company, we can see that the full tailings of this mine are neutral material and inert materials with no cementing activity. The weighted average grain size of the full tailings of Anshan Iron and Steel Company is 54.42μm, the effective grain size \( d_{10} = 6.26 \mu m \), the constrained diameter \( d_{60} = 44.94 \mu m \), the median diameter \( d_{50} = 34.9 \mu m \), \( d_{30} = 18.20 \mu m \). They belong to the finer tailings. The grain size of the full tailings of Anshan Iron and Steel Company is smaller than that of most domestic mine tailings, so they have poor permeability. As the filling aggregate, they may adversely affect the development of cementing materials\(^{10-11}\).

3. Physical and chemical properties of pozzolanic material
The activity of pozzolanic material mainly depends on its chemical composition, glassy phase content, fineness, particle shape and surface state, etc. Assess the potential activity of the pozzolanic material based on the chemical composition. When the pozzolana activity \( M_a = 0.17 \sim 0.25 \), it can be used as the active material of cementation filling.

The mineral composition of metallurgical slag in Anshan Iron and Steel Group Company is as shown in Table 2. Then the quality, alkalinity and activity of the two kinds of water quenching slags can be calculated.

| Name            | SiO\(_2\) | CaO | MgO | Al\(_2\)O\(_3\) | Fe\(_2\)O\(_3\) | S | SO\(_3\) | MFe | Caustic Soda |
|-----------------|-----------|-----|-----|-----------------|-----------------|---|---------|-----|-------------|
| Slags           | 35.15     | 43.46 | 7.57 | 12.15          | -               | 1.24 | -       | -   | -           |

1) The basicity coefficient \( M_0 \)
\[
M_0 = \frac{CaO + MgO}{SiO_2 + Al_2O_3} = \frac{43.46 + 7.57}{35.15 + 12.15} = \frac{51.03}{47.30} = 1.08 > 1.0
\]

2) The quality factor \( K \)
\[
K = \frac{CaO + Al_2O_3 + MgO}{SiO_2 + TiO_2} = \frac{43.46 + 12.15 + 7.57}{35.15 + 0} = \frac{63.18}{35.15} = 1.797
\]

3) The activity index \( M_a \)
\[
M_a = \frac{Al_2O_3}{SiO_2} = \frac{12.15}{35.15} = 0.345
\]

Thus, the quality and activity of metallurgical slags and steel slags of Ansteel Group could meet the requirements of developing cementing materials, and can be used to develop the new cementing material of full tailings core filling.

4. Experimental Analysis
According to the relevant literature and experience of previous experiments, it is shown that the core cementing material with the compound activator formula of quick lime has the same characteristics as the cement 32.5. And the economic benefits of its core cementing material are not so remarkable. When the quicklime is used as a stimulant in developing cementing materials, only high lime content could meet the strength requirements of the cementing material. Therefore, it will not only increase the cost of the new cementing material, but also be detrimental to the large-scale utilization of waste resources. In addition, the moisture absorption of quick lime may lead to the core cementing material susceptible to moisture deterioration, thereby shortening the shelf life of materials. In order to overcome the above disadvantages, lime-free composite activator is used in this study. For the activator, we mainly use three raw materials, desulfurization ash residue, cement clinker and industrial mirabilite.
4.1. Experiments of the lime-free desulfurization gypsum as the compound activator of the cementing material

4.1.1. Results and analysis of the first compound activator formula experiment of the cementing material

The experimental study of compound activator formula of the cementing material is carried out with four factors and orthogonal tables at three different levels. The cement-sand ratio is 1:6 and the concentration of slurry is 70%. Results of the orthogonal experiment and range analysis of the first compound activator formula experiment of the cementing material are as shown in Table 3.

Table 3 Results of the first orthogonal experiment of the cementing material (1:6, 70%, full tailings)

| Number | Cement Clinker % | Desulfurization Ash % | Mirabilite % | Slag Powder % | Compressive strength /MPa 28 day |
|--------|------------------|-----------------------|--------------|---------------|----------------------------------|
| A1     | 3                | 15                    | 0.5          | 81.5          | 1.907                            |
| A2     | 3                | 16                    | 1.0          | 80            | 1.664                            |
| A3     | 3                | 17                    | 1.5          | 78.5          | 1.727                            |
| A4     | 4                | 15                    | 1.0          | 80            | 2.078                            |
| A5     | 4                | 16                    | 1.5          | 78.5          | 1.960                            |
| A6     | 4                | 17                    | 0.5          | 78.5          | 2.021                            |
| A7     | 5                | 15                    | 1.5          | 78.5          | 1.448                            |
| A8     | 5                | 16                    | 0.5          | 78.5          | 1.918                            |
| A9     | 5                | 17                    | 1.0          | 77            | 1.962                            |
| A0     | 32.5 Cement Contrast Test |                   |              |               | 1.701                            |

Table 4 Results of the range analysis of the first orthogonal experiment of the cementing material

| Age     | Clinker/% | Desulfurization Ash/% | Mirabilite/% | Factors | Clinker/% | Desulfurization Ash/% | Mirabilite/% |
|---------|-----------|-----------------------|--------------|---------|-----------|-----------------------|--------------|
| 28 day  | 1.766     | 1.811                 | 1.949        | Experimen- | 3         | 15                    | 0.5          |
|         | 2.020     | 1.847                 | 1.901        | tal Level | 4         | 16                    | 1.0          |
|         | 1.776     | 1.903                 | 1.712        |          | 5         | 17                    | 1.5          |
| Range   | 0.254     | 0.092                 | 0.237        | Optimal  | 4         | 17                    | 0.5          |

According to the results of the first orthogonal test and range analysis of the cementing material, the following conclusions can be drawn:

1. In the 9 groups of orthogonal experiments, the optimum formula is group A4 which includes cement clinker 4%, desulfurized ash 15%, mirabilite 1.0% and slag powder 80%. The corresponding compressive strength at 28 d is 2.078MPa, which is 1.22 times of that of cement.

2. According to the range analysis of the orthogonal experiments, the best formula of cementing material with 28 day strength is cement clinker 4%, desulfurization ash 17%, mirabilite 0.5% and slag powder 78.5%. As the activator, cement clinker has the greatest influence on the strength, followed by mirabilite and desulfurized ash.

3. According to the quadratic polynomial regression analysis, we obtain the regression function of the strength at 8d:

$$R_{28} = 0.16759 + 2.9469x_1 - 0.3646x_1^2 - 0.0103279x_2^2 + 0.3567x_2^3 + 0.388887x_2 \cdot x_3 - 0.001x_2 \cdot x_3 - 0.07387x_3 \cdot x_4$$

(R = 0.9999)

In the above formula, $R_{28}$ means the uniaxial compressive strength at 28 d of the filling test block;
X₁, X₂, X₃, X₄ respectively refers to the lime, desulfurization ash, cement clinker and thenardite content.

(4) According to the extreme value analysis of the regression function of the strength at 28 d, the optimum proportion of cementing materials is: cement clinker 4.03%, desulfurization ash 15%, mirabilite 0.5% and slag powder 80.4% and the corresponding 28 day strength of the filling is 2.54 MPa, which is 1.4 times of that of cement.

4.1.2. Results and analysis of the second compound activator formula experiment of the cementing material

In the second experiment, the compound activator formula with a large proportion of ash slags and a small proportion of clinker is used. The results of the experiment and range analysis are shown in Table 5 and Table 6.

| Number | Ratio of Mortar to Sand | Concentration | Clinker | Ash | Mirabilite | Slag Powder | Compressive Strength |
|--------|-------------------------|---------------|---------|-----|------------|-------------|---------------------|
| B1     | 1:6                     | 70            | 3       | 17  | 0          | 80          | 0.221 3.520 3.74    |
| B2     | 1:6                     | 70            | 3       | 18  | 0.5        | 78.5        | 0.236 3.811 4.05    |
| B3     | 1:6                     | 70            | 3       | 19  | 1.0        | 77          | 0.235 3.549 3.78    |
| B4     | 1:6                     | 70            | 4       | 17  | 0.5        | 78.5        | 0.218 3.870 4.09    |
| B5     | 1:6                     | 70            | 4       | 18  | 1.0        | 77          | 0.188 3.443 3.63    |
| B6     | 1:6                     | 70            | 4       | 19  | 0          | 77          | 0.153 3.883 4.04    |
| B7     | 1:6                     | 70            | 5       | 17  | 1.0        | 77          | 0.170 3.461 3.63    |
| B8     | 1:6                     | 70            | 5       | 18  | 0          | 77          | 0.198 2.893 3.09    |
| B9     | 1:6                     | 70            | 5       | 19  | 0.5        | 75.5        | 0.207 3.537 3.74    |
| B0     | 1:6                     | 70            | 32.5 Cement Contrast Test | | | | 0.956 2.275 3.23 |

Table 6 Results of the range analysis of the second orthogonal test of the cementing material

| Age | Level | Clinker | Ash | Mirabilite | Parameters | Clinker | Ash | Mirabilite |
|-----|-------|---------|-----|------------|------------|---------|-----|------------|
| 7 day | Level 1 | 0.231 | 0.203 | 0.205 | Factor Level | 3 | 17 | 0 |
| 28 day | Level 2 | 0.186 | 0.207 | 0.186 | Factor Level | 4 | 18 | 0.5 |
| 7 day+28 day | Level 3 | 0.192 | 0.198 | 0.217 | Factor Level | 5 | 19 | 1.0 |
| | Range | 0.044 | 0.009 | 0.031 | Optimal Formula | 3 | 18 | 1.0 |
| | Relative weight | 4.9 | 1 | 3.4 | | | | |

| Age | Level | Clinker | Ash | Mirabilite | Parameters | Clinker | Ash | Mirabilite |
|-----|-------|---------|-----|------------|------------|---------|-----|------------|
| 7 day | Level 1 | 3.627 | 3.617 | 3.500 | Factor Level | 3 | 17 | 0 |
| 28 day | Level 2 | 3.732 | 3.382 | 3.718 | Factor Level | 4 | 18 | 0.5 |
| 7 day+28 day | Level 3 | 3.297 | 3.656 | 3.437 | Factor Level | 5 | 19 | 1.0 |
| | Range | 0.435 | 0.274 | 0.281 | Optimal Formula | 4 | 19 | 0.5 |
| | Relative weight | 1.6 | 1 | 1 | | | | |
According to the results of the second orthogonal experiments and range analysis of the cementing materials, the following conclusions can be drawn:

1. In 9 groups of orthogonal experiments, the strength at 7 day is lower than that of cement but the strength at 28 day is significantly higher. Therefore, the strength of the cementing material with compound activator is low at early stage and high at later stage. The strength at 28 day is greater than 2.5 MPa, which could meet the requirements of stage subsequent filling for the cementing material strength.

2. In the 9 groups of experiments, the strength of the cementing filling body of the cementing material at 7 day, 28 day and 7 day+28 day is respectively 0.153MPa, 3.883MPa and 4.04MPa. Under the same condition, the strength of cement 32.5 is respectively 0.956MPa, 2.275MPa and 3.230MPa, which is 0.16 times, 1.71 times and 1.25 times of the strength of the cement. The corresponding optimum formula is: cement clinker 4%, desulfurization ash 19%, mirabilite 0% and slag powder 77%.

3. According to the results of the second range analysis of the cementing material, it is found that the cement clinker has the greatest influence on the strength of the filling body at 28 day. Therefore, for the compound excitation of slag powder with the desulfurization ash slag, the alkaline environment produced by clinker has great impact on the improvement of the activity of the slag powder. However, this kind of compound activator has poor effect at the early stage of the stimulation, so the strength is lower than that of the cement at the early stage.

4.1.3. Analysis of the optimal formula of composite activator of the cementing material

Based on the two orthogonal experiments of the cementing material activated by the desulphurized ash slags, the optimal formula of the strength of the filling body at 28 day obtained by different mathematical statistics analysis methods is shown in Table 7. The best formula of the cementing materials is shown as follows: cement clinker 4%, desulfurization ash 19% and mirabilite 0.5%. The average strength at 28 day is 3.894MPa, which is 2.01 times of the cement strength at 28 day, 1.93MPa.

| Analytical method       | Clinker | Ash  | Mirabilite | Slag powder | The strength at 28 day | The ratio with cement strength |
|-------------------------|---------|------|------------|-------------|------------------------|-------------------------------|
| Intuitive analysis      | 4       | 19   | 0          | 77.0        | 3.883                  | 2.01                          |
| Range analysis          | 4       | 19   | 0.5        | 76.5        | —                      | —                             |
| Regression analysis     | 4       | 19   | 0.5        | 76.5        | 3.905                  | 2.02                          |
| Optimal formula         | 4       | 19   | 0.5        | 76.5        | 3.894                  | 2.01                          |
| The strength of cement (average) |        |      |            |             | 1.93                   | —                             |

5. Expansion test results of the cementing material activated by desulphurized ash

Conduct experiments to test the reliability of the optimal formula of the cementing material, and the results are shown in Table 8. It can be seen that the strength of the verification test is up to the strength of the optimum formula. Therefore, the formula of the cementing material developed by the desulfurization ash is reliable.
Table 8 Test results of the optimum formula of the cementing material activated by the desulfurization ash

| Cement sand ratio | Concentration | Optimal formula of the cementing material | Compressive strength | Sinkin g rate |
|-------------------|---------------|------------------------------------------|----------------------|--------------|
|                   |               | Clinker | Ash  | Mirabilite | Slag powder | 7 day | 28 day | 28 day |
| 1:6               | 70            | 4       | 19   | 0.5       | 76.5        | 0.504 | 3.847 | 3.77  |

6. Microstructure analysis of the core filling cementing material

In order to reveal the hydration reaction mechanism and microcosmic results of the core filling cementing material, utilize the SEM and X-ray diffraction (XRD) to reveal the hydration reaction mechanism from the micro perspective and analyze the activating mechanism of desulfurization ash for the activity of slags. In accordance with national standards, prepare the mortar specimen with the optimal ratio and take a small piece when it is conserved to the specified age and then immediately immerse it in alcohol to stop the hydration reaction. Then remove it and process the surface by spraying carbon and then test and analyze it.

6.1. Analysis of hydration products

6.1.1. Analytical methods and sample preparation

In order to study the hydration mechanism and the hydration products of the core filling cementing material, the X-ray diffractometer from Rigaku Corporation is used to analyze the phases. The purpose is to analyze the crystal material formed at different curing periods of the backfill blocks, identify some characteristic substances and judge the hydration reaction of early strength filling cementing material.

The analysis principle is to use X-ray diffraction wave. When it is projected onto the sample, as the relative position of a single atom and its scattering ability are different, there will be a discontinuous diffraction pattern. As the diffraction principle of the crystal lattice to X-ray diffraction is consistent with the Bragg equation, a material diffraction pattern can be characterized by angular position, symmetry and Bragg reflection intensity. Then use the diffraction data to analyze the crystal phase and structure [13].

6.1.2. Results analysis

Fig. 2 shows the X-ray analysis atlas of the cement paste specimen (Cement Clinker 4%、Desulfurization Ash 19%、Mirabilite 0.5% and Slag Powder 76.5%) in 3d curing period. It can be seen from the Fig. that the major main hydrated products are the ettringite, Calcium silicate hydrate gel; besides, the portlandite(Ca(OH)₂), calcite(CaCO₃) and dihydrate gypsum(CaSO₄·2H₂O) can also be observed. Therefore, Ca(OH)₂ produced by the hydrolysis of the clinker in cementing material and that in the desulfurization ash make the slurry environment gradually become alkaline, which is conducive to the dispersion and dissolution of the slag vitreous. SiO₂ and Al₂O₃ in the slag reacts with Ca (OH)₂ to form calcium aluminate and calcium silicate gel, and calcium aluminate then reacts with CaSO₄ and CaSO₄ in the desulfurized ash and sulfate ion in the mirabilite to produce hydrated calcium sulphoaluminate, that is, ettringite crystal. The formation of ettringite consumes calcium aluminate, thus promoting the hydration reaction between SiO₂ in the slag and Al₂O₃ to generate more hydration products. Besides, the strength of specimen is also becoming higher in this process. The main hydration products of cementing materials are hydrated calcium silicate gel and ettringite. Therefore, the main reason for the micro-expansivity of the core cementing material is the ettringite produced in the reaction between CaSO₄, CaSO₄ in the desulphurized ash and active Al₂O₃ and H₂O in the cementing system. The micro-expansivity of the core cementing material, coupled with scientific filling process, such as the multi-point filling and cutting technology, could be used to significantly improve the tight-filling ratio of the mine. This could ensure mine safety production.
6.2. Microscopic analysis of scanning electron microscopy

6.2.1. Analytical methods
This paper makes use of the environmental scanning electron microscope (ESEM) produced by FEI company from America to observe the microstructure and appearance of the products at different ages. Then the electron probe is used to conduct the semi-quantitative energy spectrum analysis of the characteristic matter. The principle of the analysis is to focus the electron beam (5nm) on the surface of the sample, and then carry out point-by-point scanning on the sample surface under the action of the deflection coil, which could stimulate different sub-signals. The detector will collect these signals and convert them into optical signals, which will then be enlarged by the amplifier and transmitted to the kinescope to form a three-dimensional dynamic image\cite{14-15}.

6.2.2. Results analysis
Fig. 3 shows the SEM images of the hydrated products at 7 day and 28 day of the full tailings mortar test of the core filling cementing material. Fig. 4 shows the SEM images of the hydrated products at 28 day, which indicate that the cement hydration products are mainly acicular AFT, and the hydration products of the core cementing filling material are mainly flocculent C-S-H gel. This is consistent with the XRD analysis results.

It can be seen from Fig. 3 that the hydration products of the core cementing filling material are mainly flocculent C-S-H gel, and this is consistent with the XRD analysis results. After 7 day hydration, the amorphous C-S-H gels account for a large number in the slag cementing material sample, and they are network-like and the flakes are connected to each other to form a certain initial network skeleton. However, the network structure is not complete and there are a large number of gaps where there is a certain amount of columnar materials, probably columnar ettringite. The formation of these substances produces a foundation for the compressive strength of the cement. After 28 days of curing, the slag particles are destroyed and a considerable degree of hydration reaction occurs on the surface. As the hydration reaction results in a large amount of hydration products, the materials are very dense and the pores are significantly reduced which enhance the strength of the cementing filling body.
It can be seen from Fig. 4 that the hydration products of cement are mainly acicular AFT. Compared with the hydration products of the cementing material, they have relatively sparse structure with more gaps. There is no floccule network structure. This feature reveals the reasons why the strength of cement at 28 day is lower than that of the core cementing material.

Fig. 4 SEM patterns of mortar specimen of cement

7. Conclusion
(1) To develop the core filling cementing material of full tailings with the water quenching slag powder as the main material and the desulfurization ash and other solid waste as stimulant is feasible and can significantly reduce the cost of filling mining. The raw materials of cementing materials are industrial waste, which are environmental-friendly and have low price. There is no need for secondary processing and this could also reduce the impact of industrial waste like tailings on the environment. The composite excitation formula of desulphurized ash is obtained. When the cement-sand ratio is 1:6 and the slurry concentration is 70%, the strength of the cementing filling body at 28 day could meet the requirements of stage subsequent filling.

(2) The hydration mechanism of the cementing material activated by desulphurized ash is that Ca(OH)₂ produced by the hydrolysis of the clinker in cementing material and that in the desulfurization ash make the slurry environment gradually become alkaline, which is conducive to the dispersion and dissolution of the slag vitreous. The clinker and desulphurized ash, as the activators, promote the activation of the slag activity, thus resulting in hydration reaction and forming the strength.

(3) The hydration products of the cementing material activated by desulphurized ash are mainly flocculent C-S-H gel, and the more the hydration products are, the more compact filling body structure will be, which form a network structure and significantly improve the strength of the filling body.

Acknowledgment:
The Project supported by National High Technology Research and Development Program of China(863Program No.SS2012AA062405).
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