Study on the technology of preparing foamed cement insulation board by using beneficiation tailings

Jiayuan Li*, Xiaolin Yi, Mengqi Kang, Jun Chen*, Bing Deng
Hunan Provincial Key Laboratory of Xiangnan Rare-Precious Metals Compounds and Applications, College of Chemistry & Biology and Environmental Engineering, Xiangnan University, Chenzhou, Hunan province, 423000, China
*Corresponding author’s e-mail: chenjun4174@126.com

Abstract. In order to reuse the tailings of beneficiation, a novel method which mixes and foams cement, micronized tailings powder, calcium stearate, polycarboxylic acid superplasticizer, early strength superplasticizer, HPMC and hydrogen peroxide under certain conditions is proposed in this work. The results showed that the best ratio is as follows: water temperature 50℃, hydrogen peroxide content 6.5%, water to material ratio 0.5, early strength water reducing agent 1.5%, HPMC4/10000, polycarboxylic acid superplasticizer 0.1%, calcium stearate 1.6%, 17% of tailings fine powder. The foamed cement compressive strength is 0.71 MPa, dry density less than 260 kg/m³, and no collapse phenomenon, which guarantees good performance.

1. Introduction
The utilization technology of mineral resources in our country relatively lags behind, and there are many beneficiation tailings left. Most of the solutions to abandoned tailings in our country use wasteland dams and piles, which leads to the occupation of a large number of land resources and the formation of new pollution sources [2]. Once an accident occurs, it will cause a serious burden to the country and enterprises, with extremely serious consequences. Therefore, resource utilization, resource reduction and harmless treatment of discarded tailings have become effective methods to solve the problems of environmental resource tension, and also help to achieve sustainable development of society.

2. Materials, main procedure and main equipment
The composition of tailings, tailings particle size distribution, and main reagents used in this work were listed in Table 1, Table 2, and Table 3, respectively.

The tailings were dried first, and then were grinded and sieved. Table 2 and Table 3 listed the results of grinding the tailings. The experiment uses ordinary tap water and keeps the water temperature at 50℃ to eliminate the influence of water temperature on foaming. According to the ratio, weigh the cement, tailings fine powder, calcium stearate, polycarboxylic acid superplasticizer, and early strength water reducer in sequence. Agent, HPMC, hydrogen peroxide (30% active ingredient). Experimental steps were as follows:
(1) Weigh the cement, tailings micropowder, calcium stearate, polycarboxylate superplasticizer, early strength superplasticizer, HPMC according to the ratio, put them in a stainless steel container, and mix them with a highly efficient dispersion mixer evenly.

(2) Heat the tap water to 50°C, add it to the above described solid mixture according to the ratio of water to material 1:2 and the amount of hydrogen peroxide at 6.5%, and stir through a highly efficient dispersion mixer. The stirring time should not exceed 10s. After stirring, quickly pour the slurry into the mold (100mm×100mm×100mm).

(3) After foaming, the sample is sealed and placed in an accelerated curing box (50°C). One day later, demold it and place it in a standard curing box (constant temperature 20°C, constant humidity 95%), and cure for 3 days. Then cut and shape, and perform data testing in accordance with requirements (JC/T 2200-2013).

Table 1. Chemical composition analysis table of tailings (%)

|         | SiO₂    | Al₂O₃ | Fe₂O₃  | CaO     | MgO     | K₂O    | Na₂O    |
|---------|---------|-------|--------|---------|---------|--------|---------|
| 36.06   | 10.78   | 8.356 | 22.55  | 1.626   | 1.246   | 1.246  | 0.827   |

Table 2. Tailings particle size distribution

| Name      | Sieve hole (mm) | Sieve residue (g) | Sieve mesh (%) |
|-----------|-----------------|-------------------|----------------|
| Tail sand |                 |                   |                |
| 0.38      | 5.0             | 0.5               |                |
| 0.16      | 43.8            | 44                |                |
| 0.106     | 309.0           | 31.0              |                |
| 0.08      | 96.2            | 9.6               |                |
| 0.048     | 493.4           | 49.4              |                |
| 0.045     | 41.2            | 4.1               |                |
| Sieve bottom | 9.5         | 1.0               |                |
| Total     | 998.1           | 100               |                |

Table 3. Main reagents

| Name                        | Specification model | Origin                                      |
|-----------------------------|---------------------|---------------------------------------------|
| Hydrogen peroxide           | Industrial pure (30%) | Sinopharm Chemical Reagent Co., Ltd.       |
| Calcium stearate            | Analytically pure   | Dongguan Kangjin New Material Technology Co., Ltd. |
| Polycarboxylic acid superplasticizer | QS-8020       | Shanghai Qinhe Chemical Co., Ltd.            |
| Early strength water reducing agent | HB             | Hongfu Building Additives                  |
| Hydroxypropyl Methylecellulose (HPMC) | 00              | Kehao Hydroxypropyl Methyl Cellulose HPMC HPMC |

3. Results and discussion

3.1. The influence of the number of early strength water reducing agents on the performance of foamed cement insulation board
The number of early-strength water-reducing agents will affect the foaming result. It is studied when the early-strength water-reducing agent accounts for 0.75%, 1%, 1.25%, 1.5%, 2%, 2.25%, 2.5% of foamed cement according to the experimental procedures, and the experimental results are shown in Figure 1.

It can be seen from Figure 1 that when the content of the early-strength water-reducing agent is too low (0.75%), the water-reducing effect is not obvious, resulting in poor fluidity of the slurry, the net slurry cannot be poured into a shape, and the dry density cannot be measured. As the content increases to a certain amount (1.75%), it has almost no effect on the dry density. The hydration rate of cement is relatively slow, so a certain early strength is required in the initial stage of foaming to prevent collapse. In this experiment, the early-strength water-reducing agent was added to improve the early strength. When the early-strength water-reducing agent exceeds 1.5%, the surface of the sample cracks and collapses, which seriously affects the compressive strength of the sample. Therefore, under comprehensive consideration, it is most appropriate to set the orthogonal level of early strength water-reducing agents at 1%, 1.25%, and 1.5%.

![Figure 1. Effect of early strength water reducer incorporation on dry density](image1)

![Figure 2. Effect of HPMC incorporation on dry density](image2)

3.2. The influence of HPMC content on the performance of foamed cement insulation board

HPMC (Hydroxypropyl Methyl Cellulose) as a thickening agent can change the consistency of the slurry. A suitable slurry consistency can allow bubbles to remain in the slurry stably and uniformly, and can form pores of an appropriate size; HPMC blending can increase the viscosity of the slurry, make the surface tension larger, and make the pore distribution more even [5]. Therefore, on the basis of the mixing ratio, the influence of different HPMC content on the foaming result was studied to find the suitable proportion. The result is shown in Figure 2.

Based on the basic mixing ratio, an experiment of HPMC incorporation was done to determine the level of HPMC incorporation in the orthogonal experiment). It can be seen from Figure 2 that when the HPMC content is lower than 3/10000 and higher than 6/10000, it cannot be molded. This is because when HPMC is blended, the viscosity of the slurry increases, and the cohesive force that needs to be overcome during foaming will also increase, so when the content of HPMC is too small or too large, it will have an adverse effect on the foaming of the slurry. When the content of HPMC is between 3/10000 and 5/10000, the absolute dry density of foamed cement is very close. Therefore, the level of the orthogonal experiment of HPMC content should be set to 3/10000, 4/10000, 5/10000.

3.3. The influence of polycarboxylic acid superplasticizer content on the performance of foamed cement insulation board

This experiment uses the polycarboxylic acid superplasticizer in the polycarboxylic acid series of water reducer. Compared with the traditional water reducer, this series of water reducers have a lower dosage than the traditional water reducer. Better dispersion effect [6]. In order to determine the reasonable level of polycarboxylic acid superplasticizer, the experiment used a single factor experiment method to discuss the dosage. The results are shown in Figure 3.
In this experiment, on the basis of the basic mixing ratio, the experiment of different polycarboxylic acid superplasticizer dosage was done. From Table 6 and Figure 3, it can be seen that when the polycarboxylic acid superplasticizer dosage is too low, the water reducing effect is not exerted. As a result, the water-to-material ratio is still too large, and the pure slurry cannot be poured into a shape. When the content of polycarboxylic acid superplasticizer is increased to 0.1%, the foaming effect is the best, and the dry density is the lowest. As the content of polycarboxylic acid superplasticizer increases, it will gradually negatively affect the results of this experiment. The dry density gradually increases until the inflection point is formed when the dosage is 0.2%. After that, the most extreme case of the experiment occurs, which is the pure pulp cannot be foamed. However, considering the rationality of the orthogonal experiment design, three level should be chosen. Therefore, this experiment selects 0.1%, 0.15%, and 0.2% as the polycarboxylic acid superplasticizer dosage level.

![Figure 3. Effect of different polycarboxylate superplasticizer on the dry density](image)

![Figure 4. Effect of different calcium stearate incorporation on the results](image)

3.4. The influence of calcium stearate content on the performance of foamed cement insulation board

Under the condition of other factors being fixed, adjust the dosage of the foam stabilizer calcium stearate to determine the effect of different dosages on the foaming result. The specific results are shown in Figure 4.

In this experiment, the calcium stearate dosage experiment was done on the basis of the basic mix ratio. As a foam stabilizer, calcium stearate will be adsorbed on the surface of cement particles after being incorporated, and will exhibit hydrophobic characteristics. Furthermore, it will be adsorbed on the gas-liquid interface of the bubbles, and finally make the bubbles stable and not easy to collapse [7]. Therefore, the incorporation of calcium stearate as a foam stabilizer is very necessary. It can be clearly seen from Figure 4 that with the increase of calcium stearate content, the foaming effect gradually becomes better until the content reaches 1.2%, the first inflection point appears. From 1.2% to 1.6%, the dry density tends to a stable state. It can be seen that the calcium stearate content has little effect on dry density, and more of it is the late collapse of the sample. When it reaches the limit of 1.8%, too much calcium stearate makes the sample unable to form. In order to determine the optimal dosage of calcium stearate more accurately, we choose the dosage of foam stabilizer level as 1.2%, 1.4%, 1.6% for the orthogonal experiment to make full use of the uniformity attribute of the orthogonal experiment.

3.5. Factor level distribution table

On the basis of the previous single factor experiment, select the appropriate level, and see Table 4 for the specific data of the factor level allocation table.

| Experiment number | A early strength reduction water dose/% | BHPMC amount | C polycarboxylic acid high water reducing dosage/% | D amount of calcium stearate/% |
|-------------------|----------------------------------------|--------------|-----------------------------------------------|-------------------------------|
| 1                 | 1                                      | 3/10000      | 0.1                                           | 1.2                           |
| 2                 | 1.25                                   | 4/10000      | 0.15                                          | 1.4                           |
It can be seen from Table 5 that the factor A in the first column is level 1, that is, when the content of the early strength water reducing agent is 1%, the three experiments correspond to different compressive strengths. The algebraic sum of the No.1, No.2, No.3 compressive strength tests results is denoted as AI, which is called the level 1 integrated value of the A factor. The average of the three trials is mean 1, which is recorded as K1: mean 1 = AI/3 = (0.64+0.63+0.37)/3=0.547. Similarly, mean 2 (K2) = AII/3 = (0.68+0.34+0.34) /3 = 0.453, mean 3 (K3) = AIII /3 = (0.52 +1.22+0.39) /3 = 0.710. Obviously, AI is the 1 level of factor A that appears three times, while the levels 1, 2, and 3 of the three factors of B, C, and D appear once respectively. Therefore, AI reflects the influence of the A1 level three times and the B, C, and D factors. The 1, 2, and 3 levels of each have one impact, and the same problems AII and AIII are also reflected. By comparing the sizes of AI, AII and AIII, it can be considered that the three factors of B, C, and D have basically the same impact on AI, AII and AIII. Therefore, the difference between AI, AII and AIII can be regarded as caused by the difference between the three different levels of A. This is the advantage of the orthogonal table: uniform comparability. In the same way, the average value of each level of the three factors of B, C, and D can be known. The results are shown in Table 11. Calculate the difference between the maximum value and the minimum value of the average value and record it as range, marked as R. The range results of the four factors A, B, C, and D are listed in Table 6. The impact of changes in the levels of the factors listed on the experimental results can be reflected by the range R value.

| Experiment number | A early strength reduction dose/% | BHPMC amount | C polycarboxylic acid high water reducing dosage/% | D amount of calcium stearate/% | Compressive strength /MPa |
|-------------------|----------------------------------|---------------|---------------------------------|---------------------------------|-------------------------|
| 1                 | 1                                | 3/10000       | 0.1                             | 1.2                             | 0.64                    |
| 2                 | 1                                | 4/10000       | 0.15                            | 1.4                             | 0.63                    |
| 3                 | 1                                | 5/10000       | 0.2                             | 1.6                             | 0.37                    |
| 4                 | 1.25                             | 3/10000       | 0.15                            | 1.6                             | 0.68                    |
| 5                 | 1.25                             | 4/10000       | 0.2                             | 1.2                             | 0.34                    |
| 6                 | 1.25                             | 5/10000       | 0.1                             | 1.4                             | 0.34                    |
| 7                 | 1.5                              | 3/10000       | 0.2                             | 1.4                             | 0.52                    |
| 8                 | 1.5                              | 4/10000       | 0.1                             | 1.6                             | 1.22                    |
| 9                 | 1.5                              | 5/10000       | 0.15                            | 1.2                             | 0.39                    |

| Table 6. Range Analysis Table |
|-------------------------------|
| Factor | K1     | K2     | K3     | R range |
|--------|--------|--------|--------|---------|
| A      | 0.547  | 0.453  | 0.710  | 0.257   |
| B      | 0.613  | 0.730  | 0.367  | 0.363   |
| C      | 0.733  | 0.567  | 0.410  | 0.323   |
| D      | 0.457  | 0.497  | 0.757  | 0.300   |

3.6. Experimental results table of tailings fine powder content gradient
Through the early single factor experiment and orthogonal experiment, it is determined that the basic ratio is 6.5% hydrogen peroxide content, 0.5% water to material ratio, 1.5% early strength water reducer, HPMC 4/10000, and 0.1% polycarboxylic acid superplasticizer. Calcium stearate 1.6%, this
ratio is used for the tailings fine powder dosage gradient experiment. The results of this experiment are the average of the three experimental results. The results are shown in Table 7.

| Cement | Tailings powder | Compressive strength | Dry density |
|--------|----------------|----------------------|-------------|
| 400g   | 0g             | Mean 1=0.85MPa       | Mean 1=223kg/m³ |
| 332g   | 68g(17%)       | Mean 2=0.71MPa       | Mean 2=255kg/m³ |
| 320g   | 80g(20%)       | Mean 3=0.64MPa       | Mean 3=279kg/m³ |
| 304g   | 96g(24%)       | Mean 4=0.36MPa       | Mean 4=304kg/m³ |
| 292g   | 108g(27%)      | Mean 5=0.33MPa       | Mean 5=338kg/m³ |

3.7. Primary and secondary analysis of factors affecting foamed cement

By comparing the R value of each column, it can be seen from the range analysis table in Table 6 that in this experiment, the primary and secondary relationship of the influence of each factor on the compressive strength is: B>C>D>A. The largest absolute value in the column of factor B suggested that HPMC is the main factor among the factors and has the most significant impact on the experimental results in the experiment. The amount of HPMC added should be adjusted to increase the compressive strength. Relatively speaking, the influence of C and D on the experimental results is relatively small, and the influence of factor A on the experimental results is the least significant.

3.8. Analysis of the influence of tailings fine powder content on the results

The dosages of fixed hydrogen peroxide, early strength water reducing agent, calcium stearate, HPMC, and polycarboxylic acid superplasticizer are 6.5%, 1.5%, 1.6%, 4/10000, 0.1%, respectively for the influence of foamed cement's compressive strength and dry density, make the relationship between compressive strength and fine tailings powder according to Table 7, and the relationship between dry apparent density and fine tailings powder, see Figure 5 and Figure 6.

The strength of cement depends on 3CaO·SiO₂ and β-2CaO·SiO₂. When the content of both is high, the compressive strength is high; when the content is small, the compressive strength is low [8]. After mixing tailings fine powder, the activity is not enough, and there are no compounds formed when CaO and SiO₂ react, so the strength decreases and the dry density rises is an inevitable trend. When the tailings fine powder content is small, although the strength of the foamed cement insulation board decreases, it still meets the requirements and can meet the performance requirements of the lightweight insulation board. When the dosage is increased to 20%, although the compressive strength decreases less, the dry density is already close to 300kg/m³. When the dosage is increased to 24%, the compressive strength drops rapidly, as low as 0.36MPa, and the density rises to 304kg/m³. When the dosage is increased to 27%, the compressive strength is only 0.33MPa, and the density is increased to 338kg/m³. After comprehensive consideration, the optimal dosage of tailings fine powder in this experiment is 17%.
3.9. The best ratio of foamed cement preparation

Based on the orthogonal design experiment and the tailings fine powder content experiment, it can be found from Table 6 that for factor A, the compressive strength of the early strength water reducing agent at level 3 (1.5%) is greater than level 1 (1%) is greater than 2 Level (1.25%); for factor B, the compressive strength of HPMC's level 2 (4/10000) is greater than level 1 (3/10000) and greater than level 3 (5/10000); for factor C, polycarboxylic acid is highly water-reducing, the compressive strength of level 1 (0.1%) of calcium stearate is greater than level 2 (0.15%) is greater than level 3 (0.2%); for factor D, the compressive strength of calcium stearate level 3 (1.6%) is greater than level 2 (1.4%) is greater than 1 level (1.2%); the orthogonal experiment is to select the maximum value of the comprehensive level of each factor, and its corresponding level is the experimental plan, namely A3B2C1D3. This scheme can be found in the 9 experiments in Table 10. At this time, the measured compressive strength is the largest, which is consistent with the analysis. Considering the influence of the fine tailings content on the experimental results, the optimal ratio can be determined as water temperature 50℃, hydrogen peroxide content 6.5%, water-to-mater ial ratio 0.5, early strength water reducing agent 1.5%, HPMC 4/10000, polycarboxylic acid superplasticizer 0.1%, calcium stearate 1.6%, tailing powder Add 17%.

4. Conclusion

Following conclusions are obtained:
(1) The primary and secondary factors affecting the compressive strength of foamed cement are HPMC amount>polycarboxylic acid superplasticizer>calcium stearate>early strength water reducer.
(2) The blending of Shenghong tailings fine powder after grinding into the system has an important impact on the compressive strength of foamed cement. If the amount is too much, the strength of the system will be low, which cannot meet the production requirements.
(3) The prepared sample has high quality when the water temperature is 50℃, the amount of hydrogen peroxide is 6.5%, the water to material ratio is 0.5, the early strength water reducing agent is 1.5%, HPMC is 4/10000, polycarboxylic acid superplasticizer is 0.1%, calcium stearate is 1.6% and the tailings micropowder is added at 17%. At this time, the dry density is less than 260kg/m³ and the compressive strength is 0.71MPa, which meets the requirements of JC/T 2200-2013.

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