Screening Tests of Copper Mine Tailings Flocculation with Polyacrylamide

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Abstract. The flocculation method had a good effect in treating copper tailings, and polyacrylamide (PAM) was commonly used as an effective flocculant. In this paper, a complete set of screening tests was established to effectively evaluate the flocculation performance of PAM used for copper tailings treatment. Firstly, the traditional jar tests were applied and the anionic PAM performed better for flocculation treatment of copper tailings than cationic and nonionic PAM. However, this method could not be further applied to screen anionic polyacrylamide with different molecular weights and hydrolysis degrees because of unobvious difference in settling curves of them. Therefore, the dynamic test rig was self-designed for further screening. Furthermore, the orthogonal design scheme of five factors and three levels was carried out for experimental design and results evaluation. The optimal conditions selected from dynamic rig tests were applied to the plant trial, and the treatment effect can meet the requirements after fine-tuning based on the plant operating conditions.

1. Introduction
Copper tailings are fine silty gravel solid wastes after copper ore is crushed and separated from copper concentrate[1]. For every ton of copper produced, 128 tons of copper tailings are produced[2]. According to the United States Geological Survey, in 2011, more than 2 billion tons of copper tailings were produced in the world. As one of the major producers of copper tailings, China's cumulative accumulation of copper tailings has exceeded 3 billion tons by 2014, but only about 82% of them have been recycled. At present, the tailings treatment is mostly to build tailings ponds to centralize the tailings stockpiling, which has caused great harm to the environment. Metal mine tailings are characterized by extremely fine particles, large quantities, toxicity and low concentration[7]. In addition to the harmful components and dam break caused by negligent management, tailings particles are so fine that they’re easy to blow to the surrounding areas with the wind, thereby occupying land and polluting the environment seriously affect human life.

Therefore, a large amount of work[7-8] has been implemented on finding integrated management techniques. One of these techniques is tailings flocculation[9-13]. Flocculation is used to describe the action of polymeric materials which form bridges between individual particles. Bridging occurs when segments of a polymer chain adsorb different particles thus help particles aggregate. And it is a technique in which discrete, colloidal-sized particles were agglomerated by an appropriate reagent and, as a result, settle out of suspension. Polyacrylamide (PAM) is generally used as an effective flocculant in tailings treatment, which has been widely used as process aids in the copper tailings industries to enhance solid-solid or solid-liquid separation. Their performance strongly depends on their physicochemical properties such as integrated functional groups, charge density and molecular weight (MW). Thickener performance parameters, such as underflow densities and overflow clarities, and the
The effect of feed can vary greatly for different thickener feeds. Even for the same types of application, thickener performance will vary at different sites due to factors such as particle size, the presence of clays and organic materials, water chemistry, and the effects of upstream processes such as grind size and the use of reagents. Other parameters, such as the optimal feedwell, slurry density for efficient flocculation, and the type and dosage of flocculant will also vary from application to application. These parameters will have an effect on the project’s capital and operational costs. So it is particularly important to establish a complete screening test method to screen the optimum PAM and dosage.

This paper proposes to apply traditional jar test to screen roughly because it only can be conducted with a relatively small amount of indicative feed sample, which do not account for the effects present in full-scale thickening. Furthermore, self-designed dynamic test rig is applied to screen the samples with similar performance reflected from the jar test, which can simulate the operating conditions of thickener, including feed pump, flocculant addition pump, underflow pump, and rotating rake. Eighteen groups of experiments with 3 anionic PAM samples are designed by orthogonal design[14] and tested by dynamic rig, and the optimal combination is applied to plant thickener to verify the reliability of the results.

2. Experimental

2.1. Materials and reagents
Copper tailings sample was collected from a copper mine plant in China. Anionic Polyacrylamide (APAM): APAM-A of 20% ionic degree with 12,000,000 molecular weight; APAM-B of 26% ionic degree with 12,000,000 molecular weight; APAM-C of 26% ionic degree with 18,000,000 molecular weight; APAM-D of 30% ionic degree with 18,000,000 molecular weight.

2.2. Instrumentation
Laser diffraction SEISHIN LMS-30 was used to test the particle size distribution. The underflow (U/F) and overflow (O/F) were dried at 120°C for 3 hours to obtain their solid contents.

Dynamic Test Rig: Self-designed, the flowchart of the rig was shown in Figure 1. The dynamic rig included a flocculation device and three peristaltic pumps. The flocculation device included a feed well, a flocculation tank and a rake device. Flocculants and tailings samples were respectively pumped through the mixing tee into the flocculation tank. The rake was used to drive the flocculated underflow and squeeze out the interpore water of the tailings. After the flocs settled completely, the overflow was collected from the overflow vent to test its total suspended solid (TSS), and the underflow was pumped out from the bottom of the flocculation tank through the underflow pump and collected to test its solid content.

Figure 1. Dynamic Test Rig Flowchart
2.3. Sample preparation and flocculation methods

2.3.1. Preparation of APAM solution. Powder APAM sample (0.500g) was weighed accurately, and was dissolved in 100mL of water respectively. The solution was stirred for about 1 hour. This solution was regarded as mother flocculant solution at concentration of 0.5%. The mother solution was then diluted to 0.05% with water for the flocculation tests.

2.3.2. Flocculation methods.
Jar test: The tailings sample was shaked well and was weighed 1000g in a 2L beaker. The sample was stirred with a speed of 350rpm for 3 minutes, and then was adjusted down to 160rpm with adding 0.05% flocculant solution into the beaker. Begin timing when the flocculant was injected. The duration of flocculant injection and mixing was 1min with additional 1min post-flocculate shear. Shut down the agitator and the flocculated tailings was transferred to a 1-L volumetric cylinder.

Dynamic rig test: The tailings samples were diluted to the required feed concentration with process water and stirred continuously with a mixer. The pipelines of PAM dosing pump, tailings feed pump, underflow pump and overflow vent were well connected. Process water was added into the flocculation tank to reach the overflow vent height. The PAM dosing pump was then turned on, when the PAM solution got into the tee, the tailings feed pump was turned on and the rake was started at the same time. The flow rate of PAM and tailings were set according to the requirements of this test, and the rake rotation rate was set to 1rpm. At this time, the flocculation behavior of the flocculated tailings flowing out from the feed well should be observed carefully, and minor adjustment was available for the flow rate of the dosing pump to make the flocculation performance better. The underflow pump was then turned on, until the flocculation effect improved it was turned off. And start timing, this time was considered to be feed time, during this period, overflow was collected at the vent to test its TSS, and the feed speed was also measured with a volumetric cylinder. When the feed time reached the set value, the tailings feed pump and the PAM dosing pump were all turned off, the settling curve was recorded from then. When the settlement was completed (30 minutes), the underflow pump was turned on to collect the underflow and get its solid content.

3. Results and discussion

3.1. Properties of tailings
The thickener feed of copper tailings was analyzed as mentioned in Section 2.2 and the physical properties were shown in Table 1. The average particle size was 21µm, and more than 80% of the particles were smaller than 63µm, which was too fine to naturally settle without any treatment.

| Solids concentration (wt.%) | Solids concentration (g L⁻¹) | Slurry density (t m⁻³) | Average particle size (µm) | Particle size <25µm (%) | Particle size >63µm (%) |
|-----------------------------|-------------------------------|------------------------|---------------------------|------------------------|------------------------|
| 27.4                        | 360.04                        | 1.314                  | 21                        | 67.6                   | 11.4                   |

3.2. Flocculation

3.2.1. Jar test
A series of tailings flocculation tests with cationic polyacrylamide (CPAM), anionic polyacrylamide (APAM) and nonionic polyacrylamide (NPAM) were conducted by traditional jar tests to screen the optimum PAM. The results showed that APAM performed better than the others. Further studies on the four excellent APAM samples, APAM-A, APAM-B, APAM-C and APAM-D, were developed to screen the optimum one among them.
From the settling curves of the four samples (Figure 2), APAM-A performed a little worse than the other three, but the settlement of tailings flocculated with APAM-B, APAM-C and APAM-D were hardly to distinguish their performance. In view of this, a more effective screening test was essential for further study.

Figure 2. Settling Curve for Different Samples
(a) APAM-A, (b) APAM-B, (c) APAM-C, (d) APAM-D,
(e) 4 APAMs at dosage of 14g/t, (f) 4 APAMs at dosage of 18g/t.
3.2.2. Dynamic Rig test

The self-designed dynamic rig was applied to screen the three APAM samples that were not distinguished by jar tests. Orthogonal test of five factors and three levels was designed for the screening task. Underflow density, overflow TSS and settling rate were specified as evaluation indicators to determine the optimum flocculant and dosage, as well as feed speed, feed concentration and feed time.

Table 2. Factors and levels Table

| Level | Factor          |          |          |          |          |
|-------|-----------------|----------|----------|----------|----------|
|       | A (PAM type)    | B (PAM dosage, g/ton⁻¹) | C (Feed speed, mL/ min⁻¹) | D (Feed concentration, %) | E (Feed time, min) |
| 1     | PAM A           | 14       | 450      | 11       | 15       |
| 2     | PAM B           | 18       | 475      | 12.5     | 20       |
| 3     | PAM C           | 22       | 500      | 14       | 25       |

According to the orthogonal test results shown in Table 4, the influence degrees of the five factors on the three evaluation indicators were basically the same. The most influential factors was PAM type for all these three, followed by PAM dosage and feed speed. There were slight differences between feed concentration and feed time, for underflow density, the effect of feed time was slightly greater than feed concentration, while for overflow clarity and settling rate, feed concentration had a slightly greater influence. This might be due to the fact that when the feed concentration was higher, the amount of particles requiring flocculation per unit time was relatively larger, so the overflow would be more turbid and the settling rate would be slower, but the effect on the solid content of underflow was not so obvious.

In terms of optimization level, it was worth pointing out that for underflow density and settling rate, the greater the better, while for overflow TSS, the smaller the clearer. With regard to factor A (optimum PAM), APAM-C performed the best for all the three indicators. For factor B (PAM dosage), there was no obvious difference between 18g/ton and 22g/ton because of their very close k values, the dosage of 18g/ton to 22 g/ton would thus be selected and appropriate adjustments in this range would be made during plant trial. For factor C (feed speed), there was also no obvious difference between 450mL/min and 475mL/min, and further validation trials should be conducted. The situation of factor D and E was similar to that of factor C.

The three optimal combinations were selected as A₂B₂C₁D₁E₂, A₂B₂C₂D₂E₂ and A₂B₂C₂D₂E₃ according to the three indicators. They were not included in the original orthogonal design, so the three combinations were used to conduct experiments, and the results were shown in Table 5.
Table 3. Design of experiments and responses

| No. | PAM type | Dosage (g·ton⁻¹) | Feed speed (mL·min⁻¹) | Feed concentration (%) | Feed time (min) | Response |
|-----|----------|------------------|-----------------------|------------------------|----------------|----------|
|     |          |                  |                       |                        |                | U/F density (%) | O/F TSS (mg·L⁻¹) | Settling rate (mm·min⁻¹) |
| 1   | HPA M-B  | 1 (14)           | 1 (450)               | 1 (11)                 | 1 (15)         | 55.8     | 68       | 5.36 |
| 2   |          | 1 (18)           | 2 (475)               | 2 (12.5)               | 2 (20)         | 61.2     | 43       | 10.45 |
| 3   |          | 1 (22)           | 3 (500)               | 3 (14)                 | 3 (25)         | 59.1     | 61       | 7.89 |
| 4   | HPA M-C  | 1 (1)            | 1 (2)                 | 2 (2)                  | 2 (2)          | 62.1     | 45       | 12.98 |
| 5   |          | 2 (2)            | 2 (2)                 | 3 (3)                  |                | 65.8     | 38       | 15.32 |
| 6   |          | 3 (3)            | 3 (3)                 | 1 (1)                  |                | 61.7     | 40       | 14.16 |
| 7   | HPA M-D  | 1 (2)            | 1 (2)                 | 1 (3)                  |                | 56.3     | 61       | 6.78 |
| 8   |          | 3 (3)            | 2 (3)                 | 2 (3)                  |                | 56.5     | 57       | 7.25 |
| 9   |          | 3 (3)            | 3 (1)                 | 3 (1)                  |                | 60.2     | 52       | 7.87 |
| 10  |          | 1 (1)            | 2 (3)                 | 3 (3)                  |                | 56.2     | 79       | 4.87 |
| 11  |          | 1 (2)            | 1 (2)                 | 1 (3)                  |                | 60.4     | 51       | 12.36 |
| 12  |          | 1 (3)            | 2 (2)                 | 2 (2)                  |                | 58.3     | 50       | 12.73 |
| 13  |          | 1 (2)            | 2 (3)                 | 3 (3)                  |                | 62.9     | 48       | 13.26 |
| 14  |          | 2 (3)            | 2 (3)                 | 1 (2)                  |                | 64.1     | 44       | 13.26 |
| 15  |          | 2 (3)            | 3 (1)                 | 2 (3)                  |                | 66.3     | 36       | 15.77 |
| 16  |          | 3 (1)            | 1 (3)                 | 2 (3)                  |                | 55.1     | 75       | 5.36 |
| 17  |          | 3 (1)            | 3 (2)                 | 3 (3)                  |                | 61.4     | 52       | 9.98 |
| 18  |          | 3 (3)            | 2 (2)                 | 1 (1)                  |                | 62.9     | 49       | 10.86 |

Table 4. Orthogonal results analysis

| K value | U/F density (%) | O/F TSS (mg·L⁻¹) | Settling rate (mm·min⁻¹) |
|---------|-----------------|------------------|--------------------------|
| A       | B               | C                | D                         | E                 | A       | B       | C       | D       | E       | A       | B       | C       | D       | E       |
| K: = Sum of 5 values for Level "1" | 351.0 | 348.4 | 366.2 | 361.2 | 356.6 | 352 | 376 | 304 | 313 | 315 | 53.66 | 48.61 | 64.32 | 62.78 | 62.74 |
| K: = Sum of 5 values for Level "2" | 382.9 | 369.4 | 367.4 | 359.5 | 366.7 | 351 | 285 | 289 | 306 | 312 | 84.75 | 68.62 | 69.4 | 64.54 | 60.29 |
| K: = Sum of 5 values for Level "3" | 352.4 | 368.5 | 352.7 | 365.6 | 363.0 | 346 | 288 | 356 | 330 | 322 | 48.1 | 69.28 | 52.79 | 59.19 | 63.48 |
| k=K/3 | 117.0 | 116.1 | 122.1 | 120.4 | 118.9 | 117 | 125 | 101 | 104 | 105 | 17.89 | 16.2 | 21.44 | 20.93 | 20.91 |
| k=K/3 | 127.6 | 123.1 | 122.5 | 119.8 | 122.2 | 84 | 95 | 96 | 102 | 104 | 28.25 | 22.87 | 23.13 | 21.51 | 20.10 |
| k=K/3 | 117.5 | 122.8 | 117.6 | 121.9 | 121.0 | 115 | 96 | 119 | 110 | 107 | 16.03 | 23.09 | 17.60 | 19.73 | 21.16 |
| Range | 10.6 | 7.0 | 4.9 | 2.1 | 3.3 | 33 | 30 | 22 | 8 | 3 | 10.36 | 6.89 | 5.53 | 1.78 | 1.86 |
| Optimization Level | A₁ | B₁ | C₁ | D₁ | E₁ | A₂ | B₂ | C₂ | D₂ | E₂ | A₃ | B₃ | C₃ | D₃ | E₃ |
| Ranking of impact degree | A>B>C>D=E | A>B>C=E>D | A>B>C=D=E | A>B=C>D=E | A>B=C>D=E |
All of the three combinations performed very well, and there was no distinct difference among them. Considering the economic factors, the combination of A2B2C2D2E2 was chosen for the plant trial, i.e. APAM-C at dosage of 18g/ton, 475mL/min feed speed, 20min feed time, and 12.5% feed concentration.

Table 5. Further validation tests results

| No. | PAM type | Dosage (g/ton⁻¹) | Feed speed (mL·min⁻¹) | Feed concentration (%) | Feed time (min) | Response |
|-----|----------|-----------------|-----------------------|------------------------|----------------|----------|
| 1   | 2 (HPAM-B) | 2 (18) | 1 (450) | 3 (14) | 2 (20) | 65.4 | 38 | 15.71 |
| 2   | 2       | 2 | 2 (475) | 2 (12.5) | 2 | 66.2 | 36 | 15.97 |
| 3   | 2       | 3 (22) | 2 | 2 | 3 (25) | 66.5 | 36 | 16.12 |

3.2.3. Plant trial

The plant trial conditions were determined by dynamic rig test results as follows, 18g/ton APAM-C was selected as flocculant, the tailings feed speed was set to 15000 m³/h in thickener on the basis of 475 mL/min feed speed and 20 minutes feed time, and feed concentration was 12.5%. Continuous trial was carried out under the above conditions for 7 days and the results were recorded every hour, the average value of each index was listed in Table 6.

Table 6. Plant trial results

| No. | Condition | Dosage (g/ton⁻¹) | Clear water depth (m) | Clear water TSS (mg·L⁻¹) | Bed level (%) | Bed pressure (kPa) | Underflow density (%Solid) |
|-----|-----------|------------------|-----------------------|--------------------------|---------------|--------------------|---------------------------|
| 1   | Requirement | 18 | >2.2 | <60, the lower the better | 35-45 | 112-132 | >63 |
| 2   | Combination A₂B₂C₂D₂E₂ | 18 | 2.6 | 50 | 41.2 | 127 | 61.8 |
| 3   | Adjusting dosage and dosing points in plant | 20 | 2.8 | 40 | 40.6 | 123 | 63.4 |

The trial results of the combination selected from dynamic rig tests were not up to all the requirements. So the dosage of PAM was increased to 20g/ton, and the dosage points were adjusted according to the plant conditions to make sure that the tailings and flocculants mixed better. Then the plant trial was carried out for another 7 days continuously. The average values were also listed in Table 6, which indicated that the operating conditions based on those screened by dynamic rig tests were suitable for plant operation with just fine-tuning.

4. Conclusions

Traditional jar tests were applied to roughly evaluate the performance of cationic, anionic and nonionic PAM on treatment of the copper tailings. The results showed that anionic PAM was more suitable for flocculation treatment of this copper tailings than cationic and nonionic PAM. And then the selected anionic PAM samples were further screened, but it was failed to get satisfactory results because the jar tests was incapable of distinguishing the samples with similar flocculating ability.

The APAM samples, which were failed to be distinguished by jar tests, were further screened through dynamic rig tests. The experimental design and result evaluation were carried out by the orthogonal design scheme of five factors and three levels. Flocculant and dosage, as well as feed speed, feed concentration and feed time were selected as factors, and underflow density, overflow TSS and settling rate were specified as evaluation indicators. The results were as follows: 1) APAM-C
performed the best for all the three indicators, 2) the dosage of 18g/ton to 22 g/ton was available for the flocculation, 3) the feed speed of 450mL/min to 475mL/min was suitable for the trial, 4) the optimal feed concentration was 12.5%, and 5) the optimal feed time was 20min.

The corresponding operating conditions, determined by the screened conditions based on dynamic rig tests, were applied for the plant trial to test the performance. Once the dosage of PAM was increased from 18g/ton to 20g/ton, the underflow density can be promoted to a satisfied value of 63.4%.

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