Research on Application of Centrifugal Circulating Fluidized Bed in Tin Mud Ore Separation

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Abstract. Tin mud ore separation is always the key to improve the utilization of tin resources. In view of the tin mud ore in Gejiu Yunnan province, the circulating fluidized bed is used for preconcentration. And reasonable separation parameters were determined based on the ore properties. When the tin grade in the feed is 0.3%, we can obtain the rough concentrate whose tin grade is 0.492% and recovery is 67.35%, and 60% of ore as tailings can be discarded. Then desulfurization and flotation was used, and YP-6 as collector, P86 as auxiliary collector and oxalic acid as regulator, a tin concentrate with grade of 4.38% and recovery of 77.18% was obtained by closed circuit test. The tin recovery of original mud ore was 51.02%, and the separation effect was improved.

1. Introduction
At present, tin ore mainly depends on gravity separation equipment such as shaker and belt chute for recovery in China. However, the tin mud ore in Gejiu Yunnan province is difficult to separate because of its fine crystallization strength, less cassiterite, more mud and large viscosity [1]. The circulating fluidized bed combines centrifugal separation and cyclone separation methods. The pressure of pulp can be converted into kinetic energy of high-speed rotation, thus establishing a centrifugal force field and several top-down fluidized beds. Mineral particles with different densities in pulp are separated into fluidized bed under centrifugal force. Heavy mineral particles overcome the resistance of fluidized water and are discharged from fluidized holes and drill holes at the bottom of the bed, then enter the outer large volume storage chamber and are continuously discharged from the bottom to concentrate after sedimentation and concentration. Light mineral particles cannot overcome the resistance of fluidized water. Under the action of circulating thrust of pulp, they pass through the separation fluidized beds from top to bottom in order. Then enter in central pipeline from the bottom of the separation cone and upward countercurrent discharge into tailings [2]. So circulating fluidized bed is of great significance to the separation of heavy minerals, especially like tin mud.

2. Materials
The tin mud ore for our test is obtained from a concentrator after shaking bed separation in Gejiu Yunnan province. The material contains about 0.3% tin, 3% sulfur and 8% iron. At present, it is mainly depends
on shaking bed gravity separation for comprehensive recovery in the concentrator, and the recovery effect is general. Particle size analysis of the test material is illustrated in Table 1.

From the Table 1, we can see that the cumulative yield is 70.48% which grain size between 0.019mm and 0.074mm, and the distribution rate of tin metal is 77.36%. The yield of -0.01mm grain size is 7.47%, and the distribution rate of tin metal is 4.18%. The grain size of this material is relatively concentrated, and mainly concentrated in +0.019mm grain size.

| Grain size | Productivity | Tin Grade | Metal rate | Accumulated metal rate |
|------------|--------------|-----------|------------|------------------------|
| +0.074     | 2.80         | 0.25      | 2.48       | —                      |
| 0.037      | 25.12        | 0.336     | 29.92      | 32.41                  |
| 0.019      | 45.36        | 0.295     | 47.44      | 79.85                  |
| 0.010      | 19.25        | 0.234     | 15.97      | 95.82                  |
| -0.010     | 7.47         | 0.158     | 4.18       | 100.00                 |
| Total      | 100.00       | 0.282     | 100.00     |                        |

### Table 1. Particle size composition of the materials (%).

3. Methods and Discussions

3.1. Centrifugal Separation Test of Circulating Fluidized Bed

3.1.1. Feeding density. The density of ore feeding affects mineral dispersion, while centrifugal separation of circulating fluidized bed is a kind of equipment to separate minerals according to the difference of specific gravity of materials. The quality of mineral dispersion directly affects the separation effect, and also has a certain impact on the treatment capacity [3, 4]. In our test we fixed feeding pressure 65 kPa, underflow pressure 55 kPa, overflow pressure 50 kPa, backwash water pressure 10 kPa, sand sink pressure 50 kPa, changed feeding density from 15% to 25%. The flow chart of centrifugal separation is shown in Fig 1 and the test results are shown in Table 2.

| Feeding density | Product name     | Productivity | Tin grade | Tin recovery | Enrichment ratio (multiple) |
|-----------------|------------------|--------------|-----------|--------------|-----------------------------|
| 15              | rough concentrates | 41.26        | 0.460     | 64.22        | 1.55                        |
|                 | tailings         | 58.74        | 0.180     | 35.78        |                             |
|                 | ore feeding      | 100.00       | 0.296     | 100.00       |                             |
| 20              | rough concentrates | 44.81        | 0.420     | 64.10        | 1.43                        |
|                 | tailings         | 55.19        | 0.191     | 35.90        |                             |
|                 | ore feeding      | 100.00       | 0.294     | 100.00       |                             |
| 25              | rough concentrates | 48.84        | 0.395     | 65.80        | 1.35                        |
|                 | tailings         | 51.16        | 0.196     | 34.20        |                             |
|                 | ore feeding      | 100.00       | 0.293     | 100.00       |                             |

The above table shows that with the increase of density, the concentrate yield increases gradually, the concentrate recovery rate increases little, and the concentrate enrichment ratio decreases gradually, so it is better to choose the concentrate concentration of 15%.
3.1.2. Feeding pressure. The ore feeding pressure has an important influence on the separation effect. The high centrifugal force exerted on light minerals leads to the entry of light minerals into heavy minerals, which reduces the grades of concentrates. At the same time, the low centrifugal force exerted on heavy minerals leads to the overflow of heavy minerals, resulting in metal loss [3, 4]. In our test we fixed feeding density 15%, underflow pressure 55 kPa, backwash water pressure 10 kPa, sand sink pressure 50 kPa, changed feeding pressure from 55 kPa to 75 kPa, and the test results are shown in Table 3.

Table 3. Effect of feeding pressure on centrifugal separation (%).

| Feeding pressure (kPa) | Product name     | Productivity | Tin grade | Tin recovery | Enrichment ratio (multiple) |
|------------------------|------------------|--------------|-----------|--------------|-----------------------------|
| 55                     | rough concentrates | 46.86        | 0.420     | 65.74        | 1.41                        |
|                        | tailings          | 53.14        | 0.193     | 34.26        |                             |
|                        | ore feeding       | 100.00       | 0.299     | 100.00       |                             |
| 65                     | rough concentrates | 41.26        | 0.460     | 64.22        | 1.55                        |
|                        | tailings          | 58.74        | 0.180     | 35.78        |                             |
|                        | ore feeding       | 100.00       | 0.296     | 100.00       |                             |
| 75                     | rough concentrates | 39.80        | 0.490     | 66.26        | 1.67                        |
|                        | tailings          | 60.20        | 0.165     | 33.74        |                             |
|                        | ore feeding       | 100.00       | 0.294     | 100.00       |                             |

We can see from Table 3 that with the increase of feeding pressure, the concentrate yield decreases gradually, the grade and enrichment ratio increase gradually, and the recovery changes little, so the best feeding pressure is 75 kPa.

3.1.3. Backwash water pressure. The flow of backwash water affects the dispersion of ore particles [3, 4]. In our test we fixed feeding density 15%, underflow pressure 55 kPa, feeding pressure 75 kPa and sand sink pressure 50 kPa, changed backwash water pressure from 10 kPa to 30 kPa. And the results are shown in Table 4.

Table 4. Effect of backwash water pressure on centrifugal separation (%).

| Backwash pressure (kPa) | Product name     | Productivity | Tin grade | Tin recovery | Enrichment ratio (multiple) |
|-------------------------|------------------|--------------|-----------|--------------|-----------------------------|
| 10                      | rough concentrates | 40.10        | 0.490     | 66.53        | 1.66                        |
|                         | tailings          | 59.90        | 0.165     | 33.47        |                             |
|                         | Ore feeding       | 100.00       | 0.295     | 100.00       |                             |
| 20                      | rough concentrates | 44.78        | 0.441     | 67.52        | 1.51                        |
|                         | tailings          | 55.22        | 0.172     | 32.48        |                             |
|                         | Ore feeding       | 100.00       | 0.292     | 100.00       |                             |
| 30                      | rough concentrates | 52.50        | 0.390     | 70.43        | 1.34                        |
|                         | tailings          | 47.50        | 0.181     | 29.57        |                             |
|                         | Ore feeding       | 100.00       | 0.291     | 100.00       |                             |
The results shown that with the increase of backwash water pressure, the rough concentrates rate increases, the enrichment ratio decreases, the recovery rate increases no significantly. At the same time, the equipment and energy consumption increase with the increase of backwash water pressure. Therefore, the backwash water pressure is chosen as 10 kPa.

3.1.4. Sand sinking pressure. The pressure of the sand sinking mainly affects the yield of the concentrate, and the greater the sand sinking pressure, the smaller the concentrates yield [3, 4]. We fixed feeding density 15%, underflow pressure 55 kPa, feeding pressure 75 kPa, backwash water pressure 10 kPa, changed the pressure of sand sinking. And the results are shown in Table 5.

| Sand sink pressure (kPa) | Product name     | Productivity | Tin grade | Tin recovery | Enrichment ratio (multiple) |
|-------------------------|------------------|--------------|-----------|--------------|----------------------------|
|                         | rough concentrates | 45.00        | 0.455     | 68.65        | 1.53                       |
|                         | tailings         | 55.00        | 0.170     | 31.35        |                            |
|                         | ore feeding      | 100.00       | 0.298     | 100.00       |                            |
| 50                      | rough concentrates | 40.59        | 0.492     | 67.35        | 1.66                       |
|                         | tailings         | 59.41        | 0.163     | 32.65        |                            |
|                         | ore feeding      | 100.00       | 0.297     | 100.00       |                            |
| 60                      | rough concentrates | 36.63        | 0.520     | 65.12        | 1.77                       |
|                         | tailings         | 63.37        | 0.161     | 34.88        |                            |
|                         | ore feeding      | 100.00       | 0.293     | 100.00       |                            |

From the test, it can be seen that the rough concentrate productivity decrease and concentrate enrichment ratio increase with the increase of the pressure at the sand sinking mouth, but the tin recovery decreases gradually. Comprehensive consideration the optimal pressure at the sinking mouth is 50 kPa.

According to the previous experiments, when the density of feeding ore is 15%, the pressure of bottom flow outlet is 55 kPa, the pressure of feeding ore is 75 kPa, the pressure of backwash water is 10 kPa, and the pressure of settling outlet is 50 kPa, the rough concentrate with tin grade of 0.492% can be obtained, the recovery rate of tin is 67.35%, and the enrichment ratio is 1.66. The circulating fluidized bed can be used as a pre-enrichment equipment, and about 60% tailings can be discarded. But concentrate products cannot be produced, which needs further processes to enrich.

3.2. Flotation Test
Previous experiments show that it is difficult to produce concentrate products by using only cyclone continuous centrifuge, and the effect of shaking table is poor when the size of the ore is finer. This paper intends to use flotation to further enrich, mainly to explore the optimal dosage of collector, regulator and auxiliary collector. The test material is the product after desulfurization of concentrate by cyclone continuous centrifuge, containing 0.522% tin and 0.18% sulfur, and the recovery of tin in desulfurization operation is 98.15%. Conditions of flotation were tested, and the optimum conditions were determined as YP-6 as collector, 350 g/t as roughing separation and 120 g/t as scavenging separation. The auxiliary collector is P86, the roughing separation is 100g/t, and the scavenging separation is 50g/t. The dosage of oxalic acid is 500 g/t [5].

3.2.1. Whole process test. The whole process test is carried out based on the conditional test. The process includes a roughing, a scavenging and three cleaning. The test flow is shown in Fig. 2 and the results are shown in Table 6.
Table 6. Results of whole process open circuit flotation.

| Product name | Productivity | Tin grade | Accumulated grade | Tin recovery | Accumulated recovery rate | Enrichment ratio (multiple) |
|--------------|--------------|-----------|-------------------|-------------|----------------------------|----------------------------|
| concentrate  | 4.13         | 5.91      | 49.96             | 12.09       |                            |                            |
| middling1    | 3.19         | 1.22      | 3.866             | 7.97        | 57.93                      |                            |
| middling2    | 7.86         | 0.63      | 2.191             | 10.14       | 68.07                      |                            |
| middling3    | 10.8         | 0.464     | 1.473             | 10.26       | 78.33                      |                            |
| middling4    | 9.59         | 0.728     | 1.272             | 14.29       | 92.62                      |                            |
| tailings     | 64.43        | 0.056     | 0.489             | 7.38        | 100                        |                            |
| feeding      | 100.00       | 0.489     | 100.00            |             |                            |                            |

From the above table we can see the concentrate with tin grade is 5.91% and recovery is 49.96% can be obtained by open circuit test, and tin metal loss of tailings is 7.39%.

Figure 2. Flow chart of whole process open circuit flotation.

3.2.2. Closed circuit test of flotation. According to the optimum conditions of open-circuit test, closed-circuit test of cassiterite flotation is carried out. The test flow is shown in Figure 3, and the results are shown in Table 7.
Figure 3. Flow chart of whole process closed circuit flotation.

Table 7. Results of whole process closed circuit flotation.

| Product name | Productivity | Tin grade | Tin recovery |
|--------------|--------------|-----------|--------------|
| concentrate  | 9.25         | 4.380     | 77.18        |
| middling1    | 6.13         | 1.25      | 14.60        |
| middling2    | 13.23        | 0.711     | 17.92        |
| middling3    | 3.12         | 0.634     | 3.77         |
| middling4    | 6.45         | 1.630     | 20.03        |
| tailings     | 90.75        | 0.132     | 22.82        |
| feeding      | 100.00       | 0.525     | 100.00       |

Through closed circuit test we obtained concentrate which tin grade is 4.38% and recovery rate is 77.18%. The separation effect is improved significantly.

4. Conclusion
From the above experiments, we can draw the following conclusions. The first, continuous centrifugal separation with circulating fluidized bed has a good enrichment effect in the separation of tin mud ore. It can be used as a pre-enrichment equipment to remove a large number of tailings and create good conditions for subsequent separation. The second, tin mud ore is used as material in our experiment. When the feed density is 15%, the bottom flow pressure is 55 kPa, the feeding pressure is 75 kPa, the backwash water pressure is 10 kPa and the sinking mouth pressure is 50 kPa, we obtained the rough concentrate with a tin grade of 0.492% and a tin recovery ratio of 67.35%, and about 60% tailings can be discarded. The third, the rough concentrate enriched by centrifugal separation through circulating fluidized bed is flotation with YP-6 as collector, P86 as auxiliary collector and oxalic acid as regulator after desulphurization. The tin concentrate with a tin grade of 4.38% and a tin recovery rate of 77.18% is obtained by closed circuit test includes a roughing, a sweeping and three refining. The recovery rate of tin in original tin ore is 51.02%, which achieves the anticipated purpose. The forth, through the combination of cyclone fluidized bed and flotation, the low-grade tin mud can be recovered better, which provides a new method for the recovery of tin resources.
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