DIRECT ALUMINA FLOTATION FROM A HIGH PURITY QUARTZ CONTAMINATED DURING GRINDING PROCESS

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ABSTRACT

Microflotation, bench scale and pilot plant tests were carried out for producing high purity quartz which was contaminated by particles originated from alumina balls during grinding. Direct alumina flotation was employed, using as collectors sodium salt of sulfosuccinic acid, soy oil, diesel oil and cetyl stearyl sodium sulfate. Flotanol D14, a polypropylene glycol alcohol was used as a frother. Microflotation tests of alumina from grinding balls were first performed using a Hallimond tube. Scanning Electron Microscopy (SEM) and chemical analysis through Energy-Dispersive X-Ray Detection (EDS) were applied to examine the microflotation and bench scale results. Microflotation tests presented high alumina floatability, above 95%, in the presence of cetyl stearyl sodium sulfate and sodium salt of sulfosuccinic acid at pH 5.5. The optimization of the preliminary bench flotation test results lead to a specific blending of the collectors and the frother at pH 5.5 where the mass recovered to the tailings was 6.4% with a total alumina rejection of 89.21%. The final concentrate showed 95.2% of quartz recovery. Pilot plant tests confirmed the laboratory results enabling the alumina separation by flotation with adequate quartz mass recoveries.

Key words: flotation, alumina, quartz, grinding media.
1. INTRODUCTION

Quartz is a raw material largely employed in many industrial sectors. Among its noblest applications, can be cited optical fibers, laboratory glassware, electrical and electronic applications, halogen lamp tubing, etc. Lumpy quartz fragments or high purity quartz powder are used as raw materials (Freitas, 1998). During the processing of the quartz, ball mills are frequently employed for size reduction. The correct choice of grinding balls is one of the most prominent aspects for achieving a high purity grade in the final product. The balls must have excellent abrasive properties and a high impact resistance, in such a way the contamination of the ore with their own constitutive material will be minimized. Alumina grinding balls normally have a great resistance to abrasion. These balls are produced from molten bauxite leading to the formation of artificial corundum with an Al$_2$O$_3$ content averaging 95.5% and hardness 9 in the Mohs scale (Sampaio et al., 2005).

The Santa Rosa Mining Company, a Brazilian enterprise in the segment of industrial minerals, suffered a severe alumina contamination during the grinding of lump quartz fragments. This contamination was caused by the unexpected breakage of the grinding balls inside the mill. The high cost of the final product, fine quartz, afforded for research a way to remove the alumina contamination from the ground material.

Direct alumina flotation data concerning industrial operations can barely be found in the literature. Flotation tests using a novel anionic collector known as RL produced an aluminum recovery greater than 90% and the aluminum silicates were depressed with the help of inorganic reagents (Xu et al., 2004). Reverse bauxite flotation has already been used in the aluminum production and in this case, aluminum-silicates are floated using cationic reagents, such as amine to obtain a bauxite concentrate with a low Al$_2$O$_3$/SiO$_2$ ratio in the Bayer process feeding (Wuang et al., 2004).

In the present work direct alumina flotation, using a mixture of anionic collectors was selected for the alumina removal from the contaminated mill charge. Sodium sulfosuccinic acid, soy oil, diesel oil and cetyl stearyl sodium sulfate mixed in different proportions, were the proved collectors. It was not necessary the use of depressants and the best flotation results were attained at pH 5.5.

2. METHODOLOGY

2.1. Materials Samples

The samples used in the tests were supplied by Mineração Santa Rosa. The first sample was made up of contaminated quartz by the alumina from the grinding balls. The second sample comprised some of the broken alumina grinding balls used in the grinding process. The second sample was firstly crushed in the laboratory, using jaw and roll crushers. The crushed material was screened to remove the fraction between 300µm and 106µm to be used in the microflotation tests.

2.2. Reagents

Sodium sulfosuccinic acid denominated as KE, soy oil denominated as SO, diesel oil denominated as DO and cetyl stearyl sodium sulfate denominated as CSS, were the collectors used in the alumina flotation. Flotanol D14 was used as a frother. Hydrochloric acid (5% w/v) and sodium hydroxide (5% w/v) were used for pH adjustment.

2.3. Microflotation Tests

A modified Hallimond Tube, equivalent to that tube described by Fuerstenau (1957) was used to determine the response of alumina flotation in the presence of sodium sulfosuccinic acid and cetyl stearyl sodium sulfate. One gram of the sample at the size fraction -300 µm + 106µm was used for all tests. The collector solution with a total volume of 180ml was added and the pH adjusted to 5.5. The sample was conditioned for 5 minutes and the microflotation was carried out with a nitrogen flow rate of 40ml/minute during 1 minute. The products were collected, dried and weighed. Table 1 shows the picked out conditions after several exploratory tests have been conducted.

<table>
<thead>
<tr>
<th>Test</th>
<th>pH</th>
<th>Collector</th>
<th>Concentration (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.5</td>
<td>CSS</td>
<td>0.4</td>
</tr>
<tr>
<td>2</td>
<td>5.5</td>
<td>KE 883</td>
<td>0.4</td>
</tr>
</tbody>
</table>
2.4. Bench Scale Flotation Tests

The samples used in the bench flotation tests were tested as received in the laboratory with a size distribution showing a top size of 210 μm. The flotation tests were performed in a Denver D12 sub-aerated machine with a 2 L capacity cell. A total quantity of 500 g of the contaminated quartz was used in the all bench tests. The pulp with 30% of solids was conditioned with the collectors and frother for 5 minutes at pH 5.5. Five drops of Flotanol D14 were added to promote the frothing in each test. The air was fully released at 1500 rpm and the flotation time was 3 minutes. Distilled water was used in both flotation and microflotation tests. Table II summarizes the reagents dosage of the main flotation tests. Concentrate and tailings were collected, dried and sent to chemical analysis.

<table>
<thead>
<tr>
<th>Test</th>
<th>pH</th>
<th>Collector Concentration ( g/t )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>CSS</td>
</tr>
<tr>
<td>1</td>
<td>5.5</td>
<td>300</td>
</tr>
<tr>
<td>2</td>
<td>5.5</td>
<td>300</td>
</tr>
<tr>
<td>3</td>
<td>5.5</td>
<td>400</td>
</tr>
</tbody>
</table>

2.5. Scanning Electron Microscopy and EDS studies

Energy-Dispersive X-Ray Detection (EDS) is installed in SEM equipment. It allows the spectrum analysis of X-rays generated from the specimen directly under the electron beam. Utilizing this equipment, a chemical analysis of a selected area of the sample can be determined (Klein, 2002). Microflotation and bench flotation products were studied through SEM/EDS to characterize the particles and determine the Si and Al contents.

2.6. Pilot Plant Test

The pilot plant test was carried out in two 67 liters CIMAQ flotation cells mounted in a cell to cell configuration. Grinding was not necessary at the pilot plant once the flotation feed size of minus 210 μm was associated to the original size obtained during the fragmentation performed by Min. Santa Rosa which generated the contamination of the quartz.

3. RESULTS DISCUSSION

3.1. Microflotation Tests

The microflotation tests results, depicted in figure 1, have clearly indicated a high floatability of alumina in the presence of either cetyl stearyl sodium sulfate (CSS) or sodium sulfosuccinic acid (KE) at pH 5.5. This fact collaborates to explain why the best result in the bench flotation tests was achieved when a mixture of these two collectors was used.

![Figure 1. Microflotation tests results](image-url)
Figures 2, 3 and 4 show SEM pictures from grinding ball particles floated during the microflotation tests. Three particles were analyzed using the EDS.

**Figure 2.** Particle 1 - grinding ball particle floated with KE

**Figure 3.** Particle 2 - grinding ball particle floated with CSS

**Figure 4.** Particle 2 - grinding ball particle floated with CSS

The main chemical contents of each particle, floated particles, are presented in table III. A high content of Al, which caused the quartz contamination was found in all 3 particles.
3.2. Bench Scale Flotation Tests

The best result attained with the bench flotation tests, with a 3/1 mixture of CSS/KE, is shown in Table IV.

Table IV. Bench flotation test result

<table>
<thead>
<tr>
<th>Product</th>
<th>EDS analyses (%)</th>
<th>Distribution (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Al</td>
<td>Si</td>
</tr>
<tr>
<td>Feed</td>
<td>1.21</td>
<td>59.84</td>
</tr>
<tr>
<td>Tailings</td>
<td>16.90</td>
<td>45.19</td>
</tr>
<tr>
<td>Concentrate</td>
<td>0.14</td>
<td>60.84</td>
</tr>
</tbody>
</table>

The Si and Al contents were obtained through SEM/EDS analysis. The pictures obtained during MEV/EDS analysis are depicted in figures 5, 6 and 7. As one can see, it was possible to obtain a quartz concentrate with a high content of Si and low Al content. The bench flotation confirmed the high alumina floatability previously evidenced in the microflotation tests and also a high selectivity in the process enabling a Si recovery in the concentrate larger than 94% and a rejection of 89% of the Al present at the flotation feed.

Figure 5. Flotation feed, A=alumina and Q=quartz
3.3. Pilot Plant Test

The pilot plant test was performed during 3 hours in the Santa Rosa mining site. The reagents CSS and KE were mixed in the same proportion used in the bench scale test and the flotation pH varied from 5.0 to 6.0. Composite samples from the feed, concentrate and tailings were analyzed in the Santa Rosa chemical laboratory. The plant configuration and the way the plant was operated can be considered as a single rougher flotation step. The overall result, described in the table V, shows an alumina distribution to the tailings close to 91% which is about the same result achieved in the bench scale tests. The alumina content in the concentrate was 0.10%. These results essentially confirmed both the selectivity and the recoveries found in the bench scale tests.

Table V. Pilot plant test result

<table>
<thead>
<tr>
<th>Product</th>
<th>Chemical analyses (%)</th>
<th>Distribution (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Al₂O₃</td>
<td>Weight</td>
</tr>
<tr>
<td>Feed</td>
<td>1.0</td>
<td>100.0</td>
</tr>
<tr>
<td>Tailings</td>
<td>12.1</td>
<td>7.5</td>
</tr>
<tr>
<td>Concentrate</td>
<td>0.1</td>
<td>92.5</td>
</tr>
</tbody>
</table>
4. CONCLUSIONS

Microflotation tests showed high alumina floatability in the presence of cetyl stearyl sodium sulfate and sodium salt of sulfosuccinic acid.

Bench scale and pilot plant tests presented a selective direct flotation of alumina from quartz at pH 5.5 using a mixture 3/1 of cetyl stearyl sodium sulfate and sodium sulfosuccinic acid with a total mixture dosage of 400g/t. Flotanol D14, a polypropylene glycol alcohol was used as a frother.

The quartz recovery obtained in this process was around 95% and the Al₂O₃ contamination was reduced to 0.10% from a feed grade of 1.0%. Despite the fact that the alumina grade, 0.10% in the quartz concentrate, is still high considering the market specifications, these results show a tremendous potential for cleaning the concentrate adding more flotation stages in the process.

5. REFERENCES


Fuerstenau, D. W.; Metzger, P. H.; Seele G. D. How to use this modified Hallimond tube for better flotation testing. Engineering and Mining Journal, 158:3, p. 93-95, 1957.