DIRECT FLOTATION OF APATITE FROM PRIMARY AND SECONDARY LOW-GRADE COMPLEX JHAMARKOTRA ORE, RAJASTHAN, INDIA

Anders Cassel & Elisabeth Henriksson

AkzoNobel Surface Chemistry AB
P.O. Box. Stenungsund - Sweden. S.444 85
Tel: + 46 303 86033, Fax: + 46 303 84371. E-mail: anders.cassel@akzonobel.com

RESUMO
AkzoNobel (AN) desenvolve processos para flotação direta de apatita a partir de minérios complexos de apatita contendo dolomita, calcita e sílica. Atualmente a tecnologia é aplicada globalmente. Agora AN junto com a Rajasthan State Mines & Minerals (RSMML) desenvolvem processo de flotação direta de apatita como alternativa ao processo atualmente utilizado pela RSMML de flotação reversa de apatita na mina e planta de Jhamarkotra, em Rajasthan, Índia.

O objetivo é melhorar a eficiência e a economicidade do processo, bem como a qualidade do concentrado de apatita. Apresentamos neste trabalho os resultados obtidos em testes laboratoriais com o minério primário e secundário de baixo teor de Jhamarkotra usando coletor anfotérico e aniónico para apatita, respectivamente.

Tanto o processo com o coletor anfotérico como o processo usando o coletor aniónico flotam diretamente a apatita sem deslaimagem e com água ligeiramente alcalina, produzindo concentrados com alto teor de apatita e teores relativamente baixos de MgO e SiO₂.

Propomos um fluxograma que é válido para os dois tipos de minério e que utiliza a flotação direta de apatita, mas tendo, em separado, dois coletores e dois sistemas de controle dos aditivos.

PALAVRAS-CHAVE: Apatita, dolomita, sílica, calcita, flotação direta.

ABSTRACT
AkzoNobel (AN) develops processes for direct flotation of apatite from complex apatite ores containing dolomite, calcite and silica. The technology is today globally applied.

Now AN together with Rajasthan State Mines & Minerals (RSMML) develop direct apatite flotation alternatives to RSMML’s presently used reversed apatite process at the Jhamarkotra mine and concentrator, Rajasthan, India. The aim is to improve process economy, effectiveness and concentrate quality.

We present in this paper laboratory results on primary and secondary Jhamarkotra low-grade ore using an amphoteric and anionic apatite collector, respectively.

Both the amphoteric and anionic collector systems float directly apatite without de-sliming and in slightly alkaline waters, producing high-grade apatite concentrates with relatively low MgO and SiO₂ content.

We propose a direct apatite flotation flow-sheet valid for both ore types but having two separate collectors and regulating additives systems.

KEY WORDS: Apatite, dolomite, silica, calcite, direct flotation,
1. INTRODUCTION

Akzo Nobel (AN) develops processes for direct flotation of apatite from complex apatite ores. The design of the collector systems can be adapted to different types of requirements like complexity, i.e. dolomite, calcite and silica contents, particle size distribution, regulating additives, quality of process water, etc. Direct flotation of apatite using the AN-technology is today globally applied to improve process effectiveness and concentrate quality.

AN and Rajasthan State Mines & Minerals Ltd, Rajasthan, India (RSMML) are developing direct apatite flotation processes for RSMML's dolomitic primary Jhamarkotra low-grade ore (P-LGO) and their weathered, low dolomite, high silica containing secondary low-grade ore (S-LGO). The S-LGO is today not possible to beneficiate.

The two tentative direct apatite flotation processes presented in this paper use as a basis an amphoteric collector system for the P-LGO and an anionic collector system for the S-LGO. The goal is to develop a one flow-sheet process applicable for both feed stocks and after modification of the existing plant producing an apatite concentrate of higher P2O5 grade and recovery and with lower MgO content than today's and being suitable for use in the wet phosphoric acid process. A more detailed presentation of the results from the laboratory development work with the P-LGO ore is given by Cassel and Henriksson (2008).

The plant uses today a two-step flow-sheet. The first step is a bulk flotation of apatite and dolomite using fatty acid soap as collector, rejecting silica, followed by an acidic reversed apatite flotation using sulphuric and phosphoric acid to depress the apatite. Sekhar and Jain (2006) present a recent description of the Jhamarkotra apatite concentrator. Its capacity is 3000 tons of P-LGO feed per day.

2. EXPERIMENTAL

Laboratory flotation experiments on the P-LGO and S-LGO samples were generally performed using the broken flow-line in the flow-sheet of Figure 1. 100 kg dry Jhamarkotra P-LGO ore feed from after Roller Press Crusher (= feed to wet screen) and 50 kg dry ore sample from Jhamarkotra “S-LGO ore pile” was shipped to the AkzoNobel laboratory in Stenungsund, Sweden.

The coarse materials (+1.8 mm) were re-crushed to -1.8 mm and mixed with the rest of the ore sample material. The combined ore material was split into 0.5 kg portions and packed in plastic bags. The bags were stored at ambient temperature (approx. 20°C).

Grinding conditions and flotation experimental:
- Ore feed: 0.5 kg
- Water: 0.4 kg
- Grinding time (min): 5, 7 and 12
- Rod mill charge: 3.6 kg
- Natural pH: 9-10
- Flotation machine: Denver; Cell volume: 1.5 l; Pulp density: 33%; Temperature: 20°C
- Impeller speed:
  - P-LGO: 1000 rpm (rougher) and 900 rpm (cleaner)
  - S-LGO: 1100 rpm (rougher) and 900 rpm (cleaner)
- pH at start:
  - P-LGO: 11 (NaOH)
  - S-LGO: 9.5 (Na₂CO₃)
- pH during lab flotation tests: Self regulating during rougher and cleaner steps:
  - P-LGO: After 2 cleaner steps pH is approx. 9.5 and after 4 steps pH=9
  - S-LGO: After 3 cleaner steps pH=8.3
- Collectors: Both apatite collectors, MD 20404 (anionic for S-LGO) and MD 20499 (amphoteric for P-LGO), were added as a 1% solution

In a special experimental set up the P-LGO ore feed was first separated into a +50 micron and a -50 micron material fraction, respectively, by wet sieving using as little as possible support water. Then the +50 micron material was taken to further grinding (5, 7 and 12 minutes) before being used as feed to flotation. The -50 micron material was taken directly to flotation.
Figure 1. A tentative flow-sheet for a direct apatite flotation process applied on Jhamarkotra P-LGO and S-LGO using an amphoteric and an anionic collector system, respectively. The broken lines outline performed laboratory flotation experiments. PC, SC and DC stand for Primary, Secondary and De-sliming hydrocyclones, respectively, today practiced in the plant. The notation 90% -X micron indicates that the fineness of the flotation feed is an important parameter and needs to be further optimized.
3. RESULTS

3.1 Particle size distribution curves

In Figure 2 five particle size distribution curves are presented. The coarsest material curves are represented by the laboratory mill feed of the P-LGO and by the S-LGO flotation feed. The latter material is flotation feed in Experiment JHA 208. For the flotation results for this experiment see Table II and Figure 3.

The finest material curve is represented by the -50 micron classified P-LGO mill feed material. The second finest material is represented by the +50 micron P-LGO material after 12 minutes grinding.

The fifth curve is in between and represents the flotation feed P-LGO material in experiment JHA 106 in Figure 3. This feed has a fineness of 90% <63 micron which is slightly finer than the Jhamarkotra plant feed used today.

It is to be noted that the Roller Press generates approximately 50% material <50 micron according to the black squares curve of Figure 2. This fact lead us to design the special laboratory flotation set up for the P-LGO sample giving the results demonstrated in Table I.

It is also to be noted that the S-LGO flotation feed (12 minutes grinding) represents the coarsest flotation feed material having 74% of accumulated fines material less than 90 micron in size.

3.2 Flotation experiments

The results from the first investigations of the Jhamarkotra P-LGO using a particle fineness of 90% -74 micron, which is equal to plant practice of today, indicated that it is not possible to achieve concentrates of reasonable low MgO grades. Therefore flotation experiments were run on flotation feeds of higher fineness:

---

Figure 2. The coarsest material curves are represented by the laboratory mill feed of the P-LGO and the S-LGO flotation feed. The finest material curves are represented by the -50 micron classified P-LGO mill feed material and by the +50 micron P-LGO material after 12 minutes grinding. In between is the fifth material, which represents the flotation feed of Experiment JHA 106: a non-classified P-LGO flotation feed after 7 min. grinding.
-50 micron classified P-LGO mill feed material
+50 micron P-LGO material after 12 minutes grinding

To further analyze the problems of selectivity between apatite and dolomite the final P-LGO concentrates were then wet sieved into +32 micron, 32-20 micron and -20 micron size fractions with accompanying chemical analyses of P₂O₅, MgO and SiO₂. The results are presented in Table I.

There for the -50 micron feed material MgO = 0.7% for the -20 micron fraction and 1.0% for the 20-32 micron concentrate fraction. These fractions represent 94% of the concentrate material.

For the +50 micron materials after 12 min grinding only the -20 micron concentrate materials can reach the relatively low MgO level of 1.1%, representing 63% of the material.

All other concentrate fractions, although high in P₂O₅, have MgO grades of 1.9% or higher.

Table I. P₂O₅, MgO and SiO₂ grades in the +32 micron, 32-20 micron and -20micron size fractions of the final concentrates of Experiments JHA 121 and JHA 123, respectively.

<table>
<thead>
<tr>
<th>P-LGO flotation feed</th>
<th>Size fractions (micron)</th>
<th>Weight (%)</th>
<th>P₂O₅ Grade (%)</th>
<th>MgO Grade (%)</th>
<th>SiO₂ Grade (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-50 micron fraction (not ground)</td>
<td>+32</td>
<td>6</td>
<td>36.4</td>
<td>1.9</td>
<td>2.3</td>
</tr>
<tr>
<td>(Experiment JHA 121)</td>
<td>20-32</td>
<td>18</td>
<td>39.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>+50 micron feed after grinding 12min</td>
<td>+32</td>
<td>18</td>
<td>36.8</td>
<td>1.9</td>
<td>2.4</td>
</tr>
<tr>
<td>(Experiment JHA 123)</td>
<td>-20</td>
<td>20</td>
<td>37.1</td>
<td>2.0</td>
<td>0.9</td>
</tr>
<tr>
<td>-20</td>
<td>63</td>
<td>39.0</td>
<td>1.1</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

Table II. Grade-recovery results from flotation Experiment JHA 208. Feed material: S-LGO, 12 mins grinding.

| Feed material: S-LGO | Weight (g) | Weight (%) | P₂O₅ Grade (%) | P₂O₅ Rec. (%) | MgO Grade (%) | MgO Rec. (%) | SiO₂ Grade (%) | SiO₂ Rec. (%)
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Total calculated</td>
<td>498.1</td>
<td>100.0</td>
<td>14.2</td>
<td>99.6</td>
<td>0.29</td>
<td>100.1</td>
<td>55</td>
<td>99</td>
</tr>
<tr>
<td>Rougher tailing</td>
<td>313.2</td>
<td>62.9</td>
<td>5.0</td>
<td>22.1</td>
<td>0.14</td>
<td>29.8</td>
<td>77</td>
<td>88</td>
</tr>
<tr>
<td>Cleaner 1 tailing</td>
<td>68.9</td>
<td>13.8</td>
<td>18.5</td>
<td>18.0</td>
<td>0.56</td>
<td>27.2</td>
<td>36</td>
<td>9</td>
</tr>
<tr>
<td>Cleaner 2 tailing</td>
<td>23.3</td>
<td>4.7</td>
<td>26.5</td>
<td>8.7</td>
<td>0.55</td>
<td>9.0</td>
<td>16</td>
<td>1</td>
</tr>
<tr>
<td>Cleaner 3 tailing</td>
<td>9.1</td>
<td>1.8</td>
<td>32.0</td>
<td>4.1</td>
<td>0.65</td>
<td>4.1</td>
<td>0.8</td>
<td>0</td>
</tr>
<tr>
<td>Concentrate</td>
<td>83.6</td>
<td>16.8</td>
<td>39.5</td>
<td>46.7</td>
<td>0.51</td>
<td>30.0</td>
<td>2.8</td>
<td>1</td>
</tr>
</tbody>
</table>

*(A.I.%) = Acid Insoluble

The Jhamarkotra S-LGO is completely different compared to the P-LGO. The P-LGO sample used in this study is high in MgO (12%) and relatively low in SiO₂ (4%). The S-LGO sample is very low in MgO (<0.3%) but very high in SiO₂ (55%) and weathered.

The P₂O₅ content is approximately 14% for both the P-LGO and the S-LGO sample.

Large efforts were made in our laboratory to adapt the amphoteric collector system used for the P-LGO sample also to work on the S-LGO flotation feed. However, the amphoteric system appeared too week resulting in very high collector additions and was excluded from further S-LGO flotation feed trials.

Then and under relatively mild alkaline conditions (Na₂CO₃) a direct apatite flotation route using an anionic collector system was possible to outline according to Figure 1. The results are presented in Table II and summarized in Figure 3 (Experiment JHA 208).
Figure 3. S-LGO (circles) and P-LGO (squares) flotation feed responses with regard to P$_2$O$_5$, MgO and SiO$_2$ grades versus P$_2$O$_5$ recovery using the anionic collector concept MD 20404 and the amphoteric collector concept MD 20499, respectively.

4. DISCUSSION

The results demonstrated in Figure 3 and Table II indicate that for the S-LGO feed material the anionic collector MD 20404 very selectively floats apatite and rejects silica. With the limited but successful tests performed high P$_2$O$_5$ grades (36.3%) and low MgO (0.53%) are received already in the 1st cleaner concentrate at an SiO$_2$ grade of 6.2%. Looking at the MgO grade figures there is a small enrichment of MgO in the apatite concentrates compared to the feed of the S-LGO (0.29%). However, for this S-LGO feed and using 12 minutes grinding it is not possible to receive very good apatite recovery in the final concentrate as 40% of the P$_2$O$_5$ is lost to the Rougher and 1st Cleaner tailings.

The reason to this is most probably that the apatite and silica containing mineral grains in the coarser part of the feed are not enough liberated. A re-grinding of the coarse flotation feed fraction should most probably increase the P$_2$O$_5$ recovery substantially.

For the P-LGO feed type sample it has been reported earlier by AN (Cassel and Henriksson, 2008) that by using the amphoteric collector system (MD 20499) the finer the P-LGO feed to flotation is - the better is the P$_2$O$_5$ grade and recovery and the lower is the MgO content of the corresponding concentrate. Typically in not enough fine flotation feeds non-liberated apatite-dolomite mineral grains do not allow MgO to be less than 2.0% in the concentrates without a substantial reduction in P$_2$O$_5$ recovery, see Experiment JHA 106 in Figure 3. JHA 106 represents a flotation feed fineness of 90%< 63micron, i.e. slightly finer than the flotation feed to the Jhamarkotra concentrator of today. As is demonstrated in Table I the coarse fraction (+32 micron) material of the concentrate can not contribute positively to the production of a concentrate lower than 1.5% in MgO.

Also an examination of a chemical and fractional analysis of a commercial grade Jhamarkotra concentrate as given in Table III leads to the same conclusions: The low P$_2$O$_5$ and high MgO and SiO$_2$ grades in the +40 micron part of that concentrate indicate a mineral grain liberation problem and that the P-LGO flotation feed needs to be further comminuted, if a concentrate quality real low in MgO is aimed for.

The notation 90% -X micron in Figure 1 therefore indicates that the fineness of the flotation feed is an important parameter and needs to be further investigated and optimized. This is valid for both the S- and the P-LGO feed types.
Table III. Chemical and fractional analyses of a commercial grade Jhamarkotra concentrate.

<table>
<thead>
<tr>
<th>Particle size fractions (microns)</th>
<th>Weight (%)</th>
<th>P₂O₅ (%)</th>
<th>MgO (%)</th>
<th>SiO₂ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt;125</td>
<td>0.6</td>
<td>17.5</td>
<td>8.5</td>
<td>15.7</td>
</tr>
<tr>
<td>100-125</td>
<td>1.1</td>
<td>15.2</td>
<td>8.5</td>
<td>19.5</td>
</tr>
<tr>
<td>63-100</td>
<td>6.6</td>
<td>15.0</td>
<td>8.3</td>
<td>18.2</td>
</tr>
<tr>
<td>50-63</td>
<td>3.6</td>
<td>16.2</td>
<td>8.1</td>
<td>17.3</td>
</tr>
<tr>
<td>40-50</td>
<td>4.8</td>
<td>17.9</td>
<td>7.5</td>
<td>16.9</td>
</tr>
<tr>
<td>&lt;40</td>
<td>84.3</td>
<td>34.4</td>
<td>1.7</td>
<td>6.9</td>
</tr>
<tr>
<td>Total</td>
<td>101</td>
<td>31.4</td>
<td>2.8</td>
<td>8.7</td>
</tr>
</tbody>
</table>

It should, according to the authors of this paper, be possible to combine and optimize into one system the NaOH regulating system supporting the amphoteric collector MD 20499 (P-LGO) and the NaCO₃ regulating system supporting the anionic collector MD 20404 (S-LGO). Then the direct apatite flotation flow-sheet of Figure 1 can be rationalized even further.

If the low content of MgO (≤0.53%) in the S-LGO concentrates is a general property for Jhamarkotra S-LGO then a very interesting option could be to blend the relatively high in MgO and low in SiO₂ P-LGO type concentrates with the low in MgO and relatively high in SiO₂ S-LGO type of concentrates to a concentrate blend having enough high P₂O₅ and low MgO for being an accepted raw material in the wet phosphoric acid process.

5. CONCLUSIONS

- An anionic based collector system using weakly alkaline regulating additives (Na₂CO₃) works well on the S-LGO as a selective direct apatite collector.
- Selective grinding of the S-LGO flotation feed material will most probably increase the P₂O₅ recovery substantially at good P₂O₅ grades as is demonstrated for the P-LGO.
- The low content of MgO (≤0.5%) in the S-LGO concentrates opens up an interesting option of blending the P-LGO type of concentrate with the S-LGO type. Then a concentrate blend having enough high P₂O₅ and low MgO is possible to produce and being a potential raw material into the wet phosphoric acid process.
- It should be possible to combine and optimize into one system the NaOH regulating system supporting the amphoteric collector MD 20499 (P-LGO) and the NaCO₃ regulating system supporting the anionic collector MD 20404 (S-LGO). Then the direct apatite flotation flow-sheet of Figure 1 can be rationalized even further.

6. FURTHER STUDIES

To validate the above reported indications further studies are needed. The assessed requirement of higher fineness on parts of the flotation feed for both the S-LGO and P-LGO feed materials will generate more extensive studies on both ore types on for example:
- Mineral grain liberation sizes for different ore types and feed fractions
- Classification routes
- Repetitive cleaning operations
- Effect on thickening
- Optimization of the collector and regulating system
- Power consumption

7. ACKNOWLEDGEMENTS

The authors acknowledge the support received from Rajasthan State Mines & Minerals Ltd and AkzoNobel Surface Chemistry AB.

8. REFERENCES
