Milling of the Phosphate Rock Flotation Circuit Circulating Load Aiming Production Increase and Iron Content Reduction in the Final Concentrate

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PHOSPHATE ROCK PRODUCTION INCREASE THROUGH THE MILLING OF THE APATITE FLOTATION CIRCULATING LOAD

Prof Dr André Carlos Silva,
M.F.L. Teixeira, B.P. Milanezi, A.H.P. Melo Filho,
T.D.A. Araujo, W.F. Borges Junior, E.M.S. Silva
Brazilian fertilizer supply from 2015 to 2018. Adapted from ANDA, 2018

<table>
<thead>
<tr>
<th>Year</th>
<th>Domestic production (%)</th>
<th>Importations (%)</th>
<th>Brazilian fertilizer supply (million tons)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2015</td>
<td>30.2</td>
<td>69.8</td>
<td>30.2</td>
</tr>
<tr>
<td>2016</td>
<td>31.2</td>
<td>68.8</td>
<td>28.5</td>
</tr>
<tr>
<td>2017</td>
<td>23.8</td>
<td>76.4</td>
<td>29.3</td>
</tr>
<tr>
<td>2018*</td>
<td>29.3</td>
<td>74.8</td>
<td>34.4</td>
</tr>
</tbody>
</table>

* Estimated based on data from January to March of 2018.
Situated in the alkaline complex Catalão I, Goiás, Brazil, the Chapadão mine has been ran by Copebras/CMOC International since 2016.
The Chapadão Mine has been in operation since 1976 and in 2014 was responsible for 21% of the Brazilian phosphate rock production.
In 2015, the company produced 1.1 million tons of phosphate fertilizers, 265 kt of phosphoric acid, and 147 kt of dicalcium phosphate (DCP) for animal supplementary feed.

The company products portfolio is composed in addition by sulfuric and hexafluorosilicic acids.
MP-47
Feed = 380 t/h (d.b.)

MP-76
Feed = 300 t/h (d.b.)
Phosphate rock processing flowsheet at Copebras/CMOC in Brazil

ROM

Screening
(Single inclination screen)

Crushing
(hammer crusher)

Homogenization piles
(64 and/or 80 kt)

Primary grinding
(bar mil operating in open circuit)

LIMS
(wet drum)

Secondary grinding
(ball mill operating in closed circuit)

Classification
(Hydrocyclones)

Desliming
(Hydrocyclones)

Barite flotation
(reverse apatite)

Apatite flotation
(anionic flotation)

WHIMS
(Carousel)

Chemical plant

Niobium plant

Tailings dam

*Nowadays barite concentrate is sold as a coproduct
Phosphate rock processing flowsheet at Copebras/CMOC in Brazil

**MP-76 = 170 t/h**
**MP-47 = 230 t/h**

**Rougher stage**
- pH: 10.7
- Depressant: cornstarch 500 g/t
- Collector: Lioflot 567 200 g/t
- Surfactant: Flotinor 071 100 g/t

**Scavenger stage**
- Collector: Lioflot 567 100 g/t

**Cleaner stage**
- pH: 6.5
- Collector: Lioflot 567 100 g/t

**Niobium plant**

**Circulation load (30-40%)**

**MP-76 = 59.5 t/h**
**MP-47 = 80.5 t/h**

**WHIMS**

**Tailings dam**

**Concentrate**

**Concentrate**

**Tailings**
Do we need a circulating load?

Rougher stage
- pH: 10.7
- Depressant: cornstarch 500 g/t
- Collector: Lioflot 567 200 g/t
- Surfactant: Flotinor 071 100 g/t

Cleaner stage

Scavenger stage
- Collector: Lioflot 567 100 g/t

Middling

Niobium plant

Tailings dam

Concentrate

WHIMS
Mineralogical characterization

**XRD**
Bruker D8 Discover

**XRF**
Bruker S8 TIGER WDXRF

**SEM**
JEOL JSM-IT300

**EDS**
Oxford Instruments X-MaxN

**LOI**
Calcined at 900 °C for 2 hours
Flotation tests at bench scale
Copebras/CMOC internal procedure PCT.13.001.050 for apatite flotation tests in bench scale

<table>
<thead>
<tr>
<th>Operational parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starch (g/t)</td>
<td>500</td>
</tr>
<tr>
<td>Lioflot 567 (g/t)</td>
<td>320</td>
</tr>
<tr>
<td>Flotinor 071 (g/t)</td>
<td>20</td>
</tr>
<tr>
<td>pH</td>
<td>10</td>
</tr>
<tr>
<td>Impeller speed (rpm)</td>
<td>1100</td>
</tr>
<tr>
<td>Solids (%)</td>
<td></td>
</tr>
<tr>
<td>Conditioning</td>
<td>50</td>
</tr>
<tr>
<td>Flotation</td>
<td>35</td>
</tr>
<tr>
<td>Conditioning (min)</td>
<td></td>
</tr>
<tr>
<td>Depressor</td>
<td>2.5</td>
</tr>
<tr>
<td>Collector – rougher</td>
<td>0.5</td>
</tr>
<tr>
<td>Collector – scavenger</td>
<td>0.5</td>
</tr>
<tr>
<td>Flotation (min)</td>
<td></td>
</tr>
<tr>
<td>Rougher</td>
<td>2</td>
</tr>
<tr>
<td>Cleaner</td>
<td>1.25</td>
</tr>
<tr>
<td>Scavenger</td>
<td>1.5</td>
</tr>
</tbody>
</table>

Feed
Conditioning 1
Depressant 2.5 min and collector 30 s (50% of collector dosage)
Surfactant
Rougher flotation
Flotation time 2 min
Cleaner flotation
Flotation time 1.25 min
Tailings
Weighted and sent to XRF
Scavenger flotation
Flotation time 1.5 min
Conditioning 2
Collector 30 s (50% of collector dosage)
Flotation middling
Weighted and sent to XRF
Final concentrate
Weighted and sent to XRF
## Methodology

### Experimental design for the middling rougher flotation tests

<table>
<thead>
<tr>
<th>Factors</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>$d_{95}$</td>
<td>3 (208, 150, and 74 μm)</td>
</tr>
<tr>
<td>Collector dosage</td>
<td>3 (160, 200, and 240 g/t)</td>
</tr>
<tr>
<td>Depressant dosage</td>
<td>2 (500 and 700 g/t)</td>
</tr>
</tbody>
</table>
Methodology

Experimental design for the middling rougher flotation tests

<table>
<thead>
<tr>
<th>Sample</th>
<th>Test</th>
<th>Solids % during the conditioning</th>
<th>Collector (g/t)</th>
<th>Depressant (g/t)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Middling without milling</td>
<td>1.1</td>
<td>49.4</td>
<td>160</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>1.2</td>
<td>48.8</td>
<td>200</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>1.3</td>
<td>49.1</td>
<td>240</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>2.1</td>
<td>46.4</td>
<td>160</td>
<td>700</td>
</tr>
<tr>
<td></td>
<td>2.2</td>
<td>46.9</td>
<td>200</td>
<td>700</td>
</tr>
<tr>
<td></td>
<td>2.3</td>
<td>46.1</td>
<td>240</td>
<td>700</td>
</tr>
<tr>
<td>A</td>
<td>3.1</td>
<td>50.8</td>
<td>160</td>
<td>500</td>
</tr>
<tr>
<td>After milling</td>
<td>3.2</td>
<td>49.6</td>
<td>200</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>3.3</td>
<td>52.4</td>
<td>240</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>4.1</td>
<td>55.1</td>
<td>160</td>
<td>700</td>
</tr>
<tr>
<td></td>
<td>4.2</td>
<td>38.9</td>
<td>200</td>
<td>700</td>
</tr>
<tr>
<td></td>
<td>4.3</td>
<td>39.6</td>
<td>240</td>
<td>700</td>
</tr>
<tr>
<td>B</td>
<td>5.1</td>
<td>51.1</td>
<td>160</td>
<td>500</td>
</tr>
<tr>
<td>After milling</td>
<td>5.2</td>
<td>49.8</td>
<td>200</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>5.3</td>
<td>49.1</td>
<td>240</td>
<td>500</td>
</tr>
</tbody>
</table>
Methodology

Average XRF results for the feed of the tests 1 (middling without milling) and the industrial threshold for the oxides in the final concentrate

<table>
<thead>
<tr>
<th>Feed</th>
<th>P$_2$O$_5$</th>
<th>CaO</th>
<th>Fe$_2$O$_3$</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>MgO</th>
<th>BaO</th>
<th>CPR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>22.85</td>
<td>29.30</td>
<td>18.81</td>
<td>18.62</td>
<td>0.95</td>
<td>0.83</td>
<td>0.58</td>
<td>1.28</td>
</tr>
<tr>
<td>St. Dev.</td>
<td>0.09</td>
<td>0.09</td>
<td>0.27</td>
<td>0.24</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
<td>0.00</td>
</tr>
<tr>
<td>Ind. thres.</td>
<td>≥ 37</td>
<td>-</td>
<td>≤ 0.82</td>
<td>≤ 2.90</td>
<td>≤ 3</td>
<td>≤ 0.50</td>
<td>≤ 0.50</td>
<td>≤ 1.32</td>
</tr>
</tbody>
</table>
Mineralogical characterization of the flotation feed. (a) Quantitative analysis by the Rietveld Method. Compositional map obtained by EDS from SEM image: (b) global, (c) P, (d) Ca and (e) Fe.
FLOTATION TESTS

Feed | Concentrate | Tailings | Industrial threshold | Recovery | Mass rec.

Without milling | 150 μm | 150 μm | 74 μm | 74 μm

500 g/t | 700 g/t | 500 g/t | 700 g/t | 500 g/t

Fe₂O₃ CONTENT (%) | Fe₂O₃ RECOVERY (%)
FLOTATION TESTS

- **Feed**
- **Concentrate**
- **Tailings**
- **Industrial threshold**
- **Recovery**
- **Mass rec.**

**Graph Details:**
- **SIO2 CONTENT (%)**
- **SIO2 RECOVERY (%)**

- Bars for each condition (500 g/t, 700 g/t) indicating the recovery and content levels.
- Graphs show the effect of milling size (μm) on recovery and content.
- Key markers include 500 g/t and 700 g/t feed concentrations.
**FLOTATION TESTS**

<table>
<thead>
<tr>
<th>Feed</th>
<th>Concentrate</th>
<th>Tailings</th>
<th>Industrial threshold</th>
<th>Recovery</th>
<th>Mass rec.</th>
</tr>
</thead>
<tbody>
<tr>
<td>160</td>
<td>200</td>
<td>240</td>
<td>160</td>
<td>200</td>
<td>240</td>
</tr>
</tbody>
</table>

- **Without milling**: 150 μm, 74 μm
- **500 g/t**: 700 g/t
- **700 g/t**: 700 g/t
FLOTATION TESTS

- **Feed**
- **Concentrate**
- **Tailings**
- **Industrial threshold**
- **Mass rec.**

**AL₂O₃ RECOVERY (%)**

<table>
<thead>
<tr>
<th>Feed</th>
<th>Concentrate</th>
<th>Tailings</th>
<th>Industrial threshold</th>
<th>Mass rec.</th>
</tr>
</thead>
<tbody>
<tr>
<td>160</td>
<td>200</td>
<td>240</td>
<td>160</td>
<td>200</td>
</tr>
<tr>
<td>500 g/t</td>
<td>700 g/t</td>
<td>500 g/t</td>
<td>700 g/t</td>
<td>500 g/t</td>
</tr>
</tbody>
</table>

**AL₂O₃ CONTENT (%)**

- Without milling
- 150 μm
- 74 μm

- 700 g/t
- Mass rec.
Conclusions

• Samples from Copebras/CMOC mineral processing plant were collected and mineralogical characterized.

• The results showed that the main phase present in the middling sample was **apatite** (55.24%), followed by **quartz** (23.34%) and **hematite** (7.33%).

• This result was double-checked by the XRF results.
Conclusions

• Two attractive scenarios were found.
  • The first one, for the non-milled middling, was obtained for the test 1.1, which produced with grade concentrate with *low levels of contaminants*.
  • This test was carried out with the industrially adopted depressant dosage (500 g/t), but a considerably lower collector dosage (160 g/t instead of 320 g/t).
Scenario 1: Proposed flowsheet

Feed → Rougher stage
  - \( \text{pH: 10.7} \)
  - \( \text{Depressant: cornstarch 500 g/t} \)
  - \( \text{Collector: Lioflot 567 200 g/t} \)
  - \( \text{Surfactant: Flotinor 071 100 g/t} \)

  → Cleaner stage
  - \( \text{Depressant 500 g/t} \)
  - \( \text{Collector 160 g/t} \)

  → Scavenger stage
  - \( \text{Collector: Lioflot 567 100 g/t} \)

  → Middling

  → WHIMS

  → Niobium plant

  → Tailings dam

  → Concentrate
The second scenario, for the *milled middling*, was obtained for test 4.2.

Even operating with particle size relatively smaller than the other tests a high recovery (mass and metallurgical) and relatively low level of contaminants were found.

In this particular test, a higher depressant dosage (700 g/t) and lower collector dosage (200 g/t) were used.
Phosphate rock processing flowsheet at Copebras/CMOC in Brazil

Feed → Rougher stage → Cleaner stage

- **Rougher stage**
  - pH: 10.7
  - Depressant: cornstarch 500 g/t
  - Collector: Lioflot 567 200 g/t
  - Surfactant: Flotinor 071 100 g/t

- **Cleaner stage**
  - Collector: Lioflot 567 100 g/t

- **Scavenger stage**
  - Collector: Lioflot 567 100 g/t

- **Middling**
  - Depressant 700 g/t
  - Collector 200 g/t

- **Niobium plant**
- **Tailings dam**
- **Concentrate**

- **WHIMS**
The tests 5.1 and 5.2 (middling B) did not reach the industrial threshold for $P_2O_5$ content, but they showed high metallurgical and mass recoveries and considerably low $P_2O_5$ content in the tailings.

Therefore, this sample is suitable to an additional cleaner stage, which could raise the $P_2O_5$ content in the concentrate, which will be made in a future work.
Conclusions

• The industrial implementation of a milling stage for the flotation circulation load and a subsequent flotation of this material has the potential to *increase the overall process efficiency by approximately 5.5%*, resulting in a production increase of *62 kt/year of phosphate rock concentrate*, with P$_2$O$_5$ content similar to the one currently produced.
Acknowledgements

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Influence of the impeller speed on phosphate rock flotation

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