Recovery of Apatite from Slimes of A Brazilian Phosphate Ore

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Abstract: In general terms, the flowsheet applied to apatite concentration in Brazilian phosphate industry plants involves grinding; classification in coarse and fines fractions; magnetic separation (low and high intensity); desliming to remove particles < 10 μm; barite flotation and apatite flotation. This work shows the results obtained in a study of apatite concentration from a slime sample provided by the industrial phosphate ore plant of the Copebras (China Molybdenum – CMOC International), located at Catalão (Goias state, Brazil). The feed sample containing 13.2% of P₂O₅; 14.0% of CaO; 19.7% of SiO₂ and 27.7 of Fe₂O₃, which corresponds to 32% of apatite, 25% goethite, quartz (18%) and gorceixite (5.2%). Particle size characterization showed that the characteristic diameters (d₃₂, d₁₀, d₅₀ e d₉₀) were respectively 2.6 μm, 1.0 μm, 5.7 μm and 25 μm. Considering the flowsheet with desliming in hydrocyclones (40 mm) in two stages, followed by apatite flotation in columns, it was obtained a final concentrate with 35.6% P₂O₅ and main impurities Fe₂O₃, SiO₂ around 4.0% and 5.1% respectively. Taking into account the mass and metallurgical balances of the desliming and flotation, the overall mass recovery was around 11% and the P₂O₅ recovery 30%.

Keywords: apatite; flotation; slimes; column flotation

Desliming operation with the objective to remove particles less than 20 μm is a common industrial practice in the beneficiation of some kinds of ores, for instance, niobium ores, iron ores and phosphate ores. The reason for separate the slimes fraction of the ore from the coarse particles is correlated to avoid the deleterious effects that ultrafines particles causes on the flotation performance. The difficulty in treatment of ultrafines particles is related to the small mass of these particles, high superficial area and high superficial energy, beyond the fact that usually the slimes fraction presents a large amount of clay minerals, which also causes a strong effect on flotation process. These characteristics leads a low particle/bubble collision efficiency, high reagents consumption, slime coating, high pulp viscosity, difficulty in overcoming the energy barrier between particle and particle and particle and bubble and rigidity of froth.

Desliming operation to discharge the ultrafines particles less than 20 μm prior to concentration by flotation is a common practice on the phosphate ores beneficiations plants, including those that treats sedimentary and igneous ores. Several studies report results from characterization studies and process development focused on concentrate phosphate minerals from slimes fraction of sedimentary and igneous ores.

Zhang and Bogan collected and characterized thirty phosphatic clay (slimes) from four operating plants in Florida in order to identify feasible techniques for recovering of phosphate values. The
studies showed that the mainly characteristics of the slimes are the high content of clay minerals; ultrafine particle size (35%–50% below 1 μm) and the nearly distribution of phosphate among the different size fractions. From the process point on view, the authors suggested that sizing test using 6–inch hydrocyclone showed good results, generating and underflow with particles > 10 μm with 18% P₂O₅, 0.5% MgO and 3.3% Al₂O₃. Preliminary separations tests indicates that the most viable approaches seem to be selective flocculation, sizing followed by flotation and perhaps bioleaching.

Teague and Mollback \cite{10} presents results of a method of beneficiation of ultrafine phosphate which allows the recovery of phosphate particles less than 20 μm up to 80% of the feed. The process uses conditioning with reagents at least 70% solids content and flotation with Jameson cells in a rougher, scavenger, cleaner configuration to recover at least 80% P₂O₅ at a grade of 32%.

The Brazilian phosphate industry has a long-term tradition in beneficiation of weathering igneous phosphate ores. The typical flowsheet applied involves grinding; classification in coarse and fines fractions; magnetic separation (low and high intensity); desliming to remove particles < 10 μm; barite flotation and apatite flotation. The flotation machines used in the circuits are mechanical cells to coarse fraction (d₅₀ ≈ 70 μm) and flotation columns to fine fractions (d₅₀ ≈ 20 μm). An exception of this rule is the industrial plant located at Araxá (MG) that treat both fraction, coarse and fines using columns on the flotation circuit. In terms of flotation reagents, fatty acids are saponified with NaOH to produce soluble soaps that act as apatite collector. Corn starches with different size distributions are employed as gangue depressant \cite{15}.

Regarding to production of phosphate concentrates from slimes, Guimarães and Peres \cite{7-8} resume the Brazilian industrial experience in beneficiation of the material with particle size < 44 μm. The first industrial application was in Araxá concentrator in the beginning of 80 ’s in a circuit consisting of desliming in 40 mm hydrocyclones and apatite flotation in column machines. Corn starch and fatty acids being utilized as reagents. The concept of this process was expanded to other Brazilian plants located in Catalão (Goias state), Tapira (Minas Gerais state) and Cajati (São Paulo state) are still in the 90 ’s. It is estimated that the apatite concentrate from slimes represents 11% to 13% of the overall production.

This work showed the results obtained with a slime sample collected from the plant of the Copebras (CMOC International) that process the phosphate ore from the mine located at Catalão, Goias state, Brazil. The flowsheet involves desliming in hydrocyclones in two stages, followed by apatite flotation in columns. The results include a comparison of the flotation performance between different size columns size for rougher flotation, one with 3” in diameter (2 meters in height) and the other one with 4” in diameter and 7 meters in height.

1 Experimental

1.1 Ore Sample

The slime sample was collected from the overflow of the hydrocyclones, a stage at where the material is discharged in the tailings dam. It was collected around 10 tons (dry basis) of the slime in pulp content 16% solids in weight. The pulp was storage in containers with 1 m³ and it was send 44 of these containers to CETEM in Rio de Janeiro. Fig. 1 shows the operation of the sample discharge in CETEM.

![Sample discharge in CETEM to apatite concentration](image1)

The sample characterization includes: chemical characterization (global sample); mineralogical characterization, Rietveld Method (global sample); particle size distribution; specific gravity determination.

The chemical analyses were carried out using X Ray-Fluorescence technique and the mineralogical
characterization was carried out using X-Ray-Diffraction technique associated with Rietveld refinement to identification and quantification of mineral species. The particle size distribution was determined by the laser diffraction technique using a Malvern Master sizer particle size analyzer and the specific gravity was determined by gas Helium pycnometry technique.

1.2 Reagents

Soybean oil soap was used as the collector of apatite obtained after saponification of the soybean oil for a period of 15 minutes with an fatty acid/NaOH ratio of 5:1. Gelatinized corn starch was used as the gangue mineral depressant with a starch/NaOH ratio of 4:1 and a reaction time of 10 minutes at 20% solids by weight. Both reagents were prepared with a 1% concentration of distilled water. For pH adjustment, a 10% NaOH solution was used. The tap water supply from the city of Rio de Janeiro was used to attain the correct percentage of pulp solids. Tab. 1 shows the reagents, its applications and dosages in apatite flotation.

<table>
<thead>
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<th>application</th>
<th>dosage</th>
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<td>150–350 / (g/t)</td>
</tr>
<tr>
<td>corn starch</td>
<td>depressant</td>
<td>800–3500 / (g/t)</td>
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<tr>
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<td>gelatinization, fatty acid</td>
<td>pH=9.5</td>
</tr>
<tr>
<td></td>
<td>saponification</td>
<td></td>
</tr>
</tbody>
</table>

1.3 Desliming

Each container containing 1 m³ of the slime sample was connected to a vertical pump with a system that allows pulp recirculation to the container and also to a tank with 3.6 m³ [Fig. 2 (a)]. In this 3.6 m³ tank, water was added to adjust the solids containing in the pulp to around 8%, which represent the solids to the feed of the desliming stage. From the storage tank, the pulp was pumped to the hydrocyclone apparatus [Fig. 2 (b)]. The desliming was carried out in two stages, where the underflow from the first stage feed the second stage. The underflow flow of the second stage feed the apatite flotation and the overflow from the both stages were discharge as tailings. The hydrocyclone used was supplied by Weir Minerals. The apex finder was 7 mm in the first stage and 5.5 mm in the second stage. In both stages the vortex finder was 10 mm.

1.4 Rougher flotation studies (3 inches diameter column flotation)

The rougher apatite flotation tests were carried out in a laboratory column with an internal diameter of 3 inches, a height of 2.0 m, and an effective volume of 6.8 L, consisting of three adjustable modules. The pulp with 35%–36% solids by weight was conditioned in four flotation cells at the Mini Pilot Plant (MPP-Supplied by Eriez), being two for depressant (gelatinized corn starch) and the other two for soybean oil soap as the collector. The average residence time in conditioning was around 8 minutes for both, the collector and depressant. The pH was adjusted using NaOH in 9.5. After conditioning, the pulp was diluted to 20% solids by weight and it was fed straight to the column. The height of the froth layer was set at 30 cm, and it was controlled by varying the tailings discharge flowrate. The superficial velocity of the feed flow was set at 0.66 cm/s, the air was set at 0.49 cm/s and the wash water was 0.3 cm/s. After flotation runs around 60 minutes, the sample from the concentrate and tailings was collected for 10 minutes. The samples were flocculated, oven dried at 100 °C for 12 hours, and then separated in aliquots for chemical analysis. The effects that the collector and depressant dosages had on the mass recovery, P₂O₅ recoveries, P₂O₅ grade and impurity levels (Fe₂O₃ and SiO₂) were evaluated. Fig.3 shows the column flotation applied to the rougher flotation studies.
1.5 Rougher/Cleaner apatite flotation studies
(2" and 4" diameter column flotation)

The rougher/cleaner flotation trials were performed using a column with an internal diameter of 4 inches, a height of 7.0 m, and an effective volume of approximately 46 L, composed of acrylic modules joined by flanges for rougher stage. The froth obtained in the rougher feed the cleaner stage that was carried out in a column flotation with 2 inches in diameter and 7.0 m height (13 L volume). The rougher and cleaner tailings streams were final discharge as final tailings. The pulp was conditioned with gelatinized corn starch and the pH was adjusted (9.5) with NaOH and subsequently conditioned with soybean oil soap in two cylindrical tanks. After conditioning, the pulp was diluted to 20% of solids by weight and fed at 1.26 meters from the top of the rougher column. The froth obtained in rougher feed the cleaner and both tailings were discharge as final tailings.

Bubbles were generated with controlled pressure and flow via a forced air passage in a porous pipe at the base of the column. The pulp/froth interface was controlled by a level sensor that was attached to the tailings pump. After reaching the stationary stage, product samples of the rougher and cleaner tailings and cleaner concentrate were collected, flocculated, and dried in an oven for 24 hours at 80 ℃. Then, the samples were weighed, disaggregated, and separated in aliquots for chemical analysis by X-ray fluorescence spectroscopy to determine metallurgical balance. The effect of collector and depressant dosages on mass and metallurgical recovery of apatite, P₂O₅ concentrate grades, and impurities (Fe₂O₃ and SiO₂) contents was measured. Fig. 4 shows the schematic flowsheet for the rougher/cleaner flotation tests.

2 Results and discussion

2.1 Sample characterization

Fig. 5 shows the particle size distribution of the slime sample considering the results obtained by screening and laser diffraction. The characteristics diameters D₃₂, D₁₀, D₅₀, D₉₀ were respectively 2.6 μm, 1.0 μm, 5.7 μm and 24.7 μm. Tab. 2 shows the chemical characterization results to the global sample. The P₂O₅ grade is around 13% and CaO around 14%. In terms of the major contaminants, stands out the SiO₂ and Fe₂O₃ with grades of 19.7% and 27.7% respectively. Figure 6 illustrates the results of the mineralogical characterization of the
slime sample. The main mineral phases in the sample are apatite (around 33%), goethite with 26% grade content and quartz with 18%.

2.2 Desliming

The desliming studies included the evaluation of many variables, including type of hydrocyclone (supplier), different size of apex and vortex finder and pressure of operation. Despite this extended study, only the results of the final configuration are shown in this work. The mass and metallurgical balance for P₂O₅ to the optimized condition is shown in Fig. 7. The final configuration of desliming stage includes two stages, with hydrocyclone at the same size (40 mm in diameter) but with different apex finder diameters and operation pressure.

The first stage is carried out in equipment with vortex finder in 10 mm and apex finder in 7 mm and the operation pressure is 4 kgf/cm². The feed containing 8% solids in weight produces an underflow with 16% solids in weight and an overflow with 2%–3% solids in weight, which is discharge as final tailings. The d50 of the overflow stream is around 3 μm and to underflow is 10 μm. The mass and P₂O₅ recoveries to underflow stream are 70% and 78% respectively. It is observed and enrichment in P₂O₅ in underflow from 13.3% at the feed to 14.8%. The P₂O₅,
in the overflow is 9.7%.

The underflow of the first stage feeds the second stage that is carried out in a hydrocyclone with 40 mm in diameter, vortex finder in 10 mm and apex finder with 5.5 mm. The operation pressure is in 3 kg/cm². The solids containing at the overflow of the second stage is 4%-5%, d50 5 μm and the P₂O₅ grade in 11% and is discharged at final tailings together with the overflow produced at the first stage. The solids content at the underflow of the second stage is 35%-36%, d50in 18 μm and the P₂O₅ grade 16.4%. The mass and P₂O₅ recoveries at the second stage are very similar to the first stage, reaching 70% and 77%.

The underflow obtained at the second stage feeds the conditioning circuit and after this the flotation circuit. Considering both stages, the overall mass recovery is around 50% and the P₂O₅ recovery at 60%.

2.2 Flotation studies

This topic presents the results and discussion of the apatite flotation studies considering rougher flotation in 3″ diameter column with the objective to produce a final apatite concentrate (> 34.5% P₂O₅), and also those obtained when rougher/cleaner configuration was applied.

Fig. 8 (a) shows the curve P₂O₅ grade versus recovery considering apatite rougher flotation in 3″ diameter flotation column. As observed, the P₂O₅ recovery ranged from 70% to 25% with P₂O₅ grade variation from 27% to almost 38% and mass recoveries ranged from 10.5% to 45%. It is important to mention that the results obtained refer to all the tests carried out, even with the desliming condition not optimized, which had an effect on the flotation performance. The P₂O₅ grade/recovery curves to rougher and cleaner stages are presented in Figure 8 (b). All tests were carried out with the pulp after desliming in the optimized conditions. It can be observed that the results obtained in cleaner stage showed a distribution higher than the rougher. This can be explaining by the fact that cleaner flotation had more process variables tested focused on optimization of this stage. For rougher, the P₂O₅ recoveries ranged from 85% to 40% and the variation of the P₂O₅ grade was from 20% to 35%. For cleaner, P₂O₅ grade and recoveries ranged from 25% to 37% and from 45% to values higher as 94% respectively.

![Fig. 8 Curve grade/recovery of P₂O₅: (a) Apatite flotation in 3″ diameter column flotation; (b) Apatite flotation with rougher/cleaner configuration in 4″ and 2″ diameter columns flotation respectively](image)

Comparative results of the P₂O₅ grade/recovery curve considering the rougher flotation on 3″ diameter column flotation (2 meters height – 5.5 kg/h solids) and 4″ diameter (7 meters height – 35 kg/h solids) are illustrated at Fig. 9. It is observed that the trend of the results is very similar for both scales which indicates that the flotation performance on columns can be defined by test at relatively small scales.

Fig. 10 shows the effect of depressant dosage on P₂O₅ grade and recovery considering dosages of 1 270 g/t, 1 730 g/t and 2 690 g/t and collector dosage of 120 g/t, 180 g/t and 240 g/t at pH 9.5. These flotation tests were carried out with the sample after desliming on the optimized conditions. From the Fig. 10, it can be seen that the results (grade and recovery) improve when the depressant dosage
increase from 1230 g/t to values higher than 1700 g/t. The apatite concentrates obtained when 2690 g/t depressant dosage was used, showed higher P$_2$O$_5$ grade in comparison with lowest depressant dosage. In other hand, P$_2$O$_5$ recoveries were much lower, reached 10% minus at the same collector dosage. For dosages higher than 3400 g/t and 120 g/t of collector was observed a lower grade of P$_2$O$_5$ in the concentrate.

Fig. 9 shows the relation between the P$_2$O$_5$ grade in the final apatite concentrate and the levels of impurities Fe$_2$O$_3$ and SiO$_2$. Considering the P$_2$O$_5$ grade range from 27% to 37%, it can be observed that the SiO$_2$ grade is always higher than the Fe$_2$O$_3$. To P$_2$O$_5$ grade in 27%, the SiO$_2$ content in concentrate is around 18% and Fe$_2$O$_3$ 10%. To the P$_2$O$_5$ grade around 35%, the Fe$_2$O$_3$ content in the concentrate is 4% and to SiO$_2$ the values reached is around 6%. These results indicates that the mainly contamination of the apatite concentrate it will be caused by the SiO$_2$ bearing minerals.

Fig. 11 shows the relation between the P$_2$O$_5$ grade in the final apatite concentrate and the levels of impurities Fe$_2$O$_3$ and SiO$_2$. Considering the P$_2$O$_5$ grade range from 27% to 37%, it can be observed that the SiO$_2$ grade is always higher than the Fe$_2$O$_3$. To P$_2$O$_5$ grade in 27%, the SiO$_2$ content in concentrate is around 18% and Fe$_2$O$_3$ 10%. To the P$_2$O$_5$ grade around 35%, the Fe$_2$O$_3$ content in the concentrate is 4% and to SiO$_2$ the values reached is around 6%. These results indicates that the mainly contamination of the apatite concentrate it will be caused by the SiO$_2$ bearing minerals.

Tab. 3 shows a resume of the best results achieved in apatite rougher flotation at 3” diameter column flotation. Considering the average results of five tests, mass recovery was 19%, P$_2$O$_5$ recovery and grade at 42.5% and 35.7% respectively. The impurities levels SiO$_2$ and Fe$_2$O$_3$ were 5.1% and
4.0%. Reagents consumptions in terms of feed solids rate were 141 g/t to collector, 2.300 g/t to depressant and 410 g/t to NaOH. A resume of the best results achieved using rougher/cleaner configuration is shown at Table 4. The average mass and P₂O₅ recoveries were around 23% and 52% to P₂O₅ grade on 35% (SiO₂ and Fe₂O₃).

Table 3 Resume of best results obtained in experiments at 3” diameter column flotation. Rougher flotation. Feed solids rate: 5.5 kg/h (dry basis)

<table>
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<th>test</th>
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<tr>
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<td>42.1</td>
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<td>42.4</td>
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<td>18.8</td>
<td>41.9</td>
<td>36.2 4.6 4.5 141 2.756 428</td>
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<tr>
<td>Average</td>
<td>19.1</td>
<td>42.5</td>
<td>35.7 4.0 5.1 141 2.334 411</td>
</tr>
</tbody>
</table>

Table 4 Resume of best results obtained in experiments at 4” and 2” diameter columns flotation. Rougher/cleaner configuration( Feed solids rate: 35 kg/h)

<table>
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<th>test</th>
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<tr>
<td>Average</td>
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<td>35.2 3.0 5.2 117 2.783</td>
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Different concepts has been proposed to concentrate valuables minerals from slimes of niobium, iron and phosphates ores. A similar approach applied in this study was developed by Thella et al[1] to concentrate Fe bearing minerals from iron ore slimes from India. The process is based on a combination of hydrocyclones and flotation to produce Fe concentrates with grades of 64.46% Fe, 2.66% Al₂O₃, and 2.05% SiO₂, from a feed sample assaying 55%–60% Fe and 6%–8% alumina. The authors concluded that the fine particle size, complex mineralogy and presence of locked particles determine the impossibility of direct production of hematite concentrate by froth flotation, making the desliming prior to flotation necessary. Classification of slimes with two stage hydrocyclone gives a concentrate containing mass fraction 61.99% Fe, 4.0% Al₂O₃, and 3.22% SiO₂, with Fe recovery of 54.55% in Stage–I. Concentrate from Stage–II hydrocyclone contains mass fraction 62.19% Fe, 3.45% Al₂O₃, and 2.79% SiO₂ with Fe recovery of 47.45% with respect to initial feed. Further the concentrate from Stage–II hydrocyclone is beneficiated using reverse cationic flotation by using amines and direct flotation with fatty acid. A final concentrate of mass fraction 64.46% Fe, 2.66% Al₂O₃, and 2.05% SiO₂, with Fe recovery of 69.03% in flotation stage and 34.13% with respect to the initial feed was obtained with reverse cationic flotation. The metallurgical Fe recovery obtained by these authors (34.1%) is very similar to obtained in this study (30%) using a very similar flowsheet to processing different ores. It was also observed in this study the necessity of desliming prior to apatite flotation. Mineralogical characterization showed a decrease in content of deleterious minerals from feed to underflow produced from second stage, for instance, the goethite content decrease from 33% on the feed to values around 12% at the underflow II. The same trend it is observed with kaolinite, which decrease from 4% to values lower than 1%. By the other side, quartz content on the flotation feed is higher when compare with the feed sample which explains the high SiO₂ grade in the final concentrates.

Ipek and Ozdag[2] compare the flotation performance between a column (6.25 cm cross-sectional area and 185 cm height) and mechanical cells to recover phosphate from Mazidag phosphate plant tailings. The particle size distribution of the sample showed that 80% are under 22 µm. Although a mass fraction 31.50% P₂O₅ concentration with 78.80% recovery was obtained in column flotation, a 29.58% P₂O₅ concentration was obtained with 76.69% recovery in cell flotation.

In opposition to the sustained by the works mentioned above and with the results obtained in this study, Teague and Lollback[10] discuss a novel method of beneficiation of ultrafine phosphate which allows the recovery of phosphate particles that are less than 20 µm. The authors describe a method that is in contrast with the established methods for
beneficiation of phosphates were classification by hydrocyclones is mainly used to remove ultrafines as tailings. The study includes an investigation of a number of variables and their effect on the flotation recovery of ultrafine phosphate, including the pulp density and water quality during conditioning and flotation, type of flotation machine and reagents used to depress Fe$_2$O$_3$ and Al$_2$O$_3$. The results achieved using samples containing up to mass fraction 75% less than 20 μm particles, including for example 91.2% P$_2$O$_5$ recovery to a concentrate grade of 34.7% P$_2$O$_5$ from a low feed grade of 6.46% P$_2$O$_5$ and 92.4% P$_2$O$_5$ recovery to a concentrate grade of 30.2% P$_2$O$_5$ from 10.6% P$_2$O$_5$ feed. Guar gum was found to be the most effective depressant for Fe$_2$O$_3$, whilst the Al$_2$O$_3$ was determined to be hydrophilic, resulting in low amounts being recovered to the concentrate, regardless of whether a depressant was used or not. The final process concept uses conditioning with reagents at high wt% solids (at least mass fraction 70%) and flotation with Jameson cells in a rougher, scavenger, cleaner configuration to recover mass fraction at least 80% P$_2$O$_5$ at a grade of 32% P$_2$O$_5$, or greater. The Jameson cell was found to have an advantage over conventional flotation cells when treating ultrafine particles, due to their dense mixing zone and propensity to form small bubbles. The use of de-ionised water in the process was also found to be important to minimize the concentration of hard cations that could activate silica and hence adsorb collector, thereby interfering with collector adsorption onto phosphate, decreasing its floatability. Some operations conditions used in the study of Teague and Lollhack are difficult to reproduce to the flowsheet to apatite concentration from slimes generates at the industrial plants. The maximum solids content possible to obtain to conditioning with slimes is around 35%–37%. Values higher than this means huge apatite losses on the overflow of the hydrocyclones and also is a quite difficult pulp agitation at this solids content. Another point is the use of de-ionised for their study. It is well know the effect of the process water quality under the apatite flotation as showed by Guimarães and Peres[19], Santos[18].

3 Conclusions

A conceptual flowsheet consisting in desliming in two stages followed by column flotation was established for apatite concentration from slimes using a sample from the industrial plant concentration of Copebras (CMOC International), Catalão (Goias, Brazil). From a feed containing 13.3% P$_2$O$_5$, (Fe$_2$O$_3$ and SiO$_2$ mass fraction 27% and 20%), apatite concentrates analyzing 35% P$_2$O$_5$ mass fraction (3.0% Fe$_2$O$_3$ and 6.0% SiO$_2$) were produced applying a flotation circuit with rougher/cleaner configuration. The overall mass and P$_2$O$_5$ mass fraction recoveries were 24.1% and 54% respectively. It is estimate that the earnings obtained by recovery of the apatite from slimes may represent an increase in 4% of the overall P$_2$O$_5$ recovery at the industrial plant. It is important emphasize that efforts made by the companies with the objective to improve apatite recovery from the industrial plants through the study and implementation of new circuits has a positive impact in the Brazilian phosphate industry that still import 50% of the total consumption of phosphorus fertilizers. Besides, the improvement in apatite concentrates production from the tailings of the operations plants brings the benefit of preservation of the phosphate ores reserves.

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