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## Pilot Scale Direct Flotation of a Phosphate Ore with Silicate-Carbonate Gangue

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### Abstract

The present pilot scale study addresses the direct flotation route for the concentration of a phosphate ore bearing a silicate-carbonate gangue. The target was to selectively separate apatite from a phosphate ore bearing silicate/carbonate gangue using flotation columns. Based on the results of a previous laboratory scale investigation, a reagents scheme was selected and tested, using, under alkaline conditions, corn starch and a natural collector extracted from the distillation of coconut oil. An open rougher-cleaner circuit yielded a final concentrate reaching 30.5% P<sub>2</sub>O<sub>5</sub> grade, at 80.8% recovery level.

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**Keywords:** column flotation; flotation reagents; flotation collectors; anionic flotation; phosphate flotation

### 1. Introduction

The development of process routes to concentrate phosphate ores bearing silicate carbonate gangue is one of the major challenges for the fertilizers industry. This association corresponds to a significant fraction of the Brazilian reserves from igneous origin. The performance of the available process routes is generally poor and in many instances the route is still unknown. The flotation routes applied to igneous origin phosphates are different from those used in the processing of sedimentary phosphates. This type of phosphate ore is present in reserves of the states Minas Gerais (Araxá and Tapira), Goiás (Catalão), and Ceará (Itaitaia).

The poor selectivity in flotation systems involving semi-soluble minerals is due to similarities in solubility, electrokinetic and surface chemistry properties [1].

The non-thio ionizable collectors used in phosphate ores flotation are derivatives from carboxylic acids, sulfosuccinates, sulfosuccinamates, sarcosinates, hydroxamates, among other [2]. The chain length ranges from 6 to 18 carbon atoms and the main properties are proneness to hydrolysis or dissociation (ruled by solution pH), lowering of the surface tension at the air liquid interface and trend to micelles formation in the case of long chain homologues.

Most of the industrial routes discussed in the literature consist of apatite direct flotation. The plant practice at Bunge's Cajati concentrator was described by [3]. The gangue consists basically of calcite, dolomite, and magnetite. Coarse and fine fractions of ore are floated under alkaline conditions in the presence of sarcosinate (collector) and corn starch (gangue

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depressant). The same collector is used at the Siilinjärvi concentrator, in Finland [4]. This apatite bearing igneous ore presents flogopite, calcite, and dolomite as gangue minerals.

The present investigation was preceded by a laboratory study [5] addressing apatite direct flotation with natural collectors (coconut oil and soy bran oil) and synthetic collectors (Aero 6493, hydroxamate Cytec; Eumulgin MC-711, sulfosuccinate Cognis; Berol, sarcosinate, Akzo Nobel; Flotisor 2875, sulfosuccinamate, Clariant).

Coconut oil was the most selective among those tested. Its composition expressed as carboxylic acids is lauric (40% to 50%), myristic (16% to 20%), oleic (8% to 16%), palmitic (8% to 12%), capric (4% to 8%), caprylic (3% to 7%), stearic (2% to 6%) and linoleic (15 to 3%). This composition differs from that of tall oil, soy bran oil and rice bran oil in which oleic and/or linoleic acids predominate [3], [6].

The results presented refer to column flotation pilot scale tests based on process conditions defined in the laboratory to identify a reagents schedule leading to the selective separation between apatite and the gangue minerals.

## 2. Materials and methods

The ore sample came from the Itataia reserve, Ceará state, Brazil and was processed at the Center of Nuclear Technology Development.

The size analysis was performed via a combination of wet screening (Tyler series) and the particle size analyzer Sympatec Helos (Laser principle). The determination of  $P_2O_5$  and  $SiO_2$  grades was done via X-ray fluorescence (EXD-720, Shimadzu) and  $CaCO_3$  was analyzed via calcination at  $950^\circ$ . The semi-quantitative mineralogical characterization was performed by the X-ray diffraction powder method (Rigaku equipment, model Geigerflex).

Figure 1 illustrates the flowsheet of the pilot unit used in the rougher tests. The column dimensions are 600 cm height and 5.1 cm diameter. The ore was fed at constant rate (approximately 15 kg/h) into tank CN-1 for depressant and pH modulator conditioning, flowing by gravity into tank CN-2 for collector and frother conditioning. The pulp was then fed into the column CO. The reagents used, supplier in brackets, were: non-modified corn starch (depressant from Dutch Starches International), fatty acids from coconut oil (collector from Miracema), ether polyalkylene glycol (Flotanol D-25 - frother from Clariant), sulfosuccinate (Eumulgin MC-711 - collector from Cognis), and sodium hydroxide.

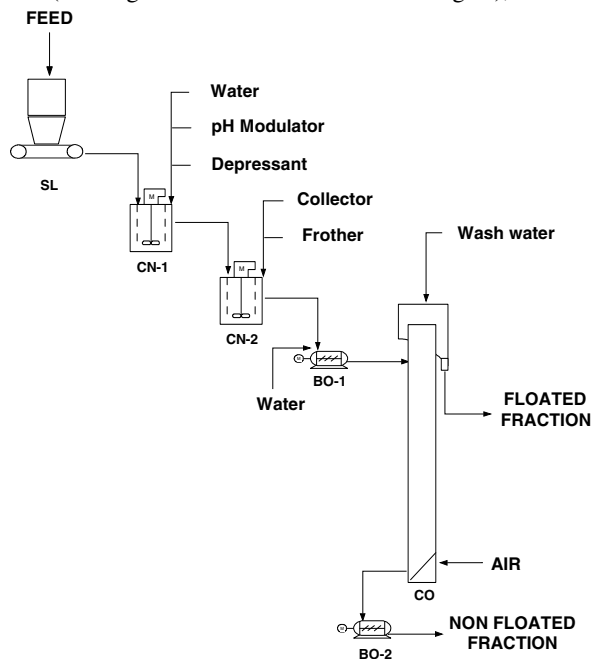


Figure 1. Rougher flotation circuit flowsheet.

### 3. Results and discussion

The size distribution, illustrated in figure 2, indicates 2.9% retention in 210  $\mu\text{m}$  and 32.7% passing 37  $\mu\text{m}$ , adequate for a high liberation degree of apatite [7].

The  $\text{P}_2\text{O}_5$ ,  $\text{CaCO}_3$ , and  $\text{SiO}_2$  contents are, respectively, 17.34%, 20.78%, and 24.14%.

The identified gangue minerals are calcite and silicates (albite, quartz, biotite, phlogopite, montmorillonite, vermiculite and amphiboles).

The first stage of the flotation tests aimed at optimizing collector (coconut oil) and depressant (corn starch) dosages. Then the additions of Eumulgin MC-711 and Flotanol D-25 were tested aiming to maximize the  $\text{P}_2\text{O}_5$  recovery. The next step was to optimize the air superficial velocity and the solids feed rate. Finally, open circuit rougher cleaner tests were performed.

The effect of coconut oil dosage on flotation response, in the range between 410 g/t and 960 g/t usage, is illustrated in figure 3. This variable affects significantly the  $\text{CaCO}_3$  recovery. For a collector dosage of 410 g/t the  $\text{P}_2\text{O}_5$  content in the concentrate reached 30.0%, at 74.2% recovery.

The effect of corn starch dosage, in the range between 589 g/t and 903 g/t is illustrated in figure 4.  $\text{P}_2\text{O}_5$  grade and recovery are not affected by this variable and  $\text{CaCO}_3$  grade and recovery decrease slightly for higher depressant dosages. For a depressant dosage of 707 g/t the  $\text{P}_2\text{O}_5$  content in the concentrate reached 27.7%, at 75.6% recovery. The selective depressant action of corn starch towards calcite was not observed in investigation on the flotation performance of the phosphate ore from Copebras [8].

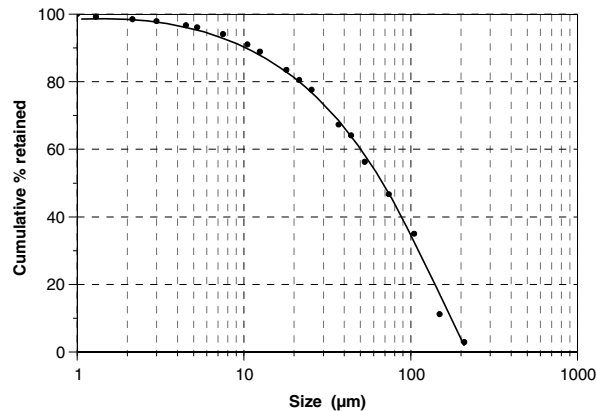


Figure 2. Head sample size distribution.

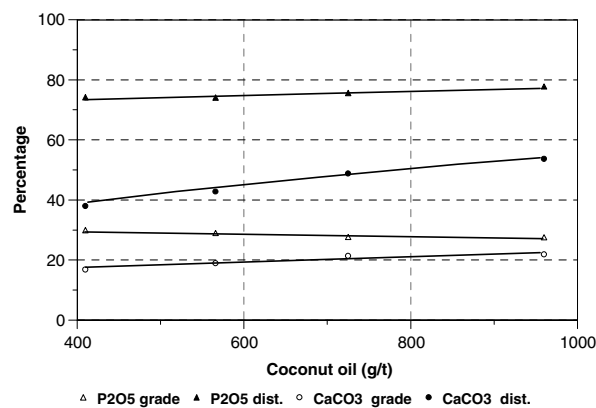


Figure 3. Effect of coconut oil dosage on  $\text{P}_2\text{O}_5$  and  $\text{CaCO}_3$  grade and recovery.

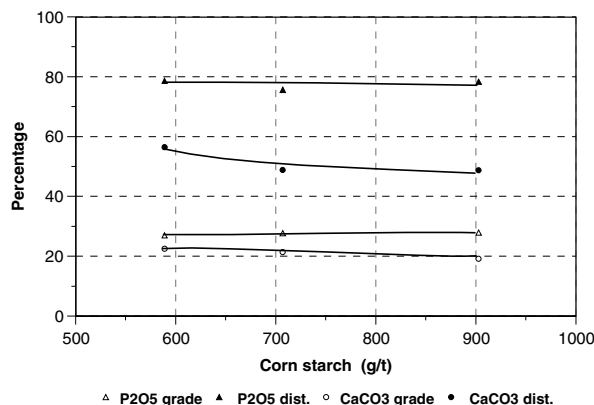


Figure 4. Effect of corn starch dosage on P<sub>2</sub>O<sub>5</sub> and CaCO<sub>3</sub> grade and recovery.

The effect of Flotanol D25 dosage, in the range between 0 g/t and 30 g/t is illustrated in figure 5. This variable increased significantly both P<sub>2</sub>O<sub>5</sub> and CaCO<sub>3</sub> recovery. The dosage increase causes a slight drop in P<sub>2</sub>O<sub>5</sub> grade leading to higher CaCO<sub>3</sub> content. For the frother dosage 16 g/t the P<sub>2</sub>O<sub>5</sub> content in the concentrate reached 31.0%, at 74.2% recovery.

The effect of Eumulgin MC-711 dosage, in the range between 0 g/t and 32 g/t is illustrated in figure 6. This variable increased significantly both P<sub>2</sub>O<sub>5</sub> and CaCO<sub>3</sub> recoveries at the expense of selectivity loss. The dosage increase yielded a significantly higher CaCO<sub>3</sub> content in the concentrate.

The effect of the air superficial velocity, in the range between 1.75 cm/s and 2.25 cm/s is illustrated in figure 7. This variable affected significantly both recoveries, with slight impact on the grades. For the velocity 2.25 cm/s the P<sub>2</sub>O<sub>5</sub> content in the concentrate reached 30.8%, at 74.9% recovery.

The effect of the solids feed rate, in the range between 9.5 kg/h and 15.5 kg/h, is illustrated in figure 8. This variable decreased significantly both recoveries. The increase in the rate resulted in higher P<sub>2</sub>O<sub>5</sub> grade and lower CaCO<sub>3</sub> grade. Higher recoveries at lower feed rates are due to the increased pulp residence time in the column. For a feed rate of 9.5 kg/h the P<sub>2</sub>O<sub>5</sub> content in the concentrate reached 29.0%, at 82.5% recovery.

The final stage was performing open circuit rougher/cleaner tests based on optimized rougher conditions. The dimensions of the rougher column are 10.2 cm diameter and 547 cm height and those of the cleaner column are 5.1 cm diameter and 580 cm height. The results, presented on table 1, show P<sub>2</sub>O<sub>5</sub> grades in the range between 30.5% and 36.1%, at recovery levels in the range between 62.8% and 80.8%. The decreases in pH from 10.0 to 8.0 and in air superficial velocity from 2.0 cm/s to 1.6 cm/s led to higher P<sub>2</sub>O<sub>5</sub> grade at the cost of decreased recovery. The cleaner stage is important to decrease the contaminants (CaCO<sub>3</sub> and SiO<sub>2</sub>) contents in the final concentrate. Figure 9 illustrates the mass and metallurgical balances of test T-04, performed under optimized conditions. The P<sub>2</sub>O<sub>5</sub> grade in the concentrate reached 30.5%, at 80.8% recovery, and CaCO<sub>3</sub> and SiO<sub>2</sub> contents, respectively, 15.2% and 7.1%.

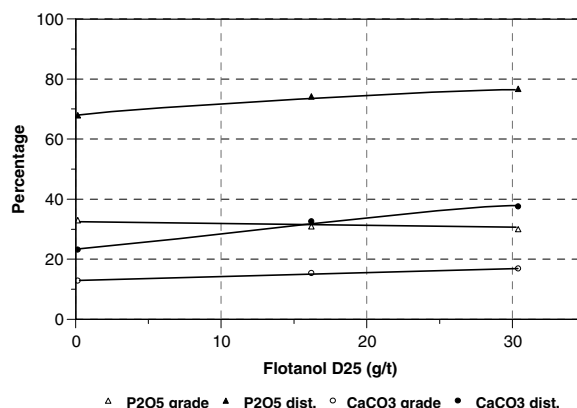
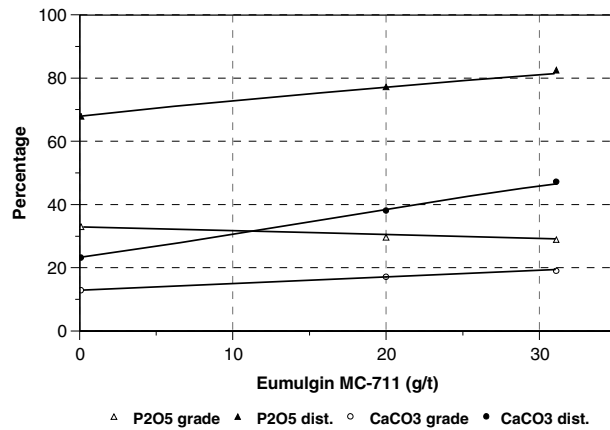
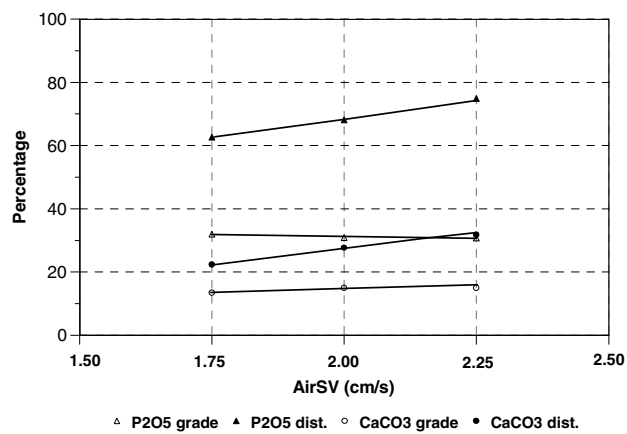
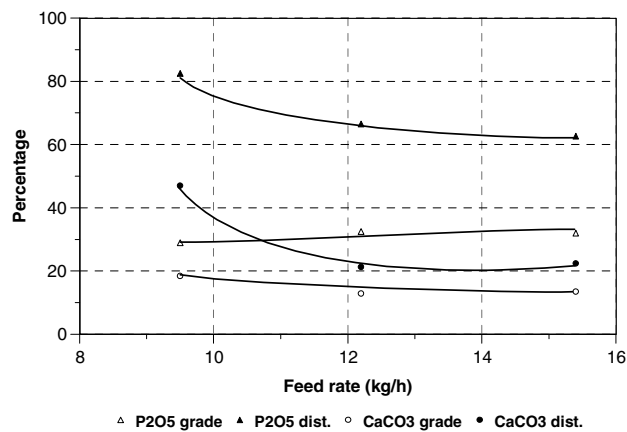


Figure 5. Effect of Flotanol D25 dosage on P<sub>2</sub>O<sub>5</sub> and CaCO<sub>3</sub> grade and recovery.

Figure 6. Effect of Eumulgin MC-711 dosage on  $P_2O_5$  and  $CaCO_3$  grade and recovery.Figure 7. Effect of the air surface velocity on  $P_2O_5$  and  $CaCO_3$  grade and recovery.Figure 8. Effect of the solids feed rate on  $P_2O_5$  and  $CaCO_3$  grade and recovery.

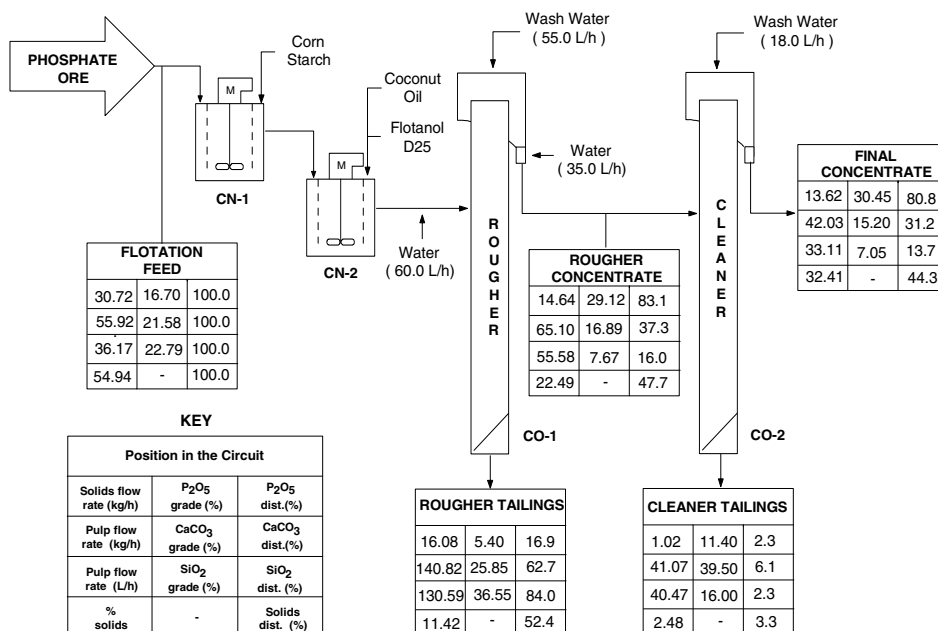


Figure 9. Mass and metallurgical balance of test T-04.

Table 1. Conditions and results of the rougher/ cleaner open circuit tests

Test	Cleaner conditions		Grade %			Recovery %			
	AirSV	pH	P <sub>2</sub> O <sub>5</sub>	CaCO <sub>3</sub>	SiO <sub>2</sub>	weight	P <sub>2</sub> O <sub>5</sub>	CaCO <sub>3</sub>	SiO <sub>2</sub>
T-01	2.0	10.0	30.5	15.2	7.0	44.3	80.8	31.2	13.7
T-02	2.0	8.0	36.1	6.8	4.6	29.6	62.8	10.4	6.1
T-03	1.6	10.0	33.0	11.1	5.0	41.1	75.1	24.0	9.6

#### 4. Conclusions

The use of reagents scheme consisting of corn starch and coconut oil was effective for the separation between apatite and the contaminants calcite and silicates present in this phosphate ore from igneous origin.

Corn starch is a selective depressant for the gangue minerals.

The operation variables solids feed rate and air superficial velocity, as well as the process variables Flotanol D25 dosage, significantly affect the flotation performance.

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