

---

## Bench scale and pilot plant flotation tests with phosphate rock from the Alto Paranaíba Region/MG, Brazil

Received: 10-08-2024 | Accepted: 15-09-2024 | Published: 23-09-2024

---

### **Sheila Silva Andrade**

ORCID: <https://orcid.org/0009-0008-7497-5181>

Especialização em Tratamento de minérios, Universidade Federal de Catalão, Brazil & RJMG Óleo Química

E-mail: [sheilasilvaandrade.eng@gmail.com](mailto:sheilasilvaandrade.eng@gmail.com)

### **Luciano José de Barros Abreu**

ORCID: <https://orcid.org/0009-0002-3055-3450>

RJMG Óleo Química

E-mail: [lucianoabreu@rjmg.com.br](mailto:lucianoabreu@rjmg.com.br)

### **Sebastião Eduardo Rezende**

ORCID: <https://orcid.org/0009-0001-3865-972X>

RJMG Óleo Química

E-mail: [sebastiaoeduardo@rjmg.com.br](mailto:sebastiaoeduardo@rjmg.com.br)

### **Alfredo Pio Coelho Oliveira**

ORCID: <https://orcid.org/0009-0004-7692-7299>

RJMG Óleo Química

E-mail: [alfredopio@rjmg.com.br](mailto:alfredopio@rjmg.com.br)

### **Elenice Maria Schons Silva**

ORCID: <https://orcid.org/0000-0003-1360-6450>

Laboratório de Modelamento e Pesquisa em Processamento Mineral, Universidade Federal de Catalão, Brazil

E-mail: [eschons@ufcat.edu.br](mailto:eschons@ufcat.edu.br)

### **André Carlos Silva**

ORCID: <https://orcid.org/0000-0002-9760-0728>

Laboratório de Modelamento e Pesquisa em Processamento Mineral, Universidade Federal de Catalão, Brazil

E-mail: [ancarsil@ufcat.edu.br](mailto:ancarsil@ufcat.edu.br)

---

### ABSTRACT

This paper highlights the significant advances in phosphate recovery in the Alto Paranaíba region, Minas Gerais, as a result of collaboration between RJMG and the local mining industry. The applied methodology focused on the development and use of the innovative AGEM B plant collector, going from laboratory tests to pilot plant tests. The results demonstrated the superiority of AGEM B compared to standard collectors, where a 14.1% increase in P<sub>2</sub>O<sub>5</sub> recovery and a 40% reduction in collector dosage were observed. On a pilot scale, the average P<sub>2</sub>O<sub>5</sub> content in the rougher tailings was 1.66 ± 0.29%, 38.40% lower than that obtained with the collector used by the mining company (a fatty acid) which presented an average content of 2.69 %. The average P<sub>2</sub>O<sub>5</sub> content in the recleaner concentrate was 32.15 ± 2.59%, 8.27% higher than that obtained with fatty acid (average of 35.05%). Increases of 43.59% in mass recovery (average of 11.20 ± 1.31%) and 36.28% in metallurgical recovery (average of 71.00 ± 4.82%) were observed. This work highlights the importance of innovation and strategic collaboration to overcome challenges in mineral processing, recommending the use of AGEM B on an industrial scale for better efficiency and sustainability in the phosphate recovery process.

**Keywords:** Phosphate rock, Vegetable collector, Flotation, Low grade ore, Innovation.

---

## INTRODUCTION

Phosphate rock represents a vital resource that is fundamental to humanity, since it is one of the main sources of phosphorus (P), an element that, together with potassium (K) and nitrogen (N), is part of the group of so-called primary macronutrients, which are essential for the growth and development of plants. In addition, P is also an important nutrient in animal feed since it participates in cell renewal processes. Phosphate rock is also used in the detergent and food industries. Apatite ( $\text{Ca}_5(\text{F,Cl,OH})(\text{PO}_4)_3$ ) is the mineral-ore of phosphorus (with  $\text{P}_2\text{O}_5$  content ranging from 5 to 15%) and is almost always present in carbonatite rocks. The growing global concern with food production, necessary to meet population growth, has shown that there is a real increase in the demand for P, making it necessary to optimize the processes of extraction, processing and recovery of the same, in addition to reducing the costs associated with the process (SILVA, 2018). Brazilian phosphate reserves are around 4,315,000 Mt, which represents only 0.5% of the global total.

Basic fertilizers can be listed as follows: MAP or monoammonium phosphate (with a  $\text{P}_2\text{O}_5$  content of 48%); DAP or diammonium phosphate (with a  $\text{P}_2\text{O}_5$  content of 45%); SSP or simple superphosphate; TSP or triple superphosphate, thermophosphate (mixtures); partially acidulated natural phosphate (phosphate rock with sulfuric acid); urea; ammonium nitrate; nitrocalcium (mixture of ammonium nitrate with limestone powder); ammonium sulfate and potassium chloride. Basic fertilizers are used to make mixtures and/or granulated products with the formula N:P:K (N:  $\text{P}_2\text{O}_5$ : $\text{K}_2\text{O}$ ) (MME, 2020). Phosphate rock cannot be replaced or recycled in agriculture, so its demand needs to be met through mining, beneficiation and chemical processing of derived materials. The United States is the world's largest producer (30%) and consumer of phosphate rock, in addition to being the leader in the production and export of fertilizers. According to data from the National Supply Company (Conab), Brazil recorded a record in fertilizer imports in January 2024, with 15% more than in the same period of the previous year. Important countries in phosphate production are Morocco, China and Russia. Most of the world's phosphate rock reserves are located in Morocco, including Western Sahara. Other large holders of phosphate rock reserves are Iraq, China, Algeria, Syria, Jordan, South Africa, the USA and Russia. Due to the growing global demand for phosphate, several producing countries have established measures to restrict the export of this mineral commodity, aiming to guarantee their own needs in the future. China, for example,

imposed a 135% tariff in 2008 to discourage exports and protect domestic supplies. According to data from the National Association for the Diffusion of Fertilizers (ANDA), more than 70% of the fertilizers used in Brazilian agriculture are imported, with the country's largest external dependencies being potassium chloride (95%), nitrogen (80%) and phosphate (60%) (RIDDER *et al.*, 2012; SIS & CHANDER, 2003; CONAB, 2024; ANDA, 2024).

Regarding phosphate deposits, they can be divided in sedimentary, igneous and biogenetic, with the first two being the most important from an economic point of view. Biogenetic deposits, according to Ridder *et al.* (2012), are nitrogenous organic concentrations, originated by bird droppings, and are of lesser economic importance. Phosphate ores originating from marine sediments are located in the United States, southeastern Mexico, Morocco, and the Northwest Sahara and Middle East. Phosphate ores originating from igneous deposits are present in South Africa, Russia, Finland and Brazil, among others. In Brazil, approximately 80% of phosphate reserves are of igneous origin with a marked presence of carbonatite rock and micaceous minerals, with low content, expressed in terms of percentage of  $P_2O_5$ . The main Brazilian deposits of phosphate rock are Araxá/MG, Catalão I/GO, Jacupiranga/SP and Tapira/MG (SILVA, 2018). Phosphates are used in the agricultural, chemical, food (including beverages) and animal nutrition sectors. Due to the high complexity of Brazilian phosphate minerals, they must undergo a series of steps in order to remove associated impurities (mainly iron oxides, silicates, calcite, barite, dolomite, micas, titanium minerals and vermiculites) and achieve the levels required for fertilizer production processes. Although essential, Guimarães *et al.* (2004) state that phosphate processing in Brazil results in significant losses of phosphorus, 15% in mining, 40% in mineral processing and between 2 and 5% in the transportation and handling of phosphate concentrate.

According to Chaves & Leal Filho (2004), the mineral processing circuit for phosphate rock in Brazil involves comminution steps (with release of around 212  $\mu\text{m}$ ), magnetic separation (to remove magnetite) and flotation steps. The presence of sludge significantly affects phosphate recovery, since it increases reagent consumption, requiring desludge removal prior to flotation. For the fertilizer industry, the phosphate concentrate must contain a  $P_2O_5$  content greater than 30%; a  $\text{CaO}/P_2O_5$  ratio of less than 1.6 and a  $\text{MgO}$  content of less than 1%.

According to Moraes *et al.* (2015), the flotation reagent scheme is a determining factor in the efficiency of the process. The use of reagents aims to increase the differences

between the original characteristics of the surfaces of the minerals present in the process. This enables separation, since the more distinct the surfaces of the species to be separated, the greater the probability of success. Flotation must be selective (BALTAR, 2008). The potential of fatty acids as collectors in phosphate rock flotation is recognized in the literature. Anionic collectors are classified as sulfhydryl or oxyhydryl. Within the group of oxyhydryl collectors, carboxylic ones stand out, among others, the group to which fatty acids belong (BALTAR, 2008). Brandão *et al.* (1994) performed microflotation tests in a Hallimond tube with pure apatite, using sodium salts of palmitic, stearic, oleic, linoleic and linolenic fatty acids as collectors, depending on the pH. The results demonstrated that unsaturated fatty acids (linoleic, oleic and linolenic) had superior performance compared to saturated ones. Costa (2012) analyzed the use of Amazonian vegetable oils in the flotation of phosphate minerals. The results obtained in the study indicate that there is a great possibility of using Amazonian vegetable oils as collectors in the flotation of phosphate minerals. The author highlights that the passion fruit seed collector represents an alternative to the reagents currently used in phosphate flotation.

Phosphate mining in Brazil faces constant challenges to optimize its mineral beneficiation processes, aiming to increase the efficiency and profitability of its operations. In this context, a partnership between the company RJMG and a phosphate rock mining company located in the Alto Paranaíba region, Minas Gerais, was established. The challenge was to achieve and guarantee the company's metallurgical recovery goal, considering a low P<sub>2</sub>O<sub>5</sub> content ore feeding the flotation circuit.

A customized vegetable collector based on fatty acids was developed by RJMG aiming to replace the collector used in mining, a commercially available fatty acid, which resulted in the development and implementation of an innovative solution for the processing of this mineral asset. It was tested not only on a bench scale, but also on a pilot scale. This work presents the development and validation of this collector.

## METHODOLOGY

Phosphate rock samples were collected at the mineral processing plant of the aforementioned mining company. The samples were collected after the grinding stages and before conditioning, in the form of pulp. The samples were then dried, homogenized and quartered into aliquots of approximately 1.0 kg for use in flotation tests. Initially, a laboratory-scale test campaign was carried out using a Denver model benchtop

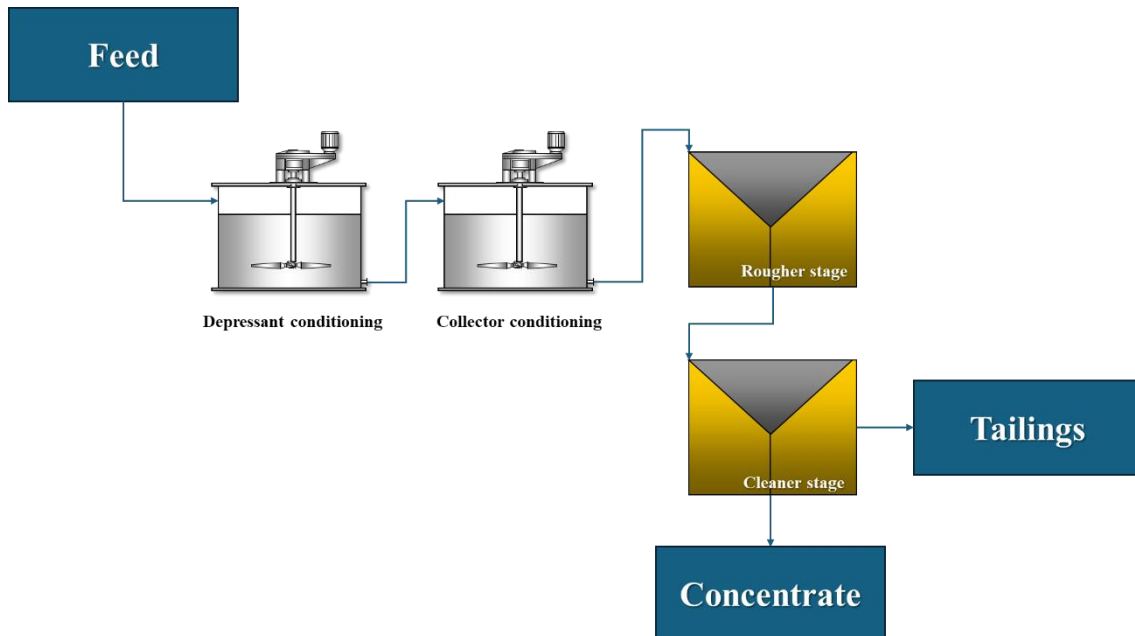
mechanical cell, aiming to customize a plant collector for the low-grade ore from the aforementioned mining operation. The tests were carried out at the RJMG mineral processing laboratory, located in Uberaba/MG.

Four collectors were tested in this stage, namely the collector used by the mining company, here called fatty acid (dosage of 250 g/t, according to the mining company's standard), AGEM A, AGEM B and AGEM R6, all from RJMG. The methodology proposed by RJMG was used to saponify the collectors, which consisted of adding 10 g of collector (temperature between 25°C and 30°C) to 20 g of distilled water (temperature between 25°C and 27°C) and 3.0 g of 50% v/v NaOH solution, kept under magnetic stirring for 3 minutes. Finally, 167 g of distilled water was added (temperature between 25°C and 27°C), keeping the solution under magnetic stirring until complete homogenization. Gelatinized cornstarch was used as a depressant in the tests (dosage of 650 g/t). Cornstarch gelatinization was performed by adding 7.0 g of cornstarch to 45 g of distilled water (temperature between 24°C and 26°C). The solution was kept under magnetic stirring until completely homogenized. After homogenization, 2.0 g of 50% v/v NaOH solution was added to the solution, which was kept under magnetic stirring for 3 minutes. Finally, 246 g of distilled water (temperature between 24°C and 26°C) were added to the solution, which was kept under magnetic stirring until completely homogenized. All tests were performed in two stages (rougher and cleaner), as can be seen in Figure 1, using tap water. Diluted solutions of NaOH and HCl, both at 10% v/v, were used to control the pH of the flotation.

The second flotation test campaign was carried out at the pilot plant of the aforementioned mining company. These tests aimed to compare the performance of the collector used in the company (fatty acid) with the best vegetable collector AGEM from RJMG, selected based on the results of the laboratory-scale test campaign. For this purpose, flotation tests were carried out in three flotation columns, forming a closed circuit composed of the rougher, cleaner and recleaner stages. Three sets of tests were carried out, one per day, varying the collector dosage (240, 280 and 330 g/t) and the conversion percentage (saponification) of the saponification collector AGEM (65% and 100%). To ensure the lowest conversion of AGEM B, the NaOH dosage was reduced by 35%. The tests were carried out in continuous flow, with a feed rate of approximately 80 kg/h. Gelatinized cornstarch was used as a depressant. All tests were carried out at pH 8.5, with process water, using a diluted NaOH solution for pH control. The sample used to feed the pilot plant tests was composed of three local lithologies: 6.6% upper

micaceous, 71.3% lower micaceous and 22.1% phosphatic, and was floated without desliming. The conditioning solids percentage was approximately 46%.

**Figure 1** – Flowchart of bench-scale flotation tests



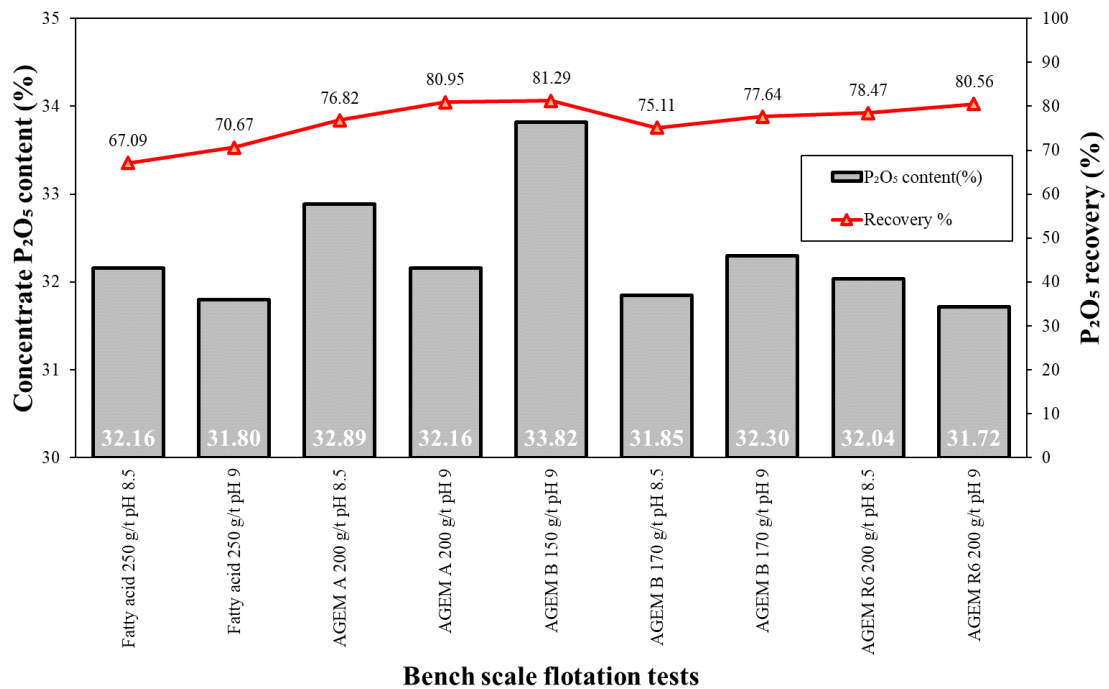
Samples of the feed, concentrates and tailings produced in the bench-scale tests, as well as samples of these two flows collected in the pilot-scale tests, were analyzed in an X-ray fluorescence spectrometer to determine the  $P_2O_5$  content by the mining company. For this purpose, the samples were filtered, oven-dried and fused with lithium tetraborate to produce pellets.

## RESULTS AND DISCUSSION

The  $P_2O_5$  content in the feed for the laboratory-scale flotation tests was 4.41%, thus classifying this deposit as low-grade. Figures 2 and 3 show the variation in the  $P_2O_5$  content and metallurgical recovery in the concentrate, and the mass and metallurgical recovery as a function of the  $P_2O_5$  content in the concentrate for the nine bench-scale flotation tests, respectively. The goal at this stage was to maximize the mass and metallurgical recoveries, with  $P_2O_5$  contents in the concentrate above 30%. It can be noted that in all tests the target for the  $P_2O_5$  content in the concentrate was achieved, with the best result obtained for the fatty acid at pH 9.0 and 250 g/t (standard dosage of the mining company), producing a concentrate with a  $P_2O_5$  content of 31.80%, with a mass recovery

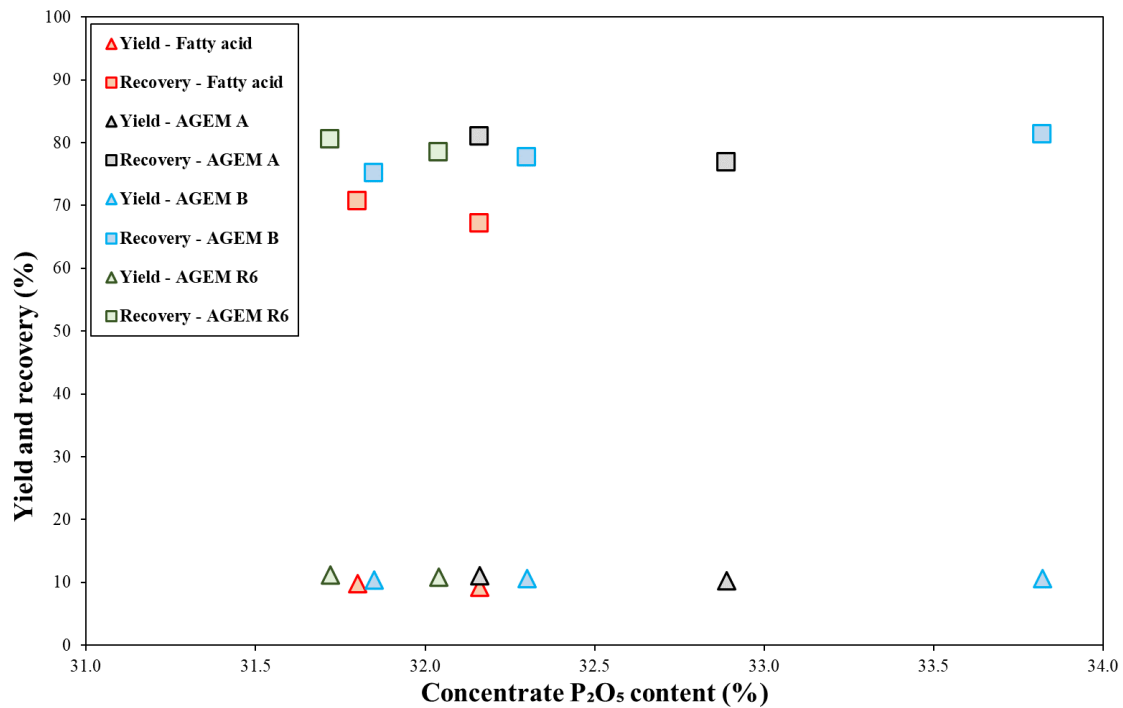
of 9.8% and a metallurgical recovery of 70.67%. Reducing the pH to 8.5 for this collector caused an increase in the P<sub>2</sub>O<sub>5</sub> content in the concentrate to 32.16% (1.13% increase), but a decrease in mass recovery to 9.2% (6.12% reduction) and metallurgical recovery to 67.09% (5.06% reduction).

**Figure 2** – Recovery and P<sub>2</sub>O<sub>5</sub> content in the concentrate obtained in the bench-scale flotation tests



With the exception of one test (AGEM R6 dosage of 200 g/t at pH 9), all tests with RJMG reagents produced higher levels than the fatty acid at the dosage of 250 g/t at pH 9. The best result was obtained with AGEM B at the dosage of 150 g/t at pH 9, with a P<sub>2</sub>O<sub>5</sub> content in the concentrate of 33.82% (increase of 6.35%), mass recovery of 10.6% (increase of 8.16%) and metallurgical recovery of 81.29% (increase of 15.03%). In addition, under these conditions there was a 40% reduction in the collector dosage, resulting in a direct reduction in flotation OPEX and an increase in final concentrate production of 8.16%. However, it is noted that AGEM R6 at a dosage of 200 g/t at pH 9 was the collector with the highest mass recovery of 11.2% (an increase of 14.29%) and metallurgical recovery of 80.56% (an increase of 14%), but with the lowest content obtained, 31.72%. Therefore, it is suggested that more tests be carried out with this collector, since the higher the mass recovery, the greater the production of phosphate rock concentrate, for the same feed.

**Figure 3** – Yield and recovery as a function of the P<sub>2</sub>O<sub>5</sub> content in the concentrate for bench-scale flotation tests

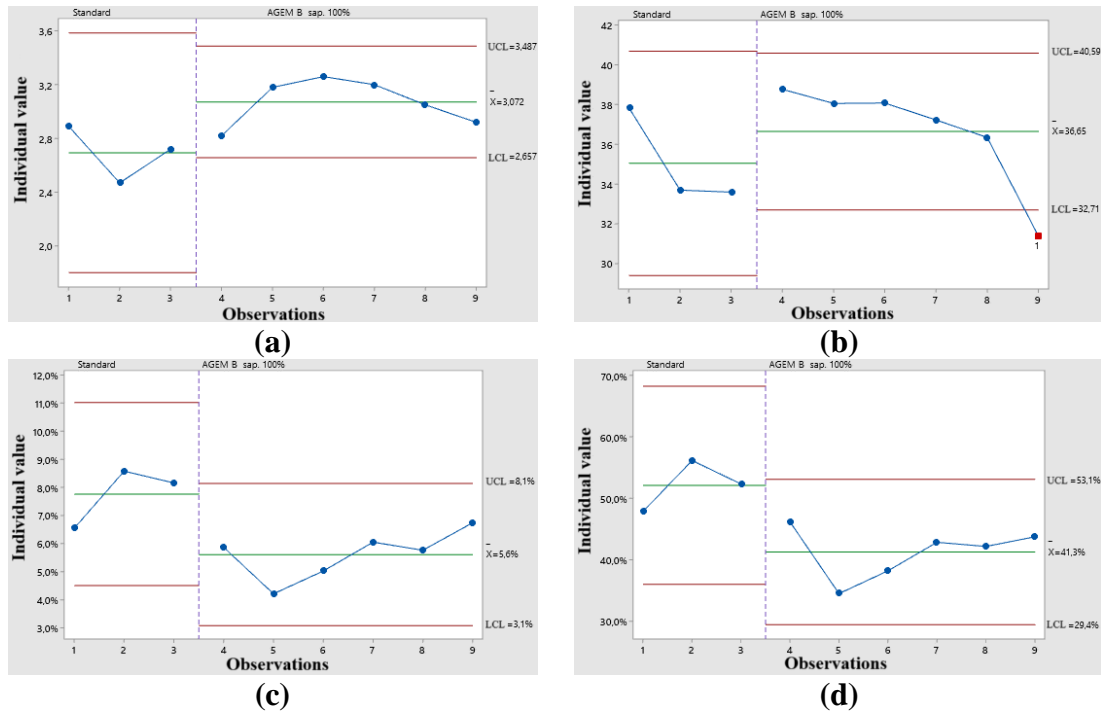


Based on the previous results, the vegetal collector AGEM B was selected to carry out flotation tests on a pilot scale and in a flotation column, compared to the fatty acid used by the mining company. For the pilot plant tests, the average P<sub>2</sub>O<sub>5</sub> content in the feed was 5.2%. Figures 4 and 5 show the results of pilot-scale tests for 100% and 65% saponification of the AGEM B collector, respectively.

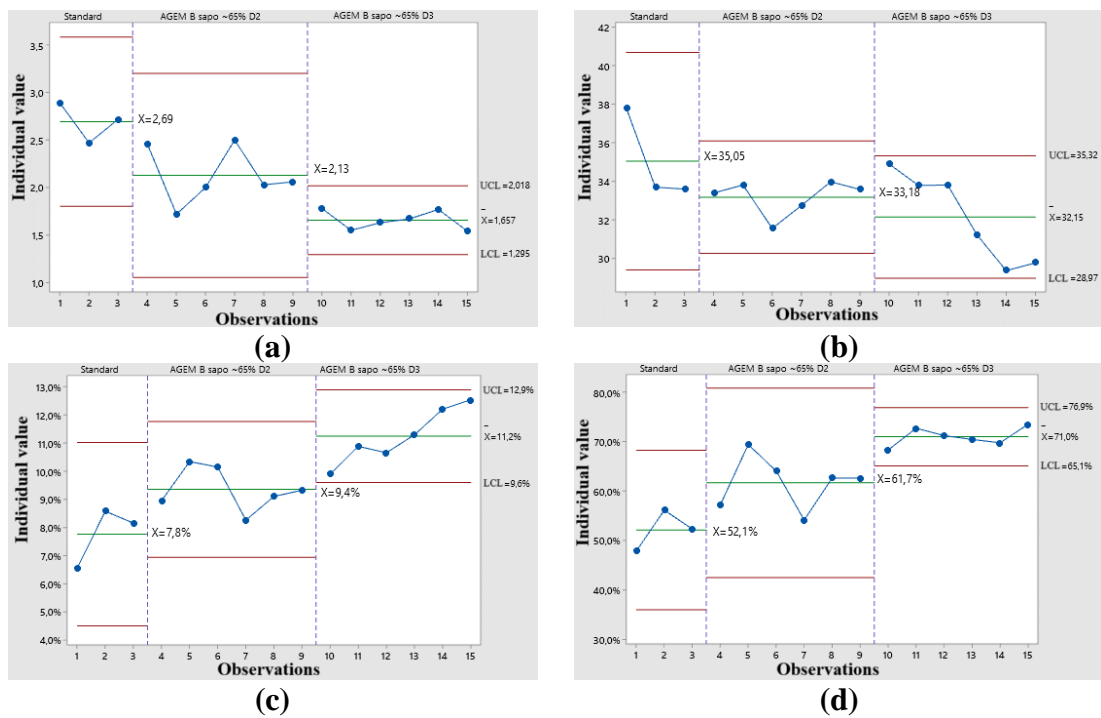
The tests performed with the standard collector used in the mining company (fatty acid) resulted in an average metallurgical recovery of P<sub>2</sub>O<sub>5</sub> of 52.1%, with an average P<sub>2</sub>O<sub>5</sub> content in the recleaner concentrate of 35.1% and in the tailings of 2.7%. On average, the 100% saponified AGEM B collector with a dosage of 330 g/t presented a higher P<sub>2</sub>O<sub>5</sub> content in the rougher tailings than the collector used by the mining company (average of 3.07 ± 0.34%). Decreases were also observed in mass recovery (average of 5.60 ± 2.04%) and metallurgical recovery (average of 41.30 ± 9.63%). However, the P<sub>2</sub>O<sub>5</sub> content in the recleaner concentrate was also higher (average of 36.65 ± 3.22%). Based on these results, the fatty acid used by the mining company would have a better performance than the AGEM B tested.



**Figure 4** – Control charts of (a)  $P_2O_5$  content in the rougher reject, (b)  $P_2O_5$  content in the recleaner concentrate, (c) mass recovery and (d) metallurgical recovery of  $P_2O_5$  for the two collectors tested. AGEM B with 100% saponification



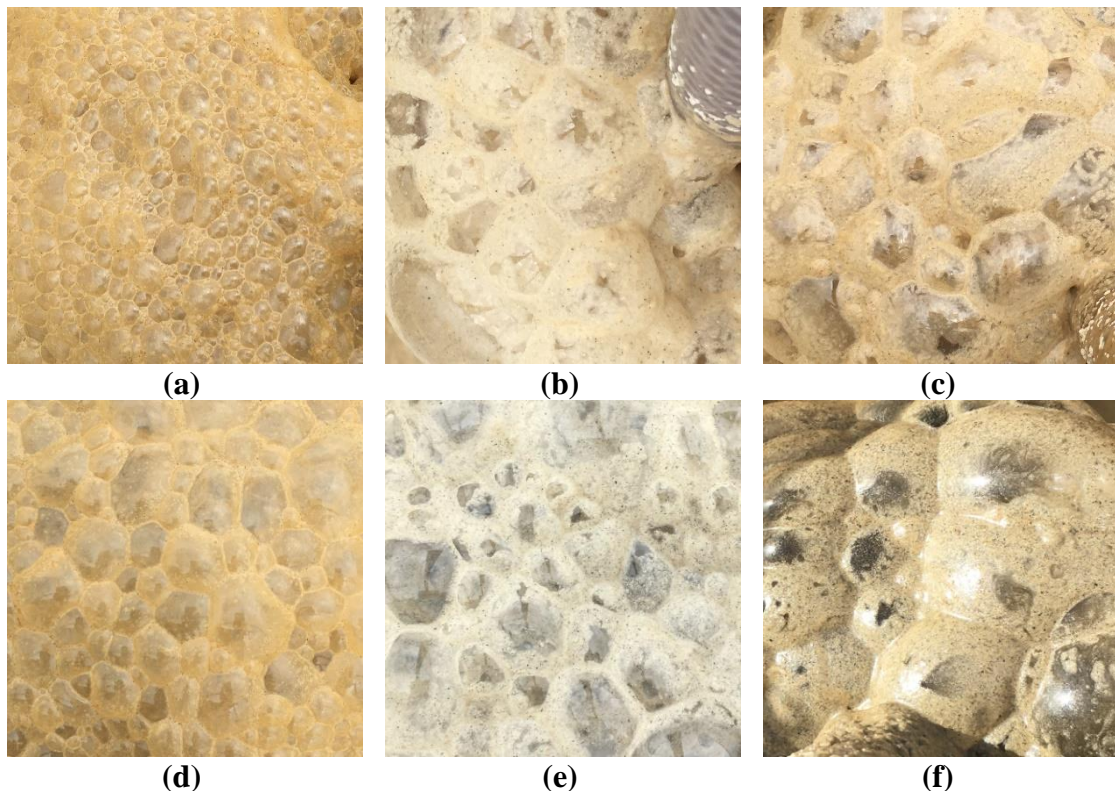
**Figure 5** – Control charts of (a)  $P_2O_5$  content in the rougher reject, (b)  $P_2O_5$  content in the recleaner concentrate, (c) mass recovery and (d) metallurgical recovery of  $P_2O_5$  for the two collectors tested. AGEM B with 65% saponification



However, the performance of the AGEM B collector changed considerably when it was tested under lower saponification conditions (65%) and dosage (240 and 280 g/t), as shown in Figure 5. Tests 4 to 9 were performed with a dosage of 280 g/t, while tests 10 to 15 were performed with a dosage of 240 g/t. In this situation, the average  $P_2O_5$  content in the rougher tailings was  $1.66 \pm 0.29\%$ , 38.40% lower than that obtained with the collector used by the mining company (a fatty acid) which had an average content of 2.69%. The average  $P_2O_5$  content in the recleaner concentrate was  $32.15 \pm 2.59\%$ , 8.27% higher than that obtained with fatty acid (average of 35.05%). Increases of 43.59% in mass recovery (average of  $11.20 \pm 1.31\%$ ) and 36.28% in metallurgical recovery (average of  $71.00 \pm 4.82\%$ ) were observed.

Figure 6 shows a mosaic of photographs taken from samples of the foams generated with the two reagents tested (fatty acid and AGEM B at a dosage of 240 g/t and 65% conversion) in the rougher, cleaner and recleaner stages. It is possible to see that the foam was well mineralized for both reagents tested, but it was noted in the tests with AGEM B that the foam layer was highly mineralized, with easy flow (low viscosity) and fast.

**Figure 6** – Mineralized foams generated during pilot-scale flotation tests with the fatty acid used in the mining company in the (a) rougher, (b) cleaner and (c) recleaner stages and with AGEM B 240 g/t and 65% conversion in the (d) rougher, (e) cleaner and (f) recleaner stages



## CONCLUSIONS

Bench-scale and pilot-scale flotation tests were conducted with low-grade phosphate rock aiming at the development of a new plant collector based on fatty acids, in order to improve flotation rates (grades and recoveries) and reduce the OPEX of this stage, which is essential for the production of phosphate rock concentrate. The results obtained demonstrate the importance of innovation and strategic collaboration in overcoming challenges in mining. The AGEM B plant collector not only significantly improved the efficiency of the phosphate rock recovery process, but also contributed to reducing environmental impact and operating costs.

Further testing is needed to determine the optimal operating conditions of this reagent in an industrial environment, with regard to pH and collector and depressant dosages.

## ACKNOWLEDGMENTS

The authors would like to thank the entire team at RJMG Óleo Química for their invaluable support in carrying out this work, and the Federal University of Catalão for the knowledge transmitted and analysis of the results.

## REFERENCES

ASSOCIAÇÃO NACIONAL PARA DIFUSÃO DE ADUBOS (ANDA). Principais indicadores do setor de fertilizantes. 2024.

BALTAR, C. Flotação no Tratamento de Minério. Recife, Brazil, UFPE, 2008.

BRANDÃO, P. R. G., CAIRES, L. G., QUEIROZ, D. S. B. Vegetable lipid oil-based collectors in the flotation of apatite ores. *Minerals Eng.*, v.7, n. 7, p. 917-925, 1994.

CHAVES, A.P.; LEAL FILHO, L.S. Flotação. In: Luz, A. B., Sampaio, J. A. e Almeida, S. L. M. (Ed.). *Tratamento de minérios*. 4ª ed. Rio de Janeiro: CETEM/MCT, pp.411-458, 2004.

COMPANHIA NACIONAL DE ABASTECIMENTO (CONAB). Boletim Logístico. 2024. Disponível em: <https://www.conab.gov.br/ultimas-noticias/5404-boletim-logistico-volume-de-fertilizante-importado-em-janeiro-e-recorde-no-pais>

MINISTÉRIO DE MINAS E ENERGIA (MME). Anuário Estatístico do Setor Transformação de Não-Metálicos. 2020. Disponível em: <https://www.gov.br/mme/pt->

br/assuntos/secretarias/geologia-mineracao-e-transformacao-mineral/publicacoes-1/anuario-estatistico-do-setor-metalurgico-e-do-setor-de-transformacao-de-nao-metalicos/anuario-estatitico-2021-setor-de-transformacao-de-nao-metalicos-ano-base-2020.pdf

MORAES, I. L. A., SILVA, A. C., SILVA, E. M. S., MORAIS, V. L., SILVA FILHO, C. M. Microflotação de apatita utilizando óleo de pinhão manso como coletor. HOLOS, 31(7), pp. 132-139.

RIDDER, M.D., JONG, S.D., POLCHAR, J.; LINGEMANN, S. Risks and opportunities in the global phosphate rock market. The Hague Centre for Strategic Studies (HCSS). Rapport n° 17/12/12, 96p., 2012.

SILVA, E.M.S. Utilização de amido de sorgo como depressor na flotação de minérios. Tese, Programa de Pós-Graduação em Engenharia Metalúrgica, Materiais e de Minas, UFMG, 325p., 2018.

SIS, H.; CHANDER, S. Reagents used in the flotation of phosphate ores: a critical review. Minerals Engineering, 16, pp.577-585, 2003.