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PRELIMINARY STUDY ON THE METALLURGICAL RECOVERY OF IRON ORE TAILINGS FROM THE UNITARY FLOTATION OPERATION

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Abstract: Mining plays a crucial role in Brazil's economy, and flotation is a fundamental operation for separating minerals. In this study, we used samples collected after a dam collapse in November 2015, as this is where iron ore tailings are found. The objective was to evaluate the potential of pequi and buriti oils, from the Cerrado, as collectors to make minerals hydrophobic during the flotation process. We conducted experiments in bench tests, varying the concentration of collectors in each test. We analyze the minerals present in the floated and non-floated materials to calculate the metallurgical recovery, which represents the fraction of iron and silicon in the stream of interest. We use statistical analyzes to evaluate the results. The results indicated that pequi oil did not perform satisfactorily, with insignificant recoveries of silicon and iron, compared to the other collector tested. On the other hand, buriti oil demonstrated statistical compliance with the proposed model and presented a better metallurgical recovery capacity. Therefore, this study investigated the application of pequi and buriti oils as collectors in mineral flotation, with buriti oil showing more promise for future industrial applications.

Keywords: Statistical analyses. Collector. Fruits from the savannah.

INTRODUCTION

Metallic iron can be extracted from iron ore rock, in a more economically viable way, generally this extraction is in the form of oxide, the most common being hematite and magnetite (1). In the present study, ore waste from the collapse of the Samarco dam, in 2015, in the city of Mariana was used, since when extracting the rock from nature and separating the iron from the waste, it is generally deposited in a dam, which are large reservoirs composed of water, sand and solid waste from the extraction of ores.

Iron ore is the raw material for use in the steel industry, for steel production. Therefore, to meet the specifications required by the industry, iron must be in a specific concentration. Therefore, the most used ore concentration process is flotation, a unitary operation that relates the liquid, solid and gaseous phases (2).

The main reagents used in the flotation process are collectors, which modify the surface of the mineral to facilitate the adsorption of the molecule to be floated, depressants, which are used to increase the selectivity of the process, and foaming agents, which are used to introduce air bubbles. in the pulp and regulatory agents, which are responsible for optimizing process conditions (3).

MATERIAL AND METHODS

The study involved the analysis of tailings from the Risoleta Neves Hydroelectric Power Plant Dam, originating from the Fundão Dam in Bento Rodrigues, MG. To prepare the samples for flotation tests, sieving was carried out to obtain a particle size below 0.149 mm, making the samples suitable for the process.

The saponification of pequi and buriti oils was carried out following the standard procedure of the Fosfertil de Tapira pilot plant. He employed a 10% (m/v) concentration of NaOH, then stirred the mixture on a magnetic stirrer at a temperature of 40°C until it was completely liquefied.

The flotation technique used was direct anionic. Initially, 20 g of the waste sample was weighed and 20 mL of water was added to create a pulp with a concentration of 50%. Adjusted the pH to 7.5 using HCl and NaOH as needed and conditioned the pulp for 5 minutes. Then added the saponified oil as collector and adjusted the pH as necessary. Conditioning was repeated for another 2 minutes (4).

For the flotation test, a teaching module from the company Up Control was used, which involved the use of a vacuum pump to pass air through a hose connected to a tube containing the sample. At the end of the process, he collected the densest and least dense material, compared to water (what floated and didn't float). A 2² factorial experimental design was used to evaluate how collector dosages (in grams per ton of ore) influenced tailings flotation.

The quantification of the chemical elements iron (Fe) and silicon (Si) was carried out using Scanning Electron Microscopy (SEM) integrated with X-ray Energy Spectroscopy (EDS) on a TM 3000 microscope with a voltage of 15 kV. Small portions of the material were fixed to double-sided tape in a 2.5 cm diameter sample holder. The equipment was calibrated and analyzes were carried out at 1500x magnification to identify the crystals in the sample.

The identification of the elements was based on the specific transition energies of each material, allowing the generation of graphs that indicate the presence of the elements and their percentages in the sample.

RESULTS AND DISCUSSION

The metallurgical recovery (RM), given by equation 1, represents the fraction of one of the components in the stream of interest. In a case where the product of interest is non-floated, it is necessary to:

$$RM = \frac{y_{flot} * M_{flot}}{y_{alim} * M_{alim}}$$
 Eq. 1

From equation 1, the results were obtained and statistically treated regarding the metallurgical recovery of iron (Tables 1 and 2) and silicon (Tables 3 and 4) from flotation using the two types of oils, pequi and buriti. Thus, it was possible to assess whether the model is statistically significant.

After statistical analyses, we evaluated the

| Source of variation | Sums of Squares | Degrees of freedom | Medium Square | F _{calc} | p-value |
|----------------------|-----------------|--------------------|---------------|-------------------|---------|
| Regression | 12,393 | 3 | 4,131 | 0,0361 | 0,986 |
| Residues | 114,433 | 1 | 114,433 | | |
| Lack of adjustment | 114,433 | 1 | 114,433 | NaN | NaN |
| Pure error | 0 | 0 | NaN | | |
| Total $R^2 = 9,77\%$ | 126,826 | 4 | | | |

Table 1 - ANOVA: metallurgical recovery of iron with pequi oil.

Source: the own authors, 2019.

Note: $Y = 5.23 + 1.46 \times 1 - 0.52 \times 2 - 0.84 \times 1 \times 2$

| Source of variation | Sums of Squares | Degrees of freedom | Medium Square | F _{calc} | p-value |
|-----------------------|-----------------|--------------------|---------------|-------------------|---------|
| Regression | 708,6 | 3 | 236,2 | 225,2 | 0,04894 |
| Residues | 1 | 1 | 1 | | |
| Lack of adjustment | 1 | 1 | 1 | NaN | NaN |
| Pure error | 0 | 0 | NaN | | |
| Total $R^2 = 99,85\%$ | 709,6 | 4 | | | |

Table 2 - ANOVA: metallurgical recovery of iron with buriti oil.

Source: the own authors, 2019.

Note:
$$Y = 10,43 + 8,37 x_1 - 7,24 x_2 - 7,40 x_1 x_2$$

| Source of variation | Sums of Squares | Degrees of freedom | Medium Square | F_{calc} | p-value |
|---------------------|-----------------|--------------------|---------------|------------|---------|
| Regression | 189,2385 | 3 | 63,0795 | 0,36713 | 0,80257 |
| Residues | 171,81522 | 1 | 171,81522 | | |
| Lack of adjustment | 171,81522 | 1 | 171,81522 | NaN | NaN |
| Pure error | 0 | 0 | NaN | | |
| Total | 361,05372 | 4 | | | |
| $R^2 = 52,41\%$ | | | | | |

Table 3 - ANOVA: metallurgical recovery of silicon with pequi oil

Source: the own authors, 2019.

Note:
$$Y = 27,38 - 2,15 x_1 + 6,46 x_2 + 1,01 x_1 x_2$$

| Source of variation | Sums of Squares | Degrees of freedom | Medium Square | F _{calc} | p-value |
|---------------------|-----------------|--------------------|---------------|-------------------|---------|
| Regression | 261,3 | 3 | 87,1 | 806,3 | 0,02588 |
| Residues | 0,1 | 1 | 0,1 | | |
| Lack of adjustment | 0,1 | 1 | 0,1 | NaN | NaN |
| Pure error | 0 | 0 | NaN | | |
| Total | 261,4 | 4 | | | |
| $R^2 = 99,96\%$ | | | | | |

Table 4 - ANOVA: metallurgical recovery of silicon with buriti oil

Source: The own authors, 2019.

Note: $Y = 25,20 - 3,68 x_1 + 6x_2 + 3,97 x_1 x_2$

values of the coefficient of determination (R²) for the metallurgical recoveries of silicon and iron using pequi oil as a collector. We found that these values did not statistically meet the proposed model, leading to their exclusion from the study.

Regarding the F test: $_{regression/residues}$, employing a significance level of 10% (p=10%) for comparison with the tabulated values. With the regression and residual degrees of freedom, a value of F_{tab} equals to 53,593.

These results indicate that buriti oil proved to be effective as a collector in the flotation of minerals of interest, showing significant metallurgical recovery. This discovery is relevant to the mining industry, as it suggests the feasibility of using this collector on an industrial scale, which can contribute to more efficient and sustainable mineral recovery processes.

CONCLUSION

Based on the results obtained, it is evident that oils derived from Cerrado fruits play a significant role as collectors in the iron ore tailings flotation process. However, while pequi oil did not prove to be statistically satisfactory in any of the flotation tests carried out within the range studied, buriti oil showed statistically satisfactory models

for the metallurgical recovery of both iron and silicon, indicating its effectiveness as a collector in this context. The variability of results observed between different oils can be attributed to several intrinsic properties of the oils, such as chemical composition, acidity, degree of saponification, among other factors. Therefore, the selection of the appropriate collector for mineral flotation must take these specific characteristics into account in order to optimize process performance. These findings have important implications for the mining industry, highlighting the potential of buriti oil as an effective collector for separating minerals of interest.

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