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Flotation performance of phosphate ore before and after dissolved air flotation water treatment

Guilherme Henrique Ferreira Silva[1], Luís Alberto Silva[1], João Victor da Silva Alves[1], Natal Junio Pires[1], Michelly dos Santos Oliveira[1]

[1]Centro Federal de Educação Tecnológica de Minas Gerais. Araxá, MG, Brasil.

e-mail: guilhermehfs06@gmail.com, luis_alberto803@hotmail.com, joao.alvesaraxa@hotmail.com, natal@cefetmg.br, michelly@cefetmg.br

ABSTRACT

Mineral flotation is a wet concentration operation that has a differentiating effect dependent on the surface properties of the minerals. Water quality is integral to the phosphate ore flotation, since the presence of certain ions in the solution can compromise the effectiveness of the operation. The objective of this study was to compare the effectiveness of phosphate ore flotation (dispersed air) before and after effluent treatment using coagulation, flocculation and dissolved air flotation (DAF) techniques. The mineral flotation tests were performed for new water (NW) and three effluents tailing dams (TD), simulated effluent with high concentrations of calcium and magnesium ions (SE) and simulated effluent after DAF treatment (SE_{DAF}). The simulated effluent underwent two stages of aggregation followed by DAF for cation removal. The results showed that the proposed technique removed 91.6% of magnesium and 66.4% of calcium from the original simulated effluent (SE). The SE_{DAF} presented better average values of mass recovery, content, and recovery of P_2O_5, confirming the effectiveness of the removing ions suggested as deleterious for mineral flotation. The flotation performance of the DAF-treated effluent was comparable to that of new water. This study underscores the significance of water quality management in optimizing mineral flotation processes.

Keywords: Dissolved air flotation; Ca^{2+} and Mg^{2+} ions; Mineral flotation; Phosphate ore; Water reuse.

1. INTRODUCTION

Water is a scarce resource, and its control has become increasingly important. Some mineral processing operations are not authorized to use new water due to environmental regulations, thus forcing companies to use relatively impure primary water sources with high salinity levels, including calcium, magnesium, and iron salts as potential precipitates [1]. This issue has led to developing strategies to improve water sustainability, including water reuse as one of the most widespread practices, especially in mineral flotation; however, this reuse tends to alter water quality [2]. Most research has focused on understanding the issues associated with water quality in flotation, where studies focus on three main aspects: (1) understanding the reasons for water quality variation, (2) quantifying the effect of water quality variation on flotation efficiency by investigating the responsible mechanisms, and (3) testing different solutions [3].

Flotation is the most commonly used operation for phosphate ore concentration. However, factors, such as the presence of ionic particles or organic matter from residual reagents in the water solution can impact process performance [4]. When recycled water containing high levels of dissolved salts and metallic ions is employed, apatite [$Ca_5(PO_4)_3$(F, Cl, OH)] recovery [5], and consequently, flotation selectivity can be affected, which is significant because flotation selectivity is critical in the operation [6].

Dissolved air flotation (DAF) is an advantageous technique for floc separation in water treatment systems that receive several different types of raw water [7]. In DAF, bubble generation is accomplished by saturating part of the effluent with air in tanks operating above atmospheric pressure, with the availability of air linked to the operating pressure of the system [8]. This process is followed by sudden decompression through a needle-type valve or flow constriction devices, resulting in the formation of microbubbles in the range of 10 to 100 μm.

This technique has been employed in several areas, particularly for treating some industrial effluents [9] and in the agricultural sector [10]. Examples of industrial applications for removing suspended solids

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and adsorbed ions in $Fe(OH)_3$ precipitates occur in Chile, where DAF plants are used, enabling the reuse of treated water for irrigation and recycling in various unit operations for ore treatment [11]. AZEVEDO *et al.* [12] conducted bench-scale and pilot studies for the removal of suspended solids and cations using the same technique, aiming to produce treated water suitable for reuse in the beneficiation of sulfide ores, achieving removals exceeding 90% for Cu^{2+} and Pb^{2+} through the precipitation of metal hydroxides and cation adsorption on the $Fe(OH)_3$ matrix. In iron ore beneficiation, PINHEIRO *et al.* [13] demonstrated the feasibility of process water reuse in iron ore flotation after treatment by DAF for the removal of Ca^{2+} and Mg^{2+} cations. OLIVEIRA *et al*. [14] achieved high turbidity removal (approximately 98%) and removal of Mn^{2+} cations (approximately 80%) and Al^{3+} (>95%) through coagulation-flocculation-DAF of process water from tailings filters and sludge thickeners in iron ore processing. FAUSTINO *et al.* [15] treated water polluted by iron ore sludge with ferric chloride ($FeCl_3$) and DAF, achieving residual turbidity of 25 NTU, with reductions of up to 99%. NUNES *et al.* [8], using a dissolved air flotation (DAF) pilot unit, treated a phosphate rock effluent with particle removal efficiencies above 90%.

However, its use in treating tailing dams or industrial wastewater for reuse in mineral flotation applications is still a challenge and requires further investigation [16]. To address this challenge, this study aimed to treatment of simulated effluent (SE) by coagulation, flocculation and DAF for subsequent application in the apatite flotation by conventional froth flotation with dispersed air. As a secondary objective, use of four types of water – TD, NW, SE and SE_{DAF} for phosphate ore flotation and evaluation of Ca^{2+} and Mg^{2+} cátions interference.

2. MATERIALS AND METHODS

The ore sample, effluents from the tailing dams (TD), and new water (NW) were obtained through a partnership with a phosphate mining company from Alto Paranaíba/Minas Gerais State – Brazil. The sample of phosphate ore was collected in the industrial flotation conditioning stage and was composed, in mass, by 85% coarse (80% less than 150 µm) and 15% fine (80% less than 74 µm) fractions. The ore was homogenized and quartered, with aliquots taken for material characterization and mineral flotation tests. The ore density was measured by gas pycnometry (ACP gas pycnometer, model Autodensity 200) at the Ore Processing Laboratory of CEFET/MG. Chemical characterization was performed by X-ray fluorescence spectroscopy (XRF) at the partner company's Chemical Laboratory. The effluents TD and NW were collected at the plant of the partner mining company during a non-rainy period. The characterization of these types of water involved determining parameters such as turbidity, pH, and concentration of calcium and magnesium ions. The turbidity of the effluents was determined using a Hanna turbidimeter, model HI93703C. The pH of the liquids was measured using a Hanna pH meter, model Edge HI2002-02. The concentration of calcium and magnesium ions was determined through titration, following the procedure detailed by ALVES [17] and was carried out at the Chemistry Laboratory of Campus IV of CEFET-MG.

A simulated effluent (SE) with a concentration of 100 mg/L of both Ca^{2+} and Mg^{2+} ions was produced to investigate the effects of the contaminants on flotation performance, more critical scenario. SANTOS *et al.* [5] showed calcium and magnesium ions react with vegetable oil soap, impairing the collection mechanism and increasing reagent consumption, especially for concentrations greater than 100 mg/L. The following reagents were added to the freshwater: calcium nitrate (hydrated) and magnesium nitrate (hydrated). Two stage coagulation, flocculation and DAF could then be conducted on the SE to achieve levels of calcium and magnesium below the maximum provided by the operators of the mining company (60 mg/L for each one). The attempt to remove the ions in a single step proved to be inefficient from a technical standpoint. In a previous study with simulated efluente (SE), ALVES *et al*. [18] optimized the levels of reagent dosage, conditioning time, pH, and recycling flow rate to achieve greater ion removal using the experimental design technique. The operational parameters of the tests are detailed in Table 1, as indicated by ALVES *et al.* [18]. The simulated effluent after DAF was denominated SE_{DAF}. The experimental system for DAF comprised a 3 L saturation tank and a 2 L flotation cell. The saturation pressure was 4 atm. The reagent's coagulant, and flocculant used in removing Mg^{2+} were aluminum sulfate ($Al_2(SO_4)_3.(14–18)H_2O$) and PAC (Magnafloc 338, anionic polyacrylamide) respectively, in the first stage of aggregation followed by DAF. The flocculant used for Ca^{2+} removal was sodium alginate ($NaC_6H_7O_6$) in the second stage. The reagents were conditioned within the flotation cell itself using an IKA RW 20 mechanical helix agitator. The removal efficiency of Ca^{2+} and Mg^{2+} ions was calculated by Equation 1.

$$E_r = \frac{100\ .\ \left[C_i - \left(C_t.(\%recycle + 1)\right)\right]}{C_i} \qquad (1)$$

Where E_r is the removal efficiency of the analyzed ion; C_i is the initial concentration of the ion in the effluent before treatment, C_t is the ion concentration in the treated effluent, and $\%_{recycle}$ is the percentage of water added to the flotation cell.

Mineral flotation tests (dispersed air) were performed twice with NW and with each of the three effluents (TD, SE, and SE_{DAF}) using a bench Denver mechanical cell and a 1250 mL vat (useful volume). The operational parameters of the flotation tests were similar to those used at the phosphate mining plant, as detailed in Table 2. The pulp was conditioned in the flotation cell tank. After the conditioning time, air was aspirated, and flotation was carried out for about 6 minutes, until the foam was exhausted.

Solutions of gelatinized corn starch, saponified vegetable oil and NaOH were used as the depressant, collector and pH regulator, in concentrations of 3% (m/m), 2.5% (m/m), and 10% (m/m), respectively. The cornmeal was sourced from a commercial brand, while the hydrogenated soybean oil, supplied by RJMG Óleo Química, was of the Agem variety. A comparative analysis of the effluents SE and SE_{DAF} was conducted to verify the influence of the DAF on the response variable values for mass recovery (yield), P_2O_5 content, and metallurgical recovery. For this analysis, the assumption of normality of the data was verified by the Anderson–Darling test. F and t hypothesis tests were then completed to compare the variance and difference between the effluents SE and SE_{DAF}. Then, a comparison between the four effluents used in the study was performed through an analysis of variance (ANOVA). The main objective of this test was to determine any differences in the averages of the analyzed response variables (mass recovery, P_2O_5 content and metallurgical recovery) for all effluents. The Tukey test was used to further validate the ANOVA test results, verifying significant differences between the response variable averages of effluents. The statistical tests were completed using the software Minitab (version 19) and a significance level of 5%. The tests were conducted in duplicate. The metallurgical recovery of each flotation test was calculated by Equation 2.

$$R_{metal} = \frac{c.C}{a.A} \times 100 \tag{2}$$

Where R_{metal} is the metallurgical recovery, A is the mass (g) fed into the flotation cell, C is the mass (g) of the floated fraction, a is the P_2O_5 content in the flotation feed sample, and c is the P_2O_5 content in the concentrate.

Table 1: Operational conditions of DAF tests.

PARAMETER FOR MG^{2+} ION REMOVAL	VALUE	PARAMETER FOR CA^{2+} ION REMOVAL	VALUE
Coagulant dosage (mg/L)	1000	Flocculant dosage (mg/L)	1000
Coagulant conditioning time (min)	1	Flocculant conditioning time (min)	5
Rotor speed (rpm) coagulant conditioning	1640	Rotor speed (rpm) flocculant conditioning	400
Flocculant dosage (mg/L)	30	pH	6
Flocculant conditioning time (min)	2	Recycle flow rate (%)	20
Rotor speed (rpm) flocculant conditioning	400		
pH	6		
Recycle flow rate (%)	20		

Table 2: Operational conditions of flotation tests.

PARAMETER	VALUE	PARAMETER	VALUE
Depressant conditioning time (min)	5	Ore mass (g)	521.12
Collector conditioning time (min)	1	Flotation time (min)	6
Depressant dosage (g/t)	200	% solids in conditioning	50
Collector dosage (g/t)	300	% solids in flotation	35
Flotation pH	9.50	Rotor speed (rpm)	800

3. RESULTS AND DISCUSSION

The specific mass of the ore was observed to be 2.86 ± 0.04 g/cm^3, not varying significantly from the values found by UMAR *et al*. [19] (2.90 g/cm^3). Table 3 quantifies the predominant oxides found in the phosphate ore sample by percentage. Notably, the sample used in this study contained high concentrations of CaO, SiO_2, and P_2O_5, contributing to 60.39% of the sample composition.

Figure 1a presents the turbidity results of the TD efluente and NW, with the TD effluent 50% more turbid than NW. Moreover, the turbidity of the tailing dam effluent (TD) was 88% lower than the value found by SANTOS *et al*. [16] (288 NTU). These authors investigated the impact of tailings dam effluent on the flotation of apatite from a phosphate concentration plant situated in a region neighboring the location of the present study, within the state of Minas Gerais. The turbidity results for the TD effluent and NW were below the maximum levels allowed by the Brazilian federal legislation for effluent return to the environment (100 NTU) [20]. Figure 1b presents the pH values of the NW and TD effluent. The pH of the effluent TD (8.10) was comparable to the pH of water from the surroundings of a phosphate mine, as stated by JAILOS *et al*. [21] (8.30), considering both analyses were performed during the non-rainy period. The pH of the NW (7.70) was similar to the average values for effluents generated by processing in phosphate ore mines in Al-Abyad (7.55 ± 0.03 and 7.58 ± 0.13) and Al-Hisa (7.65 ± 0.13 and 7.66 ± 0.03), as stated by JIRIES *et al*. [22]. Figure 1c displays the concentration of calcium and magnesium ions for the NW and TD effluent, determined through titration, indicating that Ca^{2+} and Mg^{2+} concentrations were comparable between effluents, particularly for magnesium. VINAUD [23] found lower concentrations of both Mg^{2+} ions (13.16 mg/L) and Ca^{2+} ions (1.53 mg/L) in a TD effluent, even though the effluent originates at a point near the recirculated water intake of a phosphate rock dam that receives effluents from all processes of the beneficiation plant. Figure 1d presents the removal efficiency of Ca^{2+} and Mg^{2+} ions obtained, suggesting that it is possible to remove 91.6% of magnesium (7 mg/L of Mg^{2+}) and 66.4% of calcium (28 mg/L of Ca^{2+}) from the SE effluent with treatment via coagulation, flocculation, and DAF in two stages (1st condition and 2nd condition).

Table 3: Percent composition of oxides in phosphate ore sample, produced by XRF analysis.

P_2O_5	CaO	SiO_2	TiO_2	Fe_2O_3	Al_2O_3	MgO	BaO	MrO	MnO	Nb_2O_5
13.88	25.29	21.22	9.00	12.46	3.11	5.43	0.14	0.42	0.30	0.17

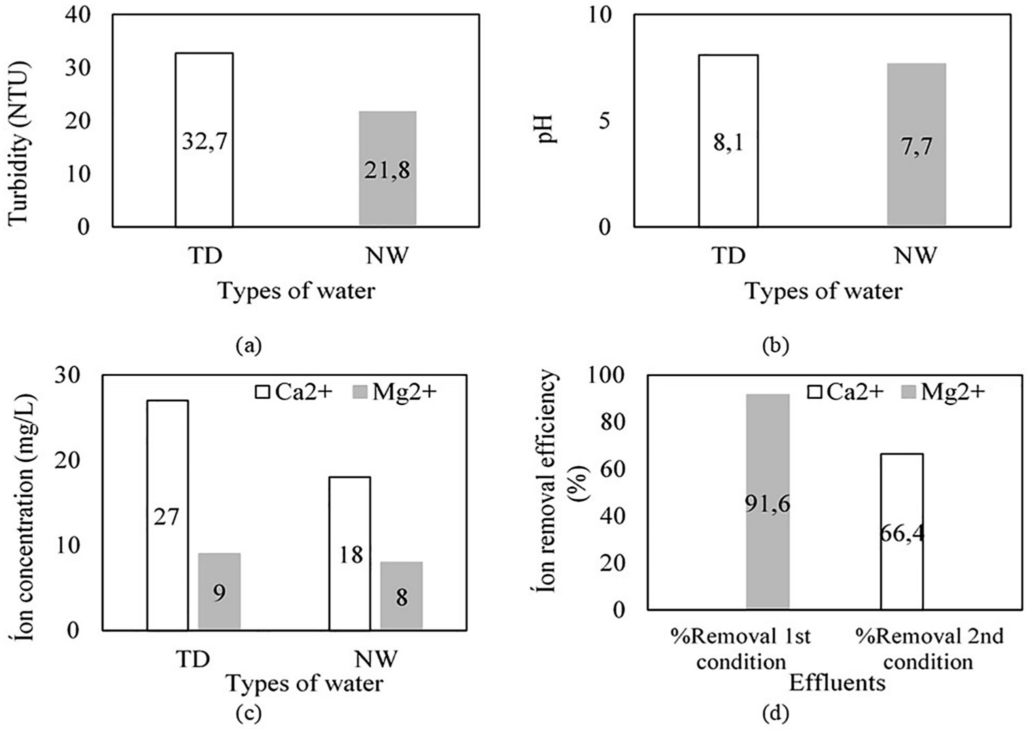


Figure 1: Characterization of TD effluent and NW by (a) turbidity (NTU), (b) pH, (c) ion concentration of Ca^{2+} and Mg^{2+} (mg/L), and (d) ion removal efficiency of Ca^{2+} and Mg^{2+} (%) from the SE effluent by DAF.

The DAF (Dissolved Air Flotation) technique reduces Ca^{2+} and Mg^{2+} content by introducing fine air bubbles into the water, which attach to the suspended particles, including ions, forming buoyant aggregates or flocs. These flocs then rise to the surface of the water, forming a froth layer that can be easily off. As a result, the concentration of ions in the treated water decreases [11].

Table 4 displays the average results for mass recovery, P_2O_5 content, and metallurgical recovery for each type of water, from the set of ore flotation tests. The SE effluent presented the least satisfactory results for all the evaluated variables. The results of a comparison between SE and SE_{DAF} are displayed in Tables 5 and 6. The effluents were analyzed under the assumption of normality of the data ($p > 0.05$). The results of the F-test proved statistical equality between effluents for the variables of mass recovery (Table 5a), P_2O_5 content (Table 5b), and metallurgical recovery (Table 5c) ($p > 0.05$). The F-test indicates that p-values above 5% exhibit equal variances However, the t-test analysis of the difference between means showed that the values of mass recovery (Table 6a), P_2O_5 content (Table 6b), and metallurgical recovery (Table 6c) were statistically different ($p < 0.05$), with SE exhibiting lower response values than SE_{DAF} for all variables analyzed. A hypothesis to explain this phenomenon suggests that the presence of calcium and magnesium ions is detrimental to the flotation process for several types of ores, such as phosphate (GUIMARÃES and PERES [24]), iron (CRUZ *et al*. [25]; OLIVEIRA *et al.* [14]), zinc (ARAÚJO and LIMA [26]), lithium (LIU *et al*. [27]), and copper (JELDRES *et al*. [28]).

Table 7 presents the results of a comparison between the four types of water evaluated for variables of mass recovery, % P_2O_5 content and metallurgical recovery. For all evaluated responses, at least one effluent presented a statistically different average value ($p < 0.05$).

Table 4: Average results from ore flotation tests for the variables (a) mass recovery (%), (b) P_2O_5 content, and (c) metallurgical recovery.

VARIABLE	TYPES OF WATER			
	NW	TD	SE	SE_{DAF}
(a)	27.99 ± 0.33	25.46 ± 1.76	14.97 ± 0.07	24.97 ± 0.15
(b)	33.04 ± 0.09	33.70 ± 0.01	31.81 ± 0.10	33.29 ± 0.29
(c)	66.61 ± 0.95	61.78 ± 4.25	34.31 ± 0.04	59.88 ± 0.19

Table 5: Parameters of F-test for SE and SE_{DAF}, and results for response variables (a) mass recovery (%), (b) P_2O_5 content, and (c) metallurgical recovery.

NULL HYPOTHESIS	Ho: $\sigma_1^2 / \sigma_2^2 = 1$			
ALTERNATIVE HYPOTHESIS	$H_1: \sigma_1^2 / \sigma_2^2 \neq 1$	RESPONSE VARIABLE	TEST STATISTICS	VALUE-P
LEVEL OF SIGNIFICANCE	$\alpha = 0.05$	(a)	0.23	0.566
METHOD	F	(b)	0.12	0.419
DEGREES OF FREEDOM 1	1	(c)	0.03	0.233
DEGREES OF FREEDOM 2	1			

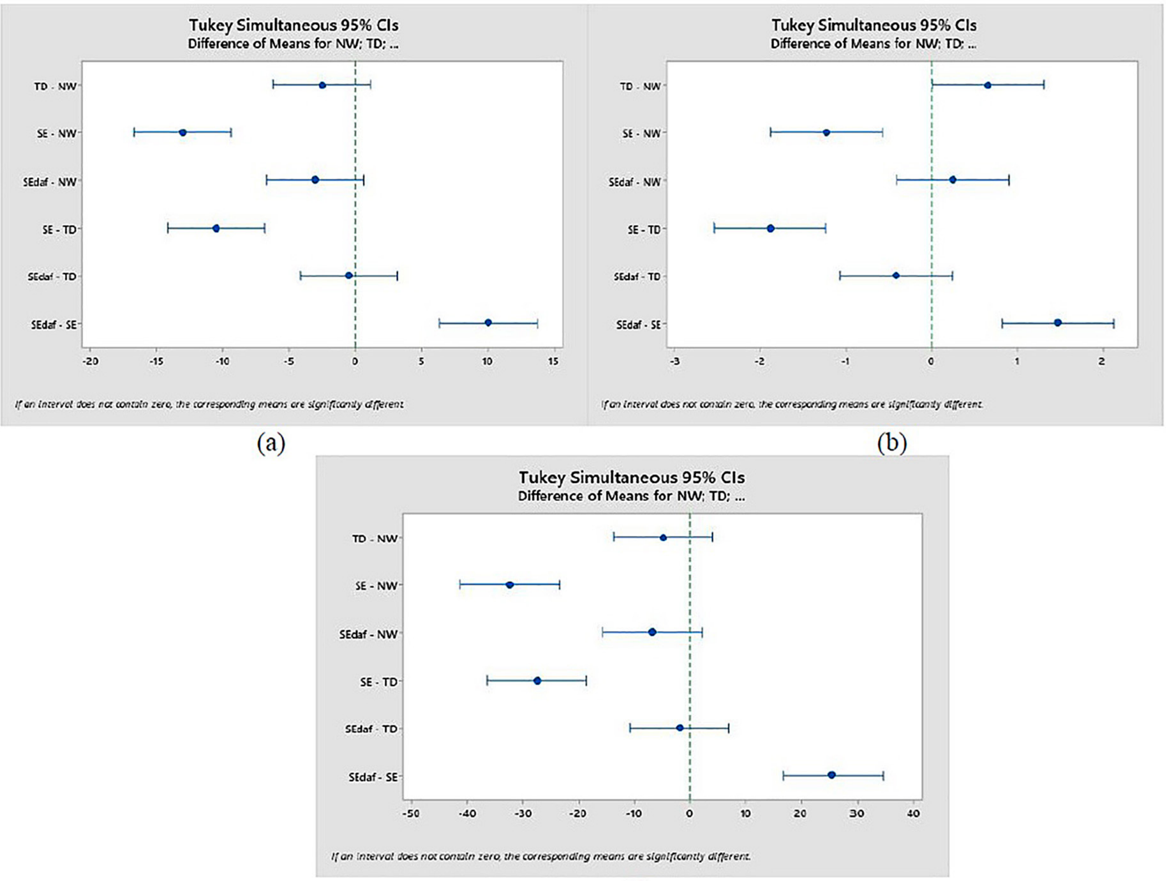
Table 6: Parameters of t-test with two means for SE and SE_{DAF}, and results for variables (a) mass recovery (%), (b) P_2O_5 content, and (c) metallurgical recovery.

NULL HYPOTHESIS	Ho: $\mu 1 - \mu 2 = 0$			
ALTERNATIVE HYPOTHESIS	H1: $\mu 1 - \mu 2 \neq 0$	RESPONSE VARIABLE	TEST STATISTICS	VALUE-P
LEVEL OF SIGNIFICANCE	$\alpha = 0.05$	(a)	–85.94	0.000
METHOD	t	(b)	–6.81	0.021
DOF	2	(c)	–186.24	0.000

Table 7: Result of ANOVA for the variables (a) mass recovery (%), (b) P_2O_5 content, and (c) metallurgical recovery.

	(a)			(b)			(c)		
SOURCE	**DOF**	**CONTRIB.**	**F**	**DOF**	**CONTRIB.**	**F**	**DOF**	**CONTRIB.**	**F**
Factor	3	98.39%	81.49	3	97.47%	51.43	3	98.52%	88.60
Error	4	1.61%	p	4	2.53%	p	4	1.48%	p
Total	7	100.00%	0.000	7	100.00%	0.001	7	100.00%	0.000

DOF: degrees of freedom; Contrib: contribution.



(a) (b)

(c)

Figure 2: Tukey test results of analyzed effluents for the variables (a) mass recovery (%) (b) content (% P_2O_5), and (c) metallurgical recovery (% P_2O_5).

Figure 2 shows the results of the Tukey test for analyzing the effluent variables of (a) mass recovery, (b) P_2O_5 content, and (c) metallurgical recovery. The Tukey test indicated that SE was the only effluent that presented statistically significant difference in average value of mass recovery (Figure 2a) and metallurgical recovery (Figure 2c). SE presented the highest concentrations of Ca^{2+} and Mg^{2+}, leading to the hypothesis that such ions impede the mineral flotation process, proven in the case of phosphate ore. For the P_2O_5 content, the Tukey test showed that in addition to the significant mean difference of SE, the TD and NW effluents also presented an unexpected difference among themselves. As the P_2O_5 content was a value obtained from quantitative chemical analysis, the difference between the TD and NW may be due to measurement error. Furthermore, the p-value is close to the level of significance used ($p = 0.048$). The statistical analysis revealed no significant difference in apatite flotation performance between TD and SE_{DAF} effluents. As depicted in Figure 2, the error bars for the three parameters assessed – mass recovery, grade, and metallurgical recovery – intersect zero, indicating their similarity.

PINHEIRO [29] states that the effectiveness of cationic collectors in mineral systems can be affected by the presence of specific cations in the water. The cations can create complex molecules with the collector, forming a precipitate and decreasing the availability of reagent molecules in the solution that are needed to promote the hydrophobization of minerals. GUIMARÃES and PERES [24] corroborated the findings of PINHEIRO [29], explaining that calcium and magnesium ions reacted with the anionic collector, forming insoluble soaps and decreasing the amount of reagent available for the collection of apatite, with this effect significant for ion concentration values above 30 mg/L. PAIVA *et al.* [30] verified that in flogopithite ore, the floatability of the apatite, calcite and dolomite minerals decreased abruptly when the solution reached concentrations of 20 mg/L of calcium and magnesium ions. For concentrations above this value, the floatability of carbonates increased, and the detrimental effect of calcium and magnesium ion concentration on apatite floatability was further proven. LANGSCH *et al.* [31] noted that one of the relevant factors in monitoring the pH value and the concentration of calcium and magnesium ions in the pulp was the precipitation of the ions in solution and adsorption on the mineral surfaces, an element that interferes with their floatability and in the selectivity of the apatite/carbonate separation. NANTHAKUMAR *et al.* [32] believed that Ca^{2+} and Mg^{2+} ions could not be avoided in industrial practice. In addition, phosphate minerals (and invariably present carbonates, such as calcite and dolomite) have poor solubility in water (salt-type minerals), with there always being a possibility of releasing small amounts of these cations in the pulp. However, such naturally low levels of cations do not pose major flotation problems.

4. CONCLUSIONS

Mining faces significant challenges due to the increasing need for primary sources of impure water with high ion levels and the utilization of recycled water, exemplified by tailings dam waters and those from solid-liquid separation operations. In the context of phosphate ore, the physical properties and solubility of apatite, the primary mineral, are crucial. Apatite releases ions into the beneficiation process water, potentially hindering its direct use in flotation as recycled water. Utilizing dissolved air flotation, along with coagulation and flocculation, may offer a viable solution for water treatment and reuse as mineral effluent.

The dissolved air flotation technique proved to be an effective alternative for treating simulated efluent, obtaining a liquid with acceptable concentrations of calcium and magnesium ions, with removal efficiencies of 91.6% and 66.4% respectively. The flotation results of phosphate ore in this study showed that higher concentrations of calcium and magnesium ions in the simulated efluent resulted in lower values for mass recovery, P_2O_5 content, and metallurgical recovery, proving the effect of water quality on the performance of the operation. Mineral flotation tests of simulated efluent treated by coagulation, flocculation, and dissolved air flotation produced average values of mass recovery, content, and metallurgical recovery that were similar to the results for NW. These results highlight the need for a water treatment stage in mineral processing plants and prove that the process presented in this study is a viable option to improve effluent quality.

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