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Feasibility study on apatite recovery from iron ore beneficiation tailings using flotation process

Mohadeseh Rajabi [a] and Parviz Pourghahramani [a, *]

[a] *Mining Engineering Department, Tabriz University of Technology, Tabriz, Iran.*

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ABSTRACT

With phosphorus demand for fertilizers and pharmaceuticals increasing and high-grade deposits diminishing, this study surveys the feasibility of recovering apatite from iron ore beneficiation tailings of the Sangan Company in Yazd Province, Iran, as a secondary resource. Optimization of the direct flotation process was examined through a full factorial experimental design considering collector dosage, pH, starch dosage, and sodium silicate dosage, with data processed by the Umetrics MODDE software and a multiple linear regression (MLR) model. The direct flotation process was designed to separate apatite; however, due to the surface similarities between apatite and carbonate minerals, such as calcite and dolomite, efficient separation could not be achieved, making an additional reverse flotation step necessary to remove carbonate minerals. To this end, the direct flotation concentrate underwent acid scrubbing to remove adsorbed collectors and was prepared with a mixture of phosphoric and sulfuric acids to depress apatite, followed by reverse flotation with sodium oleate as the collector at pH 4.5. Reverse flotation yielded a final concentrate exceeding 30% P_2O_5 content and 70% recovery. These findings show that these tailings can be processed as an apatite source, offering an economic resource while reducing tailings and reusing them for environmental preservation.

Keywords: *Apatite recovery, Secondary resource, Direct flotation, Reverse flotation, Iron ore tailings.*

1. Introduction

Phosphorus is a critical element for the production of fertilizers, detergents, and pharmaceuticals and occurs naturally in the form of phosphates, mainly as apatite ($Ca_5(PO_4)_3(F, Cl, OH)$), which is a major component of the global biogeochemical phosphorus cycle (Lishmund et al., 1982; Filippelli, 2002, 2008; Cordell et al., 2009; Vaneeckhaute et al., 2016). Apatite is a mineral found extensively as an accessory in multiple geological deposits. In specific instances, such as phosphorites and apatite-magnetite deposits, it emerges as a principal mineral, gaining economic importance (Harlov, 2015).

Effective beneficiation of phosphate ore can be realized through a multitude of methodologies, contingent upon the liberation size of phosphate and gangue minerals, as well as other phosphate ore characteristics, such as the type of associated gangue minerals. A variety of techniques, including screening, scrubbing, heavy media separation, roasting, calcination, leaching, and flotation, may be employed (Gharabaghi et al., 2010). The most common method to beneficiate phosphate ore is flotation, generating more than 60% of the world's marketable phosphate by separating minerals according to their surface properties (Abouzeid, 2008). The occurrence of carbonate gangue minerals, such as calcite and dolomite, which also have similar surface characteristics to phosphate minerals, makes their separation through flotation difficult. Anionic fatty acid collectors, employed in direct flotation, adsorb on both surfaces, lowering selectivity and efficiency (Kawatra & Carlson, 2013). To overcome this challenge, reverse flotation has been employed to remove carbonate gangue by floating carbonates while depressing apatite. This is often achieved using fatty acids as collectors for carbonates and inorganic acids (such as H_3PO_4 or H_2SO_4) as apatite depressants (Amirech et al., 2018; Huang & Zhang, 2024). Also, novel collectors and depressants have improved apatite flotation efficiency and selectivity. For instance, 2-chloro-9-octadecanoic acid (2-Cl-9-ODA) effectively separates apatite from dolomite and calcite. When combined with sodium pyrophosphate as a depressant, it achieves high recovery while reducing gangue flotation (W. Zhang et al., 2025). Carbon dioxide supplanted air during the preliminary flotation phase to selectively extract carbonates. This was succeeded by a subsequent phase to float apatite, culminating in a concentrate with an excess of 21% P_2O_5. Nevertheless, residual contaminants, such as magnetite and carbonates underscored the necessity for further process optimization (Carvalho et al., 2022). A novel collector synthesized from waste frying oils was found to exhibit selective adsorption on carbonate minerals like calcite and dolomite. This reagent showed excellent performance in separating carbonate gangue minerals from apatite, achieving over 80% recovery of calcite and dolomite, while the recovery of apatite in the carbonate concentrate remained below 10%, indicating high selectivity (El-bahi et al., 2024).

Due to an increasing demand for phosphorus fertilizers and the depletion of high-grade deposits, research and extraction efforts have focused on secondary resources. The circular economy approach is another alternative in this context to manage mineral wastes and recover valuable elements (Alsafasfeh & Alagha, 2017). Magnetic tailings from iron oxide-apatite deposits have emerged as a potential sustainable phosphorus-supplying tool, and previous experiments have confirmed that apatite is recoverable from these sources with high grade and recovery obtained via flotation (Valderrama et al., 2024). Studies on the recovery of apatite from iron ore beneficiation tailings in Iran have been neglected, particularly with respect to tailings generated by the Sangan

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* Corresponding author. *E-mail address:* pourghahramani@sut.ac.ir (P. Pourghahramani).

Company. Moreover, previous investigations have not yielded satisfactory or industrially viable results.

In this study, the tailings produced during the iron ore beneficiation process at the Sangan Company in Yazd Province, Iran were investigated to evaluate the feasibility of apatite recovery from secondary resources. The average P_2O_5 grade of this tailings deposit is around 4%. First, in order to determine the optimal operating conditions to achieve a proper grade and recovery, the effect of variables that affect direct apatite flotation (collector dosage, pH, depressant dosage) was investigated. Then, a reverse flotation stage was tested to separate carbonate minerals from apatite, in order to produce an acceptable industrial grade of concentrate (P_2O_5 grade more than 30%).

2. Material and methods

2.1. Sample collection and preparation

Random sampling was conducted on the iron ore beneficiation tailings from the Sangan Company to obtain representative samples. A total of 250 kilograms of dry, mostly agglomerated tailings was collected for laboratory studies. Fig. 1 presents the cumulative particle size distribution of a representative tailings sample, with a D_{80} of 460 µm. Approximately 39% of the particles are smaller than 37 µm, accounting for 44.85% of the total P_2O_5 content. This fine fraction may increase entrainment during flotation and reduce the selectivity of separation.

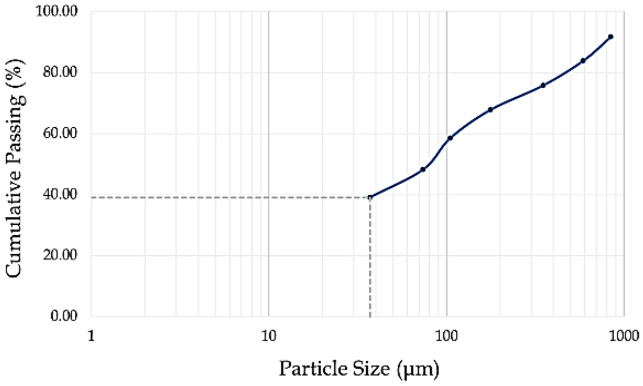

Fig. 1. Cumulative particle size distribution of the representative tailings sample.

2.2. Mineralogical studies

Mineralogical investigations on different fractions from sieve size analysis were performed with the preparation of thin sections to analyze both mineral composition and distribution. Thin sections were observed under ordinary and polarized light using a polarizing microscope to identify minerals based on optical properties. The observation of plastic, xenomorphic texture in the samples indicates the simultaneous formation of minerals under intense metamorphic conditions and high pressure and temperature. Thin section mineralogy revealed that most constituent minerals of the sample were alkali feldspar, amphibole, calcite, dolomite, and quartz. Most alkali feldspar minerals have undergone intense alteration processes and have been transformed into secondary minerals, such as clay minerals, epidote, and chlorite. The investigation results suggest that part of the calcite present in the sample was formed due to the alteration of primary minerals, while another part has a primary origin. Plagioclase occurred as widely distributed, liberated fine-grained crystals. As shown in Fig. 2, apatite grains in the coarse fractions showed internal fractures and most of them were associated with iron oxides. The degree of liberation analyses revealed that over 85% of apatite liberates in particle sizes below 75 µm.

2.3. Reagents

Procoll flo-ys20, sodium oleate, and Linaz-20 were used as collectors in this study. The collectors had frothing properties; thus, no additional frother agent was required. pH was adjusted with sodium hydroxide and sulfuric acid. Sodium silicate and starch were used as depressants, where sodium silicate depressed silicate and aluminosilicate minerals while dispersing the pulp, and starch depressed iron oxides and carbonate minerals. Sulfuric and phosphoric acids were used to depress apatite.

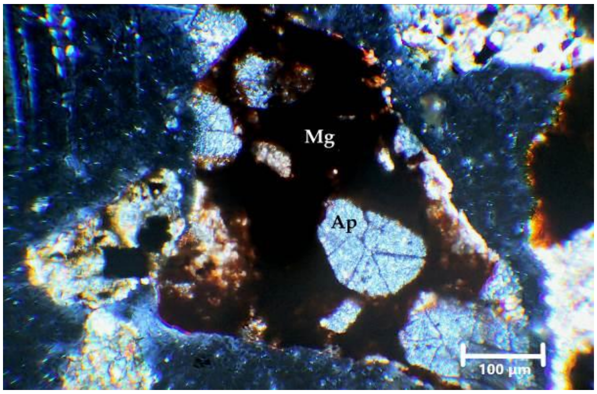

Fig. 2. Apatite grains associated with magnetite under the cross-polarized light (-350 +177 µm).

2.4. Direct flotation tests

2.4.1. Experimental design

A full factorial experimental design was employed to conduct flotation experiments for investigating the effects of several variables on the process. Four important parameters were evaluated at two levels for each factor: collector dosage (500 and 1500 g/t), pH (9.5 and 11.5), sodium silicate dosage (0 and 500 g/t), and starch dosage (0 and 400 g/t). The levels of the parameters were determined based on preliminary experiments. In total, 16 experiments were conducted along with 3 additional replicate tests at the central points of the parameters to ensure statistical reliability.

2.4.2. Conditions

Flotation tests were carried out in a Denver flotation machine with a 5-liter cell, and the flotation feed particle size was kept below 75 microns so that most of the apatite was liberated. At the start of each test, the pulp pH was adjusted according to the experimental design. If required, depressants were introduced first, followed by a combination of Procoll flo-ys20 and Linaz-20 collectors, with 70% added during the rougher stage and 30% in the scavenger stage. The solids content (25%), impeller speed (1000 rpm), aeration rate, and pulp conditioning time were held constant throughout all experiments, and the flotation time for each sample was set at 20 minutes. After completing the flotation process, the concentrate was collected, filtered, dried, weighed, and analyzed to assess its grade.

2.5. Reverse flotation test

2.5.1. Acid Scrubbing

Based on the similarity in the surface properties among calcite, dolomite, and apatite, their separation through direct flotation is difficult (Kawatra & Carlson, 2013). Accordingly, a reverse flotation stage was necessary. First, the direct flotation concentrate underwent acid scrubbing to eliminate adsorbed collectors. The concentrate, with a solids content of 50%, was agitated at pH 4.5 (adjusted with sulfuric acid) for 15 minutes to desorb the collectors. Subsequently, the pulp was washed and filtered to remove the acid and collectors, thereby improving the performance of the reverse flotation.

2.5.2. Conditions

Reverse flotation of carbonate minerals was also carried out in a Denver flotation machine with a 5-liter cell, similar to the direct flotation tests. To depress apatite, the solid recovered from the acid scrubbing was conditioned with a mixture of sulfuric and phosphoric acids at pH 4.5 and a solids content of 50% for 10 minutes. Following conditioning, the carbonate minerals were floated with 500 g/t sodium oleate as a collector at a solids content of 25%, and other conditions, such as flotation time, aeration rate, and impeller speed, were consistent with those of direct flotation. Subsequently, the concentrate was collected, weighed, and analyzed for grade determination.

2.6. Analysis and evaluation of results

The concentrates, from direct flotation and reverse flotation, were analyzed using a UV Specord 200 spectrophotometer by colorimetry. Apatite initially was dissolved in nitric acid and water solution (1:5), and then phosphorus-specific chemical reagents were added, resulting in the formation of phosphate ions in the complex phase and colored solutions. The P_2O_5 grade of the samples was then determined by comparing the color intensity of the test samples with those of standard solutions. Data from the direct flotation experiments were evaluated using the Umetrics MODDE software and the multiple linear regression (MLR) method. Model quality was evaluated using key statistical metrics (R^2 close to 1, $Q^2 > 0.5$, reproducibility > 0.5, model validity > 0.25, and R^2–$Q^2 < 0.3$) where compliance with these criteria confirms a robust fit to laboratory data, together with a reliable prediction of new outcomes.

3. Results and discussion

3.1. Direct flotation tests

3.1.1. Model fitting

Table 1 presents the experimental design along with the results of 19 direct flotation tests. The values of the MLR model evaluation metrics are provided in Table 2. The $R^2 = 0.80$ and $Q^2 = 0.57$ obtained for the grade response are representative of suitable values, with model validity (0.80) and reproducibility (0.80) values that exceed the model validity and reproducibility thresholds (0.25 and 0.5 respectively) and the R^2–Q^2 difference (0.23) within the acceptable range. For the recovery response, fitted values were strong ($R^2 = 0.93$; $Q^2 = 0.82$) and highly predictive, with model validity (0.58), reproducibility (0.97), and R^2–Q^2 difference (0.11), all within acceptable limits. All of these indicators confirm the model adequately describes the experimental data and can be employed in optimizing the direct flotation process. Relative relationships between the operational parameters and grade and recovery responses were analyzed through regression coefficients of the multiple linear regression (MLR) model at a confidence level of 95%. The outputs of this analysis are shown as plots of regression coefficients for grade and recovery in Fig. 3.

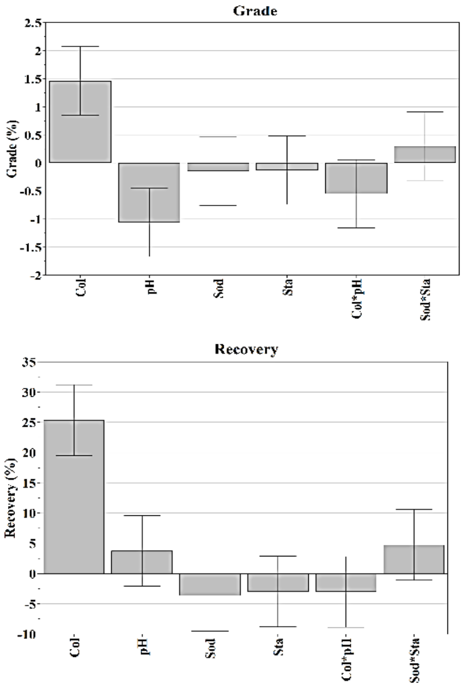

Fig. 3. Regression coefficient plots for grade (left) and recovery (right) at 95% confidence level.

Table 1. The experimental design and results of direct flotation tests (Continued on the next page).

Exp No	Factors				Responses	
	Collector (g/t)	pH	Sodium silicate (g/t)	Starch (g/t)	Grade (%)	Recovery (%)
1	500	9.5	0	0	7.32	47.46
2	1500	9.5	0	0	8.63	83.47
3	500	11.5	0	0	4.78	38.79
4	1500	11.5	0	0	6.46	87.89
5	500	9.5	500	0	4.99	12.53
6	1500	9.5	500	0	9.68	76.96
7	500	11.5	500	0	3.78	34.14
8	1500	11.5	500	0	5.15	66.97
9	500	9.5	0	400	3.75	9.09
10	1500	9.5	0	400	9.18	71.4
11	500	11.5	0	400	4.35	34.22
12	1500	11.5	0	400	6.49	81.25
13	500	9.5	500	400	5.01	14.58
14	1500	9.5	500	400	9.68	78.97
15	500	11.5	500	400	4.1	30.8
16	1500	11.5	500	400	6.2	80.52
17	1000	10.5	250	200	8.7	68
18	1000	10.5	250	200	7.8	70
19	1000	10.5	250	200	6.9	74

Table 2. Summary of fit values for the MLR model.

	R2	Q2	Model Validity	Reproducibility
Grade	0.80	0.57	0.80	0.80
Recovery	0.93	0.82	0.58	0.97

Based on the final refined model, the collector dosage is the most significant main effect affecting concentrate grade and recovery at a 95% confidence level. The factor pH appears to be the second most important statistically significant factor influencing the concentrate assay. In addition, the interaction between two depressants affects statistically the responses in a different manner. A more detailed explanation is given in the following sections.

3.1.2. *Collector dosage effect*

As depicted in Fig. 3, collector dosage had a significant (P-value < 0.05) and positive effect on both grade and recovery, making it the key parameter with the greatest influence on apatite flotation. The influence of collector dosage on flotation performance was evaluated by examining its effect on both grade and recovery, as illustrated in Fig. 4, using a main effects plot. The plot on the left-hand side illustrates a positive relationship between the grade of the final concentrate and the dosage of collector. By increasing collector from 500 g/t to 1500 g/t, concentrate assay increases from 5 to almost 8% in P_2O_5. The recovery curve on the right plot reveals that by increasing collector to 1500 g/t, the recovery of apatite increases up to 81%. The confidence intervals (dotted lines) for both grade and recovery curves suggest a statistically reliable trend. In summary, an increase in grade and recovery can both be achieved through a higher collector dosage. However, the recovery rates are more significantly improved.

One of the major reasons why higher collector dosages are imperative is because the feed comprises a considerable fraction of fine particles (<37µm). The reagent demand is reasonably high for these fine particles due to their greater surface area. The other reason which would demand more quantity of collector to improve flotation efficiency is the greater sensitivity of fatty acid collectors to dissolved ions (e.g., Mg^{2+}) present in the pulp. Due to the quality of the process water, along with mineral dissolution and the abrasion of grinding media, certain metal ions, such as Ca^{2+}, Mg^{2+}, Al^{3+}, and Fe^{3+} are inevitably present in the flotation pulp under actual operating conditions (Ruan et al., 2018). Fatty acid collectors have the tendency to easily form complexes with these ions, and thus there is greater collector consumption required for effective flotation (dos Santos et al., 2010). Also a higher collector dosage results in higher hydrophobicity of apatite particles, resulting in better recovery and grade (Santana et al., 2010; Testa, 2016). The collector concentration also has a significant effect on the flotation kinetics of apatite in alkaline environments. For example, a study found an improvement in flotation rate and recovery when collector concentration was increased (Testa, 2016).

3.1.3. *PH effect*

Based on Fig. 5, the effect of pH on apatite flotation demonstrates an inverse relationship between grade and recovery. As pH increases from 9.5 to 11.5, the P_2O_5 grade of the concentrate drops from approximately 7.5% to below 5.5%, while recovery slightly increases from around 52% to nearly 60%. However, according to the regression coefficients shown in Fig. 3, the effect of pH on recovery is not statistically significant at the 95% confidence level. Nonetheless, the declining trend in grade appears more consistent and meaningful.

This pattern can be partially attributed to the influence of solution chemistry and pH on flotation behavior. In fact, although the flotation of quartz cannot be achieved using fatty acid collectors, it may be activated using multivalent cations like Mg^{2+}, Fe^{3+}, and Al^{3+} to enhance its floatability within a pH range of 9 to 10. In addition, the addition of Ca^{2+} ions have also been proven to increase the flotation of the quartz within the pH range of 9.5 to 11.5 (Ruan et al., 2018). In addition, this effect may be explained by the impact of alkaline conditions on the behavior of fatty acid collectors. Specifically, high pH values cause the collectors to exhibit an increased frothing ability and reduced average bubble size, which enhances gas retention in the flotation cell. Under such conditions, excessive froth stability can entrap gangue particles within the froth phase (Atrafi et al., 2012). Together, these factors impair the selectivity of separation, thereby reducing concentrate purity despite the presence of a marginal increase in recovery levels. Thus, accurate pH adjustment is needed to obtain a trade-off between grade and recovery.

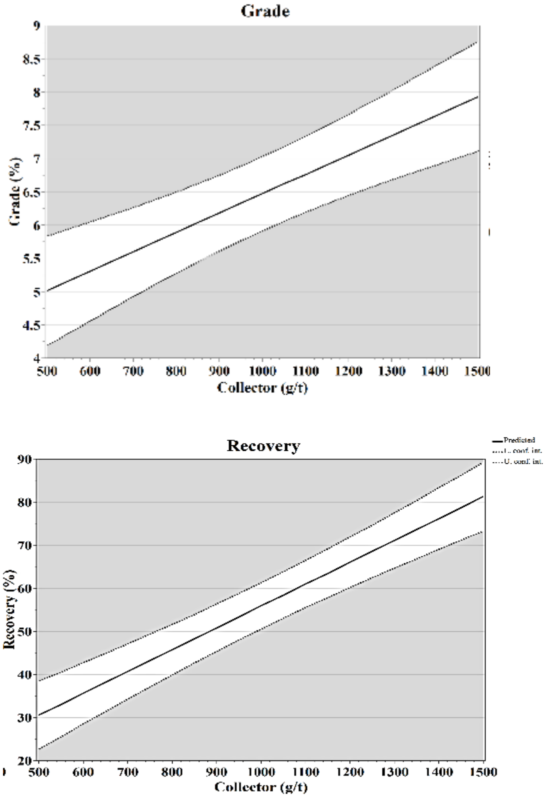

Fig. 4. The main effects plot of collector dosage on grade (right) and recovery (left) of apatite in direct flotation.

3.1.4. *Interaction effects*

According to Fig. 3, the only significant interaction affecting the process is the effect of the starch and sodium silicate interaction on recovery. Fig. 6 shows the interaction plot. At low starch dosages, with an increased amount of sodium silicate, recovery decreased sharply from approximately 67% to 50%. In contrast, at high starch dosages, recovery increased slightly from about 51.7% to 54%. This implies that at low starch concentrations, sodium silicate causes excessive depression that significantly reduces recovery, but at higher starch dosages, this effect is partially mitigated. These results highlight the importance of the interaction between these two depressants on the flotation performance.

3.1.5. *Process optimization*

Optimization of the direct flotation process was performed using response surface plots for grade and recovery (Fig. 7). The plots that are shown depict the effect of collector dosage and pH levels while keeping starch dosage constant at zero, because its effect on flotation efficiency is statistically insignificant and slightly detrimental. Sodium silicate was maintained at an optimized level of 100 g/t to support pulp dispersion. The maximum grade (9-10%) was achieved at a pH range of 9.5-10.1 and high collector dosage (1220-1500 g/t), while raising pH to 11.5 and reducing collector dosage to 500 g/t decreased the grade to its lowest value (~5%), as shown by the response surface plots. This decline in grade at higher pH levels can be attributed to the reduced selectivity of fatty acid collectors. The negative effect of increased pH on grade and positive effect of collector dosage on grade and recovery are validated from these results. Also, the significant grade drop evident at lower collector doses indicates the increased reagent consumption for this sample, suggesting that a high enough collector dosage is necessary to maintain good flotation performance. In contrast, a maximum recovery (80-90%) was achieved over a wide pH range (9.5 to 11.5) and high collector dosage (1300-1500 g/t), but decreased to 45-50% with decreasing pH to 10.5 and collector dosage of 560 g/t.

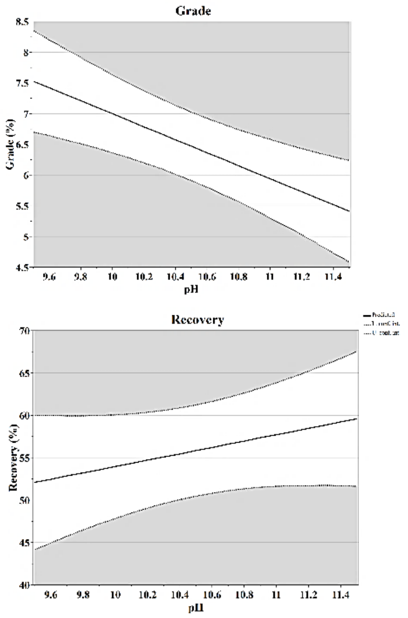

Fig. 5. The main effects plot of pH on grade (right) and recovery (left) of apatite in direct flotation.

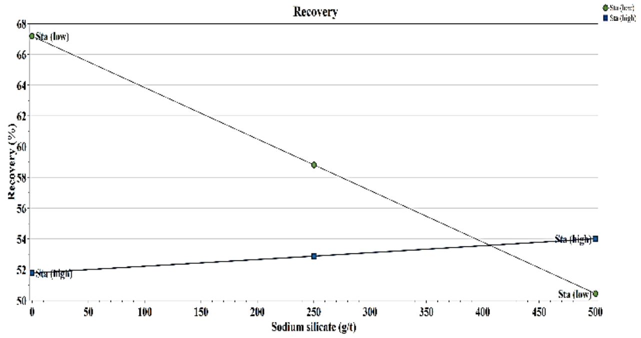

Fig. 6. The interaction plot of sodium silicate and starch on recovery.

Using a sweet spot plot, which ascertains the operational conditions under which multiple response criteria are concurrently fulfilled, the direct flotation conditions were optimized. The plot is for simultaneous high grade and recovery (≥9% and ≥80%, respectively) with sodium silicate (100 g/t) and starch (0 g/t) set as constant. The upper limits for grade and recovery are as per the experimental design outcome that showed the maximum ranges of responses. The results (Fig. 8) revealed that the desirable region (green) is located at pH 9.5-9.7 and collector dosage of 1373-1500 g/t. At higher pH and lower collector dosage, only one criterion (blue) or neither (white) is met. This plot shows the appropriate pH range between 9.5 and 9.7 and collector dosage of 1373 to 1500 g/t to achieve optimal grade and recovery for direct flotation, with sodium silicate dosage of 100 g/t and no starch addition. Using the optimizer tool in MODDE software, the precise optimal conditions were determined to be a pH of 9.5, a collector dosage of 1500 g/t, and a sodium silicate dosage of 100 g/t. Under these conditions, a concentrate of approximately 9.8% grade and 82% recovery can be obtained. Similar results in tests conducted under these optimal conditions also validate the authenticity of such data.

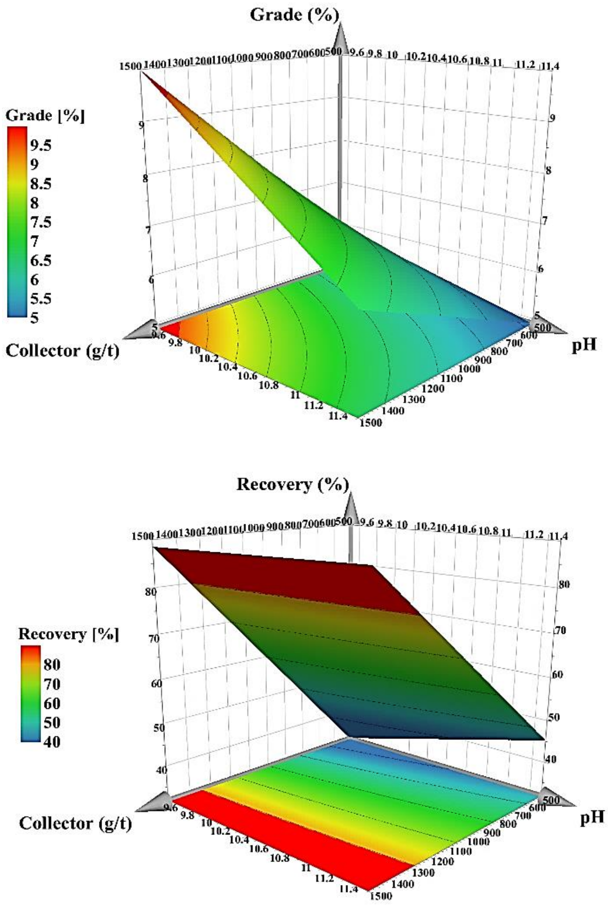

Fig. 7. The response surface plots of grade (left) and recovery (right).

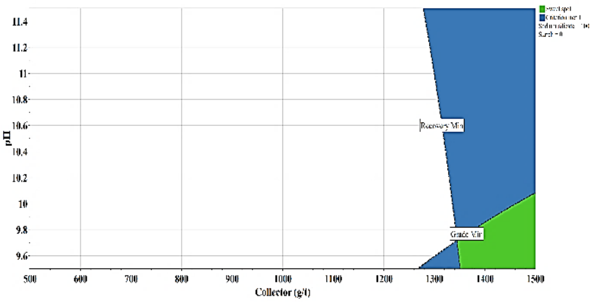

Fig. 8. The sweet spot plot for optimal grade and recovery conditions.

3.2. Reverse flotation test

As described in Section 2.5, the reverse flotation test was performed to separate carbonate minerals from the direct flotation concentrate, although due to surface similarities between carbonate minerals like calcite and dolomite and apatite, their separation via direct flotation was not feasible, necessitating a reverse flotation step to float carbonate minerals. The findings demonstrated the successful removal of carbonate minerals by reverse flotation, resulting in a final product with more than 30% P_2O_5 content and more than 70% recovery. The final process flowsheet is presented in Fig. 9. The acid scrubbing step (Section 2.5.1) enhanced the selectivity of carbonate flotation by desorbing collectors from the concentrate. It has been established that phosphoric acid and sulfuric acid are effective depressants for reverse flotation of phosphate ores under acidic conditions (Huang & Zhang, 2024).

Previous studies have confirmed that using a mixture of sulfuric and phosphoric acids is more effective for apatite depression than using either acid alone. This is why a mixed acid system was used in the present work. The mixed acid system decreases the contact angle of apatite to the lowest state, which is effective depression (Lai et al., 2023). The mechanism for the depression of apatite by phosphoric acid and sulfuric acid at pH 4.5 involves further chemical adsorption of H_3PO_4 onto apatite surfaces, leading to the formation of hydrophilic species including $CaHPO_4$, $Ca(H_2PO_4)_2$, and $CaH_2PO_4^+$, which restrict collector adsorption on apatite surfaces. Among these, $CaHPO_4$ is considered the primary species responsible for effective apatite depression (Liu, Ruan, et al., 2017; Huang & Zhang, 2024). In addition, sulfuric acid contributed further to the overall effect by forming a poorly soluble $CaSO_4$ layer on apatite surfaces, preventing collector access to the calcium sites (Liu, Luo, et al., 2017; H. Zhang et al., 2025). These results indicated that reverse flotation is a suitable complementary method to direct flotation, improving the quality of the apatite concentrate for industrial use. These grade and recovery values can be further optimized by adjusting collector consumption in this stage or by fine-tuning pH during the acid scrubbing or preparation with sulfuric and phosphoric acids for apatite depression. These findings show that these tailings can be processed as an apatite source, offering an economic resource while reducing tailings and reusing them for environmental preservation.

4. Conclusions

The feasibility of apatite recovery from secondary resources was studied using the tailings from iron ore beneficiation at the Sangan company in Yazd Province, Iran. Mineralogical investigations demonstrated that the dominant gangue minerals in the tailings, in turn, are alkali feldspar, quartz, calcite, and dolomite. A full factorial experimental design was employed to optimize the direct flotation process, and the results were processed using the Umetrics MODDE software and the MLR model. High grade and recovery (≥9% and ≥80%, respectively) in the direct flotation were achieved under optimized conditions at a pH range of 9.5-9.7 and collector dosage of 1373-1500 g/t, with sodium silicate dosage of 100 g/t and no starch addition. Microscopic examinations revealed that more than 85% of the apatite in the sample is fully liberated in the size fraction below 75 μm, indicating that selectivity, rather than liberation, is the primary challenge in this flotation process. The subsequent reverse flotation test successfully removed the carbonate minerals and improved the concentrate quality, resulting in a final grade more than 30% P_2O_5 and a final recovery more than 70% for the entire process. In reverse flotation, both phosphoric and sulfuric acids were used to depress apatite, leading to an increase in selectivity. These results demonstrate that the integration of direct and reverse flotation is indeed a potential route for producing high-quality apatite concentrates, with strong potential for industrial scale-up and further process enhancement.

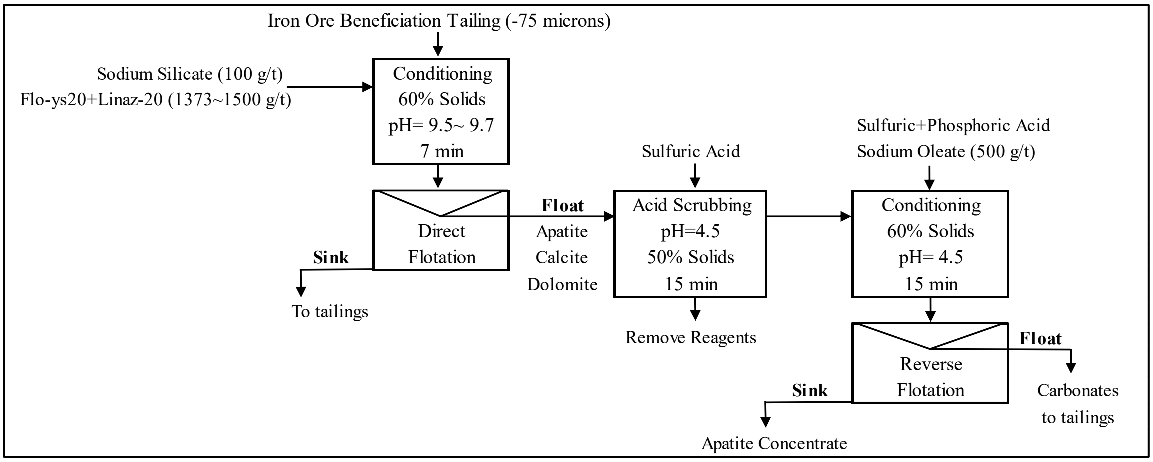

Fig. 9. Final flowsheet for the recovery of apatite from iron ore beneficiation tailings.

Acknowledgement

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