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Original Research Paper

New Insights into the Pre-Concentration of Alunite from a Siliceous ore using Froth Flotation

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Abstract: Alunite is the main non-bauxite source of aluminum. Due to the low Degrees Of Freedom (DOF) of alunite, froth flotation, as a pre-concentration method, is a viable alternative to increase selectivity in downstream processes. The current study was aimed at investigating the pre-concentration of alunite using micro-flotation. In the first step, an optical microscope studies were done to determine DOF of alunite. In the next step, flotation tests were designed. The experiments were performed based on the surface charge differential between alunite and quartz using direct and reverse flotation. In the reverse flotation, Ca^{+2} and Ba^{+2} were used to activate the quartz surface. Anionic collector was used to float the gangue minerals. The highest grade and recovery using reverse flotation were 34.96 and 81.33%, respectively. In the direct flotation, the surface of alunite was activated using Ba^{2+} , Ca^{2+} and F^- . The optimum condition was achieved using F^- as an alunite activator for particles ranged $-37+25 \mu\text{m}$ with the grade of 31.47%. The grade and recovery were 50 and 89%, respectively. All experiments were conducted in Hallimond Tube. During the experiments, several parameters were controlled.

Keywords: Alunite, Flotation, HF, NaF, CaO, BaCl_2

Introduction

Aluminum is the most abundant metal element and the third most abundant element on the earth. This metal because of its unique properties has high consumption in a wide variety of industries such as aerospace, building industries, transportation and consumer goods (Tabereaux and Peterson, 2014; Russell, 1981; Eshkalak *et al.*, 2020). One of the main sources to extract aluminum is bauxite. The depletion of economical bauxite sources in the world and increasing of aluminum price, increasing aluminum demand in the world and non-availability of bauxite sources in various countries have led to attempts to find alternative sources for it (Haraldsson and Johansson, 2018; Cullen and Allwood, 2013; Milford *et al.*, 2011). Thus, discovering and exploiting, extracting and processing of alternative sources have significant importance for the industries to provide their aluminum demands. All mentioned reasons have encouraged researchers around the world to find the solutions for this challenge.

The non-bauxite sources of aluminum are divided in two categories, namely, silicate and non-silicate. Silicate

sources include water-based silicates (clays) and non-water-based silicates (such as nepheline syenite). Non-silicates such as alunite is one of the potential and alternative sources of alumina (Dixit and Miller, 1980; Smith *et al.*, 1982).

The extraction of alumina from alunite has its own difficulties and disadvantages. They include (1) the low concentration of alumina in alunite: Alunite feed consists of two-third of gangue, mainly quartz and clay, which results in increased transportation costs as well as processing costs in hydrometallurgical circuits. (2) Significant losses of alumina and leaching agents. The most common technique for aluminum extraction from alunite is hydrometallurgical methods including calcination and leaching. Because the main gangue of alunite is quartz (around 40%), these methods lead to the formation of *sodium aluminosilicate* and *potassium aluminosilicate* complexes. These complexes cause losing alumina and leaching agents during the process. (3) Because of alunite and its gunge properties, most methods are not applicable to preconcentrate it (Dehghani *et al.*, 2019). The low differences in densities of alunite and its companion minerals such as clay and

quartz make density-based separators such as jig, shaking table, Humphrey spiral and so on non-suitable. Moreover, due to the uniform distribution of grade in different size fractions, the use of sieve bend or cyclone is not useful. Among various processing methods, froth flotation seems to be a useful method (Eskanlou *et al.*, 2019). However, the low Degree Of Freedom (DOF) of alunite makes froth flotation a complex method to purify alunite. In this study, it will be focused on the flotation as a pre-concentration method for the purification of alumina (Loest and Schaefer, 1978; Küçük and Gülaboğlu, 2002).

Koca and Koca (2000) studied the carrier flotation of alunite from kaolin in Bahkesir, Turkey. He used calcite as a carrier, sodium silicate (Na₂SiO₃) as a dispersant and sodium oleate as a collector. Under the optimized condition, 57.95% of alunite was recovered. Selective flocculation was used to separate alunite (45%) and quartz (55%) from Utah alunite reserves. In order to do selective flocculation, 1% of magnetite with an average size of 5 µm was added to the pulp. Then non-ionic flocculent of Poly Acrylamide was added to selectively flocculate quartz and magnetite. The disadvantage of this method was the significant amount of valuable mineral was mechanically trapped, especially when the percentage of solid in the pulp was more than 5%. Another challenge was the sensitivity of selective flocculation because of the presence of other ions in the pulp. However, in the case of artificial mixtures of alunite and gangue minerals using both flotation and flocculation to separate alunite from gangue mineral were somewhat successful, but, the separation of natural ores using conventional methods were not satisfactory because of low degree of freedom of alunite (Ustaer and Gürgey, 1975). Taghiyev *et al.* (2019) used flotation

method to enrich alunite ore. They improved the grade of alunite from 50-51 to 60%. Followed by, soda-alkaline method of alunite ore processing to purify alunite.

A limited number of researches have been performed in the field of pre concentration of alunite based on flotation method. None of them have reported the effect of the presence of fluoride ion on alunite flotation capability. In this study, micro-flotation was employed to process natural alunite ore using fluoride ions (F⁻). The advantage of using the micro-flotation method is no changes happen in pulp components during the flotation process. Reverse and direct flotation were performed using Ca⁺² and Ba⁺² as activator. In addition, the effectiveness of various parameters on flotation of alunite such as pH, particle size, collector type and concentration were considered.

Materials and Methods

Materials

The representative sample was obtained from Haft Sandogh alunite deposit, Iran, where the reserve was estimated to be about 500 MT of alunite with the grade above 20%. Chemical and mineralogical analyses were done by X-Ray Fluorescence (XRF) spectrometry and X-Ray Diffraction (XRD) analysis methods, respectively. The mineralogical analyses indicated the sample had more alunite than quartz and more quartz than kaolinite. The results of the chemical and mineralogical analyses of samples are given in Table 1 and Fig. 1. Table 2 represents the chemicals that were used in the froth flotation process. All chemicals except Sulfonate (manufactured by CYTEC) are manufactured by Merck Company.

Table 1: Chemical composition of the sample by the XRF technique (Wt %)

SiO ₂	SO ₃	Al ₂ O ₃	K ₂ O	Na ₂ O	CaO
44.41	20.04	19.7	4.55	0.88	0.85
MgO	Fe ₂ O ₃	MnO ₂	TiO ₂	P ₂ O ₅	L.O.I
0.74	0.6	0.46	0.25	0.24	7.2

Table 2: Type and dosage of chemicals used in the froth flotation process

Type of chemicals	The effect of chemicals	Dosage
Sodium hydroxide (NaOH)	pH modifier	Until pH adjusts
Hydrofluoric acid (HF)	pH modifier, Surface activator (it imposes negative charge on alunite surface)	Until pH adjusts
Sodium fluoride	It imposes negative charge on alunite surface	1*10 ⁻² mol/l
Armac C	cationic collector	1500 gr/ton
AERO Promoter 801 ¹	Anionic collector	300 mg/kg
Sodium Silicate ^{II}	Quartz depressant	1250 gr/ton
Sodium hex metaphosphate	Alunite depressant	
Pine oil	Frother	2-10 drops
Methyl Isobutyl Carbinol (MIBC)	Frother	2-10 drops
Barium chloride	Quartz activator	1*10 ⁻³ kmol/m ³
Calcium Oxide	Quartz activator	1*10 ⁻³ kmol/m ³

¹ These sulfonates are manufactured by CYTEC

^{II}Na₂SiO₃

^{III}All materials with the exception of Sulfonate are manufactured by Merck Co

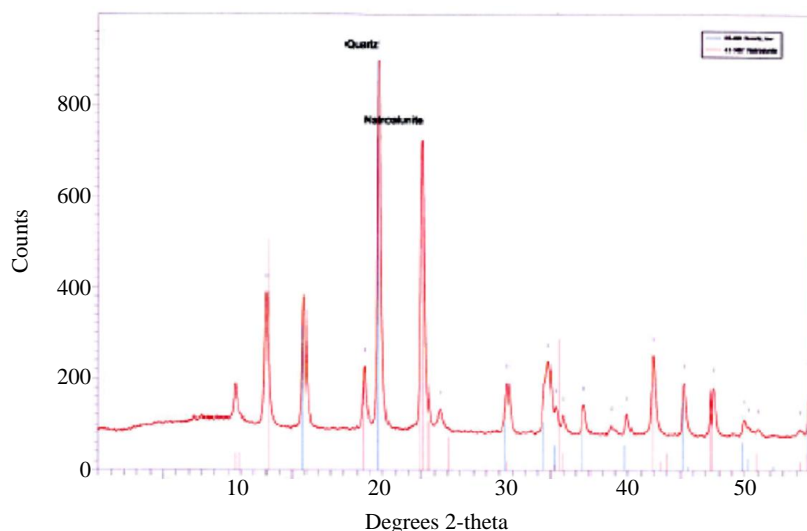


Fig. 1: XRD studies for the representative samples

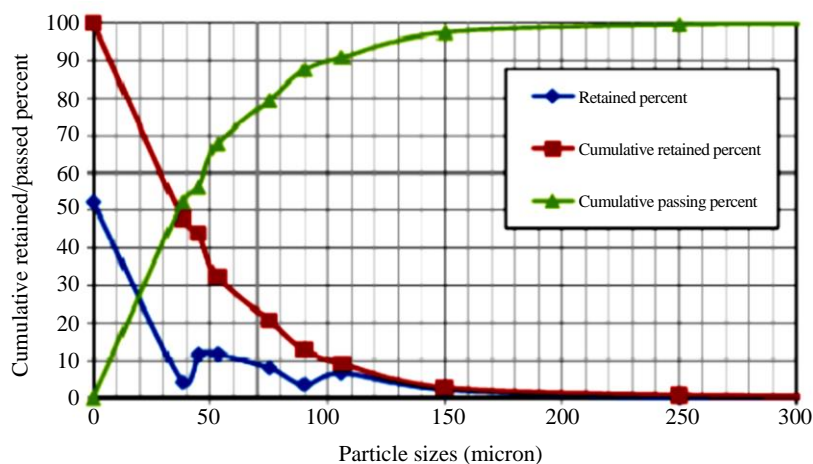


Fig. 2: Cumulative passing and retained percent of ore after 20 min grinding by ball mill

Crashing and Grinding Tests

Samples were crushed in a jaw crusher, a cone crusher and a roll crusher to produce particles with a size of -2380+841 μm . In the next step, the samples were ground in a laboratory-scale Denver ball mill of 200 mm internal diameter, the volume of 950 cm^3 , loaded with 6 steel balls with 42 mm diameter, 21 balls with 31 mm diameter and 42 balls with 25 mm diameter and a mass load of 1000 gm. The grinding time in the ball mill was 20 min. The particle size distribution of ball mill product is given in Fig. 2. The ball mill products were divided into four fractions using a shaker sieve. Fractions are including -74+44, -44+37, -37+25, -25 μm .

Optical Microscope Studies

Before preparing samples for optical microscope studies, iron was removed from the sample. Although

the iron content in the samples is only 1%, it has negative effects on the quartz surface and consequently on the flotation process. The representative samples were prepared for optical microscope studies for each size fraction. The microscope studies presented the particles <44 μm have low Degree Of Freedom (DOF) for quartz and alunite. Thus, the froth flotation process is not appropriate for these sizes. Results showed DOF for minerals of -44+37 μm fraction is about 40%, -37+25 μm is about 60% and -10 μm is 100% (Fig. 3). However, the DOF of -10 μm is 100%, but due to the problems of ultrafine particles in flotation, they are not appropriate for the froth flotation process. According to the DOF results and appropriate particle size for froth flotation, the particles with size of -37+25 μm were selected for the next step of the experiments.

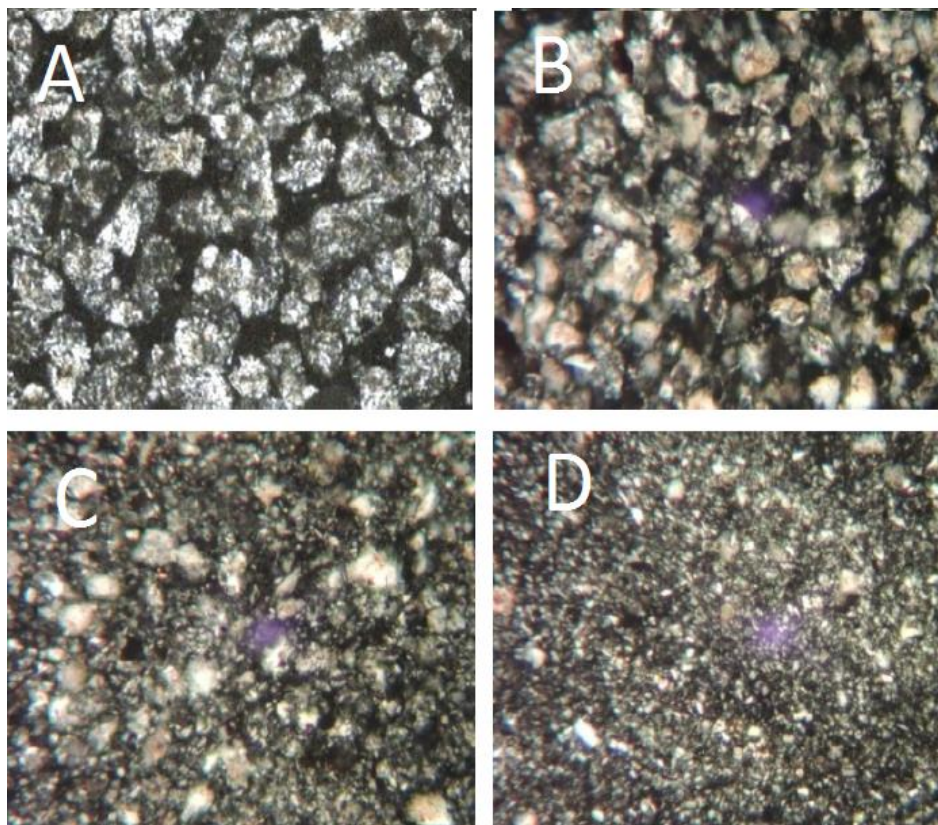


Fig. 3: Optical microscope studies, (A) -74+44 μm, (B) -44+37 μm, (C)-37+25 μm, (D) -25 μm

Methods

To perform micro-flotation tests, a modified Hallimond tube was used. This tube was made up of Pyrex glass with 100 mL volume. A filter was used at the bottom of the cell to do airflow. The advantage of this tube is that there is no change in pulp components such as changes in dosage of collector, depressant and activator during the flotation process. Before the aeration process, particles are mixed carefully with the help of a magnet. Airflow entered into the cell, the hydrophobic particles were attached to the bubbles. Bubbles carried on particles to the surface. When the bubbles arrived at the surface, they burst and the particles fell into the chamber. In the last step, they were collected from the chamber by opening the corresponding valve. Figure 4 shows the layout for the modified Hallimond tube.

A during flotation tests, solid% was 10. The pH was adjusted using NaOH or HF, followed by the addition of depressants, activators, collectors and frother. The preparation time was 10 min. The pulp aeration and the froth removal were done for 5 min. The concentration of alunite for each flotation test was dried and analyzed using XRF to get the grade. The recovery was calculated using the following equation (Rahimi *et al.*, 2012; Rezai *et al.*, 2010; Dehghani *et al.*, 2013):

$$R\% = \frac{C \times c}{F \times f} \times 100 \quad (1)$$

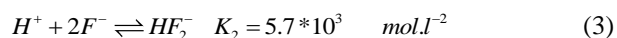
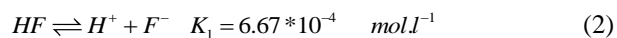
where, C is the weight of the concentration, c is the grade of the concentration, F is the weight of the feed and f is the grade of the feed.

In this study two types of experiments were performed for flotation of alunite.

Reverse Alunite Micro-Flotation using Ca^{+2} and Ba^{+2} as an Activator

In the first phase, Ca^{+2} and Ba^{+2} were used to activate the quartz surface. Anionic collector was used at pH = 12 (El-Salmawy *et al.*, 1993). The pH is adjusted using NaOH followed by the addition of $BaCl_2$. The pulp was agitated with the magnet for 20 min. The aero promoter 801 was added as a collector. The preparation time was 10 min. The aeration was done for 5 min and froth was removed.

Direct Alunite Miro-Flotation using ion F



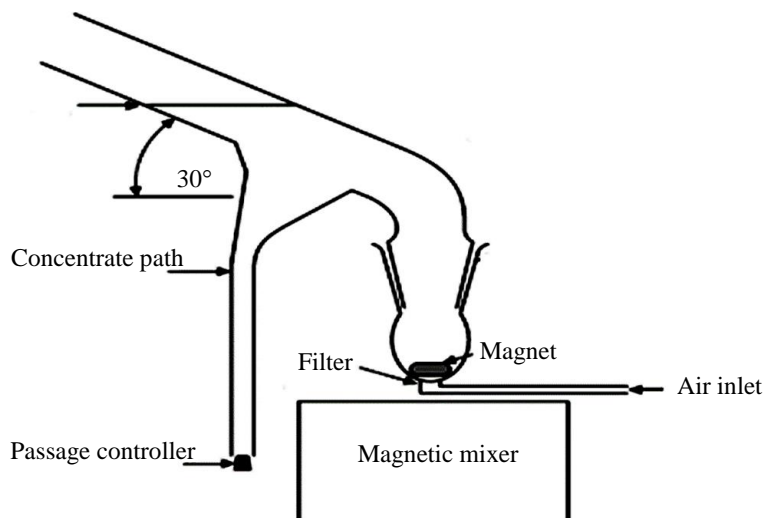
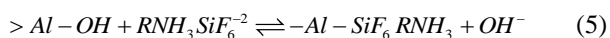


Fig. 4: Layout of the modified Hallimond tube (Bulut *et al.*, 2014)

HF and SiO₂ (quartz) react to form hexafluorosilicic acid, which in turn results in the formation of fluorosilicate ions (SiF₆⁻²). These reactions do not change the surface charge of the quartz. Therefore, quartz in the presence of a fluoride ion is not activated. Instead, SiF₆⁻² will be adsorbed by an aluminum surface and makes its surface negatively charged (Equation 4):



Finally, amine ions (collector) are absorbed on the surface layer with the help of electrostatic forces. During this process, the formation of RNH₃SiF₆⁻² is possible. This compound undergoes the ion exchange process with hydroxyl groups (Equation 5):



Both mechanisms result in the formation of similar negatively charged surface compounds (Kashkai and Babaev, 1969). In these series of experiments, F⁻ (in the form of NaF and HF) was used as the Alunite surface activator. Fluoride ion imposes negative charge on the alunite surface which makes the alunite surface ready to absorb cationic collector. One-gram of sample was mixed with 100 mL distilled water for one minute. HF was used as a pH modifier and alunite surface activator.

In the next series of experiments, in order to produce F⁻, first of all, the pH was adjusted using H₂SiF₆, followed by the addition of NaF. The pulp was agitated with the magnet for 20 min. Armac C ((Coco-alkyl-amine-acetate (C-N; R22-R34-R50)) was added as a collector and the preparation time was 10 min. The aeration was done for 5 min. Froth was removed for 5 min. Finally, concentrates and tailings were analyzed.

Results and Discussion

The main purpose of this study was to pre-concentrate alunite. Representative Samples were selected for both direct and reverse flotation methods. Sieve analysis was done for the feed and the grade was obtained for each size. The results were given in Table 3. However, the particle with sizes -25 μm has the highest grade and DOF, but they are not appropriate for flotation tests. In this series of experiments, a number of flotation tests were performed on -37+25 μm size fraction because DOF for this fraction is around 60%. Moreover, particle sizes are suitable for the flotation process (Alunite grade is 31.43%). Zeta potential was measured for alunite samples at different pH and temperature. The results are shown in Table 4. Experiments were performed for reverse and direct flotation. Finally, the effective parameters of each category were investigated and optimized.

Reverse Alunite Micro-Flotation using Ca⁺² and Ba⁺² as Activator

The purpose of this step is to sink alunite and float other minerals such as quartz. Therefore, it is expected the grades of alunite in the tailings to be higher than the concentrate (floated material) (Eskanlou *et al.*, 2018). The results are shown in the Table 5. As it can be seen in Table 5, Ca⁺² as an activator has a negative effect on the reverse flotation of alunite. The grade and recovery are 23.36 and 46.6%, respectively. Ca⁺² ions are adsorbed by both surfaces of quartz and alunite, which makes the flotation a non-selective process. Thus, both quartz and alunite behave similarly in the process (Küçük and Gülaboğlu, 2002; Apte *et al.*, 1988). In the next set of experiments, BaCl₂ was used as an activator. It had a

positive effect on the flotation as the grade increased to 34.96% and recovery to 81.33% (Table 6). Although, by adding BaCl₂, the grade and recovery increased compared to the previous step, but the result is not acceptable from an economic point of view. Therefore, the next series of experiments were performed.

Direct Alunite Micro-Flotation using ion F⁻

In this series of experiments, micro-flotation tests were performed for a variety of pH including pH = 2, 2.5, 3 and 4. HF was used as a pH modifier and alunite activator. As can be seen from Table 7 and Fig. 5, by increasing pH, the grade and recovery of the flotation process decreased. Due to more consumption of HF to achieve lower pH, the alunite surface was more negative compared to higher pH. Furthermore, at pH = 2, the surface charge of quartz is close to zero because the Zero Point of Charge (ZPC) of quartz is 1.8. Therefore, the cationic collector has a high tendency to adsorb alunite surface at lower pH. The highest grade and recovery were obtained at pH = 2, 49.9 and 88.9% respectively. On the other hand, for higher pH, the consumption of HF reduces. Consequently, the amount of F⁻ ions in the pulp will decrease. Thus, the negative charge of the alunite surface will decrease. Moreover, at higher pH, the negative charge of the quartz surface increases. As a result, quartz can float using the

cationic collector Armac C. By the flotation of quartz, final product grade was decreased.

Another series of tests were performed on coarser size fraction (-63+44 μm) which is more profitable from the industrial point of view. Two different dosages of collector were used. The results are given in Table 7. Comparing Tables 7 and 8 shows by increasing the particle size, the alunite grade declines due to the lower DOF at coarser particles. Also, Table 8 demonstrates by increasing collector dosage, alunite grade and recovery will decrease. The highest amount of grade (41.15%) and recovery (62.89%) for -63+44 μm fraction were achieved at pH = 2 with 1500 g/ton of collector.

Table 3: Alunite grade in various fractions

Fraction (μm)	Grade (%)	Weight (g)
+75	48.98	1603.81
-75+44	34.34	386.03
-44+37	38.08	132.73
-37+25	31.43	41.35
-25	71.47	364.71

Table 4: Zeta potential of the samples at different pH and temperature

No.	Temperature (°C)	pH	Zeta potential (mV)
1	2.9	21	369
2	7.2	24	-35
3	11.0	23	-160

Table 5: Alunite concentration in the present of CaO

	pH	Weight (g)	Alunite weight (g)	Quartz weight (g)	Alunite grade (%)	Alunite recovery
Conc.	12	1.71	0.55	1.16	32.36	53.4
Tailing	12	2.046	0.48	1.566	23.36	46.6

Table 6: Alunite concentration in the present of BaCl₂

	pH	Weight (g)	Alunite weight (g)	Quartz weight (g)	Alunite grade (%)	Alunite recovery
Conc.	12	1.075	0.323	0.745	30.68	19.67
Tailing	12	2.925	1.022	1.9	34.96	81.33

Table 7: Alunite grade in different pH for -25 +37 μm size fraction

	pH	Weight (g)	Alunite weight (g)	Quartz weight (g)	Alunite grade (%)	Alunite recovery
Conc.	2	2.061	1.03	1.033	49.9	88.9
Tailing	2	1.619	0.46	1.53	28.76	11.1
Conc.	2.5	1.249	0.52	0.739	41.24	50.7
Tailing	2.5	1.984	0.596	0.578	30.13	49.3
Conc.	3	1.418	0.56	0.86	39.22	50.4
Tailing	3	2.095	0.64	1.46	30.44	49.6
Conc.	4	1.536	0.56	0.98	36.32	53.46
Tailing	4	1.784	0.55	1.23	30.97	46.54

Table 8: Alunite grade in pH = 2, fraction: 44+ 63 μm with different dosage of collector

	pH	Collector (g/ton)	Weight (g)	Alunite weight (g)	Quartz weight (g)	Alunite grade (%)	Alunite recovery
Conc.	2	1500	2.43	1	1.43	41.15	62.89
Tailing	2	1500	2.00	0.59	1.41	29.49	
Conc.	2	2250	2.26	0.91	1.35	40.2	53.84
Tailing	2	2250	2.232	0.78	1.45	34.96	

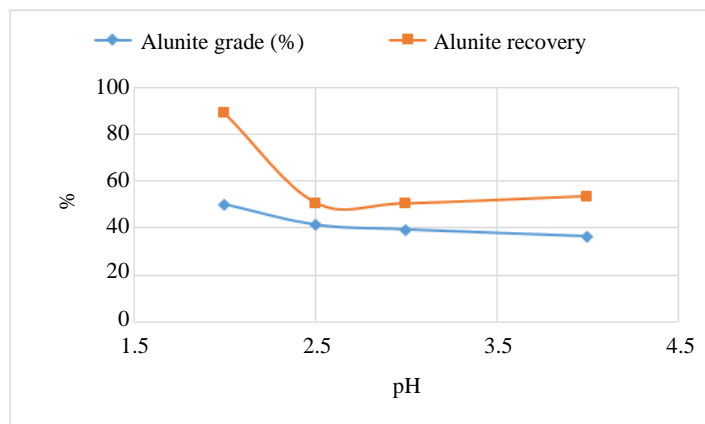


Fig. 5: Effect of pH on the grade and recovery of alunite, HF as a pH modifier and activator, -25 +37 μm size fraction

In this research, alunite ore from Haft-Sandogh mine was used for flotation tests. However, the ore composition is dependent on the geology of the deposit, hence the ore characteristics varies from one mine to other. Generally, most of the gangue minerals associated with alunite are similar but there are few instances where they show variation. In adopting the results of this research for purification of other alunite deposits, researchers should perform mineralogical and DOF studies. Based on that, they can decide how to follow our procedure. For instance, since the main gangue associated with alunite is quartz, then flotation procedure described in this research can be applied. Conversely, if the gangue is different or there are other impurities, then the researched would decide which flotation producer.

Conclusion

In this study, the main minerals of the used ore deposit were alunite, quartz and kaolinite. Around 2/3 of alunite ore is gangue minerals. Quartz was the main gangue the ore accounting for around 44.41 wt%. In order to decrease transportation and processing costs in hydrometallurgical circuits, pre-concentration methods were considered to purify alunite ore. DOF studies were done for the samples. DOF for minerals of -44+37 μm fraction is about 40%, -37+25 μm is about 60%. 100% liberation of alunite particles occurs when particles are grounded finer than 10 μm , but they are not appropriate for flotation. Alunite has a lower bond index compared to quartz, so during the comminution process, alunite is grounded to small size fractions faster than quartz. Consequently, quartz particles can be covered with alunite slimes. Slime coating of alunite particles with quartz particles significantly makes the selective flotation difficult, especially it has negative effects on reverse flotation.

Using Ca^{+2} as an activator in the flotation process had a negative effect on the result because Ca^{+2} was adsorbed by both surfaces of quartz and alunite. Consequently, it made the flotation process nonselective as both quartz and

alunite had a similar behavior in the process. On the other hand, adding BaCl_2 as an activator had a positive effect on the flotation. Using BaCl_2 floated 81.33% of alunite with a grade of 34.9%. However, by adding BaCl_2 , the grade and recovery will increase compared to using Ca^{+2} , but it is not acceptable because of the low grade of the product. Using F^- ion as an activator of the alunite surface and a pH modifier imposed negative charge on the alunite surface. As a result, cationic collector was absorbed on an alunite surface. The highest amount of grade and recovery were obtained using HF. In this condition the grade and recovery were 49.9 and 88.3%, respectively.

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Authors Contributions

Fahimeh Dehghani and Halimeh Askari-Sabzekoohi: Conceived and designed the study, conducted the study and write the manuscript.

Tathagata Ghosh: Wrote the paper, review it and give comments to improve the work.

Declaration of Competing Interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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