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The Laboratory Investigations of Reducing Iron and Silica Content of Potassium Feldspar Ore by Flotation

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Abstract: Potassium feldspar is one of the basic needs of glass, ceramic, electrical insulation and other industries. One of the challenging factors in feldspar processing industries is the presence of iron and silica in feldspar. In order to increase the quality of feldspar concentrate for use in related industries, flotation is one of the most common methods of reduce iron and silicon content in feldspar. Therefore, in this study, the ore was crushed for 15 minutes to reduce the particle size below 150 microns. Then the mineralogical and chemical composition of the crushed product were determined by conventional XRF and XRD methods. Based on the mineralogical and chemical composition of the crushed product, different flotation scenarios were designed to reduce the iron and silica content in feldspar were investigated. The amount of silica and iron in the samples used in this study were 73.31 and 0.31%, respectively. In the first stage, in order to reduce iron, reverse flotation experiments were performed. In reverse flotation experiments, using a combination of 800 g/ton of each of the two collectors Aero 801 and 845 with 100 g/ton of pine oil frother at a pH of 2.5-2.9, the iron content to a level of 0.11%, Decreased. Also, to reduce silica content up to 57.26%, 400 g/ton of fluoric acid detector with 200 g/ton methyl isobutyl carbinol (MIBC) frother was obtained at pH 2.8-3. Feldspar with this percentage of impurities is suitable for use in required industries such as ceramic and tile.

Keywords: Feldspar, Silica, Iron, Chemical Reagents, Flotation

1. Introduction

Feldspar is the most widespread type of silicate in the Earth's crust. They make up about 50 percent of the earth's crust. About 60% of them are found in igneous rocks, about 30% in metamorphic rocks, and the remaining 10 to 11% are found mainly in sandstones and conglomerates. Feldspars are the main components and minerals that make up most of the inner (plutonic), outer (volcanic) and metamorphic (metamorphic) rocks. Potassium feldspars may form in an environment of intrusive magma cooled to a relatively low temperature and slowly. In this case, they form the microcline-orthoclase series. Or formed in an environment of magma that has cooled rapidly with high temperature, in which case they are called the sanidine-invertose series and are found in volcanic rocks [1-3].

Feldspars are tectosilicates of general formula XAl (Si, Al) Si_2O_8, in which X is usually potassium, sodium or calcium [3]. Accordingly, in terms of chemical composition, they are potassium, sodium and calcium aluminosilicates (ie $KAlSi_3O_8$, $NaAlSi_3O_8$ and $CaAl_2Si_2O_8$ respectively), and rarely barium may be found in their composition [1-5].

Feldspars are a basic need of industries such as glass, ceramics, sanitary ware, electrical insulators, rubber coatings, paints, etc [2, 5-11].

Extensive research was conducted to improve the quality of feldspar rock. The most common method for this is flotation method. Prior to flotation, the feldspar ore is first crushed to the desired degree of liberation. Then the desliming process was performed. Slimes usually contain clay minerals. The presence of clay minerals causes problems in the flotation process such as changing the stability of the froth, increasing the viscosity of the pulp and excessive consumption of reagents [7, 12, 13]. The resulting sample is then passed through high-intensity magnetic separator to reduce the iron content [7, 13, 14]. Extensive research has been published on the application of various collectors in feldspar flotation. Cationic collectors of amine and hydrofluoric acid have been commonly used as feldspar activators to reduce the silica content in feldspar ores [4, 5, 7, 11, 13, 15-19].

In this research, samples of potassic feldspar from Shaygan mine in Semnan province were investigated. Shaygan feldspar mine is located approximately 130 km southeast of Damghan city and in the north of Satveh village from Sarkavir section. Its geographical coordinates are range from longitude 54° 36' 06" to 54° 41' 02" and latitude from 35° 20' 01" to 35° 21' 57". The samples were taken by network sampling method and along networks with distances of 4 meters from the ore accumulation site in the mine. In the continuation of the process, the chemical and mineralogical composition of the ore should be determined to design different flotation scenarios. The presence of iron and silica in the ore sample of the present study is a challenge that we face.

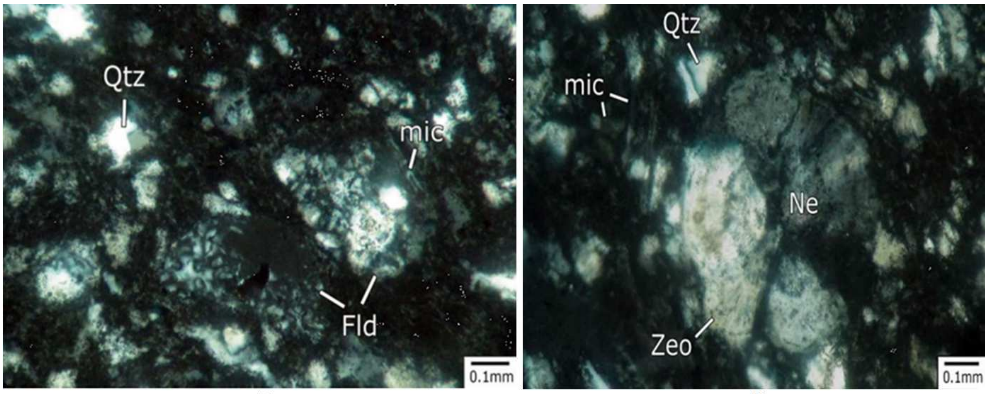
2. Research Methodology

First, XRF and XRD tests were performed on the samples taken from the mine, and then thin-section studies were performed. XRF tests were used to control the silica and iron content in feed and concentrate samples and XRD tests were used to identify the percentage of minerals in feed and concentrate samples before and after flotation tests. According to XRF results, the content of silica and iron in the feed sample was 73.31% and 0.31%, respectively (Table 1).

Table 1. *Result of XRF analysis of samples taken from the mine.*

Compound	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	Na_2O	MgO	TiO_2	MnO	P_2O_5	S	L. O. I.
Quantity(%)	73.31	14.18	0.31	0.66	9.39	0.40	0.02	0.141	0.003	0.025	0.003	1.21

Then, microscopic studies were performed on thin blades prepared from feed samples. Microscopic studies were performed by Zeiss Axioplan2 polarizing microscope in polarized light (10X-XPL). The results showed that the sample is alkaline or nepheline trachyte volcanic rocks. The basic texture of the sample is porphyry with glass paste. The main components of sample include feldspar, quartz and volcanic glass. Altered minerals such as kaolinite, quartz and zeolite have also been observed. The bulk of the sample consists of a highly altered and clayey glass paste in which feldspar microlytes are scattered. Siliceous argillite alterations can also be seen in sample.



(a) (b)

Figure 1. *(a) Dissolved feldspar phenocrysts in an altered glass paste containing feldspar microlytes. (b) Nephline polyhedral phenocryst with hybrid zoning partially replaced by zeolite filaments.*

2.1. Determining the Degree of Liberation

To determine the degree of liberation, samples taken from the mine up to dimensions less than 2 mm were crushed. This was done first by a jaw crusher and then by a roller crusher in the laboratory. Table 2 shows the results of the sieve analysis of the final product obtained from the crushing stage.

Table 2. *Results of sieve analysis of roller crusher product.*

Granulation dimensions (microns)	weight on the screen (%)	Cumulative residual weight (%)	Cumulative weight passed (%)
+2000	8.3	8.3	91.7
-2000 +1700	13.2	21.5	78.5
-1700 +850	34.34	55.84	44.16
-850 +600	13.21	69.05	30.95
-600 +300	11.75	80.8	19.2
-300 +150	9.67	90.47	9.53
-150 +75	7.23	97.7	2.3
-75	2.3	100	0

From the granulation of the final crusher product, it is clear that a small amount of soft material has been produced. Based on mineralogical studies and the grain size distribution curve of the jaw crusher, a degree of liberation of 150 microns was obtained. In order to achieve a degree of liberation of 150 microns, the product of the crushing stage must be crushed by a mill. Denver rod mill was used for this purpose.

In order to save energy and determine the optimal grinding time to achieve a degree of freedom of 150 microns, the milling operation was performed in three different time intervals. Therefore, the grinding operation was performed in three times intervals of 5, 10 and 15 minutes. Based on the sieve analysis of the products obtained in three times intervals, the best grinding time was 15 minutes until 90% of the particles were smaller than 150 microns. The average specific gravity of the samples was obtained 2.7 g/cm^3 using a pycnometer.

2.2. Flotation Tests

Flotation experiments were designed in two stages and performed in Denver cell. In the first stage, iron is removed from the sample during reverse flotation by frothing. In the next step, the silica is captured during the direct flotation operation and the concentrate is removed from the cell by frothing.

2.2.1. Reverse Flotation Tests

In these experiments, the goal is to remove the iron content from the sample. The remaining product of this process will be concentrated. In order to perform Reverse flotation operations, the most effective reagents that are effective in reducing iron are first identified. Then their optimal consumption is determined. Finally, by optimally applying each of the reagents in the reverse flotation process, the maximum reduction of iron content can be achieved.

In order for the same conditions to prevail in all reverse flotation experiments and to obtain more logical results, some parameters affecting the experiments were fixedly programmed. These parameters are summarized in Table 3. Fixed parameter values were obtained through experimental tests.

Table 3. *Same conditions in reverse flotation experiments.*

Parameter name	The unit	Content
Frother (pine oil)	g/t	100
The time of foaming	Minutes	3
Preparation time	Minutes	7
Engine speed during foaming	r.p.m.	1200
Engine speed during preparation	r.p.m.	1400
Solid pulp content	%	30
PH	-	2.4-2.5

Accordingly, in the first stage, for the purpose of flotation of iron and its purpose, samples from Aero 801 and Aero 845 collectors were used both separately and in combination.

(i). Using Aero 801 Collector

In order to determine the optimal amount of consumption of Aero 801 collector, flotation tests were performed in five different amounts of collector, including 350, 450, 750, 900 and 1100 g/t. The test results are shown in Table 4. According to the results of Table 4, the most appropriate amount of consumption of Aero 801 collector, which has the greatest effect of iron removal in concentrate, is 750 g/t.

Table 4. *Percentage of iron in concentrate in different amounts of Aero 801 collector.*

Test number	Aero 801 collector (g/t)	Iron content in concentrate %
1	350	0.28
2	450	0.253
3	750	0.17
4	900	0.19
5	1100	0.22

(ii). Using Aero 845 Collector

In these tests, in order to determine the optimal consumption of Aero 845 collector, flotation tests were performed in five different dosages of 350, 500, 750, 900 and 1200 g/t of Aero 845 collector. The results of these experiments are summarized in Table 5.

Table 5. *Percentage of iron in concentrate in five different dosages of Aero 845 collector.*

Test number	Aero 845 collector (g/t)	Iron content in concentrate %
1	350	0.26
2	500	0.231
3	750	0.17
4	900	0.203
5	1200	0.21

As can be seen from the results of Table 5, the most appropriate consumption of Aero 845 collector for iron removal is 750 g/t.

(iii). Using a Combination of Aero 801 and Aero Collector

In these experiments, two aero collectors 801 and 845 were used in combination. The weight composition and results of use of both collectors are summarized in Table 6. The results of these tests showed that if the combination of Aero 801 and 845 collectors is used, the amount of iron removal will be higher. Therefore, the maximum reduction of iron in the concentrate is when a combination of equal amounts of 800 g/ton of each of the two collectors is used.

Table 6. *Percentage of iron in concentrate in different amounts of Aero 801 and 845 collectors.*

Test number	Aero 801 collector (g/t)	Aero 845 collector (g/t)	Iron content in concentrate %
1	500	350	0.20
2	350	500	0.17
3	800	800	0.132
4	900	900	0.152

2.2.2. Direct Flotation Tests

After performing reverse flotation tests and trying to remove the maximum amount of iron from the sample, direct flotation tests were performed on the concentrate obtained from the previous step to depression of silica. In these experiments, fluoric acid was used to depression of silica and methyl isobutyl carbinol (i.e. MIBC) was used as a frother agent. Therefore, first experiments were performed to determine the optimal use of fluoric acid and MIBC in direct flotation experiments, which are as follows:

(i). Determine the Optimal Amount of Hydrofluoric Acid

In order to determine the optimal amount of hydrofluoric acid as activator for feldspar and depressor for silica in direct flotation experiments, five weights of 100, 200, 300, 400, 500, 600 and 700 g/t of hydrofluoric acid were used. In these experiments, other effective parameters were considered constant to evaluate the effect of hydrofluoric acid factor only. These fixed parameters are given in Table 7.

The test results are summarized in Table 8. As can be seen from Table 8, if the amount of hydrofluoric acid is about 400 g/t, the amount of silica depression will be increase.

Table 7. *Same conditions in reverse flotation experiments.*

Parameter name	The unit	Content
Frother (MIBC)	g/t	150
The time of foaming	Minutes	3
Preparation time	Minutes	7
Engine speed during foaming	rpm	1200
Engine speed during preparation	rpm	1400
Solid pulp content	%	35
PH	-	2.4-2.8

Table 8. *Results of the effect of different amounts of hydrofluoric acid on silica depression.*

Test number	The amount of fluoric acid (g/t)	Silica content in concentrate %
1	100	87.83
2	200	74.17
3	300	65.26
4	400	58.59
5	500	63.45
6	600	68.67
7	700	73.76

(ii). Determine the Optimal Amount of MIBC Frother

To determine the optimal consumption of MIBC frother in direct flotation tests, four different weight values were tested. The results of these experiments are summarized in Table 9. From Table 8, it can be seen that if the consumption of frother is about 200 g/t, the amount of silica removal will be the highest.

Table 9. *The effect of different amounts of MIBC on silica depression.*

Test number	The amount of MIBC (g/t)	Silica content in concentrate %
1	150	58.14
2	175	57.89
3	200	57.23
4	250	60.12

(iii). The Effect of Different pH Values

In order to determine the pH that has the greatest effect on reducing the silica content, experiments were performed at 6 different pH values. In this experiments, sulfuric acid was used as a pH regulator. In order to have the same conditions during the experiments, in all experiments, a fluoric acid inhibitor was used along with the MIBC frother in the pulp with a solid content of about 35%. Preparation time and foaming time in all experiments were 7 minutes and 3 minutes, respectively. The test results are summarized in Table 10. As can be seen, the most suitable pH value in the maximum reduction of silica content is 2.8 to 3.

Table 10. *The effect of pH changes on the reduction of silica content.*

Test number	The amount of PH	Silica content in concentrate %
1	2.4	65.12
2	2.6	64.45
3	2.8	63.12
4	3	59.24
5	3.2	60.08
6	3.3	62.43

3. Result and Discussion

Feldspar is the most abundant mineral in the Earth's crust and is generally associated with other minerals such as silicate, titanium and iron. Extraction of feldspar ores from other silicate gangues is very difficult due to their similarities in physicochemical properties. Extensive research was conducted in this regard. This study showed that factors such as crystal structure, monovalent salts, flotation reagents, and particle size distribution are important factors in the feldspar beneficiation process. Of all the methods tested, flotation is the only known method that makes it possible to separate feldspar from the associated with gangue minerals. The use of hydrofluoric acid (HF), as the main and most common regulator and HF/amine combination is the most effective reagents in selective feldspar flotation. In the present study, the effective parameters and reagents in feldspar flotation on a laboratory scale were investigated and the most optimal case is summarized in Table 11.

After examining the effective factors in the process of reverse and direct flotation experiments, the optimal conditions for reducing the iron and silica content in the ore were determined. In the first stage of the flotation process, which was reverse flotation, first 800 g of each of Aero 801 and Aero 845 collectors were prepared with 100 g of pine oil for 7 minutes. The process of reducing the iron content was performed after three minutes of frothing.

In the remaining pulp, 400 g of sulfuric acid is injected into the pulp along with 200 g of MIBC frother. After 7 minutes of pulp preparation, the final concentrate is foamed out of the flotation cell. The optimal flotation test conditions for reducing iron and silica content in feldspar ore are presented in Table 11.

Table 11. *Optimal conditions in the flotation process to reduce the iron and silica content in the sample.*

Flotation method	Effective parameters in flotation		unit	Amount
Both methods of flotation	Fixed parameters	Preparation time	minutes	10
		The time of foaming	minutes	3
		Engine speed during preparation	rpm	1400
		Engine speed during foaming	rpm	1200
		The solid amount of pulp	Percentage	30
Reverse flotation	Variable parameters	Combination of Aero 801 and Aero 845 collectors	g/t	800, each
		Pine oil frother	g/t	100
		PH value	-	2.5
Direct flotation		MIBC frother	g/t	200
		Fluoric acid depressant	g/t	400
		PH value	-	3

After applying the optimal conditions in the flotation experiments, the results of the analysis of the final concentrate samples are summarized in Table 12. This product can meet the needs of glass, porcelain, ceramic and tile industries.

Table 12. *Chemical analysis of the final concentrate.*

Compound	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	Other
Quantity(%)	57.26	16.12	0.11	0.57	13.45	12.49

4. Conclusions

In order to achieve suitable products for use in industry, reverse and direct flotation experiments were performed.

The result of reverse flotation tests to reduce the iron content in the concentrate is as follows:

By applying a combination of 800 g/ton from each of the Aero 801 and 845 collectors along with 100 g/ton of pine oil frother at a pH of 2.5-2.9, reduced the iron to a level of 0.11%.

Also, the result of direct flotation experiments to reduce the silica content of the concentrate obtained from the reverse flotation step is as follows:

Applying 400 g/ton of fluoric acid inhibitor along with 200 g/ton of MIBC frother at pH 2.8-3, reduced the silica content to a level of 57.26%.

Therefore, by performing reverse and direct flotation tests, the iron and silica content in feldspar ore decreased from 0.31 and 73.31% to 0.11 and 57.26%, respectively, which is suitable for use in various industries.

Conflict of Interest

The authors declare that they have no conflict of interest.

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