

Removal of Discoloured Impurities from Feldspar Ores by Bulk Floatation

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Feldspar ores usually contain the principal discolouring impurities of mica, titanium and iron bearing minerals, which impart colour and in turn degrade the quality of the ore. In this study, the possibility of removing mica, iron and titanium bearing mineral from feldspar ore in a single stage floatation experiment in mildly alkaline pH conditions compared to the more commonly used reverse multi-stage floatation in acidic pH conditions was investigated. For this, several collectors and their combination were tested in the single- and multi-stage floatation methods. This study has showed that the main impurities in the feldspar ore can be floated in an alkaline medium by bulk floatation, *i.e.* in one-stage, using Aero 704 collector supplied from Cytec Industries, Inc. The results of bench scale floatation tests to evaluate the performance of individual collectors and their blends will be reported along with the elaboration of the mechanism of the collector action. The parameters considered in the evaluation were grades of TiO_2 and Fe_2O_3 in the feldspar concentrate and their removal recoveries from the feldspar ore.

Key Words: Industrial minerals, Froth floatation, Floatation collectors, Mineral processing.

INTRODUCTION

Feldspar is a volcanic rock consisting of aluminum silicate mineral group containing alkali ions such as potassium, sodium and calcium. The main minerals of feldspar are albite ($\text{NaAlSi}_3\text{O}_8$), orthoclase/microcline (KAlSi_3O_8) and anorthite ($\text{CaAlSi}_3\text{O}_8$). Albite and orthoclase minerals are mainly used in the glass and ceramic industries due to their alkali and alumina content and also as fluxing agents in glass and ceramic applications.

Commercial feldspars minerals of albite and orthoclase are usually associated with quartz, clay, muscovite, biotite, iron oxide, tourmaline, hornblende, rutile and sphene minerals^{1,2}. The use of feldspar in ceramic

and glass industries necessitates the rejection of discoloured gangue minerals of mica and heavy minerals including iron oxides and titanium from the ore. In most cases, sellable quality feldspar can be produced using magnetic separation and floatation methods to remove these impurities¹⁻⁴. However, while magnetic separators are usually used in the removal of only iron bearing minerals excluding titanium, floatation process is the most selective method in the removal of both mica and heavy minerals^{1,2}.

Conventionally, feldspar can be separated from the impurities through the use of multi-stage floatation. To remove the micaceous minerals by floatation, long chain aliphatic amines are usually used at pH of 2.5 to 5.0^{2,5,6}. With amine type collectors, the use of fuel oil increases the selectivity of floatation^{7,8}. Pugh *et al.*⁹ has floated muscovite type mica minerals by applying dodecylamine collector at pH 8.

To remove the iron bearing heavy minerals, fatty acids or sulfonates are used in acidic conditions³. Non-polar oils like fuel oil can be used as an auxiliary collector to enhance the removal of iron bearing minerals^{7,10}. Albite ores from the west of Turkey contain the main impurity of titanium bearing minerals of rutile (TiO₂) and sphene (CaSiTiO₃)⁵. The rutile, containing the main titanium mineral, can be floated with the use of fatty acids at pH 4-6^{5,11,12}, petroleum sulfonates at pH 3.0-3.5 or fatty primary amine acetate at pH 2.5^{13,14}. Furthermore, titanium minerals can be floated with hydroxamates at pH 6.5, oleoyl sarcosine at pH 7.7 and potassium oleate at pH 7.7^{1,3,15}. In the floatation of titanium minerals containing sphene, oleic acid or its soaps^{5,15} or various vegetable oil soaps are successfully used^{2,5}. The use of alkyl succinamate together with petroleum sulfonate, increases the success of floatation in the removal of heavy minerals⁸.

In this investigation, in addition to a conventional beneficiation process, *i.e.*, multi-stage separation of feldspar from the impurities in acidic circuits, the feldspar concentrate was produced by a bulk floatation (*i.e.*, in one-stage) of discolouring impurities, using new collectors. In this novel process, new anionic type fatty acid collectors (Aero 704 and 727 from Cytec Industries, Inc.) have been tested in alkali circuits. In addition, for comparison purposes, different collector types and their blends were tested to remove micaceous and heavy minerals in one and multi-stages. To our best of knowledge, one-stage-floatation to remove all the discolouring impurities has been applied for the first time in an alkaline atmosphere. Bench scale floatation tests were conducted to evaluate the performance of each reagent and their blends at the same conditions, collector concentration, pH, percent solids.

EXPERIMENTAL

The feldspar ore was received from Mugla-Milas region in the west of Turkey. The main mineral of the ore is albite according to the results of XRD analysis. The ore is associated with orthoclase, quartz, muscovite, biotite, iron oxide, tourmaline, rutile and sphene. The results of chemical analysis of the ore are presented in Table-1.

TABLE-1
CHEMICAL COMPOSITION OF THE ALBITE ORE
USED IN THIS INVESTIGATION

Component	Content (%)	Component	Content (%)
SiO ₂	68.70	TiO ₂	0.23
Al ₂ O ₃	18.40	MgO	0.10
Na ₂ O	7.40	CaO	0.45
K ₂ O	3.36	LOI	0.55
Fe ₂ O ₃	0.16	—	—

The results of microscopic liberation observations based on grain size count method by an Olympus SZ-61 binocular microscope, 95 % of the albite, orthoclase, quartz and mica minerals are liberated below 0.3 mm particle size, whereas titanium is liberated below 0.1 mm.

Flootation test were conducted with different reagents individually and with their blends. These reagents are Aero 3000 C (amine type), Aero 704 and Aero 727 (tall oil fatty acid), Aero 6493 (Alky hydroxamate), Aero 869, Aero 801 and Aero 825 (petroleum sulfonate), Aero 830 (Alky succinamate). These promoters were supplied by Cytec Industries Inc. (Connecticut, USA).

In addition, as an alternative to commercial Na-oleat, in-house manufactured collector was prepared by using local olive pomage oil. It was added to the cell drop-wise by 1.8:1 volumetric ratio of 5 N NaOH into warm stirred oils. This soap was later diluted to 10% w/w with distilled water and used in the floatation tests⁵. Commercial fuel oil and kerosene (blended as 1:5 w/w) and olive pomage oil are used as auxiliary collectors. In the floatation tests, sodium silicate (Na₂SiO₃, 10 % w/w) was used as a depressant and the pH of the system was adjusted with H₂SO₄ and NaOH. These reagents are supplied by Merck, Germany. Commercial pine oil was used as frother.

Feldspar ore, received from Mugla-Milas region in Turkey, was crushed to below 2 mm by jaw and hammer crushers, respectively. Minus 0.3 mm fraction was separated by screening. The rest of the plus 0.3 mm fraction was also reduced to below 0.3 mm by using a ceramic ball mill at 82 rpm and with 4.43 kg steel ball charge. A closed system was operated with

5 min intervals to remove the ground material at 0.3 mm sieve size. Prior to floatation tests, the samples were deslimed at 45 μm with wet sieving. The amount of the material removed is 15 % by weight. All the floatation tests were conducted on -0.3 + 0.045 mm fractions with 500 g samples.

The impurities from the samples were removed by reverse floatation method, *i.e.*, mica and heavy minerals were floated. A two liter, self-aerated sub-A Denver floatation machine was used. In this investigation, all the impurities containing micaceous and heavy minerals were first floated together in one-stage using only one type of collector and the blends of different reagents. For comparison reasons, the impurities in the ore were also floated separately multi-stage-wise with different collectors at each stage and with the blends of collectors. Test conditions and the procedure followed for floatation tests are summarized in Table-2.

TABLE-2
TESTING PARAMETERS FOR FLOATATION

Conditions	Parameters	One-Stage	Multi-Stage
Conditioning	Solid (%)	50	50
	Rotor speed (rpm)	1500	1500
	Time (min)	60*	15
Floatation	Solid (%)	25	25
	Rotor speed (rpm)	1200	1200
	Time (min)	20*	5 at each stage

*Collectors added stage-wise.

For the removal of impurities by bulk floatation, various anionic reagents and their blends of anionic + anionic, anionic + cationic, anionic + oil combinations were tested. Collectors were added into the cell in four steps, 300 g/t at each step, totaling 1200 g/t. All the floatation tests were conducted at pH 9, a pH which is usually used. Both in bulk (one-stage) and multi-stage floatation tests 20 g/t pine oil was used as frother. The experimental conditions of bulk floatation (one-stage) removal of impurities were presented in Table-3.

With multi-stage removal of impurities from the ore, cationic collectors and anionic + cationic collector blends were used at different pH. First, mica was floated and the remaining ore was washed to remove the stains of the collectors from the mineral surfaces. Second, the heavy (iron and titanium bearing) minerals were removed from the ore using various anionic collectors and their blends. Sodium silicate (Na_2SiO_3) was used as a dispersant in the amount of 1000 g/t. Table-4 summarizes the multi-stage floatation test conditions. All the floatation products (feed, slime, concentrate and tailings) were analyzed by XRF to determine the best performing collector or the blends.

TABLE-3
BULK (ONE-STAGE) FLOATATION PARAMETERS FOR FLOATING
THE COLOURED IMPURITIES FROM THE ALBITE ORE

Test No.	Collector type	Blends
1	Aero 704	–
2	Aero 727	–
3	Aero 6493	–
4	Aero 704 + Aero 830	3:1
5	Aero 704 + Aero 3000C	3:1
6	Aero 704 + Olive Pomage oil	3:1

TABLE-4
MULTI-STAGE FLOATATION PARAMETERS TO REMOVE
THE IMPURITIES FROM THE ALBITE ORE

Test No.	Stage	pH	Fuel oil + Kerosene (g/t)	Collector type	Blends	Collector amount (g/t)
1	1	3	100	Aero 3000C	–	600
	2	3	–	Aero 869	–	1200
2	1	3	100	Aero 3000C	–	600
	2	3	–	Aero (801 + 825 + 830)	1:1:1	1200
3	1	3	100	Aero 3000C	–	600
	2	9	–	Aero 704	–	1200
4	1	9	–	Aero 704 + Aero 3000C	3:1	600
	2	6*	–	Na-Oleate	–	1200
5	1	6	–	Aero 704 + Aero 3000C	3:1	600
	2	6*	–	Na-Oleate	–	1200
6	1	3	100	Aero 3000C	–	600
	2	6*	–	Na-Oleate	–	1200

*Specifies the pH of the albite ore before the collector addition of second stage.

RESULTS AND DISCUSSION

Bulk (one-state) floatation results: With bulk floatation tests, all the impurities (mica and heavy minerals) were floated together in one-stage. Several anionic collectors and their blends were evaluated to measure their effectiveness for the removal of mica and heavy mineral impurities in one-floatation-stage. The results were explained on the basis of Fe₂O₃ % and TiO₂ % recoveries in tailings (floated), Fe₂O₃ % and TiO₂ % grades in albite concentrates (*i.e.* not floated or sink).

The results of floatation tests with different anionic collectors, illustrated in Fig. 1, showed that the fatty acids (Aero 704 and Aero 727) were more effective than alkyl hydroxamate (Aero 6493) type collectors for the removal of both iron and titanium containing minerals at the conditions analyzed.

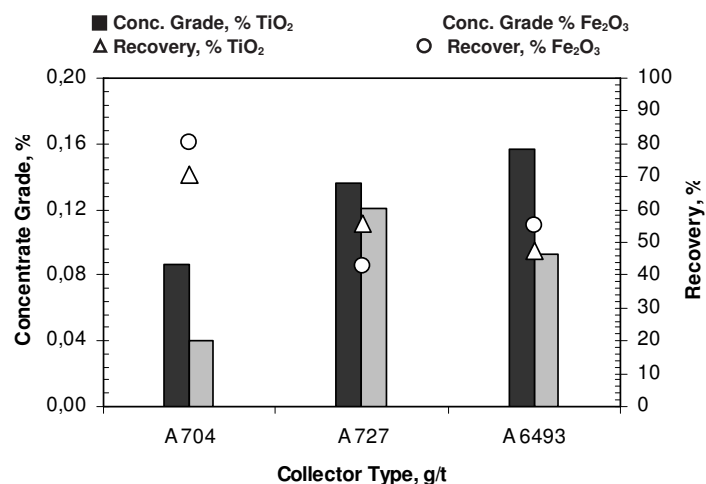


Fig. 1. Effect of different anionic collectors on the removal of both mica and heavy minerals in one stage

Aero 704 collector gave better results in the removal of iron and titanium bearing minerals. Albite concentrate (sink) was produced with 0.087 % TiO₂ and 0.040 % Fe₂O₃ grades. The recoveries of TiO₂ and Fe₂O₃ in tailings (float) were 70.50 and 80.50 %, respectively.

The performance of collector blends consisting of Aero 704 and various other collectors were tested at pH 9 and the results are shown in Fig. 2. The results were evaluated on the basis of TiO₂ and Fe₂O₃ grades in the albite concentrate and recoveries of the coloured impurities from the albite ore (*i.e.* float).

Based on the floatation results shown in Fig. 2, Aero 704 blended with Aero 3000C gave the best results. The albite concentrate contains 0.108 % TiO₂ and 0.036 % Fe₂O₃ grades with the removal of coloured impurities of 65.25 % TiO₂ and 83.35 % Fe₂O₃. Similarly, the tests performed with Aero 704 blended with olive pomage oil (OPO) at pH 9, gave an albite concentrate with the grades of 0.130 % TiO₂ and 0.068 % Fe₂O₃ and with the recoveries of coloured impurities of 57.68 % TiO₂ and 68.18 % Fe₂O₃. The blend of Aero 704 with Aero 830 gave the poorest performance at pH 9, as seen from the Fig. 2.

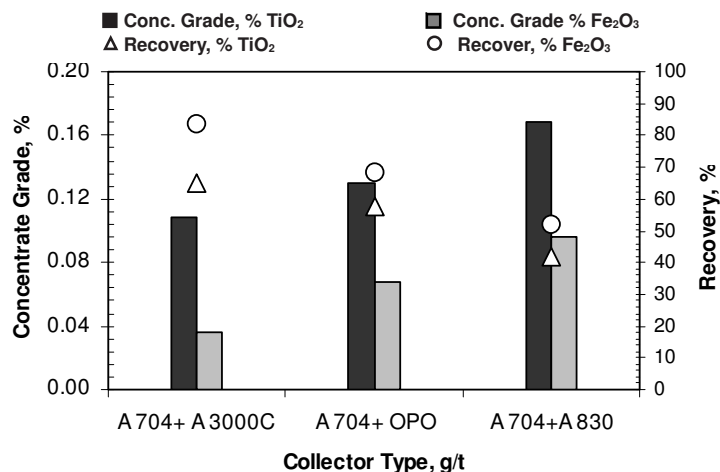


Fig. 2. Performance of Aero 704 blended with other collectors for the removal of mica and heavy minerals

Multi-stage flotation results: The coloured impurities in the albite ore were floated stage-wise, *i.e.*, multi-stage flotation. In the first stage, mica flotation and in the second stage, heavy minerals flotation were applied. Micaceous minerals were floated with Aero 3000C cationic collector at pH 3. Then, heavy minerals were floated with Aero 704 (tall oil fatty acid) at pH 9 and with Aero 869 (petroleum sulfonate) and the blends of Aero 801 + 825 + 830 (petroleum sulfonates + alkyl succunamate) separately at pH 3. The results of the flotation tests were presented in Fig. 3. The results were again evaluated on the basis of TiO₂ and Fe₂O₃ grades in the albite concentrate and recoveries of the coloured impurities from the albite ore.

As seen from Fig. 3, all three collector combinations gave about similar results. The recoveries of removal of TiO₂ from the albite ore ranged from 58.55 to 64.19 % and for Fe₂O₃ ranged from 86.46 to 88.14 %. Similarly, the grades of TiO₂ and Fe₂O₃ in the albite concentrate decreased to 0.116 and 0.026 %, respectively.

The effect of pH for the removal of mica and heavy minerals were also investigated using blends of fatty acid-amine-oleate and amine-oleate combinations. Na-oleat in natural circuit (pH 6) was used in second flotation stage. The results are shown in Fig. 4.

As shown in Fig. 4, in first flotation stage, with the use of Aero 704 blended with Aero 3000C at pH 9, albite concentrate with 0.103 % TiO₂ and 0.029 % Fe₂O₃ grades was produced. Mica and heavy minerals were removed from the albite ore with the recoveries of 68.65 % TiO₂ and 87.31 % Fe₂O₃.

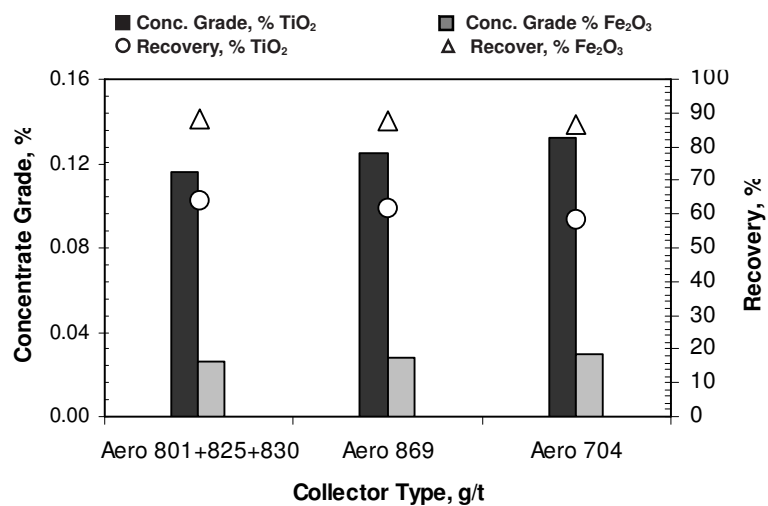


Fig. 3. Performance of various collector blends in multi-stage flotation of mica and heavy minerals

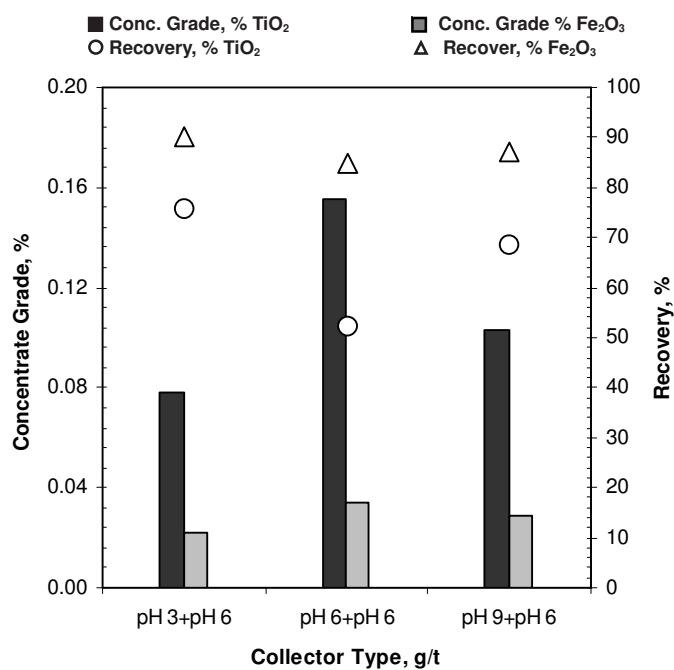


Fig. 4. Effect of pH with the blends of fatty acid-amine-oleate and amine-oleate in multi-stage flotation system for the removal of mica and heavy minerals

In addition, with the use of Aero 3000C at pH 3 in first floatation stage, an albite concentrate with 0.078 % TiO₂ and 0.022 % Fe₂O₃ grades was produced. The removal recovery of TiO₂ from the ore was 75.58 % and of Fe₂O₃ was 90.10 %.

Mechanism of collector action: Fatty acids particularly oleate are the most common collectors used for the removal of coloured impurities from feldspar ores³. Albite has an iep of around 1.5¹⁶. Rutile is the major titanium mineral in albite and the reported iep of natural rutile vary from 3.5 to 5.5¹⁷. The mechanism of oleate adsorption onto salt-type minerals such as fluorite and calcite is regarded as chemisorption and has been covered in several comprehensive reviews¹⁸⁻²⁰. Similarly, the adsorption mechanism of oleate on titanium minerals such as rutile is also regarded as chemisorption with titanium³.

Lui *et al.*¹² in rutile floatation fatty acid soaps and sodium dodecyl sulphate collectors is determined to have strong collecting power for rutile. Oleate adsorption on rutile minerals like that of calcite and fluorite minerals is also obtained in chemisorption ways. Purcell *et al.*¹¹ the rutile pzc value is obtained at pH 6.7 but rutile floatation is determined to be more dependent on Na-oleate concentration than that of pzc. At 10⁻⁶ M and 10⁻⁷ M Na-oleate concentrations, rutile floatation is obtained below rutile's pzc values, whereas higher concentration of Na-oleate in rutile floatation is obtained at higher pH values above the pzc.

Conclusion

The results of bench floatation tests studies for the removal of micaceous and heavy minerals from an albite ore received from the Mugla-Milas region in Turkey are presented below:

- The ore contains albite, orthoclase, quartz, muscovite, biotite, iron oxide, tourmaline, rutile and sphene. Chemical analysis of the ore reveals 7.40 % Na₂O, 3.36 % K₂O, 68.7 % SiO₂, 0.16 % Fe₂O₃, 0.23 % TiO₂, 0.10 % MgO, 0.45 % CaO, 18.4 % Al₂O₃, 0.55 % LOI grade values.
- The use of Aero 704 promoter was proved to be effective in the removal of coloured impurities (micaceous and heavy minerals) in an alkali pH in one-stage, providing benefits of reduced reagent and acid consumption and non-degradation of tank walls. It also provides easy of control of operations due to a one-stage floatation system.
- The results of conventional multi-stage-floatation tests performed with single and blends of collectors revealed that Aero 3000C was effective for the removal of micaceous minerals. Na-oleate proved to be more effective than other collectors in the removal of both iron and titanium minerals.

It is noted that the favourable results with Aero 704 collector was applied to a plant practice and a sellable feldspar product with impurities below specifications was produced with a bulk floatation in an alkaline medium.

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