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# Multi-stage flotation of colored impurities from albite ore in the presence of some cationic and anionic collectors

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**Abstract:** Mica and heavy minerals containing iron oxides and titanium oxides such as rutile and sphene are floated using cationic and anionic collectors, respectively. In this study, separation of colored impurities including mica and heavy minerals from the albite ore obtained from the Aydin region in Turkey was investigated by multi-stage flotation. The effects of the new cationic and various anionic collectors and their dosages in either acidic or natural circuits were investigated to upgrade the albite ore. In the case of the flotation stage of mica minerals, the results obtained from this study showed that the stearylamine collector (Flotigam-S) gave the best performance and an albite concentrate with 7.58% Na<sub>2</sub>O grade was produced with 81.40% Na<sub>2</sub>O recovery at 400 g/Mg Flotigam-S under the acidic condition at pH 3. Additionally, in the heavy minerals flotation stage, the use of Naoleate in the natural circuit (pH 6) was the most effective for removal of iron and titanium oxides, and an albite concentrate with 0.042% Fe<sub>2</sub>O<sub>3</sub> and 0.061% TiO<sub>2</sub> grades was produced with 91.89% Fe<sub>2</sub>O<sub>3</sub> and 88.56 %TiO<sub>2</sub> recoveries, respectively, with the use of 900 g/Mg Na-oleate. Under the optimum conditions, the colored impurities containing mica minerals, iron oxides and particularly sphene as well as rutile could be effectively removed from the albite ore in the presence of Flotigam-S and Naoleate collectors by multi-stage flotation.

Keywords: albite, colored impurities, collectors, multi-stage flotation

# 1. Introduction

Commercial feldspars minerals of albite and orthoclase are primarily used in glass and ceramic industries, whereas the main minerals of feldspar are albite (NaAlSi<sub>3</sub>O<sub>8</sub>), orthoclase/microcline (KAlSi<sub>3</sub>O<sub>8</sub>) and anorthite (CaAlSi<sub>3</sub>O<sub>8</sub>). A 60% of world feldspar production is used in the ceramic industry and 35% in the glass industry as a source of alumina or as a flux (Kursun and Ipekoglu, 2000; Hacifazlioglu et al., 2012). Turkey is one of the prominent albite producers in the world despite possessing 14% of the world feldspar reserves (Hacifazlioglu et al., 2012). In particular, Menderes Massif, south Aegean region of Turkey, is among the largest albite (Na-feldspar) deposits in the world (Celik et al., 1998).

Mica and heavy minerals containing iron and titanium oxides are the primary source of impurities in albite ores, and they cause coloring in the final products of ceramic and glass. For this reason, these impurities must be removed from the albite ores using mineral processing methods in order to increase the quality and value of feldspar ore. One of the most effective and common methods for removing colored impurities containing mica and heavy minerals is flotation (Celik et al., 2001; Seyrankaya, 2003; Orhan and Bayraktar, 2006; Kilinc-Aksay, 2008; Terzi and Kursun, 2015). In general, feldspar can be separated from the impurities through the use of multi-stage flotation. In this method, mica minerals were floated prior to flotation of heavy minerals. Recent applications of flotation flowsheets for removing discoloring gang minerals from feldspar were presented elsewhere (Bayraktar et al., 1997; Gulsoy et al., 2004; Heyes et al., 2012). In the first stage, long chain aliphatic amines were used at pH 2.5-5 to remove the mica minerals by flotation (Baarson et al., 1962; Bayraktar et al., 1997; Orhan and Bayraktar, 2006). These studies indicated that mica minerals could be separated effectively from a feldspar ore by flotation with tallow amine acetate (Orhan et al., 2006), primary tallow amine acetate salt (Bayraktar et al., 1997; Akar et al., 2000) and primary fatty ammonium acetate salt (Sekulic et al., 2004; Kilinc-Aksay, 2008) at pH 3. Moreover, the use of fuel oil along with amine type collectors increased the selectivity of flotation (Rau, 1985; Akar, 1994). After flotation of mica minerals, the use of either classifier or cyclone with the purpose of decreasing the effect of amine type reagents in water increased the selectivity of anionic reagents used during heavy mineral flotation (Rau, 1985; Orhan and Bayraktar, 2006). In the second stage, flotation of heavy minerals consisting of iron and titanium oxides were possible with the use of fatty acids at mildly acidic and alkaline medium, petroleum sulfonates and alkyl succinamates at acidic medium, hydroxamates, oleoyl sarcosine and potassium oleate at about natural circuit and mildly alkaline medium, sodium oleate and various vegetable oil soaps at about mildly acidic medium (Bayraktar et al., 1997; Bayat et al., 2006; Celik et al., 1998; Celik et al., 2001; Kurcan et al., 2007; Kilinc-Aksay, 2008; Kilinc-Aksay et al., 2009; Kaya et al., 2012; Terzi et al., 2013). In the recent years, novel flotation techniques such as the cyclojet, Jameson cell and dissolved air flotation have been applied for removal of colored impurities in fine-sized feldspar ores (Karaguzel, 2010; Karaguzel and Cobanoglu, 2010; Hacifazlioglu et al., 2012). If desired, separation of feldspar from quartz can be provided after flotation of heavy minerals. Firstly, feldspar is activated by hydrofluoric acid, and then floated by amines at pH 2.5-3.5 (Gulsoy et al., 2004). Also, the non-HF process for flotation of feldspar was developed by using diamine acetate plus sulfonate and di-oleate alkylpropylene diamine as collectors (Celik et al., 1998).

In this study, the colored impurities containing mica and heavy minerals in the albite ore supplied from the Aydin region in Turkey were removed by multi-stage flotation. New amine type collectors in the acidic circuit in the first stage and different collector types depending on pH in the second stage were tested to remove mica and heavy minerals containing iron and titanium oxides, respectively. The effects of these collectors on removal of colored impurities from the albite ore were investigated based on its dosages.

#### 2. Materials and methods

#### 2.1 Materials

The albite ore sample used for the experiments was obtained from the Aydin region in Turkey. The mineralogical properties of the sample determined by the X-ray diffractometer (XRD) indicated that the main mineral of the ore was albite (NaAlSi<sub>3</sub>O<sub>8</sub>) along with ortoclase (KAlSi<sub>3</sub>O<sub>8</sub>), quartz (SiO<sub>2</sub>), muscovite (KAl<sub>2</sub>(AlSi<sub>3</sub>O<sub>10</sub>)(F,OH)<sub>2</sub>), biotite (K(Mg,Fe)<sub>3</sub>(AlSi<sub>3</sub>O<sub>10</sub>)(F,OH)<sub>2</sub>), sphene (CaTiSiO<sub>5</sub>) and rutile (TiO<sub>2</sub>) (Fig. 1). A chemical analysis of the sample (Table 1) carried out by X-ray fluorescence (XRF), showed that TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> contents of the sample were determined as 0.25 and 0.22%, respectively. According to the results of microscopic liberation observations based on a grain size count method (Aytekin, 1979; Saklar et al., 2000) by an Olympus SZ 61 binocular microscope, 95% of the albite sample was liberated below 125 µm particle size.



Fig. 1. XRD results of sample

Component	Content (%)			
SiO <sub>2</sub>	68.11			
$Al_2O_3$	18.15			
Na <sub>2</sub> O	7.24			
K <sub>2</sub> O	2.76			
Fe <sub>2</sub> O <sub>3</sub>	0.22			
TiO <sub>2</sub>	0.25			
MgO	0.09			
CaO	1.33			
LOI*	1.43			
*Loss-on-ignition				

Table 1. Chemical composition of sample

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The albite ore was crushed to below 2 mm by a laboratory jaw crusher, and ground to below 125  $\mu$ m by using a porcelain ball mill. The particle size distribution of the sample was determined using wet sieving. The results of sieve analysis showed that  $d_{80}$ ,  $d_{50}$ , and  $d_{20}$  sizes of the particles were found to be 69.80, 45.70 and 30.10  $\mu$ m, respectively.

#### 2.2 Methods

Prior to the flotation tests, the samples were de-slimed by repeated (4 times) decantation. The particles below 25  $\mu$ m ( $d_{80}$ =15  $\mu$ m) size were separated as slimes. The amount of the material deslimed was 7% by weight. All the flotation tests were conducted on -125+25  $\mu$ m fractions with 500 g samples.

The impurities, mica and heavy minerals, were removed from the samples by a reverse flotation method. First, flotation of mica was carried out prior to flotation of heavy minerals due to considerably higher amounts of muscovite. Second, the heavy minerals consisting of iron oxide and titanium oxide were removed from the ore using anionic collectors. The anionic collectors were added into the cell stage-wise. A self-aerated sub-A Denver flotation machine (2 dm<sup>3</sup>) was used. The multistage flotation test conditions and procedure are presented in Table 2. All the flotation products were analyzed by XRF to determine the best performing collector. As can be seen in Table 2, 50% solids were used for the conditioning stage prior to mica and heavy minerals flotation. It is well known from literature that fatty acid collectors and petroleum sulfonates are usually conditioned at 50 to 70% solids, whereas amines, amine acetates or amine dispersions are usually conditioned at 30% to 50% solids (Baarson et al., 1962). Similar applications are seen in previous studies (Akar, 1994; Bayraktar et al., 1997; Gulsoy et al., 2004; Orhan et al., 2006).

Fable 2.	Multi-stage	flotation test	t conditions	and	procedu	ıre

Conditions	Parameters	Mica minerals flotation	Heavy minerals	
Conditioning	Solids, %	50	50	
	Time, min	15	15	
	Impeller speed, rpm	1500	1500	
	Na <sub>2</sub> SiO <sub>3</sub> , g/Mg	1000	1500*	
	Collector, g/Mg	Cationic	Anionic*	
	Fuel oil+kerosene, g/Mg	100		
	Dowfroth 1012, g/Mg	75	75*	
Flotation	Solids, %	20	20	
	Time, min	10	10	
	Impeller speed, rpm	1200	1200	

\* in second stage, collectors were added into the cell in 2 steps as 1:1 w/w.

The flotation tests were conducted with different cationic and anionic reagents. These reagents were Flotigam-S (stearylamine), Flotigam-T (tallow fatty amine), Aero promoter 801 (petroleum sulphonate), Aero promoter 830 (alkyl succinamate), and Na-oleate. Flotigam and Aero series were

supplied by Hoechst AG (Germany) and Cytec Industries Inc. (USA), respectively. Na-oleate was prepared with similar way by Bayraktar et al., 1997. Local olive oil was used for preparing Na-oleate. A 5 N NaOH was added drop-wise by 1.8:1 volumetric ratio of into warm stirred olive oil and diluted to 10% w/w with distilled water. Besides, fuel oil and kerosene (blended as 1:4 w/w) were used as auxiliary collectors. In the flotation tests, the pH of the system was adjusted with H<sub>2</sub>SO<sub>4</sub>. Sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) and Dowfroth 1012 were used as a dispersant and a frother, respectively.

### 3. Results and discussion

The colored impurities in the albite ore were floated stage-wise, i.e., multi-stage flotation. In the first and second stages, the mica flotation and heavy minerals flotation were applied, respectively. The results were explained on the basis of Na<sub>2</sub>O% grade and recovery in albite concentrates. Mica minerals were floated with stearylamine (Flotigam-S) and tallow fatty amine (Flotigam-T) at pH 3. The performances of Flotigam-S and Flotigam-T collectors were tested with various collector dosages at pH 3. Flotigam-S and Flotigam-T were used as cationic collectors in the amount of 200, 400, 600, and 800 g/Mg. The results of the flotation tests are shown in Fig. 2.



Fig. 2. Effect of cationic collectors and its dosage on mica minerals flotation at pH 3

As shown in Fig. 2, stearylamine (Flotigam-S) was more effective than tallow fatty amine (Flotigam-T) for removal of mica minerals from the albite ore. It was found that the optimum dosage of Flotigam-S was 400 g/Mg at pH 3. The albite concentrate with 7.58% Na<sub>2</sub>O grade and 81.40% Na<sub>2</sub>O recovery was obtained with the use of 400 g/Mg Flotigam-S. For Flotigam-T, the highest performance was provided with 600 g/Mg at pH 3, and the albite concentrate containing 7.35% Na<sub>2</sub>O grade was produced with 71.50% Na<sub>2</sub>O recovery. As known from literature, feldspar, guartz and some ironbearing minerals as gang minerals are associated with mica minerals. The point of zero charge (pzc) of muscovite is about pH 1.0, and muscovite is readily floated using a cationic collector in an acidic system and an anionic collector in a basic system (Fuerstenau et al., 2007). Many papers on the application of collectors in flotation of mica minerals from feldspar have been focused on primarily to cationic amine collectors at pH 3 (Celik et al., 1998). The hydrogen ion concentration allowing activation and flotation of the mica and some of the iron-bearing minerals at pH of about and below 3.5 is sufficient to prevent attachment of the amine collector onto the feldspar and silica surfaces (Baarson et al., 1962). On the other hand, the pzc of feldspar is similar to that of quartz which is about pH 2 (Karaguzel, 2010; Heyes et al., 2012). It was stated that feldspar can be floated by cationic amine collectors at pH 2.5-3.0 in the presence of soluble fluoride ions (Heyes et al., 2012). The presence of the fluoride ion activates the feldspar surfaces for bubble-particle attachment by the amine collector, and hence depresses quartz (Baarson et al., 1962, Celik et al., 2001).

In this purpose, in the second stage, flotation of heavy minerals from the albite ore was carried out using various anionic collectors. The iron and titanium bearing minerals containing sphene and rutile in the albite ore were floated by anionic collectors at pH 3 and 6 (natural). Petroleum sulfonate (Aero 801), alkyl succinamate (Aero 830) and Na-oleate (Na-Ol) were used as anionic collectors for the flotation tests. The performances of the collectors were tested at 500, 700, 900 and 1100 g/Mg. The results were evaluated on the basis of  $Fe_2O_3$  and  $TiO_2$  grades in the albite concentrate and recoveries of the colored impurities from the albite ore. The results are shown in Figs. 3 and 4.



Fig. 3. Effect of anionic collectors and its dosage on flotation of heavy minerals at pH 3

As clearly seen in Fig. 3, the alkyl succinamate promoter (Aero 830) exhibited the best performance at pH 3. It was determined that the optimum dosage of Aero 830 was 900 g/Mg. The albite concentrate containing 0.056% Fe<sub>2</sub>O<sub>3</sub> and 0.096% TiO<sub>2</sub> grades was obtained with the recovery of 90.03% Fe<sub>2</sub>O<sub>3</sub> and 83.60% TiO<sub>2</sub>, respectively. The use of Aero 801 substantially removed iron bearing minerals at pH 3. However, it failed to show an adequate performance in terms of titanium bearing minerals, and an albite concentrate with 0.061% Fe<sub>2</sub>O<sub>3</sub> and 0.149% TiO<sub>2</sub> grades was produced with the recoveries of 87.23% and 74.91% at 900 g/Mg, respectively. Additionally, Na-oleate gave the poorest performance at pH 3.

As seen from Fig. 4, Na-oleate at natural pH (pH 6) gave the best results for removal of iron and titanium bearing minerals from the albite sample. It was found that the optimum dosage of Na-oleate was 900 g/Mg at pH 6 (natural). The albite concentrate with the grades of 0.042% Fe<sub>2</sub>O<sub>3</sub> and 0.061% TiO<sub>2</sub> was produced with the recoveries of 91.89% Fe<sub>2</sub>O<sub>3</sub> and 88.56% TiO<sub>2</sub>, respectively. In the case of Na-oleate, the recoveries of colored impurities in the albite concentrate increased with increasing to pH 6 from pH 3, and the best quality concentrate was obtained at natural pH (pH 6). On the other hand, flotation of iron and titanium minerals from the albite ore was not achieved using petroleum sulphonate (Aero 801) and alkyl succinamate (Aero 830) collectors in natural pH (pH 6). The albite

concentrate with the high values of  $Fe_2O_3$ % and  $TiO_2$ % grades and the low recoveries of iron and titanium minerals from the albite ore was produced using Aero 801 and Aero 830 collectors at pH 6 (natural). For instance, while the concentrate with the grades of 0.133%  $Fe_2O_3$ , 0.162%  $TiO_2$  was obtained with the recoveries of 45.11%  $Fe_2O_3$ , 34.21%  $TiO_2$  using 900 g/Mg Aero 801, the concentrate with the grades of 0.127%  $Fe_2O_3$ , 0.152%  $TiO_2$  was produced with the recoveries of 46.83%  $Fe_2O_3$ , 36.33%  $TiO_2$  using 900 g/Mg Aero 830.



Fig. 4. Effect of anionic collectors and its dosage on flotation of heavy minerals at pH 6

As can be seen from Figs. 3 and 4, Na-oleate in the natural circuit (pH 6) gave better results than Aero 830 in the acidic circuit (pH 3) in terms of the removal of titanium bearing minerals containing particularly sphene and rutile as well as iron bearing minerals from the albite ore. It is known that while albite has the iep of around 1.5 (Celik et al., 1998, Bayat et al., 2006), the iep of rutile and sphene as titanium minerals in albite is 3.5-5.5 (Parks, 1965) and 3.5 (Kosmulski, 2009), respectively. Fatty acids are commonly used in order to remove the colored impurities from feldspar ores (Celik et al., 1998, Lui and Peng, 1999). In particular, fatty acids and fatty acid soaps containing oleic acid are strong collectors for flotation of rutile at pH 4-6 (Bayraktar et al., 1997; Lui and Peng, 1999).

# 4. Conclusions

This study investigated separation of colored impurities containing mica, iron and titanium bearing minerals in the albite ore received from the Aydin region in Turkey by multi-stage flotation. It was determined from the XRD analysis that while the sample mostly contained albite, it also contained muscovite, biotite, iron oxide, tourmaline, sphene and rutile as colored impurities. In addition, it was observed that the liberation size of albite was below 125  $\mu$ m. Based on the results from the flotation experiments, in the first stage, the use of a stearylamine collector (Flotigam S) was found to be more selective than tallow fatty amine (Flotigam T) for flotation of mica minerals from the albite ore. Under

the optimum conditions, the albite concentrate was obtained at conditions of 400 g/Mg dosage of Flotigam S at pH 3. The Na<sub>2</sub>O% grade of the concentrate increased to 7.58% from 7.24% and 81.40% Na<sub>2</sub>O recovery was obtained. In the second stage, Na-oleate in the natural circuit (pH 6) was found to be effective in the removal of both iron and particularly titanium minerals containing sphene and rutile. The albite concentrate with 0.042% Fe<sub>2</sub>O<sub>3</sub>, 0.061% TiO<sub>2</sub> grades was obtained with the recoveries of 91.89% Fe<sub>2</sub>O<sub>3</sub>, 88.56 %TiO<sub>2</sub>, respectively. At pH 3, the use of Aero 830 was more effective than Aero 801 in removal of iron bearing minerals and its optimal dosage was found to be 900 g/Mg. The albite concentrate with 0.056% Fe<sub>2</sub>O<sub>3</sub> and 0.096% TiO<sub>2</sub> grades was obtained with the recoveries of 90.03% Fe<sub>2</sub>O<sub>3</sub> and 83.60% TiO<sub>2</sub>, respectively. Finally, the albite concentrate containing 69.92% SiO<sub>2</sub>, 18.85% Al<sub>2</sub>O<sub>3</sub>, 7.58% Na<sub>2</sub>O, 2.96% K<sub>2</sub>O, 0.042% Fe<sub>2</sub>O<sub>3</sub>, 0.061% TiO<sub>2</sub>, 0.07% MgO, 0.23% CaO, and 0.11% LOI was produced using 400 g/Mg dosage of Flotigam S at pH 3 and 900 g/Mg dosage of Na-oleate at pH 6 (natural). In the presence of Flotigam-S and Na-oleate collectors, the results obtained from this study showed that the mica minerals and heavy minerals containing particularly sphene and rutile as well as iron oxides in the albite ore could be effectively removed under the optimum conditions by multi-stage flotation.

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