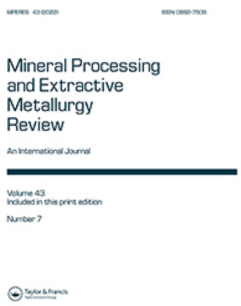

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Enes Kalyoncu & Fırat Burat

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Selective Separation of Coloring Impurities from Feldspar Ore by Innovative Single-stage Flotation

Enes Kalyoncu [a] and Fırat Burat [b]

[a]Department of Machinery and Metal Technologies, Istanbul Gelisim University, Istanbul Gelisim Vocational School, Istanbul, Turkey; [b]Faculty of Mines, Mineral Processing Engineering Department, Istanbul Technical University, Maslak, Turkey

ABSTRACT

The classical use of an anionic collector does not lead to a satisfactory flotation response for feldspars with high content of mica, iron, and titanium oxides. Therefore, amine-type cationic reagents are preferred together with an anionic reagent to remove especially problematic mica minerals. In this study, main colored impurities in the albite ore were efficiently removed by single-stage flotation using newly developed anionic-cationic mixed reagents. 2408 collector alone achieved much greater results at 1000 g/t dosage and a salable feldspar concentrate assaying 0.03% Fe_2O_3 and 0.07% TiO_2 was successfully produced with 93% Na_2O recovery at natural pH. The synergistic effects of 2404 and 2408 collectors, which were successful in removing iron and titanium oxides, were investigated and a feldspar concentrate with 0.04% Fe_2O_3 and 0.03% TiO_2 was obtained at a slightly basic pH medium. With this novel and innovative approach, the increased capacity, easier control, less unit cost, and low collector dosage can be easily achieved using a single collector without the need for any frother or pH adjusting agents.

HIGHLIGHTS

- CustoFloat 2408 has been singled out to be the best performer among all other potential collectors.
- A salable feldspar concentrate assaying 0.03% Fe_2O_3 and 0.07% TiO_2 was successfully produced in single stage flotation.
- 1000 g/t collector concentration succeeded in removing 95% of mica minerals.

KEYWORDS

Feldspar; flotation; collector; synergistic effect

1. Introduction

Turkey hosts the largest Na-feldspar reserves in the world and is by far the leader in feldspar mining. Despite their high sodium content, impurities such as mica, iron, and titanium oxides, limit the marketing of feldspars (Görken et al. 2019 ; Gulsoy and Kademli 2006; Gülgönül et al. 2012; Kangal and Guney 2002; Pariyan et al. 2020). A salable feldspar concentrate typically contains 11–13% Na_2O+K_2O, less than 1.5% CaO+MgO, 0.07–0.3% Fe_2O_3+ TiO_2, and free quartz up to 8–10% (Karaguzel et al. 2006). While high-intensity magnetic and electrostatic separators are used for feldspar ores with low iron content (Moura et al. 2019; Peretti, Serci and Zucca 2012; Xu et al. 2021), the flotation method is indispensable for many ores with relatively high titanium and iron content (Burat, Kangal and Önal 2006; Kangal et al. 2017).

Conventionally, mica minerals are removed using cationic reagents (especially long-chain aliphatic amines) at around pH 2.5–3.5 (Burat et al. 2007; Gulsoy et al. 2004; Heyes et al. 2012; Kangal et al. 2018; Xie et al. 2020). After removal of mica-containing tailings, iron and titanium oxides are floated using fatty acids in an acidic medium (pH 4–6) or basic conditions (pH 9). For feldspar ores containing high amounts of mica minerals, an increasingly common practice is to mix synthetic reagents with fatty acids. In the study of Orhan and Bayraktar (2006), mica minerals were floated with 100 g/t tallow amine acetate, and then iron and titanium bearing oxide minerals were removed by 1000 g/t sodium oleate. Co-adsorption of oleate on feldspar occurred in the presence of the residual amine and high feldspar loss resulted in the second stage. Because of the challenges in multi-stage flotation more efficient collectors have been developed to remove coloring impurities by reducing the number and the amount of collectors (Aksay et al. 2009).

A wide variety of applications have been presented by different authors to explain the fact that reactive mixtures provide a higher flotation performance than each reagent. Much smaller critical micelle concentration (CMC) could be produced compared to each component and the cationic/anionic systems have the greatest synergistic effect among the various binary collector systems (Bradshaw, Harris and O'Connor 1998; Kume, Gallotti and Nunes 2008; Wang et al. 2016). Electrostatic head-head repulsion between adjacent surfaces of cations reduces in the presence of anionic collectors. In contrast, the number of lateral tail-tail hydrophobic bonds increases. Finally, the charge repulsion between the cations decreases, and the density of the mixed micelles is eliminated (Lu et al. 2008). In this way, lower dosage requirements, improved selectivity and recovery rates, and the floatability of coarse particles can be achieved (Bozkurt et al. 2006; Rao and Forssberg 1997).

CONTACT Fırat Burat buratf@itu.edu.tr Faculty of Mines, Mineral Processing Engineering Department, Istanbul Technical University, Maslak, Turkey
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Considering the above-mentioned information, single-stage experiments were carried out with newly developed mixed collectors to solve the challenges of the multi-stage feldspar flotation system. A single collector alternative to replacing standard multi-stage flotation using various and high amounts of collectors became the main objective of this work and the potential synergistic effects of the tested collectors were discussed by adopting various flotation flowsheets.

2. Materials and methods

2.1. Materials

Approximately 100 kg of comminuted and de-slimed albite ore sample was supplied from Esan Company in the Milas district of Muğla province, Turkey. The chemical analysis was performed using a Panalytical Axios X-ray fluorescence (XRF) in the accredited Esan laboratory. The samples were annealed for 1 h at 950°C using borate fusion and the ratio of sample to flux was defined as 1:8. The feldspar ore assayed 67.64% SiO_2, 18.84% Al_2O_3, 9.73% Na_2O, 1.22% CaO, 0.69% MgO, 0.66% K_2O, 0.45% TiO_2, 0.32% Fe_2O_3, and 0.45 LOI. The mineralogical analysis was determined by a Panalytical X'pert Pro X-ray diffraction (XRD) spectroscopy (see Figure 1Sa).

Microscopic studies helped to identify muscovite, biotite, and phlogopite-type mica minerals. Minerals such as titanious (rutile, anatase, and sphene) and ferruginous (garnet, tourmaline, and hornblende) found in typical Menderes massive in the Aegean region were below the XRD analysis limits. Akar et al. (2017) reported that the albite ore in the Milas district was liberated by 90% from other minerals below 180 μm. The sieve analysis indicated that the ore was below 300 μm, the d_{80} and the d_{50} sizes were 220 μm and 160 μm, respectively.

The reagents with the brand names of CustoFloat 2404, CustoFloat 2408, and CustoFloat 2410 were supplied by Arkema-ArrMaz (USA) to investigate the effect of mixed collectors with different compositions. 2404 is an oil-based collector and formulated with tall oil fatty acid (TOFA) with anionic surfactants. 2408 and 2410 are water-based (emulsion) anionic-cationic mixed collectors and have a synergistic effect of chemisorption and hydrogen bonds adsorption on especially on mica and iron minerals. 2408 was formulated with pre-saponified fatty acids/surfactants and with a higher total amine value (TAV). 2410 was formulated mixed carbon chain-length fatty acids with lower TAV. Sulfuric acid (H_2SO_4) (Merck) and sodium hydroxide (NaOH) (Sigma-Aldrich) were used as pH modifiers. No frother was required during the experimental studies. Tap water was used for batch flotation tests.

2.2. Methods

A self-aerated Denver flotation machine, equipped with a 2.5 L stainless steel cell, was used in flotation tests. 500 g sample was mixed with 2000 ml tap water and the impeller speed was fixed to 1100 rpm. The conditioning time of 5 min for each stage of collector addition was kept constant and a flotation time of 3 min was selected. The natural pH of the pulp was measured as 7.5. The adsorption of the reagents on the solid particles was improved by preparing each collector as a solution. To evaluate the results of flotation tests Fe_2O_3 + TiO_2, MgO, and Na_2O contents were taken into consideration for the representation of heavy metal oxides, mica, and feldspar minerals, respectively.

3. Results and discussion

3.1. Single-stage flotation tests

The preliminary flotation experiments were carried out to determine the optimum collector type and concentration. The conditions of the flotation tests are given in Table 1. The effect of collector type and dosage on Fe_2O_3+ TiO_2 content and Na_2O recovery are presented in Figure 1.

A continuous increase in the collector dosage caused a significant decrease in the Fe_2O_3+ TiO_2 value. 2404 and 2408 collectors easily reduced the iron contents below 0.07% and 0.03% with around 85–91% removal efficiency at 1000 g/t. Similarly, a feldspar concentrate (with 10.4% Na_2O content) containing 0.04% and 0.07% TiO_2 was produced with an average of 89% TiO_2 removal efficiency. Further increases in dosage resulted in a noticeable decrease in iron and titanium contents, however, feldspar particles were easily activated and transported to the float zone. An interesting point is that Na_2O recovery dropped dramatically from 98.8% to 79.7% as the dosage of 2408 increased from 500 g/t to 1500 g/t. The reason is that the TAV of 2408 is the highest compared to other competitive collectors. The co-adsorption of fatty acid together with other components, mainly amine, on feldspar surfaces creates the interactions of oppositely charged heads and tails that increase the floatability of feldspar particles (Figure 2S). As the feldspar surfaces are covered with collector molecules at 2000 g/t dosage the mixed micelle formation gradually increased and the feldspar particles started to agglomerate. When 2410 was selected the amount of floated products remained low and the increased collector concentration did not cause feldspar loss. Especially after 1000 g/t collector concentration, 2404 and 2408 succeeded in removing 84% and 95% of mica minerals, respectively (Figure 3S). 2408 leads to better performance and provided significantly better Fe_2O_3 reductions by collecting slow floating coarse mica particles.

The dependence on pH of Fe_2O_3+ TiO_2 grades and recoveries for each collector are presented in Figure 2. The pH value of the medium has a great influence on the removal of metal oxide impurities and the recovery of Na_2O. 2408 has been singled out to be the best performer among all other potential collectors both for the removal of Fe_2O_3+ TiO_2 and Na_2O recovery, however, the selectivity is slightly impaired at higher pH levels. The results also indicated that iron and titanium-bearing minerals

Table 1. Constant and variable parameters in bench-scale flotation.

Operating parameters	
Particle size	−300 + 20 microns
pH	6, 7.5, and 9
Collector type	2404, 2408, and 2410
Collector concentration	500, 1000, 1500, and 2000 g/t
Conditioning time	5 min (for each stage)
Flotation time	3 min (for each stage)
% solids (by weight)	20%

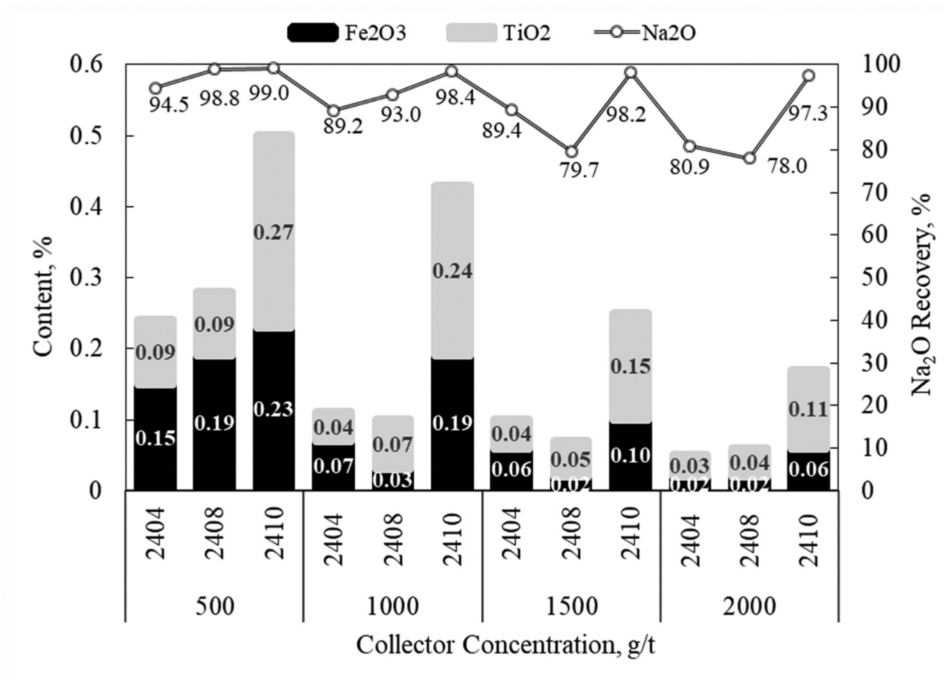

Figure 1. The effect of collector type and dosage on Fe_2O_3+ TiO_2 content and Na_2O recovery.

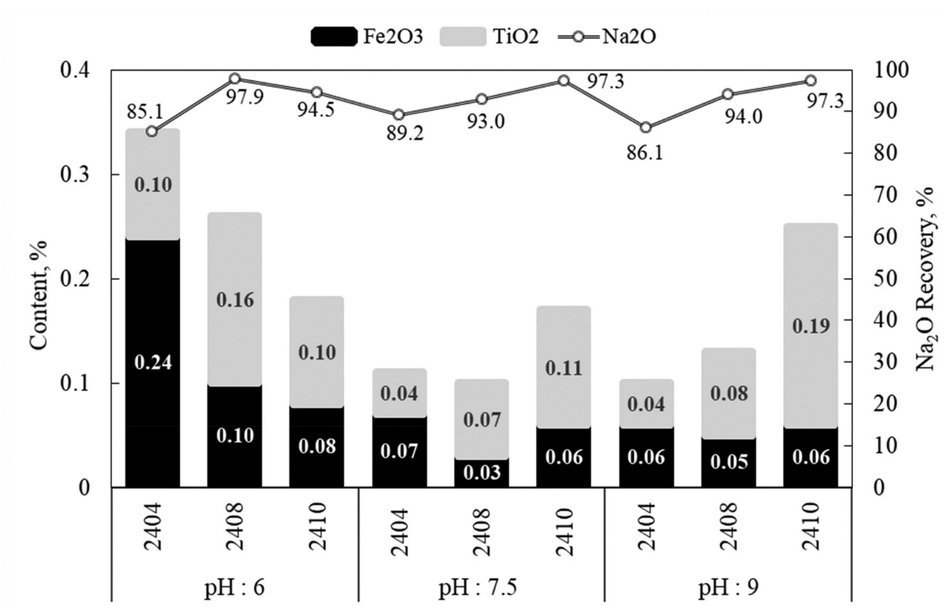

Figure 2. The effect of pH on Fe_2O_3+ TiO_2 content and Na_2O recovery.

were very sensitive to acidic pH values and substantially removed from feldspar ore at pH 7.5 and 9. Adsorption of oleic acid and oleate on salt-type minerals such as fluorite and calcite is achieved by chemical adsorption and has been addressed in many reviews (Hanna and Somasundaran 1976; Rao and Forssberg 1991). It has been reported that the chemical adsorption of oleic acid on fluorite reaches its peak around pH 9. Similar results were obtained with barite and calcite minerals (Aplan and Fuerstenau 1962). Congruently, the adsorption mechanism of oleate on titanium minerals such as rutile is considered as chemisorption with titanium (Celik, Can and Eren 1998). Since a high-quality feldspar concentrate could be produced without the need for pH adjusters, the natural pH was selected in the next tests. 2410 collector containing less TAV in its formulation activated a limited number of feldspar particles even at high collector concentrations and pH values. However, 2410 was not used in the subsequent studies since it has failed to produce a salable feldspar concentrate. The variation of curves in Figure 4S clearly shows the importance of pH in the removal of mica minerals. The low MgO recovery of 2404, which is also insufficient in removing iron impurities at pH 6, reveals that the iron content is largely dependent on mica minerals.

3.2. Multi-stage flotation tests

2404 and 2408 were chosen for their superior success in removing colored impurities and obtaining high Na_2O recovery. Since 2404 contains TOFA and anionic reagent mixture in its formulation it easily floats titanium minerals, while 2408 enables the flotation of mica minerals due to its amine content. For this reason, these two collectors were added to the flotation cell with different combinations and the experiments were carried out by following the flowsheets given in Figure 5S. The effect of binary collectors on the content and recovery of feldspar concentrate was investigated at various pH values by keeping the total collector concentration to 1000 g/t. The effect of the sequence of collectors on content and recovery is shown in Figure 3.

Figure 3 indicated a dramatic decrease in Fe_2O_3+ TiO_2 contents as the pH increased from 6 to 7.5. Na_2O recovery decreased due to the high amount of misplaced feldspar particles in the floated product. When 2408 was used alone it caused lower feldspar loss compared to 2408(1°)-2404(2°), however, this combination allowed the production of a very high-quality feldspar with 0.04% Fe_2O_3 and 0.03% TiO_2 contents at pH 9. 89% of iron oxides and 95% of titanium oxides were removed from feldspar ore by this way. The synergistic effects of collectors on the removal of colored impurities were also investigated by following the flowsheets in Figure 5Sb and 5Sc. Initially, 2404 and 2408 were added individually in the first and second flotation stage at a total concentration of 500 g/t (2404 + 2408). Differently, 2404 and 2408 reagents were mixed for 15 minutes in a beaker and then entrained to the flotation cell at 500 g/t in two stages (2404&2408). The synergetic effect of collectors on Fe_2O_3+ TiO_2 content and Na_2O recovery are illustrated in Figure 4.

Fe_2O_3+ TiO_2 grades gradually decreased with increasing pH and the selectivity was improved for iron and titanium oxide minerals when 2404 + 2408 was implemented at pH 9. However, the amount of floated materials increased and the Na_2O recovery dropped dramatically from 93.8% to 73.6%. 2404&2408 caused a significant feldspar loss at natural pH compared to 2404 + 2408. It can be concluded that the synergetic effect could not be observed when the collectors were premixed before the addition and synergism may depend on the sequence of adding collectors more than the presence of a mixture. The results in Figure 6S show that the 2408 collector, which was first added to the flotation cell, could be adsorbed more easily on mica minerals and enhance floatability at slightly acidic and basic pH values. On the other hand, 2404 gains its ability to float mica minerals at natural pH values. When 2404 and 2408 were added to pulp together, they were able to remove mica minerals to an extent as the average of their individual results. Figure 5 shows the results of single and multiple flotation studies at natural pH. The lowest Fe_2O_3 + TiO_2 content (0.07%) could be obtained when 2408(1°)-2404(2°) suit was selected in pH 9. However, the Na_2O recovery was 6% less than 2408, and a pH adjusting agent was required (NaOH). Consequently, a high quality feldspar concentrate assaying 68.49% SiO_2, 19.27% Al_2O_3, 10.46% Na_2O, 1.06% CaO, 0.04% MgO, 0.26% K_2O, 0.07% TiO_2, and 0.03% Fe_2O_3 was successfully produced using a single collector (2408). The XRD analysis was subjected to this feldspar concentrate (Figure 1Sb) and the mica band, which was previously seen in Figure 1Sa was disappeared.

Esan Co. has huge alkali feldspar reserves and ranks first in the world in feldspar production (a million ton/year). In the plant, anionic and cationic collectors are added in two stages as 1300 g/t and 150 g/t, respectively. To compare the results of single and multi-stage flotation, and to simulate the data in the plant laboratory-scale tests were designed and performed using these collectors at the optimized dosages. A total of 86.5% Fe_2O_3 and 90.7% TiO_2 were removed and a feldspar concentrate assaying 0.05% Fe_2O_3 and 0.05% TiO_2 was obtained with 85% Na_2O recovery, whereas 1000 g/t 2408 singly produced a feldspar concentrate

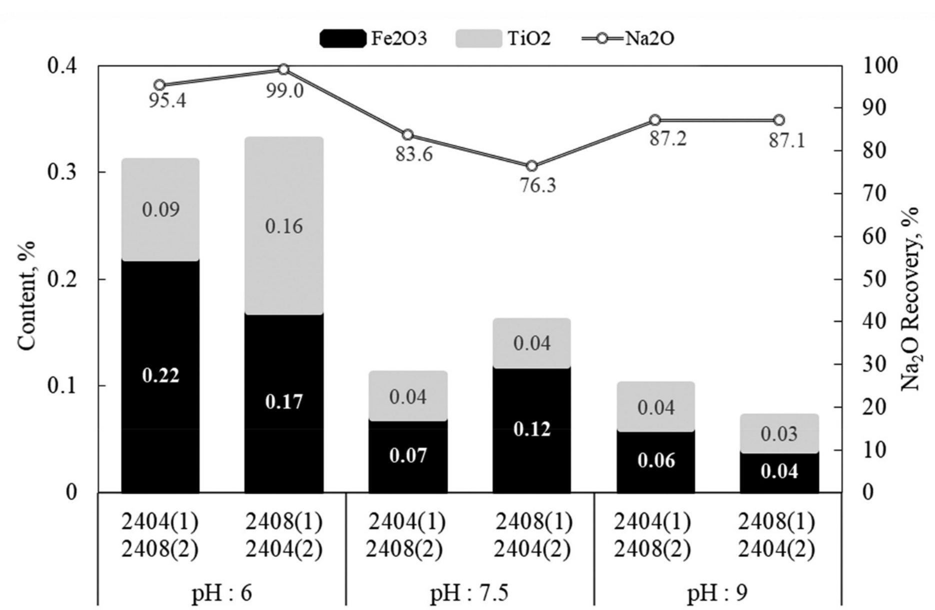

Figure 3. The effect of the sequence of collectors on Fe_2O_3+ TiO_2 content and Na_2O recovery.

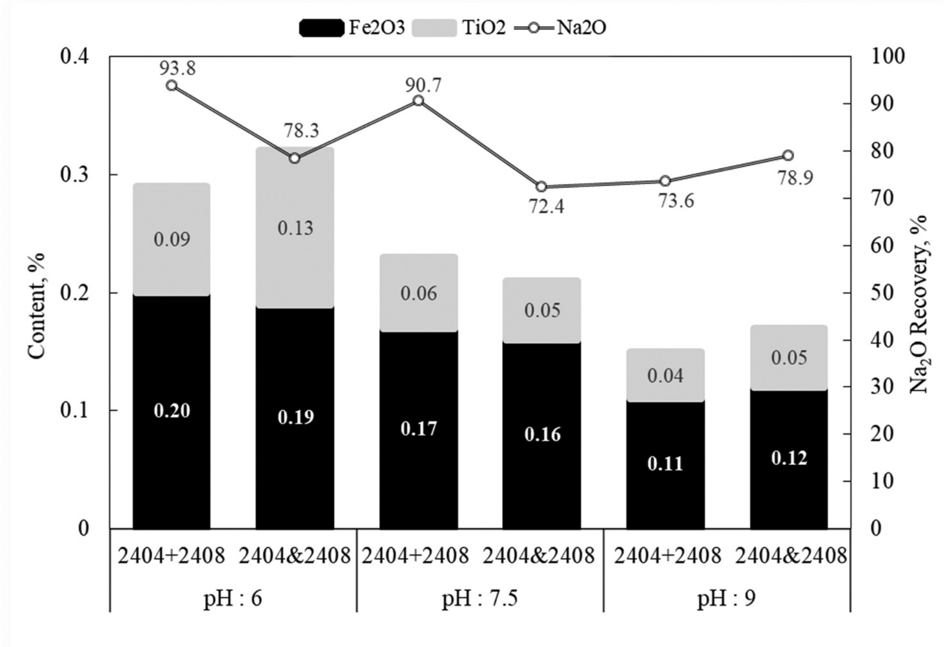

Figure 4. The synergetic effect of collectors on Fe_2O_3+ TiO_2 content and Na_2O recovery.

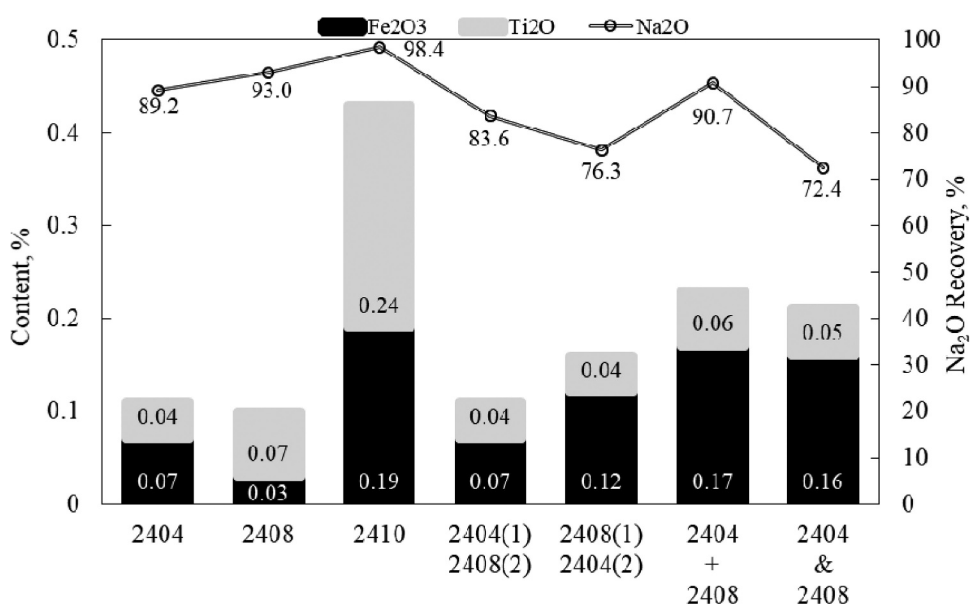

Figure 5. The combined results of single and dual collector systems on Fe_2O_3+ TiO_2 content and Na_2O recovery.

with similar metal oxide content (0.03% Fe_2O_3 and 0.07% TiO_2) and 8% higher Na_2O recovery compared to the standard collector suite. In this way, 80.000 tons of feldspar concentrate will not be lost during a year of production. An innovative process can be developed that uses a single collector without the need for pH adjusters and frothers by converting multi to single-stage flotation. Even if the results of the laboratory studies performed with the reagents currently used in the factory are in line with the plant data, it must be emphasized that single batch flotation tests do not necessarily simulate plant performances due to complicated plant operations. Therefore, the current approach based on laboratory studies must be followed by actual plant trials.

4. Conclusions

Increasing demand for raw materials, economic and environmental concerns necessitate the use of less number and amount of reagents in the feldspar sector. In this study, colored impurities in feldspar, which do not allow its marketing, were successfully removed by single-stage flotation. A high-quality and salable feldspar concentrates with 0.03% Fe_2O_3 and 0.07% TiO_2 grades were obtained at 1000 g/t dosage of 2408 in natural pH by a single-stage flotation. Mica and heavy minerals were removed from the ore with the recoveries of 91.3% Fe_2O_3 and 86.9% TiO_2. Excess collector dosage caused feldspar particles to float and the Na_2O recovery dropped. 2408(1°)-2404(2°) collector suit produced a feldspar concentrate with 0.04% Fe_2O_3 and 0.03% TiO_2 grades at alkali pH, however, Na_2O recovery decreased by 6%. The results of this study presented a step-change for feldspar flotation. In this way, many benefits will be provided both economically and environmentally, i.e., lower reagent dosage, reduced unit cost with the decrease in the number of flotation cells, no need for acid, base, and frother reagents, easy control of operations, and the increased capacity.

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Disclosure statement

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ORCID

Enes Kalyoncu http://orcid.org/0000-0001-9500-1898
Fırat Burat http://orcid.org/0000-0001-7051-0063

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