Mineral beneficiation of nepheline syenite with combination of dry magnetic separation and flotation methods

Mustafa Çınar 1, Emrah Durgut 2

1 Department of Mining Engineering, Faculty of Engineering, Çanakkale Onsekiz Mart University, Çanakkale, Turkey
2 Çan Vocational School, Çanakkale Onsekiz Mart University, Çanakkale, Turkey

Corresponding author: emrahdurgut@comu.edu.tr (Emrah Durgut)

Abstract: Nepheline syenite is an important raw material for ceramic body composition as a melting agent. However, impurities such as iron, titanium, mica, and calcite minerals in nepheline syenite can lead to quality problems on the surface of floor tile because of different sintering properties. Therefore, these impurities should be eliminated from syenite before sintering process to increase its quality while reducing economic and environmental impacts. In this study, it was aimed to reduce impurities of nepheline syenite using dry magnetic separation and flotation methods. The dry magnetic separation results showed that the amount of TiO$_2$+Fe$_2$O$_3$ in the sample decreased from 2.50% to 0.06%. In addition to this, optimum flotation conditions were determined while separating calcite, mica, and further iron bearing minerals from the nepheline syenite by using sequential flotation method. The optimum flotation conditions for calcite flotation were determined as natural pH: 7.9 and 500 g/Mg DER NA7 collector dosage; for mica removal pH: 3.1 and 500 g/Mg Custamine 9024 or A4 collector dosage. After overall mineral beneficiation experiments, albite+microcline mineral content increased from 78% to 97%. In conclusion, a clean concentrate for ceramic body and frit composition quality was gained from nepheline syenite containing high amount of Fe$_2$O$_3$ and TiO$_2$ with dry magnetic separation and flotation experiments.

Keywords: mineral beneficiation, nepheline syenite, dry magnetic separation, flotation, ceramics

1. Introduction

Feldspars are the most abundant mineral group that constitutes almost 60% of all rocks in the earth crust. Syenite is a holocrystalline magmatic igneous rock basically, and the chemical formula of nepheline is Na$_3$KAlSi$_4$O$_{16}$. When a little amount of nepheline takes place of quartz or/and feldspar inside the syenite, nepheline syenite is formed. Nepheline syenite is combined of 48-54% albite, 18-23% microcline, and 20-25% nepheline. The rock can also contain muscovite, biotite, corundum, hornblendt, and magnetite minerals (Potter, 2003).

Nepheline syenite is mostly used in the ceramic and glass production leadingly in terms of high content of aluminium and alkaline as an industrial raw material. In the porcelain stoneware bodies, nepheline syenite enhances body densification and mullite formation by reducing sintering temperature (Pekdemir, 2008; Kamseu et al., 2013). Feldspathic minerals also play an important role in the frit and glaze compositions as melting agents (Dondi, 1994). Raw materials used in the ceramic production are obtained from the nature, and can contain impurities which affect the production quality negatively. Such impurities are specified as Na-amphibole, Na-pyroxene, biotite, muscovite, and melanite in literature (Bayhan and Girgin, 1993; Biswal et al., 2004). Additionally, the calcite was formed in the intrusive as impurity for nepheline syenite with the alteration of marble (Moyd, 1949). In the ceramic production, iron and titanium bearing minerals have an adversely effect on the color of the product (Amaireh ve Alijaradin, 2014). Besides calcite and mica minerals can make pinholes on the glaze surface
because of the expansion and outgassing in the sintering process due to the high quantity and coarser grain size (Suleiman et al., 2013).

Mineral processing methods are applied to the raw materials according to mineral types and particle size to remove impurities. Iron bearing minerals are the major impurities which affect the color of product negatively, and magnetic separation is the most widely used method to eliminate such minerals. Dry/wet and high/low intensity magnetic methods are applied to nepheline syenite with regard to the mineral type and particle liberation size (İbrahim et al., 2002; Jena et al., 2014). Dry magnetic separation method is used to separate magnetic minerals from nepheline syenite ore in order to use in ceramic bodies mainly (Abouzeid and Negm, 2014). It is also applied by the reason of pre-enrichment because this method is applied to relatively higher particle size distributions, and the concentrates which are taken after dry magnetic separation can be enriched further with wet magnetic and flotation methods due to the better mineral particle liberation (Gougazeh et al., 2006; El-Rehiem and Abd El-Rahman, 2008). Flotation method is applied to fine grain sized feldspathic minerals, besides slimes have negative impact on the flotation recovery. Therefore, ore must be ground and deslimed before the flotation process. Burat (2017) found that feldspar flotation recovery decreased almost 10% in the presence of slime. In the feldspathic mineral flotation, mica minerals are removed with amine type collectors at acidic pH: 3-3.5, then sulphonate and oleic acid collectors are used in order to separate coloring impurities without changing pH. Lastly sodium/potassium feldspar separation is executed in the presence of salt by using amine type collectors following quartz separation with HF at pH 2.5-3 typically (Karagüzel et al., 2006; Demir 2010; Boulos et al., 2015). Besides, metallic minerals can be removed from the ore in the desliming process; this situation shows that the metallic minerals are disseminated in the ore with fine grain sized (Ahmed et al., 2016). In addition to these, magnetic separation and flotation are used together alternately in terms of feldspar beneficiation (Saklar et al., 2003; Seyrankaya, 2003). Moreover, Fe₂O₃ content of nepheline syenite can be further decreased by leaching with oxalic acid following magnetic separation and flotation to have better quality (Kangal et al., 2018).

In this study, it was aimed to eliminate impurities of nepheline syenite by physical and physico-chemical methods. In this regard, raw material was firstly crushed, ground, and sized for high intensity dry magnetic separation experiments. Concentrates obtained from optimum magnetic separation were then ground to -212 µm, and deslimed to -38 µm. -212+38 µm of optimum magnetic concentrate were applied to calcite, mica, and metal oxide flotation experiments, respectively.

2. Materials and methods

2.1. Materials

Nepheline syenite sample used in this study was obtained from Kırşehir Region/Turkey. The chemical analyses of the sample were done with Panalytical Axios Max XRF device, and Panalytical X’pert Pro MPD diffractometer was used for mineralogical analyses.
The mineralogical analysis is seen in Fig. 1, and muscovite, albite, microcline, montmorillonite quartz was determined in the sample. The chemical composition of the sample is presented in the Table 1, and Fe$_2$O$_3$, TiO$_2$, CaO, and MgO contents were found as 0.93%, 0.17%, 2.26%, 0.23%, respectively as impurities.

2.2. Methods

The nepheline syenite sample was firstly crushed to -10 mm with a laboratory jaw crusher, then classified into two groups of particle sizes as -10+2 mm and -2 mm. -10+2 mm sized group seemed to have lighter color compared to -2 mm. Therefore, -10+2 mm was crushed to -2 mm, and named as -10+2 mm.

As a feed material for magnetic separation -10+2 mm (namely) and -2 mm particle sizes of nepheline syenite were sieved to -2+1, -1+0.425, -0.425+0.250, and -0.250+0.150 mm ranges, and high intensity roller magnetic separator that has 18000 Gauss (for 0.5 mm belt thickness) magnetic induction property was used for magnetic separation experiments. -2+1, -1+0.425, -0.425+0.250, and -0.250+0.150 mm size groups of nepheline syenite were fed to high intensity dry magnetic separator, and Fe$_2$O$_3$+TiO$_2$ contents of concentrates were analyzed by changing belt speeds (100, 200, 300, and 400 rpm). The chemical analysis of -2+1, -1+0.425, -0.425+0.250, and -0.250+0.150 mm feed sizes for -10+2 (namely), and -2 mm crushed materials are given in Tables 2 and 3, respectively.

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<th>Size interval (mm)</th>
<th>Amount (%)</th>
<th>LOI</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>TiO$_2$</th>
<th>Fe$_2$O$_3$</th>
<th>CaO</th>
<th>MgO</th>
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<td>-1+0.425</td>
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<td>-0.250+0.150</td>
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<th>Amount (%)</th>
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<th>Al$_2$O$_3$</th>
<th>TiO$_2$</th>
<th>Fe$_2$O$_3$</th>
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<tr>
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<td>0.3</td>
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<td>16.9</td>
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<tr>
<td>-0.250+0.150</td>
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<td>56.2</td>
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Table 1. Chemical composition of nepheline syenite sample

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<tr>
<th></th>
<th>LOI</th>
<th>SiO$_2$</th>
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<th>TiO$_2$</th>
<th>Fe$_2$O$_3$</th>
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<th>MgO</th>
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<td>0.23</td>
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*LOI: loss on ignition

Table 2. Chemical analysis of -10+2 mm feed materials

Table 3. Chemical analysis of -2 mm feed materials

Flotation tests were carried out with three steps using a laboratory type Denver flotation machine which has 1.5 dm$^3$ of cell capacity, and Iso Laborgerate GmbH pH meter was used to measure the pH values. Firstly, calcite minerals, secondly mica minerals, and lastly metal oxide minerals were aimed to separate with the flotation experiments. 5 min of conditioning time, 3 min of collector mixing, and 2 min of froth collecting were applied. The flotation experiments were performed at the same impeller.
speed of 1100 rpm, and pulp in solid ratio was kept as 15%. Sulphuric acid and caustic were used as pH modifiers.

The optimum concentrates from the magnetic separation experiments were used for the calcite, mica, and metal oxide mineral flotation. -212+38 µm ground and sieved material was used for the flotation experiments. R801 and R825 (CYTEC) as sulphonate type and DER NA7 (DERBOTEKS) as fatty acid type collector were used for the calcite mineral flotation in acidic, natural, and alkaline pH. In the second stage, mica mineral flotation experiments were performed with amine type collectors named as Custamine 9024 (ARRMAZ) and A4 (DERBOTEKS) at acidic pH. In the last stage metal oxide mineral flotation experiments were done with anionic collector named Flotinor SM15 (CLARIANT) at acidic pH.

3. Results and discussion
3.1. Dry magnetic separation

Particle size of the feeding material is an important topic for separating minerals in dry magnetic method. Finer particle size affects separation efficiency negatively due to the electrical forces on the mineral by covering the surfaces. Therefore, it is important to eliminate finer particles from materials before dry magnetic beneficiation tests (Gülsoy et al., 2004; Burat et al., 2005). Magnetic, gravity, and centrifugal forces are effective for separating particles with high intensity roller magnetic separator. Particles are moved by resultant force, and left the magnetic separator. Magnetic separation is based on the magnetic intensity, grain size of particles, and belt speed. On one hand, same sized particles act to attach with the drum or leave drum from close-range under the influence of magnetic and gravity forces in case of having high intensity. On the other hand, particles leave drum with the projectile motion under the influence of just gravity force due to the low magnetic intensity. Centrifugal force will be enhanced with belt speed increment for the same magnetic intensity and particle size interval in comparison to other forces. Particles with the low magnetic intensity will move in the direction of centrifugal force and emerge from the magnetic field, thus the iron content of the nonmagnetic product will be increased. Belt speed and amount of feeding must be kept lower for maintaining particles with the low magnetic intensity in the magnetic product. In this study, it was seen that iron content of the non-magnetic product was increased when the belt speed was enhanced. The detailed parameters which affect the magnetic separation efficiency were given by Özdemir et al. (2011).

Dry magnetic separation experiments were done in order to reduce iron and titanium contents which can cause color and pinhole problems for the ceramic tile production. Two different groups were used namely -10+2 mm and -2 mm as basic feed materials. Then, these two groups were sized into -2+1, -1+0.425, -0.425+0.250, and -0.250+0.150 mm intervals by crushing and sieving. These size intervals were used as final feed materials in the dry magnetic separation tests one by one with different belt speed conditions. Fig. 2(a) and (b) shows the Fe₂O₃+TiO₂% variation for feeds and concentrates at each condition, respectively.

![Fig. 2. Fe₂O₃+TiO₂% contents of concentrates for (a)-10+2 mm and (b) -2 mm magnetic separation](image)

As seen from Fig. 2(a) and (b), Fe₂O₃+TiO₂% contents of the feed materials increased with the size reduction, and Fe₂O₃+TiO₂% contents of the concentrates could be reduced to less than 0.36% until
0.06%. It can be inferred that magnetic separation could be easy with finer liberation size. On the other hand, it is clear that slower belt speed, which gives the lower $\text{Fe}_2\text{O}_3$+$\text{TiO}_2$% contents, affects dry magnetic separation positively.

Figure 3 shows the $\text{Fe}_2\text{O}_3$+$\text{TiO}_2$ removal recovery for different particle sizes and belt speeds. $\text{Fe}_2\text{O}_3$+$\text{TiO}_2$ removal recoveries of -10+2 mm and -2 mm were 93.9% and 97.3% for -0.250+0.150 mm feeding particle size and 100 rpm belt speed, while 36.6% and 48.2% for -2+1 mm feeding particle size and 400 rpm belt speed, respectively. It is obvious that $\text{Fe}_2\text{O}_3$+$\text{TiO}_2$ removal recovery declines as the feeding particle size increases, and belt speed increment also decreases the $\text{Fe}_2\text{O}_3$+$\text{TiO}_2$ removal recovery for each particle sizes. The dry magnetic separation experiments and analysis showed that finer particle size and lower belt speed should be applied to have better nonmagnetic concentrate.

![Fig. 3. $\text{Fe}_2\text{O}_3$+$\text{TiO}_2$% removal recoveries for (a)-10+2 mm and (b)-2 mm magnetic separation](image)

The magnetic material was also analysed with XRD to figure out which minerals had magnetic material from the magnetic separation experiments. In Fig. 4, it is seen that magnetic material contained with iron-bearing mineral, syderophilite, which is a kind of biotite mineral.

![Fig. 4. Magnetic material taken from magnetic mineral separation experiments](image)

### 3.2. Flotation experiments

Flotation experiments were carried out in order to remove calcite, mica, and iron minerals from the nepheline syenite. In the first stage, it was intended to find optimum calcite mineral separation conditions. Then, in the second stage, feed material was applied to find the optimum conditions for mica removal following optimum calcite mineral separation. After obtaining ideal condition for calcite and mica flotation, it was tried to find best iron flotation conditions for the third stage.
3.2.1. Calcite mineral flotation

Calcite as a calcium mineral is decomposed by thermal treatment with degasification. Decomposition temperature of the calcite increases with coarser grain size (Suleiman et al., 2013). This situation causes pinhole problems on the ceramic tile surface depends on the glaze melting conditions (Dondi et al., 2014). In literature, it was found that fatty acids and sulphonate type collectors were efficient in the flotation of calcite (Ren et al., 2017). Feldspathic mineral beneficiation was applied respectively mica, heavy mineral, quartz-feldspar separation in acidic atmosphere with the flotation method as known from literature. However, it was observed from our preliminary laboratory experiments that there were some difficulties to apply conventional methods with the proper order for feldspar flotation. It was determined that nepheline syenite sample contained high amount of CaO from the chemical analysis. At the same time, it was difficult to adjust pH values to the acidic atmosphere (pH: 3.5-4), and thus acid consumption increased excessively. On the other hand, collector consumption also increased in the mica flotation after pH adjustment. Taking into account all of these negative factors, it was decided to remove calcite with flotation method, and one more level was added to conventional feldspar flotation. It was indicated that calcium carbonate dissolved in the acidic compounds, and the pH of the atmosphere was neutralized (Huminicki and Rimstidt, 2008; Bouchelaghem, 2010). Therefore, it was aimed to determine the acid consumption of CaCO₃ in the nepheline syenite pulp by measuring pH as a function of time. Therefore, pH values were adjusted to 1, 2, and 3 by adding H₂SO₄ to distilled water, after pH adjustment -150+38 µm sized material was used as the feed material. The feed mineral was floated for the specified collector types and dosages at pH 9.5, 7.9 (natural pH), and 4.5, and the sink mineral was taken as a concentrate. In Fig. 6(a), (b), and (c), CaO% content variations are shown for each condition.

Table 4 presents CaO% removal recoveries of calcite mineral flotation at different pH values, collector and dosage conditions. 52.2% CaO content of tailing was removed with highest recovery (83.5%) by using 500 g/Mg of DER NA7 at natural pH: 7.9. On the other hand, CaO removal recovery was very low at acidic pH compared to the natural pH with DER NA7, R825, and R801 collectors. The reason for that was to dissolution of calcite in the acid while decreasing pH to 4.5.

It was found that CaO content of the floated mineral increased while that of sink minerals decreased with the flotation experiments. However, optimum result was gained with DER NA7 as fatty acid compared to the sulphonate type collectors. The optimum values were achieved with 500 g/Mg DER NA7 at natural pH 7.9, and, 4.4% of the total nepheline syenite ore was floated as the tailing product with optimum calcite flotation conditions. It was seemed to have good results with acidic pH values but the acid consumption increased excessively due to the calcite dissolution in the H₂SO₄. Thus, the acid

![Graph](https://via.placeholder.com/150)

**Fig. 5.** pH variation by time based on the acid consumption of syenite
consumption increased, and CaO content of the concentrate was lower. Calcite was found as CaO mineral from mineralogical analyses that was taken floated mineral in the conditions of 500 g/Mg DER NA7 at natural pH (Fig. 7).

![Fig. 6. CaO% change for collector types and amounts at different pH values (a) pH: 9.5 (basic), (b) 7.9 (natural), (c) 4.5 (acidic)](image)

**Table 4. CaO% removal recoveries for calcite mineral flotation experiments**

<table>
<thead>
<tr>
<th>CONDITIONS</th>
<th>Acidic (pH: 4.5)</th>
<th>Natural (pH: 7.9)</th>
<th>Alkaline (pH: 9.5)</th>
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<tr>
<td></td>
<td>1000 g/Mg</td>
<td>2000 g/Mg</td>
<td>500 g/Mg</td>
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<tr>
<td>R825</td>
<td>9.3 9.9</td>
<td>14.2</td>
<td>26.9 71.8</td>
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<td>R801</td>
<td>4.7 5.7</td>
<td>18.8</td>
<td>12.7 30.5</td>
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<td>DER NA7</td>
<td>15.8 19.8</td>
<td>14.0</td>
<td>83.5 86.4</td>
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</table>

3.2.2. Mica mineral flotation

As a result of laminar shape, mica minerals may not be ground enough, and passed through sieve surface without any dimensional control. Mica minerals like muscovite and biotite can cause surface defects and color problems in the ceramic production because of coarser grain size and iron content. In literature, acidic anionic and alkaline anionic/cationic methods are used for separating mica minerals (Browning, 1973). Biotite minerals are pushed down to flotation cell base in the alkaline anionic/cationic method; herewith it was preferred acidic anionic method in order to take biotite minerals with floated material.

It is known from literature that albite, microcline, quartz, and muscovite minerals have 20.35%, 18.32%, 0%, and 38.36% Al₂O₃ content, respectively. Therefore, mica removal from the material was investigated by means of Al₂O₃% change and mineralogical analysis. In the flotation test, it was aimed to find optimum collector dosage conditions at acidic pH: 3.1 after the feed material was treated with 500 g/Mg DER NA7 for calcite separation, and the results are shown in Fig. 8. The results in Fig. 8 indicated that Custamine 9024 and A4 collectors have similar Al₂O₃% and Na₂O+K₂O% change. The Al₂O₃ content of feed material to mica separation was 19.77%. Al₂O₃ values increased to 23.07% and
23.27% until 500 g/Mg dosage for Custamine 9024 and A4, respectively, then was diminished. It is clear that feldspar minerals were taken with mica minerals in the floated material after 500 g/Mg collector dosage. Thus, it is determined 500 g/Mg Custamine 9024 or A4 collector dosage at acidic pH: 3.1 for mica mineral flotation.

![Fig. 7. XRD results of floated mineral in the conditions of 500 g/Mg DER NA7 at natural pH](image)

![Fig. 8. Results for mica mineral flotation with Custamine 9024 and A4 at acidic pH: 3.1 following calcite removal with flotation](image)

Table 5. Results for iron mineral flotation

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<tr>
<th>Collector</th>
<th>Dosage (g/Mg)</th>
<th>pH</th>
<th>Amount (%)</th>
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<th>SiO₂</th>
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<td>0.2</td>
<td>0.6</td>
<td>0.1</td>
<td>4.7</td>
<td>8.7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
3.2.3. Iron mineral flotation

Anionic type Flotinor SM15 collector was used at acidic pH for further iron-bearing mineral treatment after separating calcite and mica mineral at the optimum conditions. As seen from Table 5, Fe₂O₃% content was reduced from 0.2% to 0.1%.

3.3. Total Mineral Beneficiation Experiments

In consequently, the raw material was applied to mineral beneficiation tests with the best conditions that were gained from the magnetic separation and flotation experiments. Firstly, -10 mm raw material was crushed and sieved to -2+0.150 mm, and given to magnetic separation tests. The nonmagnetic material of this part then was ground and sieved to -212+38 μm for fractional flotation procedure. Formerly, -212+38 μm nonmagnetic material was applied to calcite, mica, and iron mineral flotation, respectively. The concentrates obtained from the experiments were given to quantitative mineralogical analysis to see the mineral variation, and the results are shown in Fig. 9. It is seen from Fig. 9 that the raw nepheline syenite’s total albite and microcline mineral content increased from 78% to 97%, and mica impurities as muscovite and biotite content decreased from 12% to 2%.

![Fig. 9. Results for total mineral beneficiation experiments mineralogical results](image)

In Fig. 10, a mineral beneficiation flowsheet for nepheline syenite ore was proposed. In the flowsheet, the dry magnetic separation should be used to eliminate iron bearing minerals at coarser grain sizes. However, it is known from literature that wet high intensity magnetic separation must be used to remove gangue minerals from the nepheline syenite ore at finer particle sizes, and the Fe₂O₃+TiO₂ contents and removal recoveries showed that wet magnetic method should be suggested to separate such impurities. Then, the nonmagnetic product can be recovered using flotation methods. In the case of calcite, it should be floated at natural or alkaline pH with fatty acids before the mica or iron bearing mineral flotations which are done at acidic pH for nepheline syenite. After the calcite removal, the mica mineral flotation should be applied at acidic pH: 3-3.5 with amine collector (Custamine 9024 or A4), then iron oxide minerals can be floated at acidic pH: 3-3.5 again with a phosphoric ester collector (Flotinor SM15).

4. Conclusions

In this study, it was aimed to produce suitable raw material for ceramic industry by promoting alkaline content of the Kırşehir nepheline syenite ore, and removing impurities such as calcite, quartz, mica, and iron-bearing minerals.

Primarily, iron-bearing minerals which have negative impacts on ceramic bodies were removed with rare earth type high intensity roller dry magnetic separator, and Fe₂O₃+TiO₂ content was reduced to 0.06% from 2.50%. It was observed from dry magnetic separation that the Fe₂O₃+TiO₂ removal recovery was affected negatively as the feeding particle size and belt speed increased. High content of calcite in the floor tile body composition causes pinhole defect on the ceramic tile surface. Meanwhile, it was seen
from the flotation experiments that mica minerals affected the flotation process negatively due to the high amine type collector consumption in the presence of calcite. Apart from that mica minerals were floated in the acidic pH levels, and it was observed that the acid consumption increased as a result of high calcite content of the ore. In view of the environmental impacts, separating calcite in the first stage of flotation process at natural pH also affects the subsequent stages positively. Calcite flotation was done at three different pH values and with three different collectors, and it was obtained from chemical analysis that CaO content of the material was reduced from 3.1% to 0.4% with 83.5% calcite removal recovery as the optimum conditions of 500 g/Mg amount DER NA7 (fatty acid collector) and at natural pH: 7.9. In the second stage of flotation, mica minerals were tried to float with two different types of amine type collectors at acidic pH: 3.3 and, Na₂O+K₂O alkaline content was enhanced effectively from 13.4% to 13.9% with 500 g/Mg of both collectors (Custamine 9024 and A4). In the last flotation stage, Fe₂O₃ content of the ore was reduced to 0.1% from 0.2% with 300 g/Mg of Flotino SM (anionic collector) at acidic pH: 3.4.

Raw nepheline syenite ore was composed of 21.54% Al₂O₃, 2.26% CaO, 1.10% Fe₂O₃+TiO₂, 10.78% Na₂O+K₂O chemically, after mineral beneficiation experiments the concentrate was gained with 19.28% Al₂O₃, 0.09% CaO, 0.37% Fe₂O₃+TiO₂, 14.01% Na₂O+K₂O content. It was obtained from mineralogical analysis that calcite, quartz, syderophyllite, muscovite etc. were separated from the ore with mineral beneficiation experiments, and a clean concentrate was gained with 50% albite and 47% microcline content.

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