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Characterization and production of Turkish nepheline syenites for industrial applications

Murat Olgaç Kangal, Gülay Bulut, Zeynep Yeşilyurt, Hüseyin Baştürkcü, Fırat Burat

Istanbul Technical University, Faculty of Mines, Mineral Processing Engineering Department, Maslak, 34469, Istanbul, Turkey

Corresponding author: gbulut@itu.edu.tr (Gülay Bulut)

Abstract: Nepheline syenite, which is a silica-poor crystalline rock, competes with feldspar in applications such as glass, ceramic filler, and pigment industries. While its appearance is medium coarse granular like granite, main differences between them are nepheline syenite is silica poor, and contains high alumina and alkali content. Turkey has considerable nepheline syenite deposits in Kırşehir Region, and they contain 1.3% Fe_2O_3 on average, thus cannot be used unless beneficiated by flotation or/and magnetic separation. In this paper, physical and physicochemical experiments were carried out to improve the quality of nepheline syenite ore. After determinations of chemical, mineralogical, and properties of the sample, three different technologies such as flotation, magnetic separation, and leaching were applied on the ore sample, separately and combined. The obtained results showed that the magnetic separation alone could not produce a nepheline syenite concentrate assaying 0.45% Fe_2O_3. It was not also possible to obtain a nepheline concentrate less than 0.25% Fe_2O_3 adapting optimum flotation conditions. The best results were found in combination of the high intensity wet magnetic separation and flotation between -212+63 µm particle size, and a final concentrate with 0.20% Fe_2O_3 and 0.01% TiO_2 was obtained. The leaching experiments were conducted to further decrease Fe_2O_3 content. Finally, a saleable nepheline syenite concentrate for tile, sanitary ware, electrode, glass, and fiberglass industries was obtained with 6.63% K_2O, 9.02% Na_2O, 0.15% Fe_2O_3, and 0.01% TiO_2 by the weight of 63.9% at the end of the experiments.

Keywords: Nepheline syenite, flotation, leaching, glass, ceramics

1. Introduction

Nepheline syenite is a feldspathic rock, and contains major minerals like nepheline, microcline, albite, and minor minerals like mica, hornblende, and magnetite (Bolger, 1995). In glass and ceramics industries, nepheline syenite, like feldspar, provides alkalis that act as a flux to lower the melting temperature of a glass or ceramic mixture, prompting faster melting and fuel savings, lower viscosity, faster growth of glassy phase and high reactivity against quartz. In glass, nepheline syenite is a source of alumina, which gives increased resistance to breaking, increased thermal endurance, and improved chemical durability (Özpeker, 1999; Tait et al., 2003; Esposito et al., 2005).

Main nepheline syenite reserves are located in Russia, Norway, Canada, and Turkey. Reserves of the rock in Turkey are recently estimated over 1 Pg (pentagrams or billion tons) (Gülsoy et al., 1994). Canada and Norway produce nepheline syenite for glass and ceramic use. An estimated 70% of the output goes into glass, especially container glass and glass fiber. About 15% is used in ceramic applications, and 15% in pigments and fillers. Nepheline syenite used for glassmaking should be a sandy sized product falling within the range of 420 to 75 µm. Its iron content should not exceed 0.1% Fe_2O_3 while alumina and alkali should be as high as possible, typically at least 23 and 14%, respectively. On the other hand, nepheline syenite for the ceramics industry should be finely ground, typically into products of 75, 53, and 38 µm (Harben, 1995).

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In order to produce glass and ceramic grade nepheline syenite, the ore is first crushed and milled then enters a separation stage. Usually nepheline syenite ores contain some magnetic minerals (magnetite and hematite) which darken the color of the final powder and manufactured products. Therefore, magnetic separation is used to remove such particles (Burat et al., 2006).

In the other studies conducted on Egyptian nepheline syenites upgrading the ore applying a single technique either on dry basis (magnetic separation) or wet basis (flotation) could never lead to a marketable concentrate product, however cleaning of the magnetic separation concentrate by flotation resulted in a final concentrate of high quality regarding its iron, alumina, and alkali contents (0.09% Fe_2O_3, 23.58% Al_2O_3, 16.47% Na_2O + K_2O) (Ahmed, 2011; Abouzeid and Negm, 2014). In the so called Russian Process, nepheline syenite is used to produce alumina, Portland cement, and other chemical products by sintering. Flotation methods are used to remove the nepheline syenite from the apatite tailings. The nepheline syenite is calcined with limestone, producing a mixture of $CaSiO_3$ and $Na_2O.Al_2O_3$. Leaching by caustic soda and treatment with CO_2 produces alumina and by-products (El-Roudi and Ismail, 1993).

Kırşehir, Turkey nepheline syenites represents one of the largest and unaltered alkaline intrusive body in Central Anatolia having Na_2O/K_2O ratio between 0.89-2.66% which makes them very suitable for ceramic and glass industries (Deniz and Kadıoğlu, 2017). Burat et al. (2006) investigated the beneficiation possibilities of nepheline syenite ore obtained from Orhaneli Province, Bursa, Turkey using the magnetic separation followed by flotation method. They succeeded to produce a concentrate meeting the specification for glass industry with 0.08% Fe_2O_3 and 0.09% TiO_2 contents. Also another study was carried out by Demir et al. (2001 and 2003). In their study, the flotation separation of Na-Feldspar and K-Feldspar from nepheline syenite with monovalent salts and bivalent salts were investigated, and they obtained the best results using NaCl salts with the floatability difference of 72% between Na-Feldspar and K-Feldspar.

In this study, after determinations of the chemical, mineralogical, physical properties of the ore syenite, the magnetic separation, flotation, and leaching methods were applied separately and combined on the nepheline syenite to produce an alternative raw material to feldspar using tile, sanitary ware, electrode, glass, and fiberglass industries.

2. Materials and methods

2.1. Materials

The representative nepheline syenite ore sample was obtained from the Buzlukdagi Region, Kırşehir, Turkey. The chemical contents of the sample were determined by the Inductively Coupled Plasma (ICP) method, and the results are presented in Table 1.

Table 1. Chemical composition of representative ore sample

Compound	Content, %	Compound	Content, %
SiO_2	56.85	CaO	1.59
Al_2O_3	23.31	MgO	0.09
Na2O	9.46	MnO	0.07
K_2O	6.43	TiO2	0.05
Fe_2O_3	1.29	LOI	0.96

X-ray diffraction analysis was made with Cu X-ray sourced Panalytical X’Pert Pro diffractometry. For mineral characterization, PDF4/Minerals ICDD software was used. Crystals’ mineral phase ratio was determined by the Rietveld method. Modal mineralogical analysis was made with FEI MLA 650F device and back-scattered electron (BSE) images were taken by Bruker 5010 SDD device.

The XRD patterns of the sample seen in Fig. 1 showed that nepheline, albite and microcline minerals were observed with strong peaks. In addition, fluorite, muscovite, and corundum were traced containing weaker peaks.

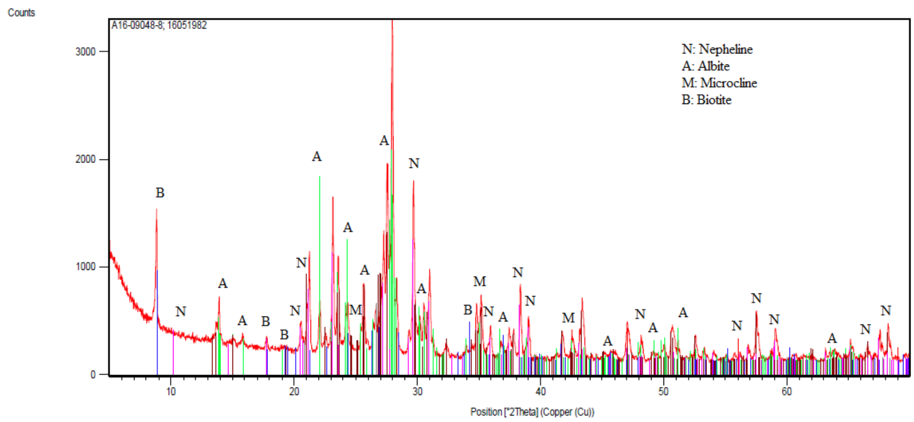

Fig. 1. XRD patterns of the nepheline syenite sample

Fig. 2 shows the results of the modal mineralogical analysis in which particle counting and liberation tests are performed. According to the results, 90% of the nepheline syenite are liberated below 200 µm. BSE image in Fig. 3 clearly shows nepheline (feldspathoid), albite, thorianite, and zircon surfaces. Table 2 presents mineral types and ratios generally. The sample contains high amount of nepheline (feldspathoid) (35.42%), albite (28.85%), and potassium feldspar (29.72%) as expected. Minor components have been detected as muscovite, biotite, iron bearing minerals, and clays.

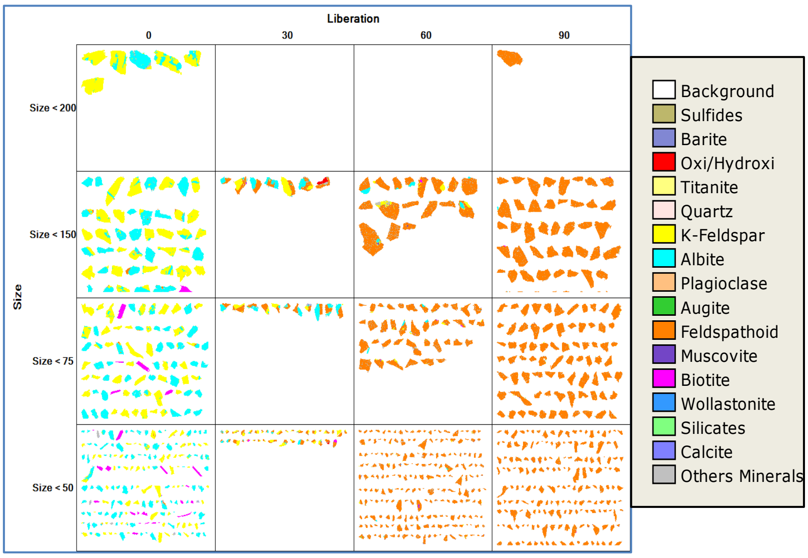

Fig. 2. Modal mineralogical analysis of the nepheline syenite sample by liberation

In order to determine the particle size distribution of the representative ore sample, wet sieve analyse was conducted. According to results, d_{80} and d_{50} sizes of the ore were found as 1.5 mm and 0.5 mm, respectively. In order to prepare the sample for the proper size for magnetic separation and flotation experiments, the ore with the maximum size of 4 mm was wet ground below 300, 212, and 106 µm. 26×20 cm alumina ball porcelain mill was used for the grinding tests.

Dodecylamine hydrochloride (DAH) from Acros Organics (Geel, Belgium), derivatives of petroleum sulfonates namely R801-R825 from Cytec (Woodland Park, NJ, USA), Derna 7, A4 from Dermoteks (Turkey), SM15 and SM35 from Clariant (Muttenz, Switzerland) were used as flotation collectors. Sodium silicate (Na_2SiO_3) and methyl isobutyl carbinol (MIBC, DOW Chemical Company) was used as a depressant and a frother, respectively. Technical grade sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH) were used as pH adjustment. In the case of the leaching experiments, the effect of oxalic acid and sulfuric acid (H_2SO_4) were investigated.

Table 2. Modal mineralogical analysis of the nepheline syenite sample by mineral contents

Components	Mineral	Content, %
Sulfides/Sulphates	Sphalerite	0.02
	Galena	0.03
Oxi/Hydroxi	Magnetite/Hematite	0.13
	Goethite	0.09
Silicates	Quartz	0.08
	K-Feldspar	29.72
	Albite	28.85
	Plagioclase	0.05
	Feldspathoid	35.42
	Muscovite	0.34
	Biotite	2.71
	Wollastonite	0.04
	Si-Al Clays	0.43
Others		2.10

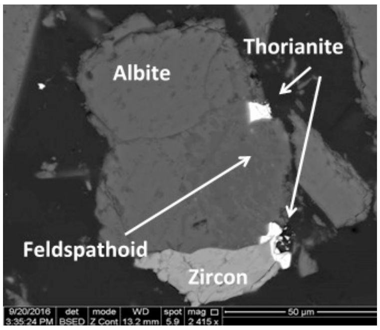

Fig. 3. BSE images of the nepheline syenite sample Albite: $NaAlSi_3O_8$, Feldspathoid: $(Na,K)AlSiO_4$, Thorianite:ThO_2; Zircon: ZrSiO4

2.2. Methods

By adapting direct flotation studies, the effects of feed size, reagent types, and amounts was investigated in order to find the maximum removal of colored impurities from nepheline syenite ore. As a second group testing, the original ore was subjected to magnetic separation using a high intensity wet type Jones magnetic separator. Finally, the magnetic separation followed by flotation tests was performed successively.

The flotation experiments were conducted in a self-aerated Denver flotation machine equipped with a 1.5 dm^3 cell at a constant impeller speed of 1300 rpm. The percentage of the solids was kept constant as 20 wt.%. As known from literature, in feldspar flotation, the gangue minerals associated with feldspar like mica and other iron bearing minerals are floated initially by the addition of suitable amine at acidic pH range of 2.5-3.5. After the flotation of mica, other iron-bearing oxide minerals are floated using sulfonate type collectors at acidic pH range as 3-4 (Kangal et al., 2017).

By adding H_2SO_4 to the medium pH was successfully adjusted to 2.5 for mica flotation and 3.5 for oxide flotation. Based on the results of previous studies on the flotation of similar materials (Kangal et al., 2017), DAH for mica flotation, R801 and R825 for oxide minerals were selected for removing of colored impurities from nepheline syenite ore. Following the aforementioned stages and conditions for each type of mineral, the effects of different parameters on flotation process as particle size, reagent type on the flotation separation were investigated.

DP40 type high intensity wet type Jones magnetic separator was used in magnetic separation experiments. Jones magnetic separator generates a magnetic field at 6.8 amps at around 16,000 Gauss (1.6 Tesla).

3. Results and discussion

3.1. Effect of particle size

In the flotation experiments, the samples ground below 300, 212, and 106 µm were separated from 63 µm and 38 µm fractions, respectively. The general flowsheet of the experiments is seen in Fig. 4, the experimental conditions and results are presented in Table 3 and Fig. 5, respectively.

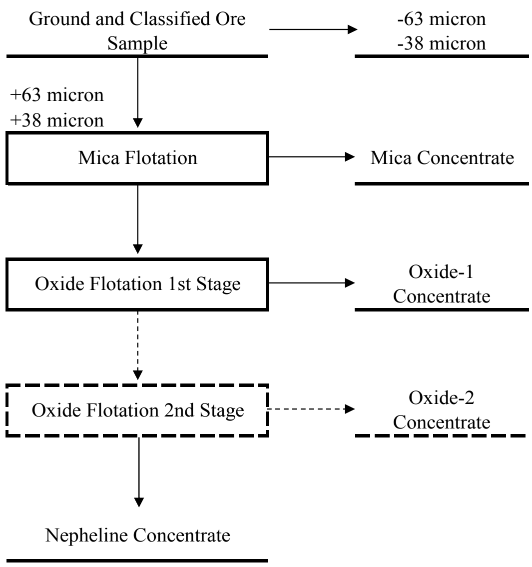

Fig. 4. General flowsheet for the flotation experiments

Table 3. The experimental conditions for determination of optimum particle size

Mica Circuit		Oxide Circuit	
pH	2.5	**pH**	3.5
DAHC	100+100 g/Mg	**R801**	2000+2000 g/Mg
MIBC	30+0 g/Mg	**R825**	2000+2000 g/Mg
Conditioning time	5+3 min	**MIBC**	0
Flotation time	1+1.5 min	**Conditioning time**	5+3 min
		Flotation time	2+2 min

As can be seen from Fig. 5, at -106+63 µm size fraction, a nepheline syenite concentrate with 0.30% Fe_2O_3 content was obtained with 16.2 wt%, while the concentrates with 0.20% Fe_2O_3 and 0.40% Fe_2O_3 contents were produced for -212+63 µm and -300+63 µm fractions, respectively. Fe_2O_3 contents of concentrates at over 38 µm were found higher compared to over 63 µm fractions. Generally, iron containing minerals are accumulated in finer sizes. Therefore, obtaining a concentrate with low iron content becomes harder. It is also clear from the tests results that Fe_2O_3 content was very high because of the insufficient liberation of particles below 300 µm. In the flotation experiments carried out in the range of -106+38 µm, an increase in Fe_2O_3 content was observed. This can be explained by the increase in the slime fraction and the deterioration of the flotation in the fine size groups.

As it is well known that the particle size and the liberation degree play an important role in flotation, as demonstrated by many flotation studies of various types of ores (Gaudin et al., 1931; Trahar, 1981; Kangal and Güney, 2002; Sekulic et al., 2004; Gülsoy et al., 2005; Miettinen et al., 2010 Kangal et al., 2017). The recovery of fine size particles (<20 µm) in flotation is mainly low due to their low collision efficiency (EC) with bubbles. The EC values can be increased by reducing the bubble size and by enlarging the sizes of the fine particles (Miettinen et al., 2010). As can be seen from the test results, the proper conditions for the flotation was not obtained due to the insufficient liberation in large particle sizes and because fine particles did not sufficiently interact with the bubbles. For these reasons all subsequent experiments were carried out with samples below 212 µm.

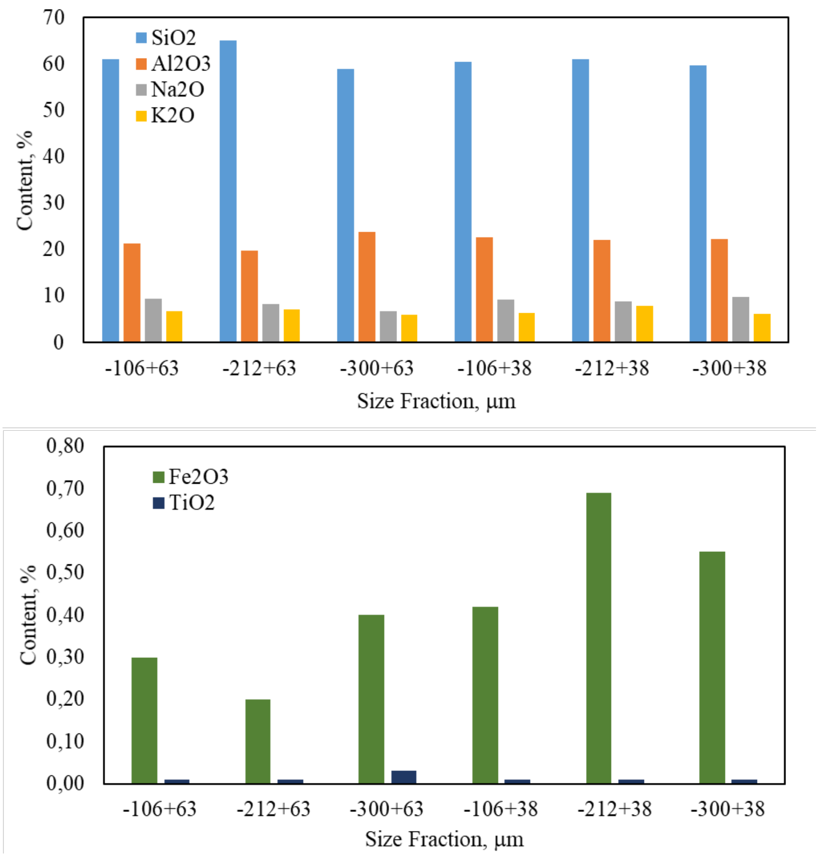

Fig. 5. Effect of particle size on flotation

3.2. Effect of reagent type and amount

The effects of reagent type and amount for selective flotation of nepheline ore were investigated using collectors such as DAH, Derna-7, SM-15+SM-35, and R-801+R-825 for the fractions of -212+63 and -212+38 µm. The flotation conditions are presented in Table 5, and the results are shown in Fig. 6.

After the flotation separation test in which particle size, reagent type, and amount were investigated the Fe_2O_3 content of the feed material from about 1.30% was successfully reduced to 0.20%. All TiO_2 contents were reduced under 0.01%. Na_2O content was increased to 10.50% while K_2O content was 6.50%. In order to produce a concentrate with lower iron content and decrease the amount of reagent for subsequent flotation tests highly intensive Jones magnetic separation was conducted at further tests.

3.3. Magnetic separation followed by flotation

To reduce the content of iron oxides of nepheline syenite, the material ground below 300 µm and 212 µm was sieved from 38 µm, and deslimed material was subjected to high intensity Jones magnetic separation. In the experiments, the effects of current intensities of 0.5, 3, and 6.8 Amp. were tested (Fig. 7). The non-magnetic product obtained from first low intensity magnetic separation stage was fed again by increasing current intensity. The final non-magnetic product was then subjected to the flotation. The results of magnetic separation tests are given in Fig. 8.

As clearly seen in Fig. 8 that the iron and titanium oxide contents of non-magnetic products are very similar. The direct flotation tests indicated that -212+38 µm particle size was more efficient for the separation. Therefore, the removal of impurities was further investigated using the magnetic separation followed by flotation using this fraction by keeping optimum flotation conditions (Tian et al., 2017; Li and Gao, 2017; Gao et al., 2018). The conditions of the flotation experiments are presented in Table 8, and the results are shown in Fig. 9.

As can be seen from Fig. 9, non-magnetic product having 0.40% Fe_2O_3 and 0.02% TiO_2 contents was reduced to 0.25% and 0.01% as a result of flotation separation. In terms of Fe_2O_3 and TiO_2 contents and recoveries the best results were obtained when using Derna-7 alone. As a result of magnetic separation followed by flotation process, lower iron and titanium containing concentrates were produced and the flotation regents were consumed in a smaller amount than the direct flotation enrichment. As compared to direct flotation test results, a nepheline syenite concentrate with higher iron content (0.25%Fe_2O_3) was produced.

Table 5. Flotation conditions for the effect of reagent type and amount

	1st Group		2nd Group
Size fraction	-212+38 μm	**Size fraction**	-212+63 μm
	Mica Circuit		**Mica Circuit**
pH	2.5	**pH**	2.5
DAH	100+100+100 g/Mg	**DAH**	100+100+100 g/Mg
MIBC	30+0+0 g/ Mg	**MIBC**	30+0+0 g/Mg
Conditioning time	5+3+3 min	**Conditioning time**	5+3+3 min
Flotation time	1+1.5+2 min	**Flotation time**	1+1.5+2 min
	Oxide Circuit		**Oxide Circuit**
pH	3.5	**pH**	3.5
R801	500+500 g/Mg	**R801**	500+500 g/Mg
R825	500+500 g/Mg	**R825**	500+500 g/Mg
MIBC	0	**MIBC**	0
Conditioning time	5+3 min	**Conditioning time**	5+3 min
Flotation time	2+2 min	**Flotation time**	2+2 min
	3rd Group		**4th Group**
Size fraction	-212+63 μm	**Size fraction**	-212+63 μm
	Mica Circuit		**Mica Circuit**
pH	2.5	**pH**	2.5
DAH	100+100+100 g/Mg	**DAH**	100+100+100 g/Mg
MIBC	30+0+0 g/Mg	**MIBC**	30+0+0 g/Mg
Conditioning time	5+3+3 min	**Conditioning time**	5+3+3 min
Flotation time	1+1.5+2 min	**Flotation time**	1+1.5+2 min
	Oxide Circuit		**Oxide Circuit**
pH	9.5	**pH**	3.5
DERNA7	500+500+500 g/Mg	**SM15**	250+250 g/Mg
MIBC	0+30+0 g/Mg	**SM35**	125+125 g/Mg
Conditioning time	5+5+5 min	**Conditioning time**	3+2.5 min
Flotation time	2+1+1 min	**Flotation time**	1+1.5 min
	5th Group		**6th Group**
Size fraction	-212+38 μm	**Size fraction**	-212+63 μm
	Mica+Oxide Circuit		**Mica+Oxide Circuit**
pH	9.5	**pH**	9.5
DERNA7	500+500+500+500+500 g/Mg	**DERNA7**	500+500+500+500+500 g/Mg
MIBC	0+0+0+0+30 g/Mg	**MIBC**	0+0+0+0+30 g/Mg
Conditioning time	5+3+2+2+2 min	**Conditioning time**	5+3+2+2+2 min
Flotation time	1+1.5+1+1+1 min	**Flotation time**	1+1.5+1+1+1 min

3.4. Leaching experiments

For the production of high-quality ceramics/glass material, iron components in feldspar, clay or silicate ores are required to be removed. The iron content is generally desired to be reduced below 0.1% to achieve an acceptable level of whiteness. Within the scope of chemical enrichment, the leaching tests were carried out using different types of acids. In the literature, there have been many studies conducted using inorganic and organic acids (Arslan and Bayat, 2008; Vapur et al., 2017; Santi et al., 2017; Zhang et al., 2017; Lin et al., 2018;). Sulfuric and hydrochloric acids have been tried out of inorganic acids, but most of the work has been concentrated on organic acids because of cost, pollution of product sulfate and chlorine ions, and environmental concerns for solution to be obtained after leaching. The use of organic acid has a higher rate of dissolution and higher yield of iron and allows working at a wide pH range. Until now, although acetic, formic, citric and ascorbic acid have been tried in many scientific studies of organic acids, the most efficient results were achieved using oxalic acid for iron oxide removal (Gupta and Mukherjee, 1990; Taxiarchou et al., 1997; Akçıl et al., 2007; Lee et al., 2007; Lazo et al., 2017; Adebisi et al., 2018).

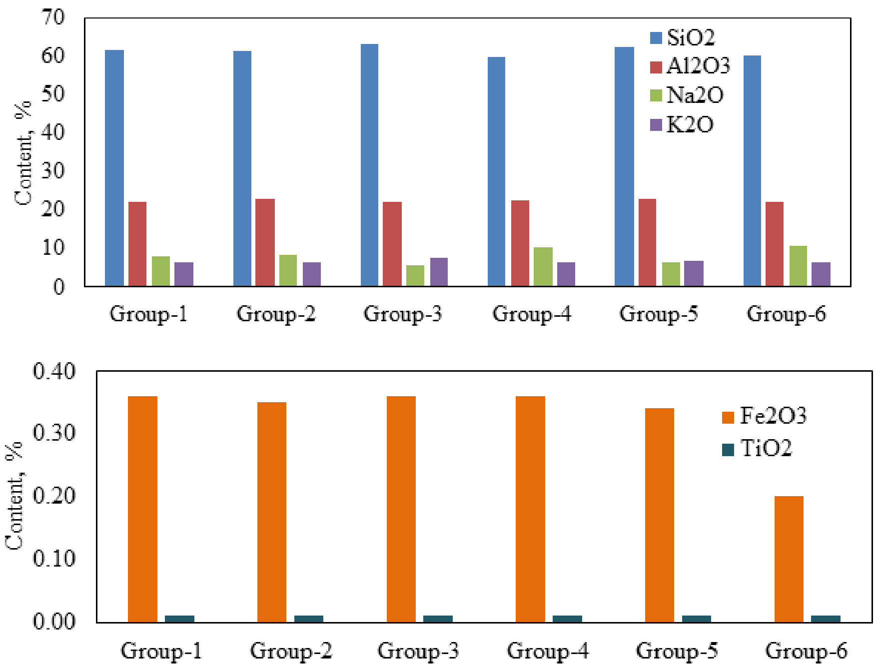

Fig. 6. Effect of reagent type and amount on flotation

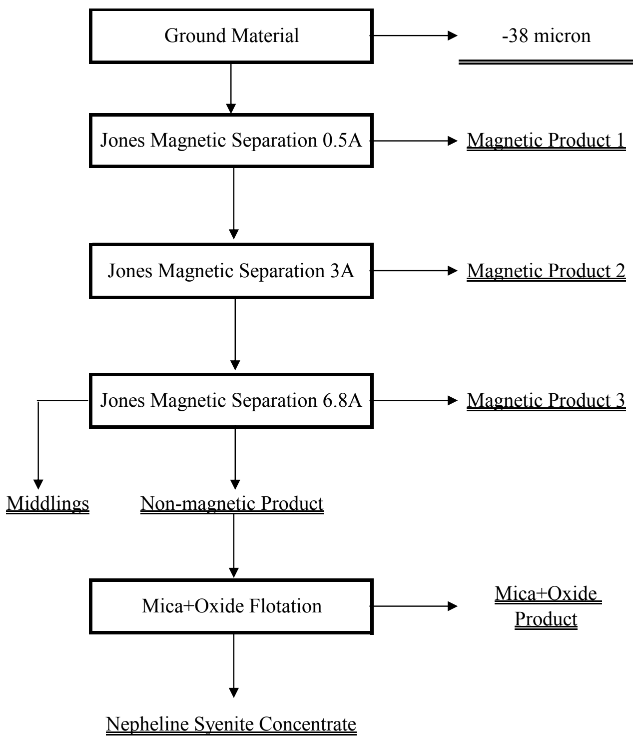

Fig. 7. Flowsheet of the flotation experiments following Jones magnetic separation

Fig. 8. High intensity Jones magnetic separation results in different size fractions

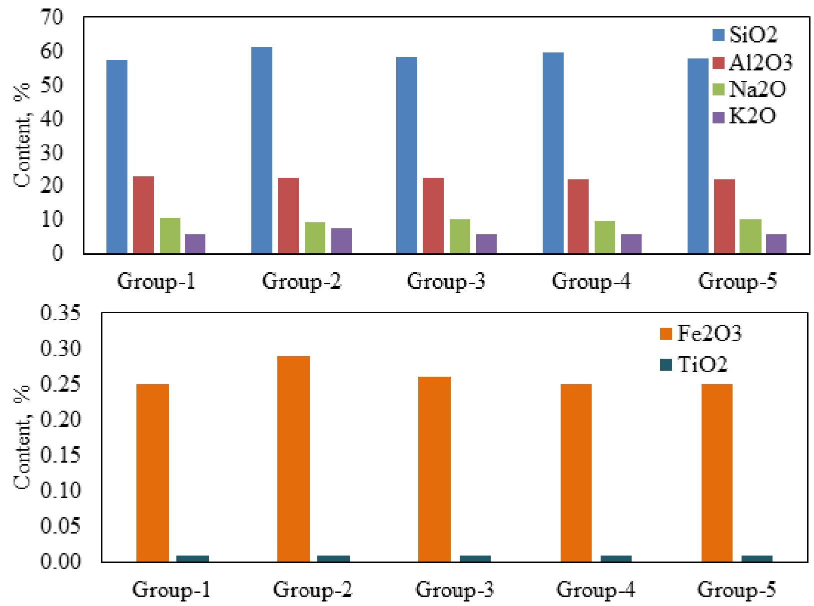

Fig. 9. Results for magnetic separation followed by flotation tests

Table 8. Experiment conditions for magnetic separation followed by flotation tests

1st Group		2nd Group		3rd Group	
Mica+Oxide Circuit		Mica+Oxide Circuit		Mica+Oxide Circuit	
pH	9.5	**pH**	9.5	**pH**	9.5
DERNA7	500+500+500 g/Mg	**DERNA7**	500+500+500 g/Mg	**DERNA7**	500+500 g/Mg
MIBC	30+0+0 g/Mg	**Na_2SiO_3**	1000+0+0	**A4**	25+25 g/Mg
Conditioning time	5+1+3 min	**MIBC**	0+0+15 g/Mg	**MIBC**	30+0 g/Mg
Flotation time	1+1+2 min	**Conditioning time**	10+ 3+3 min	**Conditioning time**	5+3 min
		Flotation time	2+ 2+1 min	**Flotation time**	2+2 min
4th Group		**5th Group**			
Mica+Oxide Circuit		Mica Circuit		Oxide Circuit	
pH	9.5	**pH**	9.5	**pH**	9.5
Na_2SiO_3	1000+0	**DERNA7**	500+500+500 g/Mg	**A4**	25+25 g/Mg
DERNA7	500+500 g/Mg	**MIBC**	0+0+15 g/Mg	**MIBC**	0+0 g/Mg
A4	25+25 g/Mg	**Conditioning time**	10+ 3+3 min	**Conditioning time**	5+3 min
MIBC	30+0 g/Mg	**Flotation time**	2+ 2+1 min	**Flotation time**	2+2 min
Conditioning time	5+3 min				
Flotation time	2+2 min				

In addition to being an effective reagent in the leaching stage of oxalic acid, it also has the advantage that it does not show a risk for the contamination of the processed material. It is also possible to convert pure hematite by calcination after iron dissolved in oxalic acid precipitates in the form of iron oxalate. The mineralogical composition in the ore desired to be removed has a great influence because it affects its dissolution behavior in acidic media. As hematite slowly dissolves, iron hydroxide and oxyhydroxide compounds such as goethite and lepidocrocite dissolve more rapidly. The dissolution reaction of the iron oxides in the oxalic acid solution takes place as follows:

$$H^+ + Fe_2O_3 + 5HC_2O_4^- = 2Fe(C_2O_4)_2^{2-} + 3H_2O + 2CO_2^- \quad (1)$$

In the leaching tests following flotation and magnetic separation, it was aimed to remove the iron selectively by leaching. The effect of H_2SO_4 and oxalic acid on iron removal was investigated for varying leaching times, while the temperature (± 25°C) and solids ratio (20 wt.%) were kept constant. The experimental flowsheet is illustrated in Fig. 10, the leaching parameters applied in the experiments and the test results are given in Table 10 and 11, respectively.

When the leaching results are taken into consideration, Fe_2O_3 content of concentrate decreases to 0.15% when 0.4 mol/dm³ oxalic acid and sulfuric acid are used in 2 h the leaching time. As a result of the leaching experiments using oxalic acid, the amount of Fe_2O_3 in the nepheline syenite sample was reduced to 0.15%, giving a recovery of 44.6% of iron removal.

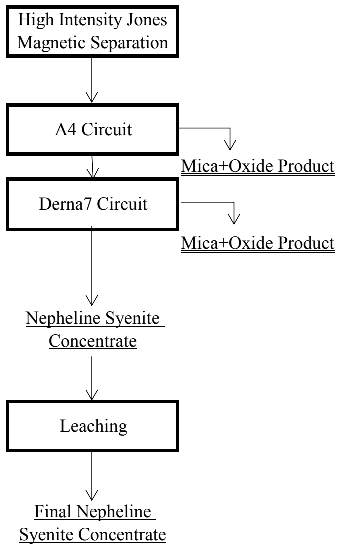

Fig. 10. Experiment flowsheet for magnetic separation and flotation followed by leaching

Table 10. Leaching experiment conditions with oxalic acid

Code	L1	L2	L3
Acid Type	Oxalic Acid	Oxalic Acid	Oxalic Acid+H_2SO_4
Acid Concentration, mol/dm³	0.4	0.8	0.4+0.4
Temperature, °C	Room Temperature (24±1)		
Time, h	2	2	2+2

Table 11. Leaching experiment results with oxalic acid

Code	Cake Amount,%	Fe_2O_3, %	Fe Removal Recovery, %	TiO_2, %	Al_2O_3, %	Na_2O ,%	K_2O, %
L1	91.6	0.18	34.1	<0.01	18.93	9.21	6.48
L2	90.8	0.17	38.3	<0.01	19.07	9.03	6.59
L3	92.3	0.15	44.6	<0.01	19.18	9.02	6.63
Feed	100.0	0.25	0.0	<0.01	22.16	10.57	6.33

4. Conclusions

Nepheline syenite is becoming increasingly important as a source of potential feldspar for our country. Kırşehir Buzlukdağı Region nepheline syenite ore was investigated in this paper to produce a high value added concentrate for many industrial purposes. Therefore, the parameters such as particle size, reagent type and dosage were investigated in direct flotation tests and then magnetic separation followed by flotation tests were performed to minimize colored impurities. Direct flotation and magnetic separation followed by flotation experiments were subjected at -212+63 µm size range and concentrates assaying 0.20% and 0.25% Fe_2O_3 with very similar iron removal rates (~80%) were successfully produced, respectively.

Considering the results of the dissolution tests carried out with oxalic acid in order to further reduce the Fe_2O_3 content, a nepheline syenite concentrate was obtained with 0.15% Fe_2O_3 content when 0.4 mol/dm^3 oxalic acid and sulfuric acid were used for 2 h leaching.

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