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Effect of calcite and mica contents in nepheline syenite samples on the ceramic body sintering behaviours and surface roughness

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Abstract: In this study, the effects of calcite and mica contents in nepheline syenite (NS) samples beneficiated by high intensity dry magnetic separation and flotation methods on ceramic bodies were investigated in detail. The NS samples were, first, sintered to observe the physical and surface roughness properties, and characterized based on the change of NS samples such as color, shrinkage, water absorption, and surface roughness after the sintering process. L-a-b color and Ra, Rz, Rt values for the NS samples were determined. The decrease in calcite and mica contents affected the surface of sintered specimens positively by reducing roughness values. Additionally, the water absorption values were found to be directly proportional to Ra values for unglazed surfaces. In conclusion, calcite and mica minerals in NS samples negatively affected surface quality by forming pinholes due to dehydroxylation and outgassing reactions in the sintering process.

Keywords: nepheline syenite, calcite and mica, ceramic, sintering, surface roughness

1. Introduction

Ceramic bodies are comprised of industrial raw materials with a combination of clays, quartz, earth, and alkaline earth minerals basically (Manfredini and Hanuskova, 2012). Clays are plastic materials that are used for giving shape and dry bending strength to the ceramic body. Quartz is not a plastic material and gives the body strength after thermal treatment. Feldspathic minerals are utilized to reduce sintering temperature and duration as a fluxing agent (Finkelnburg, 2012; Obstler, 2012).

Nepheline syenite is a light-colored, and low-free silica industrial raw material that is comprised of albite, microcline, and nepheline minerals basically (Potter, 2000). It also may contain muscovite, biotite, and magnetite in small quantities as impurities (Gulgonul et al., 2014; Deniz, and Kadıoglu, 2018). In the traditional ceramic composition, nepheline syenite is being used newly due to the wasting feldspar reserves, and high alkaline content as a melting agent. Nepheline syenite also develops mechanical characteristics such as flexural strength, water adsorption in the porcelain bodies, and microstructure is homogenized as compared to albite (Esposito et al., 2004; Salem et al., 2009). However, impurities such as iron-bearing minerals, calcite, and mica minerals that are a by-product of the source can cause quality defects in the production of ceramic. Iron-bearing minerals are the reason for the darker body color and black spot defect on the ceramic tile surface (Dondi et al., 2014; Radojevic et al., 2015). Calcite can make a pinhole defect on the ceramic tile surface while the sintering process if the glaze melts before the body, does not finish degasification (Tunalı, 2014). These impurities must be cleaned from nepheline syenite to eliminate production problems.

Magnetic separation is a mineral beneficiation method that is used to separate iron-bearing magnetic minerals particularly. The magnetic separation method can be performed in the dry or wet medium to the extent of the mineral liberation size with applicable magnetic separators. The dry magnetic separation method is applied to coarse-grained magnetic minerals for reducing the iron content of feldspar ores (Ibrahim et al., 2002; Dondi, 2003; Saklar and Oktay, 2003).

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Flotation is another mineral beneficiation method that is applied to remove fine particle-sized impurities by using chemicals such as frothers, collectors, and pH modifiers in an aqueous medium with flotation machines. On the other hand, dry magnetic separation and flotation methods are used in combination with each other for further feldspar beneficiation (Burat et al., 2006; El-Rehiem and Abd El-Rahman, 2008; Karaguzel et al., 2009).

In Turkey, the final product of the ceramic tile process is controlled by TSE EN 14411 standard in terms of physical properties; on the other hand, surface defects are checked by visual inspection. This study aimed to analyze the physical characteristics change of nepheline syenites such as color, shrinkage, water absorption, and surface roughness after the sintering process.

2. Materials and methods

2.1. Materials

In this study, the nepheline syenite sample obtained from Kırsehir Region/Turkey was used for the experimental studies. The chemical (X-ray fluorescence, XRF) and mineralogical analyses (X-ray diffraction, XRD) of the sample were done with Panalytical Axios Max XRF and Panalytical X'pert Pro MPD diffractometer, and the results are shown in Table 1 and Fig. 1, respectively. As presented in Table 1, Fe_2O_3, TiO_2, CaO, and MgO contents of the sample were found as 0.82%, 0.35%, 1.61%, 0.26%, respectively. The mineralogical analysis of the raw nepheline syenite sample seen in Fig. 1 indicated that the muscovite, albite, microcline, montmorillonite, and quartz were determined in the sample.

Table 1. Chemical analysis of the raw nepheline syenite sample

Sample Name	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	*LOI
Raw nepheline syenite	61.33	20.57	0.35	0.82	1.61	0.26	4.82	7.70	2.13

*LOI: loss on ignition

In our previous study, a suitable material for the ceramic industry was produced from the nepheline syenite ore obtained from the Kırsehir region of Turkey using magnetic separation followed by a flotation process to remove impurities such as calcite, quartz, mica, and iron-bearing minerals (Cinar and Durgut, 2019). And, several samples having different minerals contents were obtained from these processes, and these samples were used for the sintering studies along with raw nepheline syenite samples. These samples were, namely, non-magnetic product (-2+0.150 mm), deslimed non-magnetic product (-212+38 µm), *Concentrate 1*, and *Concentrate 2*.

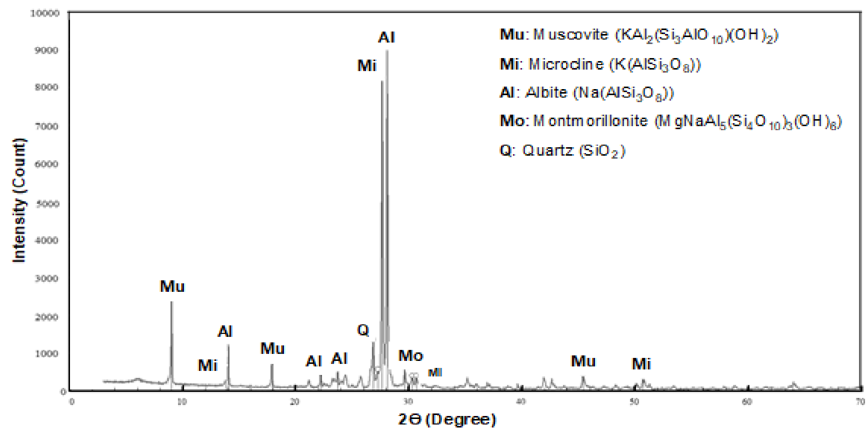


Fig. 1. XRD results of raw nepheline syenite sample

2.2. Methods

2.2.1. Sintering process

The samples before and after the mineral beneficiation were checked to see whether they were suitable for the sintering properties. Firstly, the samples were ground in a wet laboratory ball mill and sieved to the fineness of 2% residue for +63 μm, and then the ground sample was given 6% of moisture to have granules. The granules were pressed to have 5 cm diameter samples at 360 kg/cm^2. Then, the samples were dried at 105°C in a laboratory oven and sintered at a maximum of 1170°C for 48 min in the glazed porcelain production kiln. The technical properties of the samples such as water absorption and linear shrinkage were determined according to ISO Standards 10545-2. The color values of the sintered samples were determined using MINOLTA CR 300 colorimeter which operates on the CIELab method and in reflection mode.

2.2.2. Surface roughness

Surface roughness is a significant criterion managed to check the suitability of a surface for different purposes. The abnormalities on the surface affect the quality and performance of the end product. There are different kinds of parameters to evaluate surface roughness. R_a is the arithmetic average of the absolute values of the profile heights over the evaluation length. R_z is calculated by measuring the vertical distance from the highest peak to the lowest valley within five sampling lengths, then averaging these distances. R_t is the vertical distance between the highest and lowest points of the profile within the evaluation length. In this manner, the roughness analyses were applied to the sintered specimens, and the Mutitoyo Surftest SJ210 device was used to measure roughness parameters according to ISO 1997.

3. Results and discussion

3.1. Characterization of the samples

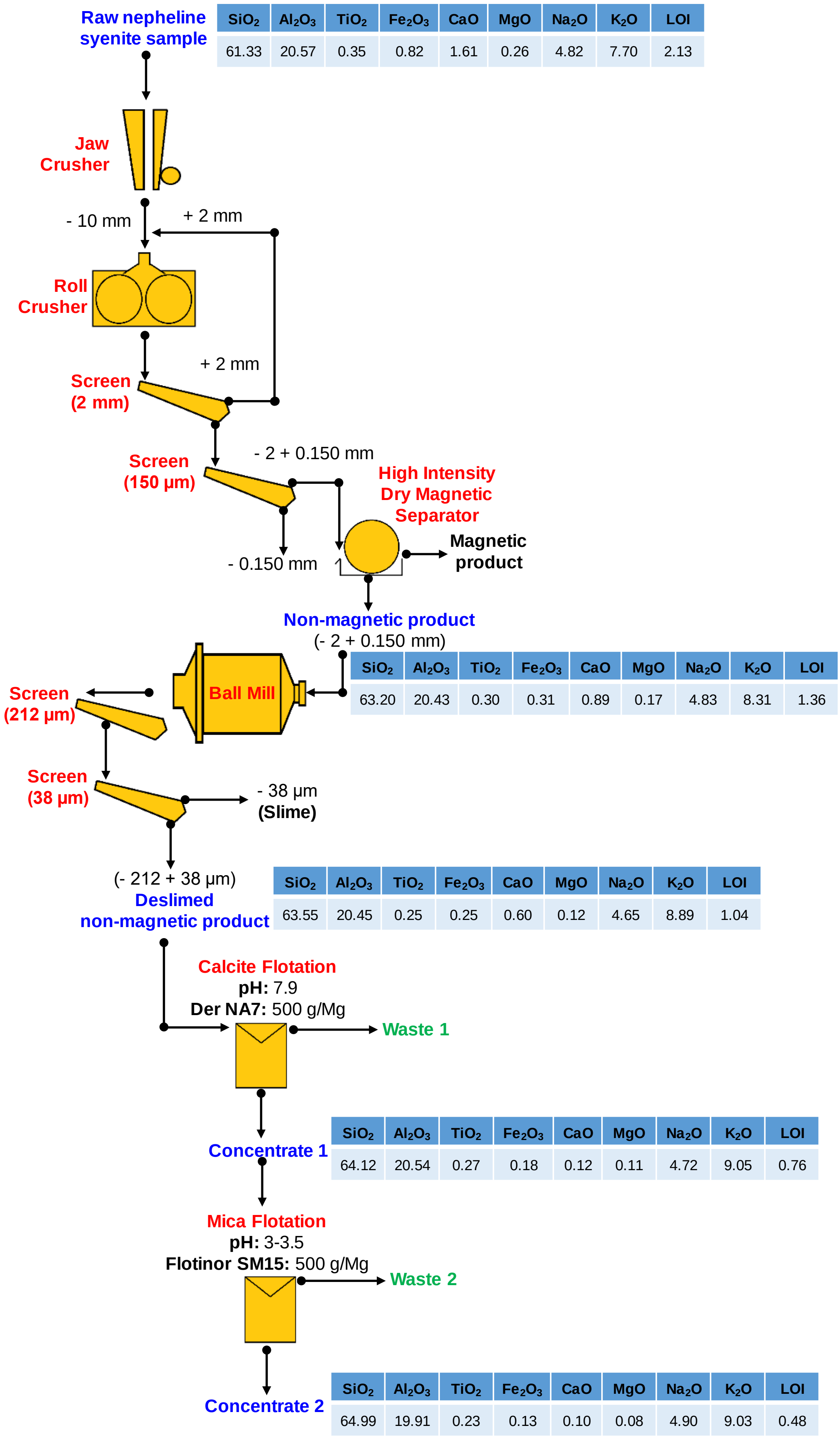
The preparation and mineral beneficiation processes to produce a high-quality nepheline syenite along with the conditions and chemical analysis results of the samples are shown in Fig. 2. As seen in Fig. 2, *Raw nepheline syenite sample* was first crushed to -2 mm using a laboratory-type jaw crusher, and then the crushed sample was sized to -2+0.150 mm for the dry magnetic separation experiments using a high-intensity roller magnetic separator to remove iron-bearing minerals from the sample. Next, *Non-magnetic product* from the magnetic separation was ground by a laboratory ball mill, then sieved and deslimed to obtain a -212+38 μm size fraction for the flotation experiments to remove further impurities.

The flotation experiments were carried out using a laboratory-type Denver flotation machine which had 1.5 dm^3 of cell capacity. The tap water was used for the flotation experiments, and the analytical grade of H_2SO_4 was used to adjust the pH. In the first step of the flotation experiments, *Deslimed non-magnetic product* (-212+38 μm) was floated by using 500 g/Mg of a commercial fatty acid type collector (DER NA7, DERBOTEKS) at natural pH 7.9 for CaO mineral removal, and sink material was taken as calcium mineral flotation concentrate (*Concentrate 1*). Secondly, *Concentrate 1* was floated by using 500 g/Mg of amine type collector (Custamine 9024, ARRMAZ) at acidic pH 3-3.5 for mica mineral removal, and sink material was received as a mica mineral concentrate (*Concentrate 2*).

The chemical and mineralogical analyses of the samples obtained from the beneficiation processes are presented in Table 2. As seen in Table 2, the Fe_2O_3 content of the raw nepheline syenite was reduced from 0.82% to 0.31% with the dry magnetic separation. Before the flotation experiments, slime was removed from the nonmagnetic product because of the negative effects on the flotation (Karaguzel et al., 2006; Burat, 2017). It was seen from the chemical analysis that TiO_2, Fe_2O_3, CaO, MgO, and Na_2O contents were reduced importantly in the slime removal process. CaO content of the nonmagnetic product was decreased from 0.60% to 0.12% in calcite mineral flotation. Additionally, it was seen that *LOI* decrease and pH variation over time based on acid consumption (Cinar and Durgut, 2019) support the calcite mineral presence in the calcite flotation feed.

Potash feldspar and soda feldspar standards for the glass and ceramic industry which were adopted by the Bureau of Indian Standards (BIS) are presented in Table 3. As seen in Table 3, the raw nepheline syenite has no class grade due to the high loss on ignition and iron oxide amount. However, the dry magnetic separation and flotation processes brought the material into Grade 3 was a suitable potash feldspar source for the pottery industry other than whiteware. K_2O and Na_2O values just cannot meet the Grade 1, this is because of the mineralogical form of the ore.

The mineralogical analysis was applied to the magnetic part that was taken from dry magnetic



Raw nepheline syenite sample

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
61.33	20.57	0.35	0.82	1.61	0.26	4.82	7.70	2.13

Non-magnetic product (- 2 + 0.150 mm)

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
63.20	20.43	0.30	0.31	0.89	0.17	4.83	8.31	1.36

Deslimed non-magnetic product (- 212 + 38 µm)

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
63.55	20.45	0.25	0.25	0.60	0.12	4.65	8.89	1.04

Concentrate 1

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
64.12	20.54	0.27	0.18	0.12	0.11	4.72	9.05	0.76

Concentrate 2

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
64.99	19.91	0.23	0.13	0.10	0.08	4.90	9.03	0.48

Fig. 2. Preparation and mineral beneficiation processes to produce nepheline syenite

Table 2. Chemical analysis of the samples before and after the mineral beneficiation

Sample Name	**SiO_2**	**Al_2O_3**	**TiO_2**	**Fe_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**	***LOI**
Raw nepheline syenite	61.33	20.57	0.35	0.82	1.61	0.26	4.82	7.70	2.13
Nonmagnetic	63.20	20.43	0.30	0.31	0.89	0.17	4.83	8.31	1.36
Deslimed nonmagnetic	63.55	20.45	0.25	0.25	0.60	0.12	4.65	8.89	1.04
Concentrate 1	64.12	20.54	0.27	0.18	0.12	0.11	4.72	9.05	0.76
Concentrate 2	64.99	19.91	0.23	0.13	0.10	0.08	4.90	9.03	0.48

*LOI: loss on ignition

Table 3. Requirements of potash and soda feldspar for glass and ceramics (BIS, 2007)

Characteristics	**Requirement**			
	Grade 1	**Grade 2**	**Grade 3**	**Grade 4**
Loss on ignition. the percent by mass. Max	0.6	0.6	0.8	1.0
Silica (as SiO_2). the percent by mass. Max	67	67	68	68
Alumina (as Al_2O_3). the percent by mass	17-20	17-21	17-21	19-22
Ratio of silica to alumina	3.4-3.6	3.4-3.6	3.5-3.8	–
Iron oxide (as Fe_2O_3), the percent by mass. Max	0.20	0.35	0.50	0.20
Calcium and magnesium oxides (as CaO + MgO). the percent by mass. Max	0 75	1.0	1.0	4.5
Alkalis (as K_2O). the percent by mass. Min	10.0	9.0	7.0	–
Alkalis (as K_2O). the percent by mass. Max	–	–	–	3.0
Alkalis (as Na_2O). the percent by mass. Max	4 0	4.0	6 0	–
Alkalis (as Na_2O). the percent by mass. Min	-	-	-	8.0

separation experiments due to identify the mineral type. As seen in Fig. 3, the magnetic part contained siderophllite ($KFe_2^{2+}Al(Al_2Si_2O_{10})(OH)_2$) which was a kind of iron-bearing mica, biotite mineral. In the XRD diagram, it was seen that there was no other iron-bearing mineral except siderophllite mineral, however, it was thought that the other iron mineral sources contained amphiboles and iron oxide minerals (hematite, limonite). TiO_2 content of the raw material was reduced from 0.4% to 0.2% with the dry magnetic separation because the mica mineral was removed from the ore. In the feldspathic ores, primer titanium mineral sources are rutile and sphene, but muscovite and biotite minerals can contain Ti^{4+} in the crystal lattices. It was seen from the literature that an important amount of titanium could be included in mica minerals, and TiO_2 values could reach 0.8% and 0.4% for muscovite and biotite, respectively (Roger and Van Dyk, 1994).

The contents of albite, microcline, mica, and quartz minerals in the products are shown in Fig. 4. As seen in Fig. 4, the raw nepheline syenite was comprised of 44% albite, 34% microcline, 12% mica, and 9% quartz minerals. After the magnetic separation of the feed sample, the amount of albite and microcline in the non-magnetic product increased to 47% and 41%, respectively, and, the amount of mica and quartz mineral ratio decreased to 7% and 4%. After the grinding and desliming operation, the amount of albite and microcline in the deslimed non-magnetic product was 47% and 45%, and the mica and quartz were 5% and 2%. It was thought that 2% of mica minerals in the fine fraction was removed with the desliming operation. Finally, the deslimed nonmagnetic material was used for the calcium and mica mineral flotation experiments, and the total albite+microcline mineral content increased to 97% whereas the mica mineral ratio was 2% and there was no quartz left in the final product.

The *L*, *a*, and *b* color values for the nonmagnetic sample increased to 84.89, 5.03, and 9.05 from 54.61, 3.69, and 5.86, respectively. As known from the literature that the metal oxides which are comprised of iron and titanium affect the *L*, *a*, and *b* values while the sintering process (Chandrasekhar and Ramaswamy, 2002; Chandrasekhar and Ramaswamy, 2006; Tatar, 2012). The *L* value of the deslimed nonmagnetic material decreased from 84.47 to 82.63 after the calcium flotation. Meanwhile, the calcite

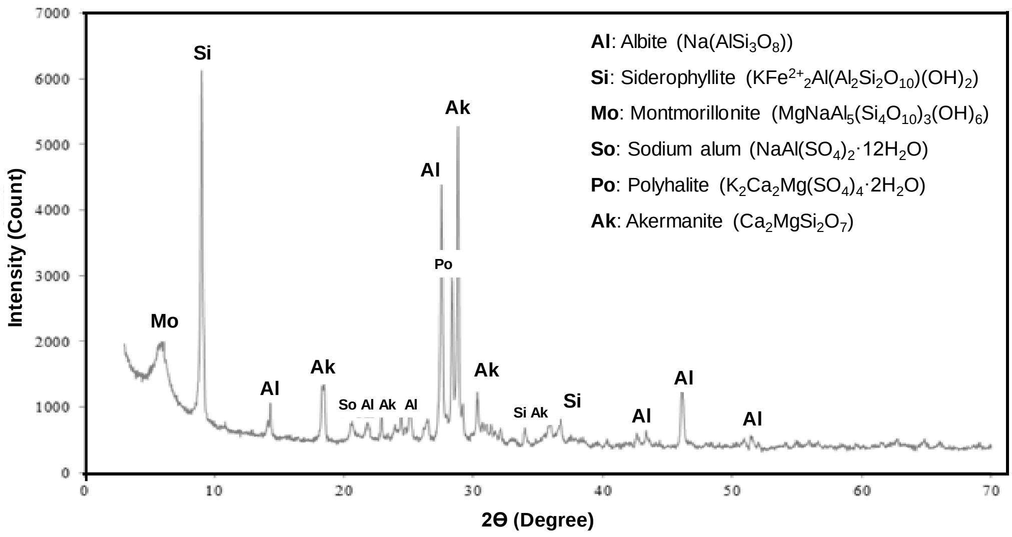


Fig. 3. XRD diagram of magnetic mineral

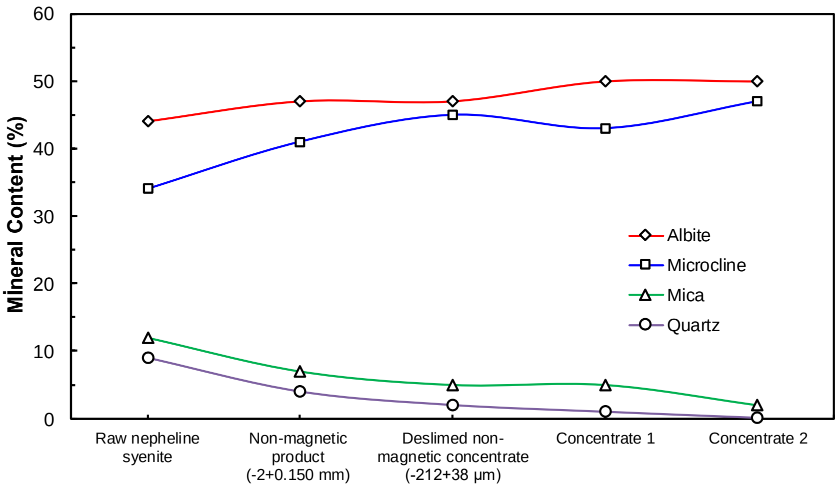


Fig. 4. Quantitative mineralogical analysis of the samples before and after the mineral beneficiation

Table 4. Sintering properties before and after mineral beneficiation

Samples	Shrinkage (%)	Water absorption (%)	Color values		
			L	*a*	*b*
Raw nepheline syenite	7.8	0.04	54.61	3.69	5.86
Nonmagnetic	2.4	16.93	84.89	5.03	9.05
Deslimed nonmagnetic	-1.4	23.47	84.47	3.95	8.19
Concentrate 1	-1.5	22.18	82.63	5.71	11.64
Concentrate 2	-0.9	26.41	84.31	5.41	10.75

addition to the ceramic body enhances the whiteness *L* value, therefore it is inferred that the calcite mineral was removed from the deslimed nonmagnetic material by the calcium flotation (Ersoy, 2015). The color variation of the samples is seen clearly in the pictures of sintered specimens before and after mineral beneficiation (Fig. 5).

After the magnetic separation, the shrinkage decreased to 2.4%, and the water absorption increased to 16.93%. In the case of K_2O, Na_2O, and free silica is together in the ceramic composition while the sintering, eutectic was formed by lowering melting temperature thusly vitreous phase forming was promoted (Pekdemir, 2008). Besides, mica minerals acted as fluxing material in the ceramic bodies due to the alkaline content in the formula (Hortling et al., 1997). On the other hand, the shrinkage decrease and water absorption increase of the sintered specimens via removal of impurities from ore can be explained by the increment of total alkaline minerals and an excessive amount of potash in contrast with soda content in the composition (Kyonka and Cook, 1954). Because particles cannot attach and form a glassy phase in the way of excess alkaline feldspars and lack of free silica in the composition while sintering at 1170°C, 48 min. Eutectic points and temperatures diagram of quartz-albite-microcline is shown in Fig. 6.

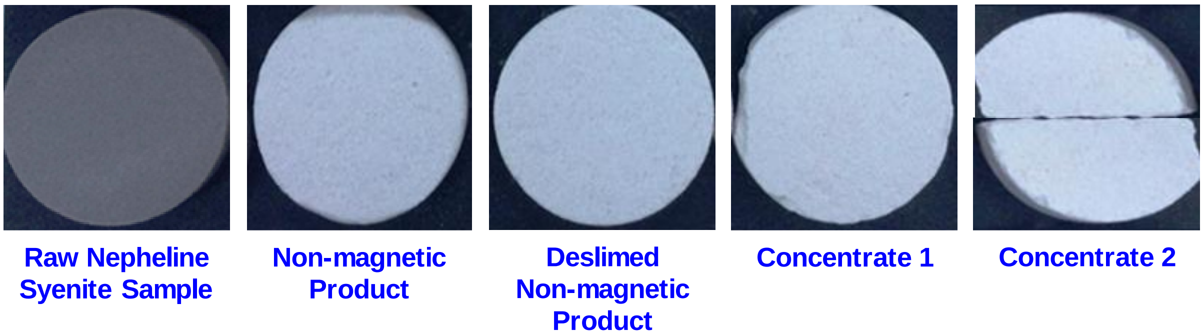


Fig. 5. Pictures of sintered specimens before and after mineral beneficiation

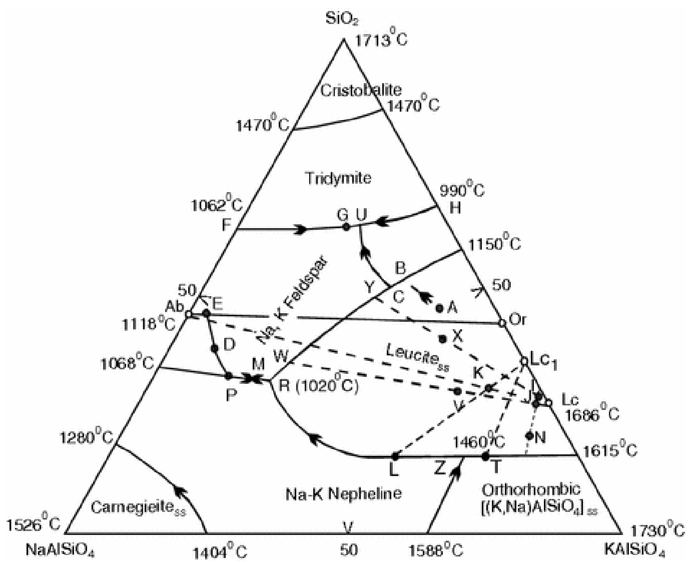


Fig. 6. Eutectic diagram of albite, microcline, and quartz (Gupta, 2015)

Differential thermal analysis (*DTA*) and thermogravimetric analyses (*TG*) of materials taken from after and before the mineral beneficiation experiments were done with NETSCH thermal analyzer at 10°C/min heating speed (Figs. 7 and 8). Until 500°C the materials showed endothermic reactions because of moisture, and chemically bonded water that existed in montmorillonite. The exothermic reactions occurred at 500°-600°C due to dehydroxylation of biotite mineral, and α – ß quartz phase transformation at 573°C. Another exothermic reaction occurred after 700°C, and calcite was being decomposed. Then, dehydroxylation of muscovite also occurred between 820-920°C (Foldvari, 2011; Suleiman et al., 2013). It is seen from *TG* analysis in Fig. 8 that the weight loss of materials reduced with the mineral beneficiation experiments.

Especially enriched materials were not sintered in the floor tile sintering conditions on their own due to the high alkaline content, and surface roughness analysis could not be done. As for that new compositions were made up by adding a 50% amount of clay to each material one by one after the grinding and sieving processes to have ceramic bodies. Then, new compositions were sintered in the floor tile sintering conditions with unglazed and glazed forms. The water absorption and surface rough-

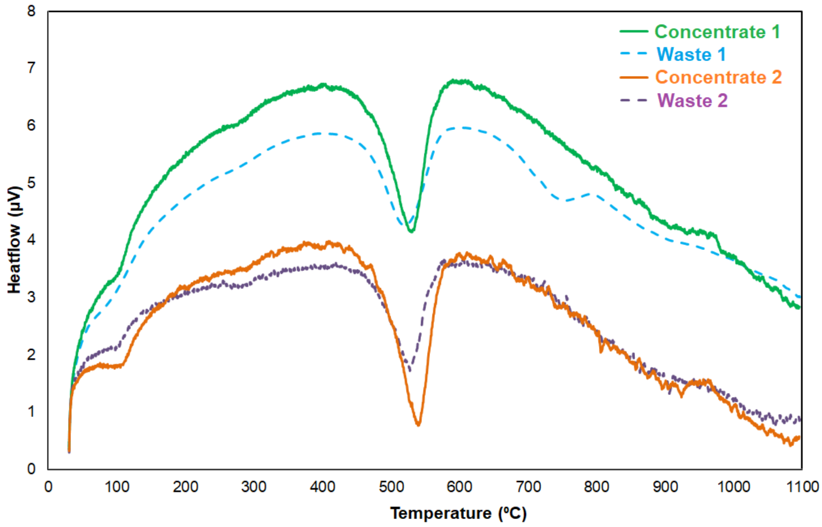


Fig. 7. *DTA* analysis of the samples before and after mineral beneficiation

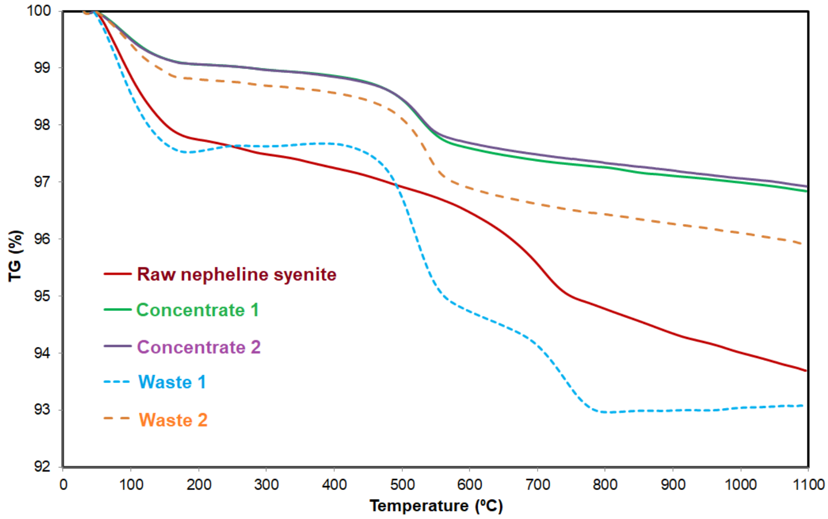


Fig. 8. *TG* analysis of the samples before and after the mineral beneficiation

ness properties of the unglazed and glazed specimens were analyzed, and the results are presented in Table 5.

It is seen from Table 5 that the water absorption values of unglazed specimens increased directly proportional to surface roughness results. The water absorption value of the ceramics is defined as the absorbed water into the entire open pores. It is related to body combination, processing, and sintering conditions which are directly proportional to apparent porosity (Kamseu et al., 2013; Gultekin et al., 2016).

In the ceramic sintering process, thermal reactions such as dehydroxylation, and outgassing must be finished before the glaze melting process; otherwise, such reactions could cause defects on the glazed surface. On the other hand, surface roughness is a significant factor in the polished surface of porcelain bodies (Hutchings et al., 2005). It is found that from the literature dehydroxylation occurs for muscovite minerals at 820-920°C, iron that is built-in biotite is oxidized at 500-600°C, and dehydroxylation occurs for phlogopite mineral as another variation of biotite at 900-1200°C (Gupta, 2015). The particle size of calcite is inversely proportional to decomposition temperature by thermal treatment. It is noticed that decomposition reaction could be continued above 1000°C depending on the particle size of calcite mineral; hence outgassing is advanced (Suleiman et al., 2013). As a consequence, the calcite content of the material makes surface roughness results clear for the glazed specimens. The pictures of unglazed and glazed sintered specimens are also shown in Fig. 9.

Table 5. Surface roughness analysis of sintered specimens

Sample information / Surface roughness analysis	Unglazed				Glazed		
	Water absorption (%)	R_a (µm)	R_z (µm)	R_t (µm)	R_a (µm)	R_z (µm)	R_t (µm)
50% Clay + 50% Raw nepheline syenite	6.35	2.587	15.667	20.946	0.177	0.858	1.385
50% Clay + 50% Nonmagnetic	0.45	1.541	9.946	12.378	0.185	0.983	1.493
50% Clay + 50% Deslimed nonmagnetic	1.86	1.759	10.859	14.418	0.178	0.850	1.253
50% Clay + 50% Concentrate 1	4.87	2.396	15.549	21.292	0.170	0.932	1.419
50% Clay + 50% Concentrate 2	4.46	2.286	14.957	21.483	0.165	0.925	1.490
50% Clay + 50% Waste 1	12.00	2.853	17.082	22.942	0.248	1.278	1.901
50% Clay + 50% Waste 2	0.85	1.622	8.826	12.249	0.212	1.088	1.724

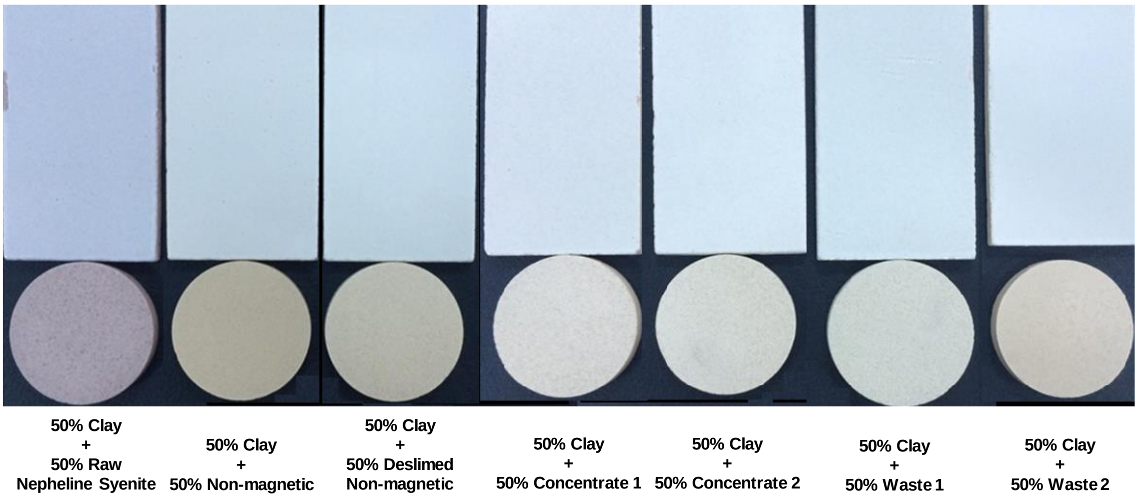


Fig. 9. Pictures of unglazed and glazed specimens

Overall, this study aimed to investigate the effects of impurities in the nepheline syenite sample, which is a ceramic raw material, on the final product. The experiments were carried out systematically in the light of the results obtained from Çınar and Durgut (2019). The products obtained from the beneficiation steps consisting mainly of magnetic separation and subsequent flotation processes were used in the ceramic body.

While it was stated that nepheline syenite used in ceramic products without enrichment caused roughness, pinhole shapes cavities, and color problems in the body, it was observed that the problems disappeared in the products with beneficiation. Pinhole-shaped cavities, which would normally occur due to calcium-induced gas escape, are made in ceramic factories by adjusting the firing regime. Here, gas escape is provided by extending the cooking temperature before glazing. However, this will increase energy costs.

Therefore, *Concentrates 1* and *2* can be used in floor tile body compositions. The nepheline syenite can also be used in frit production in the case of Fe_2O_3+TiO_2<%0,2, so Concentrate 2 should be further beneficiated with physical and physicochemical mineral processing methods such as wet magnetic separation and leaching, respectively.

4. Conclusions

The total alkaline ratio (Na_2O+K_2O) of the composition was taken from 12.52% to 13.93% with mineral beneficiation experiments, and the quantitative mineralogical analysis showed that the albite and microcline content increased from 47% to 50% and from 41% to 47%, respectively.

L-a-b color values for the raw nepheline syenite sample, dry magnetic separation concentrate, and flotation beneficiation concentrate were gained as 54.61-3.69-5.86; 84.89-5.03-9.05, and 84.31-5.41-10.75

respectively. Dry magnetic separation has a significant impact on the *L, whiteness value* of sintered specimens while calcite and muscovite minerals that were separated from the ore; hence balanced the whiteness value.

Thermal analysis showed that the calcite mineral caused outgassing which could bring pinholes on the glazed surface. Mica and calcite minerals showed endothermic reactions, thus the sintering process could be affected negatively in terms of energy consumption.

Surface roughness analysis was applied to see the effects of impurities on the sintered specimens' glazed surface. It was seen that dry magnetic separation affected glazed surface roughness positively. R_z and R_t values of nonmagnetic concentrate decreased to 0.850 and 1.253 µm from 0.858 and 1.358 µm compared to raw nepheline syenite respectively.

The surface roughness was also affected positively after the flotation experiments. Calcite and mica content decrement in the flotation experiments affected the surface of sintered specimens positively by reducing roughness values. R_a value decreased from 0.178 µm to 0.170 and 0.165 µm with the calcite and mica removal in flotation experiments respectively for the unglazed surface.

It was seen that the water absorption values were directly proportional to R_a values for unglazed surfaces. R_a should be an indicator of water absorption and porosity.

In conclusion, calcite and mica minerals affect surface quality negatively by forming pinholes due to dehydroxylation and outgassing reactions in the sintering process. Magnetic mineral removal with dry magnetic and calcite-mica mineral removal with the flotation methods showed an important impact on the surface roughness.

Acknowledgments

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