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DRY VERSUS WET UPGRADING OF NEPHELINE SYENITE ORES

Received April 20, 2010; reviewed; accepted July 10, 2010

Under strict specifications regarding its iron, alumina, and alkali contents, nepheline syenite has a wide range of applications as filler, pigment and extender besides its usage in glass and ceramic industries. Kingdom of Saudi Arabia has huge nepheline syenite deposits in SAWDA mountain. Unfortunately, the ore is of low grade as to its high iron content (7.68% Fe₂O₃) and low alumina (17.38% Al₂O₃), and thus cannot be used in any of the previously mentioned industries as mined. This paper aims at investigating the amenability of processing the ore to meet market specifications. In the investigation two different technologies, dry and wet, are considered. The first is magnetic separation as a dry upgrading technique while the second is flotation as the wet upgrading technique. In applying magnetic separation technique the cross belt dings magnetic separator was used. The main studied variables were applied field intensity, separator belt speed, feed rate, and feed size, while the collector dosages were tested for upgrading the ore by flotation technology in a Denver D-12 flotation cell. The obtained results showed that magnetic separation can never produce nepheline concentrates having Fe₂O₃ less than 0.85%. It was also found that at optimum flotation conditions the nepheline concentrates have Fe₂O₃ content not less than 0.40%. However, combining the two techniques i.e. applying flotation under optimum conditions for cleaning of the previously obtained magnetic concentrate resulted in a final concentrate of 0.09% Fe₂O₃ with Al₂O₃ content of 23.58%. The produced concentrate can be used in many industrial applications, especially in glass and ceramics production.

keywords: nepheline syenite, magnetic separation, flotation, glass and ceramics

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1. BACKGROUND

Nepheline syenite is an igneous rock. It may be formed in alkali-rich rock magma deficient in silica or by metasomatic nephelinization processes. Nepheline syenite rocks are composed essentially of nepheline, sodic plagioclase (usually albite or oligoclase) and microcline but in varying proportions, with small amounts of biotite, hornblende, magnetite, pyroxene, muscovite, sodalite, garnet, zircon, apatite, ilmenite, calcite, pyrite and zeolites or some of them as impurities or iron-bearing minerals (Guillet, 1994). The main nepheline syenite reserves are located in the former USSR, Norway, Canada, and recently Turkey (Harben, 1995). In Norway the proven nepheline syenite reserves exceed 400 Tg (teragrams, million tons). In Canada, Idusmin Co. has published a reserve figure of 240 Tg of nepheline syenite found in Blue Mountain area. Reserves of the rock in Turkey, was recently estimated and was found over 1 Pg (pentagrams or billion tons) (Gulsoy, 1994). Reserves of other countries are typically much smaller; however, there is a little information about their possible economic significance. In Egypt for example, the main nepheline syenite deposits are located in the southern sector of the Eastern Desert, south Idfu-Mersa Alam road with estimated reserves of approximately 100 Tg (El Ramely et al., 1971; Dardir et al., 1994). In the Kingdom of Saudi Arabia nepheline syenite deposits exist mainly in SAWDA mountain which is located 7 km to the east of EL Aqbaa Gulf and 35 km south of ALhaql port. The proven reserves of the ore have not been evaluated but the report prepared by Collin (1994) for the Saudi Ministry of Petroleum and Mineral Resources showed that the ore deposits cover an area of 7 square kilometers. Considering this fact, one can expect an average ore reserve of not less than 90 Mg assuming an ore thickness of 3 m.

In many industrial applications, feldspar and nepheline syenite are largely interchangeable (Burat et al., 2006). Compared to feldspars, nepheline syenite has a higher alkali/alumina ratio and therefore is considered as a challengeable competitor for feldspars (Gulsoy et al., 1994). In this aspect Esposito et al. (2005) stated that *“Compared to pure feldspars, the advantages coming from the use of nepheline-syenite are: (i) the content of potassium and sodium is higher, $K_2O + Na_2O$ is about 9–12% in feldspars, whereas it is larger than 14% in nepheline syenite, and (ii) the melting temperature is generally lower than that of potassium-feldspar, which always contains other phases, such as quartz, which shifts the melting point to higher temperatures”*. In ceramics, the low fusing temperature and high fluxing capacity of the nepheline syenite allow for a vitrifying agent by producing an early glassy phase that binds other constituents of the mix. Applications for nepheline syenite as extender, pigments and fillers, have been pioneered in Canada. Finely-ground nepheline syenite is especially used as inert filler in paints, both latex and alkalid systems, for use in high traffic areas, as metal primers, wood stains, sealers and undercoats (Guillet, 1994). In plastics, nepheline syenite is used as inert, low cost

mineral filler in polyvinyl chloride (PVC), epoxy, and polyester resin systems. Because it exhibits a low resin demand, high filler loadings are possible, permitting reduced requirements for more expensive components (Guillet, 1994).

Regarding the required specifications for each application one can say nepheline syenite used for glassmaking should be a sandy sized product falling within the range of 40 to 200 mesh (Harben, 1995). Its iron content should not exceed 0.1% Fe₂O₃ while alumina and alkali should be as high as possible, typically at least 23 and 14%, respectively (Harben, 1995).

On the other hand, nepheline syenite for the ceramics industry should be finely-ground, typically into products of 200, 270, and 400 mesh (Harben, 1995). Summary of the above background reflects the wide range of industrial applications of nepheline syenite deposits after achieving the required specifications.

Unfortunately, the Saudi nepheline syenite at Sawda Mountain contains high iron content and low alumina assay which makes it not suitable for industrial applications. As a result, this investigation is devoted to study the amenability of the ore to upgrading to meet specifications needed for different industrial applications.

2. EXPERIMENTAL

2.1. FEED PREPARATION

Nepheline syenite feeds were prepared to suit the applied upgrading technique. The sample was firstly subjected to primary and secondary crushing leading to a product of 100% -3.36 mm. In the subsequent size reduction stages a “Wedag” rod mill was used to produce -0.25 or -0.125 mm as separate feeds for magnetic separation, where the mill was fed with the -3.36 mm secondary crushed ore. It was operated in a batch closed-circuit with a screen (0.25 mm or 0.125 mm) according to intended produced feed. The operating conditions of the mill were as follows: dry bases, 9 grinding rods and 15 min batch grinding time. On the other hand, preparation of the finer feeds (-0.075 mm) for flotation purposes was conducted by regrinding the rod mill fine product (-0.125 mm) in a ball mill working in wet bases at 50% solid by weight. To achieve the targeted size, the mill was operated in a closed circuit with a 0.075 mm screen. The produced flotation feed was deslimed using a rig hydrocyclone giving a cut size of 25 µm. Finally, grinding of the cleanest magnetic concentrate for further cleaning by flotation was achieved by using a porcelain planetary mill.

2.2. CHARACTERIZATION OF ORE SAMPLES

The ore was characterized physically and chemically. The first was accomplished by size analyses of the crushed and ground products while the second was achieved by running complete chemical analyses. Complete chemical analysis of the original and

final produced samples for determining Fe_2O_3 , SiO_2 , CaO , MgO , Al_2O_3 , Na_2O and K_2O was conducted by X-ray fluorescence. For routine chemical analysis in the optimization tests Fe in the form of Fe_2O_3 was determined applying a standard method of dissolution of the samples using HCl or HNO_3 . Iron contents in the dissolved samples were determined using Perkin Elmer Atomic Adsorption (Suzan et al., 2002).

2.3. MAGNETIC SEPARATION TESTS

These tests were designed to check the possibility of dry upgrading of the ore, where water was used neither in the previous feed preparation steps nor in the separation stage. Two series of magnetic separation tests were carried out. The first series was performed on samples ground to 100% -0.250 mm while the second series were carried out using feed samples 100% less than 0.125 mm. The “Dings” cross-belt magnetic separator was used in both cases. The separator used in this investigation is a “pick-up” separator with an auxiliary permanent magnet for the separation of ferromagnetic material. The head of the electromagnet reaches a maximum magnetic field of 1.3 T (13 kGauss) at a minimum air gap of 3 mm. Optimization of the main parameters affecting the separation process included verification of magnetic field strength, feed rate and feed size. In studying the effect of feed rate mono or, sometimes multiple particle layer were applied. The feed rate was calculated from equation adopted by Negm et al. (2000). Moreover, cleaning of the concentrate was repeated sometimes.

2.4. FLOTATION TESTS

The $-0.075 + 0.025$ mm prepared flotation feed was cleaned in a “Denver D-12” flotation machine at a pulp density of 67-70% solid and motor speed of 2500 rpm for 10 min. Reverse flotation of iron-bearing contaminants from the nepheline syenite samples were then carried out in the laboratory using a “Denver D-12” subaeration flotation cell. The used collector was a mixture of the “Cyanamid” aeropromoters 801 and 825 (1:1 by weight mixture) of commercial grade from local market. For each test, 250 g batches of the $-0.075+0.025$ mm feed were used and conditioned with H_2SO_4 or NaOH for 5 min for pH control. Other operating conditions were adjusted at their optimum values determined by Abouzied et al. (2000), where the optimum conditions were as follows: 25% solid pulp density, pH~4, frother Aerofrother 65 at a dosage of 0.1 kg/Mg. After adjusting all the parameters, the aeration was started, followed by manual skimming of the froth for about 10 min, which usually denotes demineralized froth. Both floated and sink products were collected, dried and analyzed for their iron contents.

2.5. COMBINED MAGNETIC SEPARATION-FLOTATION OF SAMPLES

Cleaning of the non-magnetic nepheline syenite concentrate was carried out by flotation, under the predetermined optimum dosage of the studied collector mixture. Cleaner concentrate were collected, dried, weighed and analyzed. The final product from this series was subjected to complete chemical analyses.

3. RESULTS

3.1. CHARACTERIZATION OF NEPHELINE SYENITE SAMPLE

Table 1 shows results of the complete chemical analysis of the original nepheline syenite sample. It indicates that the ore is of low grade and out of market specifications for glass and ceramics production. This is because of its low alumina content (17.38% compared with at least 23%), its high iron content (7.68% Fe_2O_3) compared to minor iron contents for glass and ceramics manufacture. Similarly, its alkali content is 14.59% on the border line to market specifications, which is 14%. As a result its upgrading becomes a must.

Table 1. Complete chemical analysis of the considered nepheline syenite sample

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O
%	56.82	17.38	7.68	0.97	0.20	9.23
Constituent	K ₂ O	TiO ₂	P ₂ O ₅	S	Cl	L.O.I
%	5.36	0.09	0.01	0.02	0.05	1.55

Figure 1 shows some of the physical and chemical characteristics of the primary crushed nepheline syenite sample. From this Figure it is clear that a general unimodal representation is exhibited with d_{50} of 2 mm. Meanwhile, chemical analysis of the different size fractions shows, more or less, an even distribution of Al₂O₃ and Fe₂O₃ indicating no separation of individual mineral components due to preferential hardness.

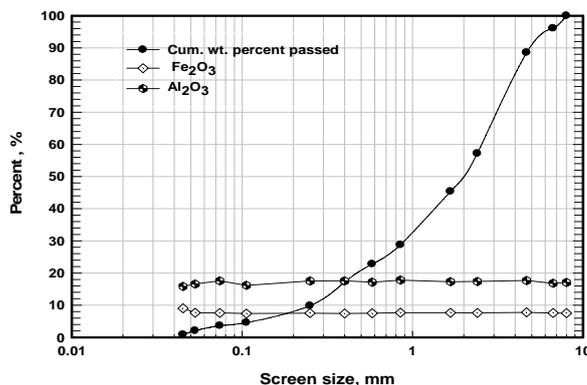


Fig. 1. Size distribution and chemical analyses of primary crushed nepheline syenite sample

Results of further size reduction of the sample are shown in Table 2. It shows that near 50 % of the sample is in the size cut of $-0.211 + 0.125$ mm. Chemical analyses of the different size fractions indicated that the finest fraction (-0.045 mm) has the highest iron content (10%).

Table 2. Size distribution and chemical analyses of the ground nepheline syenite considered sample

Screen size, mm	Wt. percent retained	Cum. Wt. percent passed	Assay in a given fraction, %		Recovery in a given fraction, %	
			Fe ₂ O ₃	Al ₂ O ₃	Fe ₂ O ₃	Al ₂ O ₃
-0.25+0.211	12.84	100	6.99	17.59	11.81	13.10
-0.211+0.125	44.7	87.16	7.27	17.67	42.74	45.82
-0.125+0.106	12.44	42.46	7.29	16.93	11.94	12.22
-0.106+0.075	12.51	30.02	8.05	16.84	13.24	12.22
-0.075+0.053	7.03	17.51	8.24	16.74	7.63	6.83
-0.053+0.045	5.56	10.48	8.42	16.55	6.16	5.34
-0.045	4.92	4.92	10.00	15.61	6.47	4.45
Head	100		7.60	17.24	99.99	99.98

3.2. MAGNETIC SEPARATION RESULTS

The magnetic separator belt speed is an important parameter because it determines the residence time of the feed particles in the magnetic field. Figure 2 shows the effect of changing belt speed of the "Dings" magnetic separator on the separation process, considering a feed rate as single particle layer of the first feed (100% -0.25 mm) at applied magnetic field of 1 T. It illustrates that increasing the belt speed leads to improvement of the nonmagnetic fraction yield but this is in expense of its quality as its iron content increases. However, at a belt speed of 2 m/min, which is the optimum, only 42.6% of the iron is recovered in the non magnetic product (concentrate). This means that about 47.4% of the iron content was rejected in the tail product (magnetic fraction). The overall poor results obtained in this series maybe attributed to the wide size range of the feed, and thus, the calculated monolayer particle is not accurate.

Changing the feed rate (number of feed layers) on the belt at its optimum speed and under the same operating conditions applied in the previous series resulted in deterioration of the separation process due to the shielding effect (Fig. 3). This has, in fact, a major effect on the quality of the concentrate and the capacity of separator as well. Results indicate that at a four-particle layer feed (48 kg/hr), a concentrate having 6.73% Fe₂O₃ was obtained, i.e. only 15.34% of iron-bearing impurities were rejected. This indicates that the optimum feed rate for this size is 12 kg/hr, which implies a monolayer feed. However, the hitherto unsatisfactory obtained results lead to investigating the effect of increasing the magnetic field strength to its maximum of

1.3 T by increasing the applied current to 3 A, under the predetermined optimum conditions and entering the previous feed in two modes. The first is as it is (100% - 0.25 mm) and the second is after its desliming using a 0.045 mm screen followed by repetitive cleaning until no change in the concentrate weight. The obtained results are shown in Table 3. It can be noticed that a relative improvement in the quality of the concentrate is achieved. This is because the iron content the first feed (100% -0.25) was decreased to 2.63% Fe₂O₃, i.e about 70.8% removal of magnetic impurities and the iron content of the second feed (-0.25+0.045 mm) was 1.96 % without cleaning achieving ~ 73% removal of the original feed iron. It is also clear that the fifth cleaning cycle of the second product lead to a remarkable improvement in its quality where the iron content of this product is 1.33% which means iron removal of approximately 81%. The improved obtained results confirm the shielding effect of coarse particles and necessity using much narrower size feeds with this kind of separators.

However, this was taken into consideration when studying the finer feed (-0.125). Figure 4 shows that using the deslimed fine feed (-0.125 + 0.045 mm) at monolayer feed rate and a magnetic field intensity of 1 T an optimum belt speed of 4 m/min is noticed. At this speed, a nonmagnetic fraction of ~1% Fe₂O₃ can be obtained achieving an overall iron removal of 73%, with an iron recovery in the non-magnetic fraction of 11% and the rest of the iron was previously rejected with the slimes (-0.045 fraction).

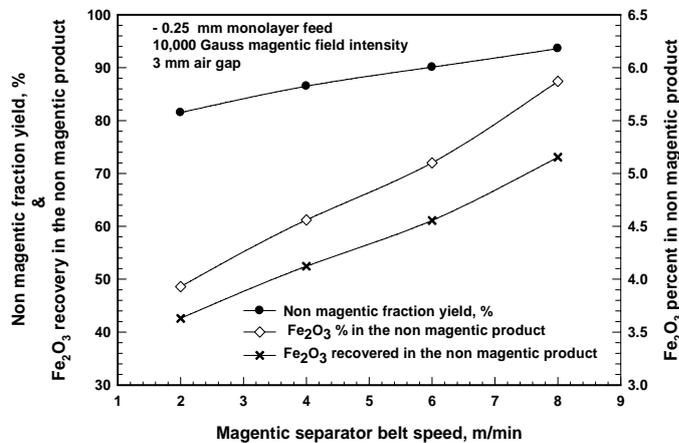


Fig. 2. Effect of the main belt speed of the "Dings" cross belt magnetic separator on the quantity and quality of nepheline syenite concentrate

Using this deslimed fine feed i.e. -0.125 +0.045 mm, under a monolayer feed rate, belt speed of 4 m/min, and maximum field strength of 1.3 T with three repetitive

cleaning cycles resulted in an appreciable decrease in the iron content of the concentrate to 0.85% Fe_2O_3 , corresponding to air removal of 77% (Table 4). The obtained results are in agreement with some published results regarding upgrading of a similar Egyptian nepheline syenite ore (Abouzeid et. al., 2000; Suzan et al., 2002)

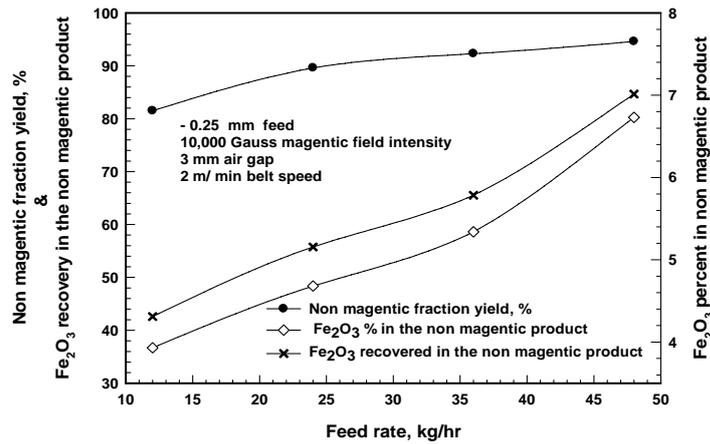


Fig. 3. Effect of the feed rate of "Dings" cross belt magnetic separator on the quantity and quality of nepheline syenite concentrate

However, one can conclude that, within the aforementioned limits of experimentation, it was not possible to produce high quality nepheline syenite concentrates by dry upgrading using magnetic separators only.

Table 3. Separation of the considered nepheline syenite sample (-0.25+0.045mm) at maximum field strength (1.3 T) using the "Dings" cross belt magnetic separator

Feed size, mm	Product	Wt., %	Fe_2O_3	
			Ass., %	Rec., %
-0.25	Conc.	83.67	2.63	29.22
	Tail	16.33	32.64	70.78
	Total	100	7.53	100
-0.25+0.045	Conc.	79.83	1.96	20.78
	Tail	15.25	36.06	73.03
	Total	95.1	7.43	93.81
-0.25+0.045 With 5 cleaning cycles	Conc.	74.76	1.33	13.2
	Tail	20.34	29.85	80.63
	Total	95.1	7.43	93.83

Table 4. Separation and cleaning of fine deslimed nepheline syenit feed at maximum magnetic field strength of "Dings" cross belt separator

Feed size, mm	product	Wt. %	Fe ₂ O ₃	
			Ass. %	Rec. .%
-0.125+0.045 with 3 cleaning cycles	Conc.	70.63	0.85	7.97
	Tail	18.9	30.6	76.8
	Total	89.53	7.13	84.77

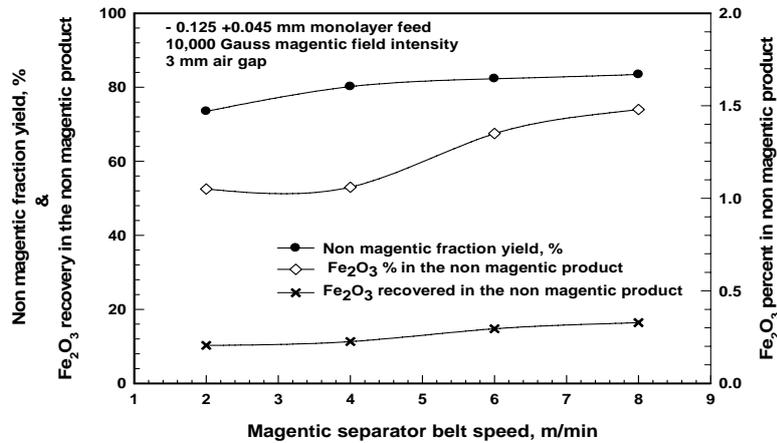


Fig. 4. Effect of main belt speed of "Dings" cross belt magnetic separator on the quantity and quality of nepheline syenite concentrate considering deslimed finer feed (-0.125+0.045 mm)

3.3. FLOTATION RESULTS

Figure 5 illustrates the effect of dosage of Cynamid 801 and 825 as a collector on the quantity and quality of the nepheline syenite concentrate obtained by flotation under the operating parameters listed in the bottom left corner of the graph. It can be easily noticed that the greater the collector dosage the better is the produced concentrate regarding its quality and quantity. However, collector dosages higher than 2.5 kg/Mg have no credible effect either on the quality or quantity of the obtained concentrate. At this dosage, approximately 61% by weight of the original sample is yielded as concentrate having 0.4% Fe₂O₃. In this way, the iron recovered in the concentrate is only 4% of the iron contained in the original sample. The amount of iron rejected in the flotation tailings and slimes represents a good success regarding the separation process. But still the targeted specifications for marketable nepheline concentrates are not met yet. As a result further upgrading will be introduced in the next section.

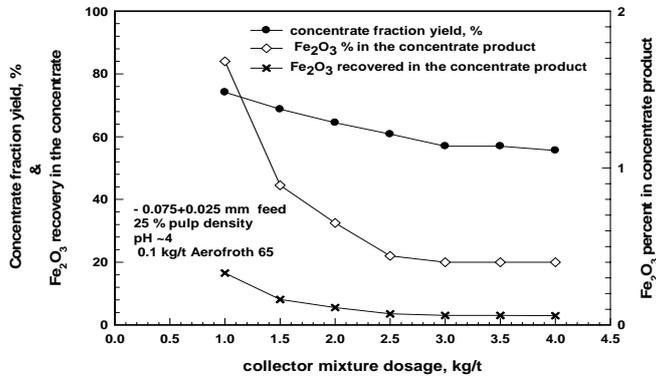


Fig. 5. Effect of dosage of Cynamid 801 and 825 as a collector on the quantity and quality of nepheline syenite concentrate

3.4. COMBINED MAGNETIC SEPARATION-FLOTATION OF SAMPLES

The cleanest nepheline syenite concentrate obtained from the finer feed (-0.125+0.045 mm), shown in Table 4, was ground in a planetary mill and was considered as a new flotation feed. It was tested under the same optimum conditions mentioned in the flotation section. The Cynamid mixture was added stepwise. The final obtained concentrate results obtained at collector dosage of 2 kg/Mg is shown in Table 5. It reflects a concentrate of high quality regarding its iron content (0.09% Fe₂O₃).

To ensure the quality of the produced concentrate regarding its content from other constituents, it was subjected to complete chemical analyses. Results are shown in Table 6. The results confirm the high quality of the produced concentrate and its amenability to be applied in different industries.

Table 5. Combined magnetic separation-flotation results of nepheline syenite

Feed size, mm	product	Wt. %	Fe ₂ O ₃	
			Ass. %	Rec. %
-0.075 at 2 kg/Mg Cyanamid mixture (step wisely added)	Conc.	55.43	0.09	0.66
	Tail	15.1	3.62	7.31
	Total	70.53	0.85	7.97

Table 6. Complete chemical analysis of the cleanest nepheline syenite concentrate obtained by combined magnetic separation - flotation

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	K ₂ O
%	59.43	23.58	0.09	10.16	6.31

4. CONCLUSIONS

From the presented results the following conclusions can be drawn:

- the Saudi nepheline syenite from the Sawda Mountains, is a low grade ore and as mined beyond market specifications due to its high iron and low alumina contents,
- upgrading the ore applying a single technique either on dry basis (magnetic separation) or wet basis (flotation) can never lead to a marketable concentrate product,
- the cleanest concentrate that can be obtained in case of dry upgrading of the ore, by magnetic separation only, contains not less than 0.85% iron in the form of Fe_2O_3 ,
- wet upgrading of the ore applying flotation technology and using 2.5 kg/Mg of Cyanamid 801 and 825 mixtures as a collector can lead to a concentrate having 0.4% Fe_2O_3 or more,
- cleaning of the concentrate obtained at the optimum conditions of magnetic separation, by flotation results in a final concentrate of high quality regarding its iron, alumina and alkali contents (0.09%, 23.58%, 16.47% for Fe_2O_3 , Al_2O_3 and $\text{Na}_2\text{O} + \text{K}_2\text{O}$, respectively).

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Nefelin sjenitowy ma zastosowanie jako wypełniacz, pigment oraz jako składnik szkła i ceramiki. Królestwo Arabii Saudyjskiej ma duże złoża nefelinu sjenitowego, które występują w Górach Sawda. Są one jednak niskiej jakości, ze względu na wysoką zawartość żelaza (7.68% Fe₂O₃) oraz niską zawartość Al₂O₃ (17.38%). W pracy badano ich wzbogacanie metodą magnetyczną na sucho i flotacyjną na mokro. Badano wpływ natężenia pola magnetycznego, prędkość taśmy, prędkości podawania nadawy oraz uziarnienia, a flotację prowadzono w maszynie Denver D-12 badając zużycie kolektora. Separacja magnetyczna pozwala na otrzymywanie koncentratów nefelinowych posiadających jednak więcej niż 0.85% Fe₂O₃, podczas gdy flotacja mogła dostarczać koncentratów zawierających nie więcej niż 0.40% Fe₂O₃. Łącząc obie techniki tj. dokonując flotacji w optymalnych warunkach uprzednio wzbogacanego na sucho materiału, otrzymano końcowy koncentrat zawierający 0.09% Fe₂O₃, przy zawartości Al₂O₃ wynoszącej 23.58%. Otrzymane koncentraty mogą być użyte w wielu przemysłach, zwłaszcza do produkcji szkła i ceramiki.

słowa kluczowe: nefelin sjenitowy, separacja magnetyczna, flotacja, szkło, ceramika