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Upgrading of Libyan Feldspar Ore Using Different Separation Techniques

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ABSTRACT

The demand for feldspar as a raw material for various industrial applications is continuously increasing. It is a valuable raw material in the manufacture of glass, ceramic, fillers, enamel frits and welding electrodes. Feldspar is abundant in the earth's crust and is generally associated with other silicate, titanium and iron oxide minerals. The present paper aimed to reduce the iron oxide content from feldspar ore of Libya, as well as, to obtain an optimal grade of feldspar concentrate for industrial applications. It should be mentioned that this is the first time that Libyan feldspar ores subjected to upgrading studies. Dry and wet magnetic separation techniques followed by leaching processes were carried out, in order to decrease the iron contamination and increase the feldspar content. From a feed containing 2.48% Fe_2O_3 a non-magnetic concentrate of 0.28 % Fe_2O_3 with feldspar yield of 84.5 % was obtained at size fraction – 0.032 mm. Such concentrate contains about 21.05% Al_2O_3% and Na_2O, K_2O of 18.24% in comparison to 12.18% in the feldspar ore. Leaching process successfully reduced the iron oxide content up to 0.07 %. At the same time, the total contents of Na_2O + K_2O increased to about 16.73% in comparison to 12.18% in the original feldspar ore.

1. Introduction

Feldspar is one of the most common minerals in the world where it forms about 60% of the rocks of the earth's crust [1,2]. Orthoclase (K-feldspar), albite (Na-feldspar) and anorthite (Ca-feldspar) are the most widespread feldspar minerals. The most associated minerals into feldspar ore are clays, mica's minerals (i.e. biotite and muscovite), tourmaline, rutile, and sphene [2].About 60% of world feldspar production is used in the ceramic industry and 35% in the glass industry and 5% as a source of alumina or as a flux [3]. As for ceramics (including electrical insulators, tableware, pottery, tile, and sanitary ware), the alkalis in feldspar (CaO, K_2O and NaO) are used as grog components in the first stage of producing porcelain and act as fluxes to form a glassy phase at the softening point (1150–1350°C) in the production of ceramic and manufacture of glazes and enamels [4].Presence of coloring materials such as iron oxides and rutile decreases its quality due to forming a black spot in the product body\during firing process. Due to the lack of advanced processing and mechanism to separate feldspar from gangue minerals, the production of feldspar with high quality is relative low.[5,6]. Several studies were conducted to improve the feldspar products to use in the ceramic industries in many countries [7,8]. It is also a source of alkalies that make the products more transparency and reduces the melting temperature this make less energy consumption for the glass and ceramics production. However, the specifications of feldspar to be used as a raw material for these industries are SiO_2, <70%, Al_2O_3>17%, Fe_2O_3<0.1% with K_2O and Na_2O both >5% **[**9].To this end, feldspars are mostly beneficiated by means of magnetic separation, flotation or combination of both methods [10]**.**Magnetic separation technology is inevitable for feldspar beneficiation because non-magnetic feldspar is associated with magnetic gangue minerals, as a result of low magnetic properties of its gangue minerals. Therefore, beneficiation can be improved only by using high-intensity magnetic separation equipment or magnetic separation followed by flotation. [11,12,13].Feldspars are inherently more floatable than quartz under acid conditions and this difference in flotation behavior can be greatly accentuated over the pH range 2.0 - 3.5 by the addition of HF [14].Using quaternary ammonium salt as a cationic collector for feldspar flotation, and comparing between different types of cationic collectors like Flotigam DAT, Armo flot 64, and Aero 3030 for flotation of feldspar and mica. They revealed that Aero 3030 was more selective and preferred in feldspar and mica's mineral flotation [15,16]. Many papers on the application of collectors in the flotation of feldspar have been published. Feldspar has been traditionally separated from quartz using amine type cationic collectors and hydrofluoric acid as activator for feldspar [17].

The objective of this work is to reduce the iron oxide content of Libyan feldspar from about 2.5 % to less than 0.30%. For this purpose dry and wet high intensity magnetic separatorsas well as leaching processes were performed to obtain a feldspar product matching the industrial requirements.

2. Experimental Methods

2.1. Preparation of feldspar sample

The feldspar sample represents a feldspar ore from feldspar mines in South Libya. The feldspar sample was crushed, using a Jaw crusher, followed by Roll crusher, in closed circuit with screen, to 100 % less than the required size. Samples with different mesh sizes were prepared for beneficiation experiments.

2.2. Evaluation of feldspar sample

X-Ray Diffraction Analysis (XRD) was carried out using A Philips PW 1730 powder X-ray diffractometer with Fe-

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filtered Co (K-alpha) run at 30 kV and 20 mA. Routine chemical analysis of samples was conducted using standard method, iron oxide was determined using Atomic Absorption instrument, Analyst 200. Complete chemical analysis of samples was conformed using "Philips" X-ray fluorescence (XRF) instrument.

2.3. Magnetic separation experiments of kaolin sample

The experiments of magnetic separation of feldspar were conducted using two different techniques; dry "Carpco" magnetic separator and wet "Boxmag" magnetic separator.

2.3.1. Carpco magnetic separator

It is a high-intensity electromagnetic separator which is widely used to separate paramagnetic materials from non-magnetic ones. Adry feed of +45 micron is fed via a top-feeder. The feed material is passed through the magnetic field, and the magnetic iron oxide particles are attached to the roll and separated from the nonmagnetic feldspar stream. Three parameters could be studied which are feed rate, roll speed and field intensity.

Figure 1. Dry Carpco Magnetic Separator

2.3.2. Boxmag Rapid" LHW magnetic separator

It is a wet high intensity magnetic separator which is efficient for size -75 micron. The separating box (canister) was packed with stainless steel wool. The feed was conditioned to avoid the presence of agglomerate particles. The slurry was fed from topand the magnetic iron oxide was retained on the stainless steel wool where the non-magnetic feldspar fraction is collected at the bottom. The magnetic fraction was taken out of the separating grid by washing with water in the absence of the magnetic field. The field intensity and feed pulp density (solid to liquid ratio) were studied.

Figure 2. Boxmag High Intensity Magnetic Separator

2.4. Acid leaching of feldspar concentrates products

The acid leaching experiments were carried out in a Perspex reactor using oxalic acid as bleaching agent at different conditions of temperature, time and dosage percent from 1 to 5 kg/t. At the end of the test, the slurry was filtered and dried in a dryer at 40 ^{0}C. Then, the products were chemically analyzed.

3. Results and Discussion

3.1. Evaluation of the Feldspar Sample

3.1.1. X-ray diffraction analysis (XRD)

Figure 3 shows the XRD of the feldspar ore sample. The XRD patterns showed that feldspar is present in three forms of feldspar minerals. They are in decreasing order: Albite (NaK-feldspar ~ 40.5 %) ,Sanidine (KNa-felspar ~ 37 %) , and Analcime (Na feldspar ~ 22.5%).

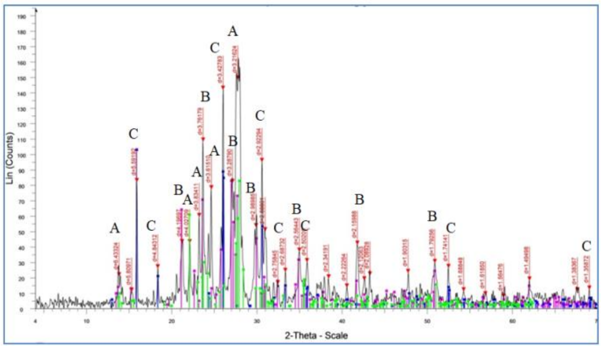

Figure 3. XRD patterns of feldspar where: A) represents albite, B) represents sanidine and C) represents analcime

3.1.2. X-ray fluorescence analysis (XRF)

Chemical analysis of the feldspar ore sample was conducted using the X-Ray Florescence (XRF), the results of which are presented in Table 1. The sample contains 21.43 % Al_2O_3 and high silica (60.38 %). The alkali contents are 7.04 % Na_2O, and 5.14 % K_2O with sum of 12.18%. The Fe_2O_3 impurities are about 2.48 %.

Table 1. Chemical analysis of the Feldspar ore sample

Component	Al_2O	SiO_2	Na_2	K_2	Mg	Ca	Fe_2O
%	21.43	58.3	7.04	5.14	0.01	0.92	2.48

3.2. Size Analysis of the Feldspar Sample

Table 2 showed the size and chemical analyses of the feldspar ore sample. It is shown that feldspar sample is crushed to about 4.0 mm and the fines below 0.125 mm are 2.84 % only. The d_{50} of the crushed ore sample is 1.5 mm.

Table 2 also indicated that the contents of silica (57.18 – 59.16 %) and iron oxide (2.4 - 2.9 %) are evenly distributed among the different size fractions.

Table 2. size and chemical analyses of the feldspar ore

Sieve,	Wt.	Cum. Wt%	SiO_2	Fe_2O_3
+4.00	1.18	1.18	57.18	2.66
+2.80	9.92	11.10	57.53	2.72
+2.00	22.52	33.62	58.24	2.77
+1.00	47.76	81.38	57.39	2.89
+0.50	10.35	91.73	57.23	2.86
+0.250	3.50	95.23	56.85	2.51
+0.125	1.93	97.16	59.16	2.42
-0.125	2.84	100	56.67	2.69
total	100			

3.3. Magnetic Separation of Feldspar Sample

Two magnetic separators are tested:-

1-Dry "Carpco" high intensity magnetic roll separator

2-Wet "Box Mag" high intensity magnetic separator

3.3.1. Carpco High Intensity Magnetic Separator

Carpco Induced-Roll magnetic separator is used as a high gradient dry magnetic separator.The magnetic separation of feldspar using "Carpco" magnetic separator is performed at maximum field intensity to get the best separation. The experiments are conductedatdifferent size fractions;(-0.5 mm +0.045),(-0.25 mm +0.045 mm) and (-0.100 mm +0.045

mm). The samples were deslimed at 0.045 mm, as the carpco magnetic separator has poor separation efficiencybelow this size, Table 3.

Table 3. Results of "Carpco" magnetic separation for different feldspar fraction sizes

Feed size, mm	Magnetic Fraction			Non Magnetic Concentrate		
	Wt, %	SiO_2 %	Fe_2O_3 %	Wt, %	SiO_2 %	Fe_2O_3 %
-0.5+0.045	47.16	60.38	2.25	52.84	59.09	1.69
-0.25+0.045	53.79	58.49	2.83	46.21	58.81	1.60
-0.1+0.045	14.63	54.61	5.56	85.37	58.11	1.58

The iron oxide impurities are reduced from 2.48 % to 1.58 %. Improvement of feldspar weight recovery and iron oxide removalis appeared with decreasing the feed size from -0.5mm to -0.1mm. Therefore,size reduction is recommended to reach a considerable degree of liberation.Figure 4 (a & b) shows the response to get highiron oxide content in the feldspar of size fraction -0.100mm +0.032 mm at different values of roll speed and feed rate at maximum field intensity. Low values of roll speed and feed rateare preferable for iron oxide removal. Slow feed rate and roll speed enables the iron oxide particles to be separated from the feldspar.

The optimum parameters of "Carpco" magnetic separator are: roll speed (80 rpm), feed rate (100 g/min) and at maximum field intensity. With these optimum parameters a feldspar concentrate of 85.3 wt % containing 1.51% $Fe_2O_3$and 59.3% SiO_2 was obtained.

It is indicated that there is no efficient reduction of iron oxide impurities with dry high intensity magnetic separation even after reduction the feed size to 0.1 mm. Thus, feldspar sample will be subjected to wet high intensity magnetic separation.

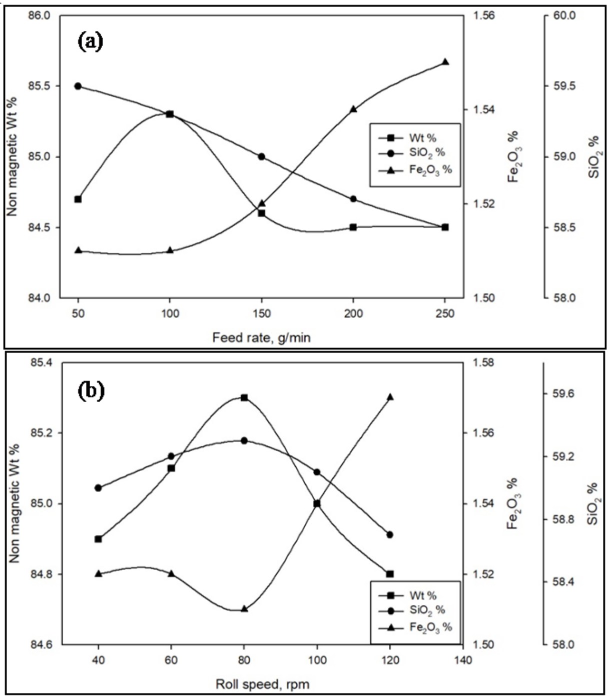

Figure 4. Effect of a) feed rate and b) roll speed on "Carpco" magnetic separation of feldspar of size fraction (-0.10+0.032 mm)

3.3.2. Wet "Box Mag" High Intensity Magnetic Separator

A "Box Mag" high intensity wet magnetic separator is used to improve the removalof iron oxide content. The experiments were performed at the highest field intensity and low solid- water ratio (of about 15 % solids). The following four size fractions were tested inorder to have the best liberation size: (-0.1+0mm), (- % 0.075 + 0 mm), (- % 0.045 + 0 mm) and (-0.032+0 mm).

The results in Figure 5 show the effect of feldspar size fraction on the magnetic separation efficiency. For the feldspar size fraction -0.100 + 0.00 mm; the iron oxide content of the non magnetic concentrate is decreased from 2.48 % in the feed sample to 0.94 % in non-magnetic concentrate. However, the yield of product is low (40 wt. %) due to that the majority of sample (~60 wt. %) is reported as magnetic fraction, probably due to the feed sample is still not liberated enough. The same trend is found with the size fraction -0.0750 + 0.00 mm where the Fe_2O_3 content is reduced to 0.73% with a poor feldspar yield of ~ 30 wt. %.

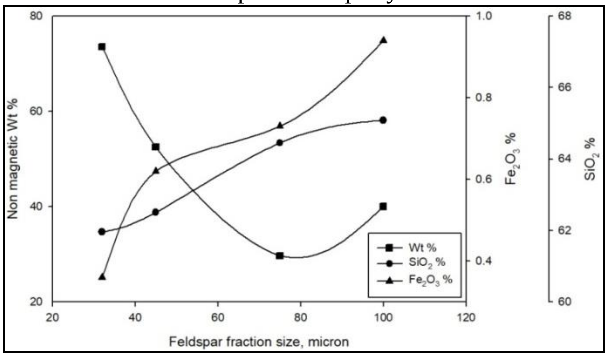

Figure 5. Results of "Box Mag" magnetic separation at different feldspar size fractions

It seems that reduction of iron content is related to the degree of grinding (i.e., liberation degree). This means that further grinding of the feldspar ore sample, is a must that leads to betterliberation degree between iron oxide and feldspar minerals, and in turn, may give concentrates of higher yield and grade. So, the reduction of feed size to less than 0.045 mm leads to a significant reduction of the iron oxide contentto 0.62 % with a moderate yield concentrate (52.50 %).

On the other hand, the results indicated that the magnetic separation of feldspar at size fraction less than 0.032 mm yields the lowest iron oxide contents, where a concentrate with 0.36 %$Fe_2O_3$and high yield (~73.5 % by weight) is produced. This is because the degree of liberation between feldspar and iron oxide reached its maximum value at size less than 32 micron.

Figure 6(a&b) shows the responses of feldspar concentrate yield and iron oxide content at size fraction -0.032 mm at different values of feed pulp density and matrix loading capacity. Moderate values of feed pulp density and matrix loading capacity lead to efficient iron oxide removal. High values of feed pulp density cause crowding of the particles inside the Boxmag canister and hence decrease the iron oxide removal from feldspar mineral. Also, high matrix loading capacity decreased the feldspar concentrate yield to 71%.

The optimum parameters of "Boxmag" magnetic separator are: feed pulp density (15%) and matrix loading capacity (20%) andat maximum field intensity. At these optimum parameters a feldspar concentrate yield of 74.5 wt % containing 0.28% Fe_2O_3 and 62.3% SiO_2 was obtained.

These results suggested that application of the wet high intensity magnetic separation is essential for such type of feldspar ore to produce salable product.

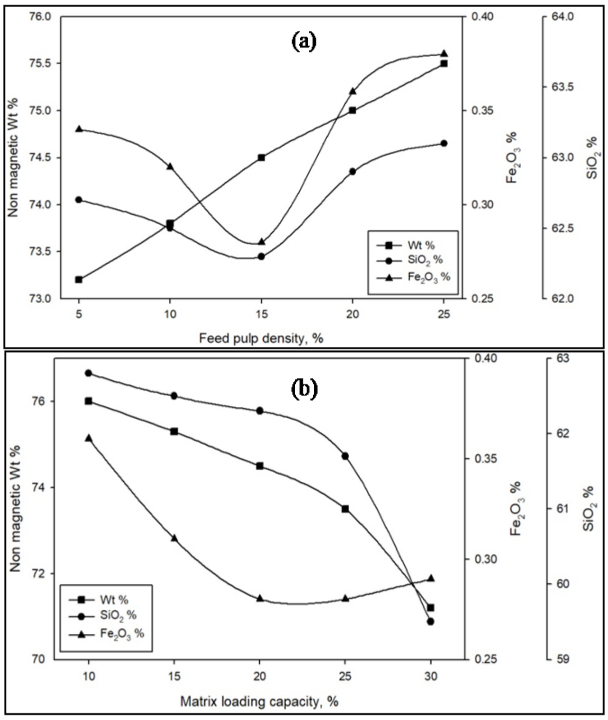

Figure 6. Effect of a) feed pulp density % and b) matrix loading capacity on boxmag magnetic separation of feldspar of size fraction (-0.032 mm)

3.3.3. Characterization of the nonmagnetic feldspar concentrate

The XRF analysis of the boxmag non-magnetic concentrate of fraction size -0.032 mm displays that it contains 0.28 % Fe_2O_3, 21.05 % Al_2O_3, 54.75 % SiO_2, 11.56 % Na_2O and 6.68 % K_2O. The total alkali's (Na_2O + K_2O) became 18.24 % instead of 12.18 % in the ROM feldspar sample.

Table 4. XRF analysis of non-magnetic feldspar product compared to original sample

%	Original ROM	Non-Mag. Concn.
Na_2O	7.04	11.56
K_2O	5.14	6.68
Al_2O_3	21.43	21.05
SiO_2	58.38	62.30
Fe_2O_3	2.48	0.28
CaO	0.92	1.18

In the mean time, the XRD diffraction of the "Boxmag" non-magnetic concentrate contains mainly feldspar minerals which are present as Albite, Sanidine and Analcine minerals, Figure 7.

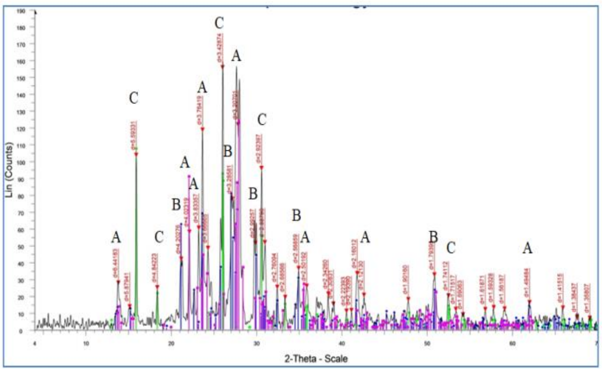

Figure 7. XRD analysis of the non-magnetic feldspar product where A) represents albite, B) represents sanidine and C) represents analcime

3.4. Bleaching of the Cleaned Non-Magnetic Product (of -0.032 mm)

Bleaching experiments are also conducted, in a trail to improve the grade of the produced feldspar concentrate of 0.032 mm in size, which obtained after treatment with "Boxmag" magnetic separation.

Using oxalic acid as leaching agent, the experiments were performed for 3 hrs at 90 ^oC at pH 3.7. Addition of high amounts of oxalic acid (5-20 kg/ton) did not lead to any significant reduction of iron contents. However, addition of very large amount of oxalic acid (~30 kg/ton) decreased the iron oxide to from about 0.28 %Fe_2O_3 to about 0.19 %Fe_2O_3 only (Table 6).

However using diluted HCl as a leaching agent, leads to the highest iron oxide removal (0.07 % Fe_2O_3) obtained so far.

Table 5. Results of bleaching of the non-magnetic "Boxmag" product of feed size - 0.032 mm

Bleaching Agent	Final Product	
	Wt., %	Fe_2O_3, %
Oxalic acid (30 kg/ton)	70	0.19
HCl (diluted)	70	0.07
Feed (Boxmag non magnetic concentrate)	73.56	0.28

Table 6. XRF analysis of bleached product in comparison to the original feldspar sample

%	Original ROM	Bleached product
Na_2O	7.04	5.08
MgO	0.01	0.09
Al_2O_3	21.43	15.90
SiO_2	58.38	63.19
CaO	0.92	0.34
K_2O	5.14	11.65
Fe_2O_3	2.48	0.07

3.5. Characteristics of the bleached feldspar products

Complete XRF analysis for thefeldspar product bleached with HCl is shown in Table 7.Interestingly, the chemical analysis showed that the bleached product has lower contents of alumina (15.9 % Al_2O_3) and Na_2O (5.08 %) with higher content of silica (~ 63.19 % SiO_2) when compared to the original feldspar sample. At the same time, the content of K_2O increased from about 5.14 % in the original sample to 11.65 % K_2O in the bleached product. On the other hand, the bleached product became containing potassium feldspar more than sodium feldspar as in the original sample. It seems that the mineral acid (HCl) solublized some of the sodium feldspar minerals present in the sample.

In the mean time, the results of XRD analysis of the feldspar bleached product showed, interestingly, that the bleached feldspar product contains only feldspar minerals as Albite and Sanidine minerals, Figure 8. Feldspar phase presents as Analcime [$Na_{14.96}AL_{15.36}O_{96}(H_2O)_{16}$] was disappeared. This means that such phase is dissolved by HCl. This may explain the significant reduction of Na_2O (5.08%) and alumina (Al_2O_3 15.90 %) and in turn the content of K_2O and SiO_2 are raised to 11.65 % and 63.19 % respectively.

Comparing of the distribution of different phases of feldspar minerals in different products to that of the original ROM sample is shown in Table 7 based on the XRD analyses in Figures 3, 7 and 8. These results confirm the disappearance of Analcimephase in the bleached product due to its dissolution as it mainly contains high Na_2O and the only phase containing H_2O in its structure.

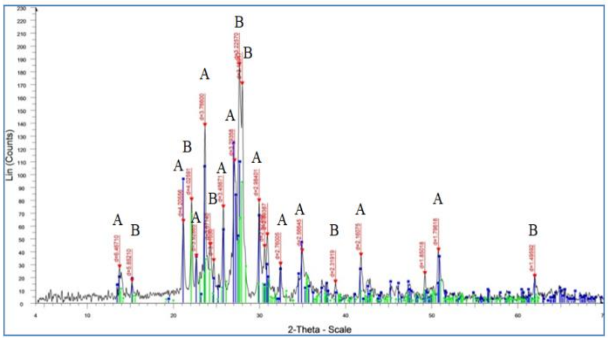

Figure 8. XRD analysis of the bleached feldspar product where A) represents sanidine and B) represents albite

Although such high grade bleached product, of very low iron oxide content (0.07 %Fe_2O_3), represents a great achievement from the scientific and technical points of views, However, using mineral acids (such as HCl) in bleaching process may cause many environmental problems in addition to the problems of corrosion of differentmetallic equipments. Moreover, using such mineral acid will add extra cost for treatment of the used water (during the bleaching process) before drainage to the sewage. So, it is not recommended to conduct bleaching process (due to its technical, environmental and economic aspects), and production of final feldspar with about 0.28 % can represent the final feldspar concentrate.

Table 7. Distribution of feldspar minerals in feldspar products based on semi-quantitative XRD

	ROM	Non-mag. product	Bleached product
Albite($K_{0.22}Na_{0.78}$)($AlSi_3O_8$)	40.6	50.6	45.1
Sanidine[$K_{0.65}Na_{0.35}AlSi_3O_8$]	37.0	30	54.9
Analcime[$Na_{14.96}$ $AL_{15.36}Si_{32.64}$ $O_{96}(H_2O)_{16}$]	22.5	19.4	0

Conclusions

This work aims at upgrading of Libyan feldspar sample.The feldspar ore sample contains high (2.48 %Fe_2O_3)in comparison to the maximum iron oxide which should not exceed 0.3 %to be suitable for industrial applications. Appling "Carpco" dry high intensity magnetic separator decreased the Fe_2O_3 content to 1.15 %.This technique is not effective and in turn wet high intensity magnetic separator (WHIMS)is used. "Boxmag" high intensity magnetic separator gave more promising results. Theproduct grade was found to be related to the size reduction degree. So, successive reduction of feed size to -0.075 mm yielded a product of 0.73 %Fe_2O_3but at the expense of yield (~41.5 wt%) of the product. The best grade for feldspar sample with WHIMS was obtained size fraction -0.032 mm where the iron oxide content decreased to 0.28 % Fe_2O_3 with yield of about 74.5 wt %.

Leaching of non-magnetic product usingeither oxalicacid or HCl was also tested. Oxalic acid gave a concentrate of 0.19 % Fe_2O_3. Leaching using HCl leads to further reduction in iron oxide to 0.07 %Fe_2O_3. However, such bleaching process with HCl indicated that part of the feldspar minerals in the form of Analcine (Na feldspar containing water) was disappeared from the XRD patterns.

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