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PURIFICATION OF FELDSPAR FROM IRON OXIDE IMPURITIES USING NITRIC ACID IN PRESENCE OF AMMONIUM BIUORIDE

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ABSTRACT

The main objective of this study is focused on the mechanical preparation of Algerian crude feldspar with a view to extract the information about the comminution and the coloring iron rate distribution, on the one hand, and to remove the iron oxides, on the other hand, main impurities of crude feldspar by magnetic separation, flotation and more particularly by chemical leaching. In this process, the nitric acid and dithionite as reducing agent are used to replace sulfuric acid whose the application tests have yielded negative results. Research results shows that using a nitric acid in a reducing medium as ammonium bifluoride (HF, NH_4F) process is effective and pushes the degree of iron oxides (Fe_2O_3) removal around 0.01\%. The hydro-metallurgical process applied especially to size fractions [-0.8 + 0.1] mm intended for the glass industry previously undergo a high intensity magnetic field reduces iron levels up to 0.054 \% with a yields in terms of weight of 58.32\% compared to those of all comers.

Keywords: Leaching; magnetic separation; Flotation; nitric acid; ammonium bifluoride.

1. INTRODUCTION

Feldspars are essential constituents of igneous rocks, plutonic or extrusive (59.5% of the minerals of magmaticrocks). Their chemical composition varies with the nature of the rocks : acidic rocks contain alkali feldspar, intermediaterocks of alkali feldspar and plagioclase and basic rocks calcic plagioclase. Feldspars belong to the tectosilicates family. Analysis of feldspars allows them as more or less homogeneous mixtures of three basic components :$KAlSi_3O_8$: Orthose or Sanidine ou microcline, $NaAlSi_3O_8$: Albite (Ab) and $CaAl_2Si_2O_8$: Anorthite[1–6]. They generally occur together with quartz, mica, iron oxides, rutile and hornblende. Basically, the two properties which make feldspars useful for down stream industries are their alkali and alumina content. Feldspars play an important role as fluxing agents in ceramics and glass applications, and are also used as functional fillers in the paint, plastic, rubber and adhesive industries and involved in interesting economical and environmental processes, especially for removing dissolved metals from waste water [1,7–12]. However, the presence of iron oxides in feldspar is not desirable as it imparts color to the final product, and therefore, iron content has to be kept below a certain value, 0.10% for glass making and 0.07-0.08% for ceramics [8,13]. Several conventional enrichment techniques, such as hand sorting, comminution, magnetic separation and flotation, are employed to remove coloring impurities including iron bearing minerals from feldspar [4,14–18]. These techniques are used for the treatment of particles larger than 40 microns. Other technics are applied for the ultra-fine particles treatment (slimes), less than 40 micron such as flocculation [19]. The mechanical properties of feldspathic rock have a direct impact on weight yields for different size fractions for purification and marketing; therefore they affect the selection and sizing of comminution and screening equipment. Any method of treating requires purification treatments to improve the quality demanded by customers. Studies supported in this work are mainly based, first, on the mechanical preparation of the raw feldspar, to produce the main grain-size slices for the glass industry and on the other hand, the elimination of the iron oxides main impurities of crude feldspar by three methods: magnetic separation, flotation and chemical leaching by nitric acid in presence of ammonium bifluoride as reducing medium in order to obtain a whitening product for the glass industry and ceramics.

1.1. Materials and methods

A representative sample of the Ain Barbar deposit located in the east of Algeria received in the ENP laboratory, whose mineralogical composition and chemical analysis are presented in Table 1 and Table 2. The sample was previously crushed in a jaw crusher followed by grinding in an impact mill of Ain Barbar unit. In this work, we aim to produce the size fraction [-0.8+0.1] mm for the glass. In view of the glass market application, we screened the sample E0 in [0.8 mm] size, were the fines [-0.1 mm] are extracted by sieving to avoid over-grinding. Grinding and sieving are carried out as shown on the Figure 1. The desired results of this diagram are the production of three fractions sizes [-0.8+0.4], [-0.4+0.25] and [-0.250] mm taken as the basis for subsequent recovery treatments as shown on the Figure 1. The sample grain size distribution is shown in table below Table 3.

In a second step, we conducted the high magnetic intensity separation experiments to reduce the iron oxides (Fe_2O_3) content in the feldspars. This is achieved by the magnetic separation of the different slices obtenaid using the previous diagram (Figure 1) in two passes on the intensive brand CARPCO magnetic separator. The next step is to treat the particles from magnetic separation by flotation ; the other part will be leached. The requirements for effective flotation is to achieve a grinding thus enabling to release a maximum quantity of minerals ; and a suitable release of the particles to float. In some cases, it is necessary to desliming pulp before the float and to avoid that the recovery of the mineral particles surfaces. In our cases, dry milling was performed in a ceramic ball mill to avoid iron contamination, the milling time and the grinding massare determined on the basis of tests. For this type of test, the grinding time is estimated about 8min with a mass gainof 300 g. After grinding, we conducted a screening to remove particulates, having a size of less than 0.04 mm. The purpose of the grinding is to achieve the size less than 0.250 mm to release the most of the mixed iron oxide particles.This operation is controlled by a sieve whose opening is smaller -0.250 mm as shown on the Figure 2. At the end of the operation desliming refusals and passersby were dried and weighed. The operating mode used during the flotation is as follows: a pulp containing between 5 and 50% solids mixed with water, the pulp is stirred in a conditioning tank to ensure the medium homogeneity. In a first time, AP 801 and AP 840 dispersants are added to avoid any kind of agglomeration before floating the concentrate

(feldspar), then the collector Dodycelamine agent and hydrofluoric acid are added to modify the feldspar particles surface to be driven to the surface by the air bubbles. This process is repeated 3 times and the concentrates were filtered washed and dried as shown on   the Figure 3.

Table 1. Mineralogical composition of sample

Size fraction (mm)	Pourcentage (%)
Orthose	52.66
Quartz	37.42
Phyllites	7.02
Albites	1.70
Anorthite	0.58
Iron oxides	0.53

Table 2. Chemical analysis of feldspar sample from Ain Barbar deposit

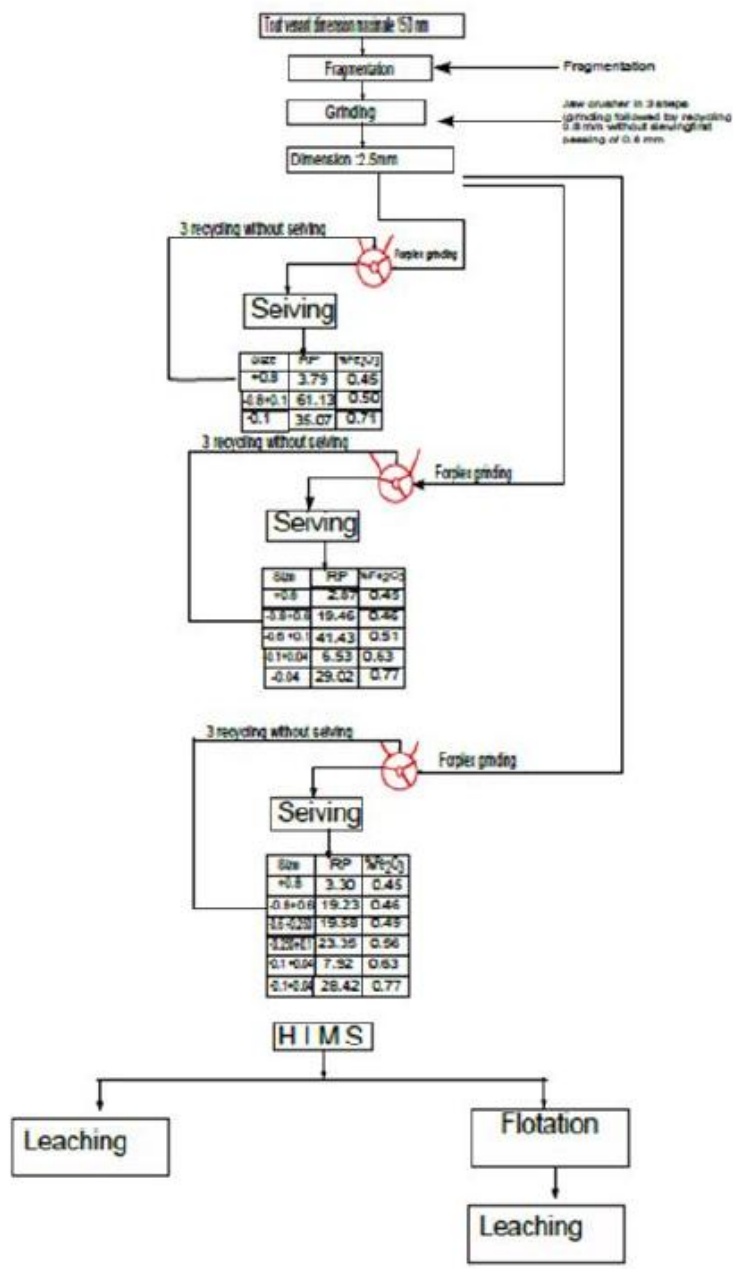| Size fraction (mm)	Pourcentage (%)
SiO_2	75
Al_2O_3	12.45
Fe_2O_3	0.57
TiO_2	<0.05
CaO	0.30
MgO	0.20
Na_2O	0.30
K_2O	<0.20
MnO	<0.20
P.F	0.70
P_2O_5	0.20

Fig.1. Illustration of feldspar flow-sheet leaching highlighting the screening effect

Table 3. Grain size distribution of sample

Size fraction (mm)	weight yield (Rp %)	Iron oxides (Fe_2O_3 %)
[+0.8]	3.30	0.45
[-0.8 +0.6]	19.23	0.46
[-0.6 +0.250]	19.58	0.49
[-0.250 +0.1]	23.35	0.56
[-0.1 +0.04]	7.92	0.63
[-0.4]	28.42	0.77
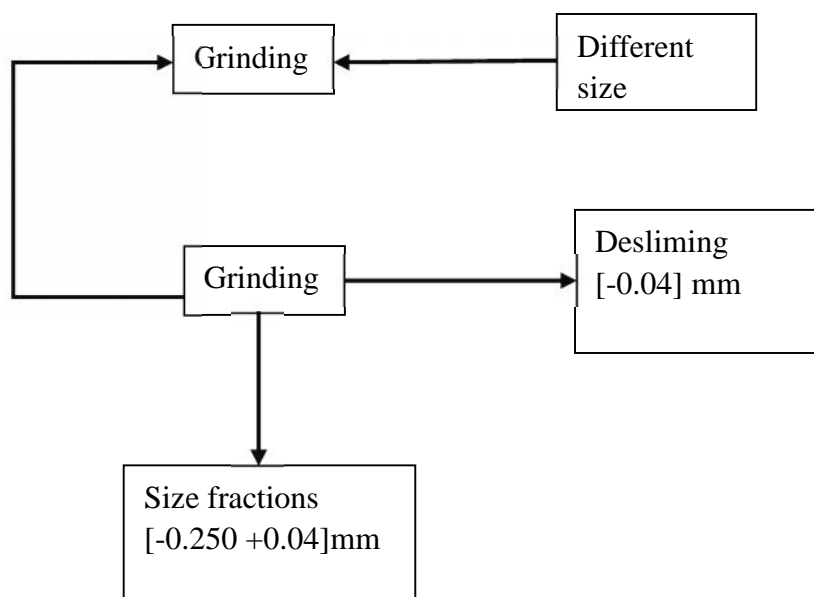
Fig.2. Illustrative diagram for sample preparation to the flotation process
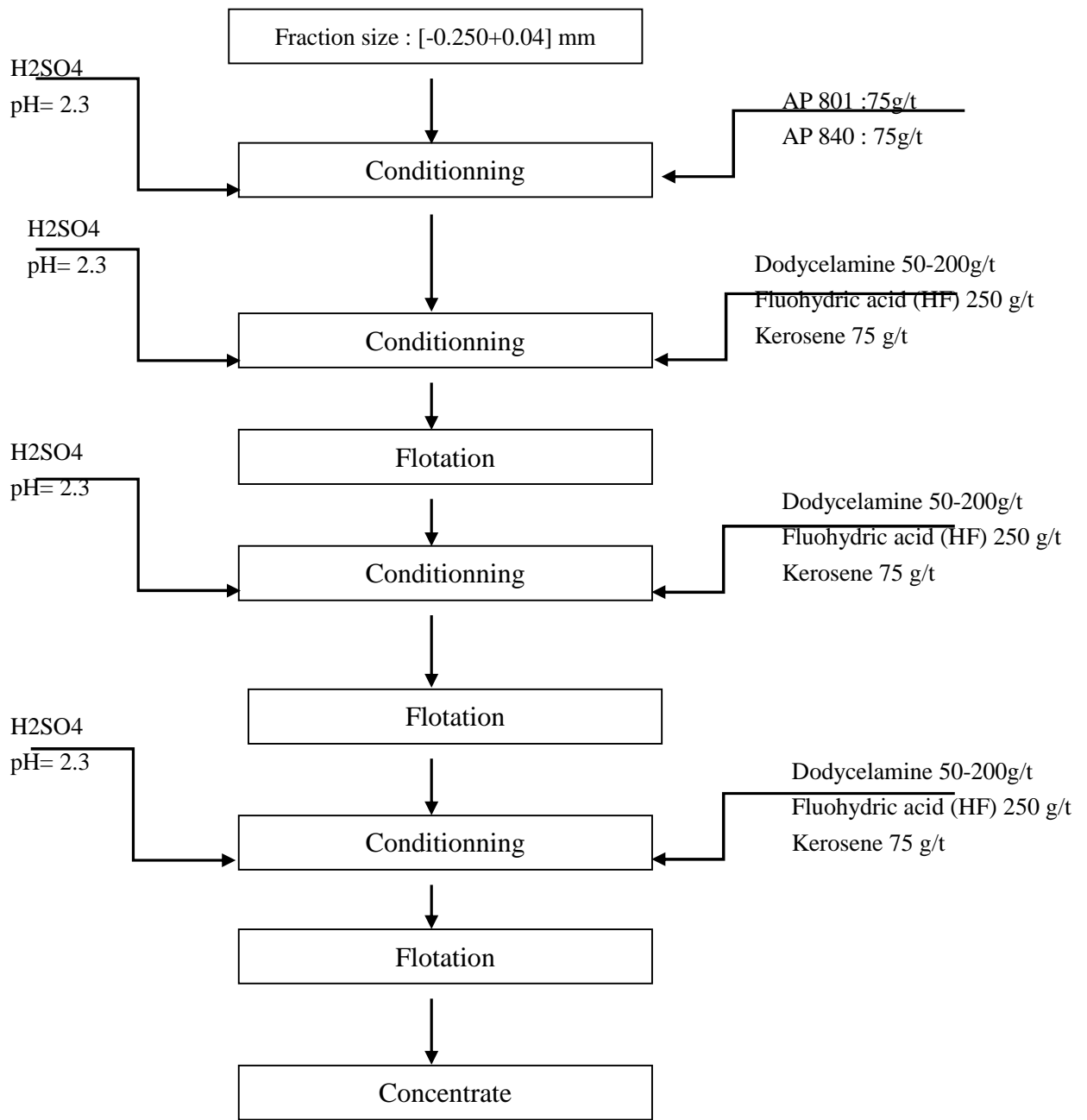
Fig.3. Flotation flowheet highlighting the feldspar purification steps

2. RESULTS AND DISCUSSION

2.1 The feldspar preparation scheme

Feldspar fragments having a maximum dimension of [150] mm are first crushed with a mass to be able to introduce it in a laboratory jaw crusher, whose maximum adjustable opening is [50] mm. First, a sample is inserted into the jawcrusher. The crushed product is recycled again without sieving after reducing the outlet opening of the crusher. The[0.8] mm sieved residues was crushed in a FORPLEX mill to reduce it to [0.8] mm, the [+0.8] mm go over 3 times in FORPLEX mill. Three samples were selected separately and carefully mixed, the results of crushing and grinding process are shown on the following figure Figure 1. The chemical analysis of different particles was performed for all threes creening stages, then the results of grinding and screening show that the level of fines [-0.04] mm decreases as the screening level recycling increase. This results can be explained by the fact that the weaker particles crumble on the first pass on the grinder whereas the harder produce less fines [20]. The crushing model applied in this study responds to market demand. The knowledge of the physical properties of the size fractions required by the customers, helps to choose, on the basis of the results of this study and the quality and the types of equipment most adapted to the envisagedproduction unit.

2.2. High intensity magnetic separation to remove iron oxides

In order to remove iron oxide particles from feldspar, a high intensity magnetic separation has been applied. For that purpose, different slices fractions subsequently prepared by the mechanical diagram (Figure 1) have undergone two passes on an intensive magnetic laboratory separator by varying the magnetic field. The obtained results are shown on the Figure 4. It should be noted that the magnetic purification reduces the iron oxide content of feldspar to 0.34 with weight yield of 99.62% relative to the all comers for the slices fractions [-0.8 + 0.1],[-0.6 + 0.1], [-0.6 + 0.250] and [-0.250 + 0.1] mm respectively, except for the slice fraction [-0.8 +0.1] mm where the iron oxide content is reduced from 0.50 to 0.39%. The results obtained are not effcient, this is explained by the fact, on the one hand, that a large irregular grains produce particles with different magnetic susceptibilities and different particle size distributions, resulting in a slight decrease of the iron oxide content[21], and secondly, this is because the degree of release increases as the size fraction decreases [22]. For a better

release of iron oxides generated by a finer grinding, there is a better treatment for size fractions greater than 0.1 [mm]. For particle size fractions [-0.1] mm, removal of iron oxides is needless, caused by the ultra-fine particles that the magnetic field strength is no longer able to attracted and retain them on the drum machine [23] (Figure 4). These ferrous dust mixed with those of finely ground feldspar, which cover the receivable oxides, they reduce the driving forces generated by the magnetic field on these oxides, however favorable to their extraction feldspar dimensions [24–26]. Purification of the particle size range [-0.8 +0.1] mm by high gradient or high intensity magnetic field resulted in only an iron residual of 0.34 % and 0.39 % values very close who do not justify the high investment in the use of high gradient separator. For products used in the ceramic industry, the high intensity magnetic purification gives iron oxide contents of about 0.34% and 0.31% for size fractions [-0.2+ 0.080] and [-0.25+0.1] mm respectively. It is best to treat several size fractions separately and then mix them to reconstruct the portion that meets the required criteria for use.
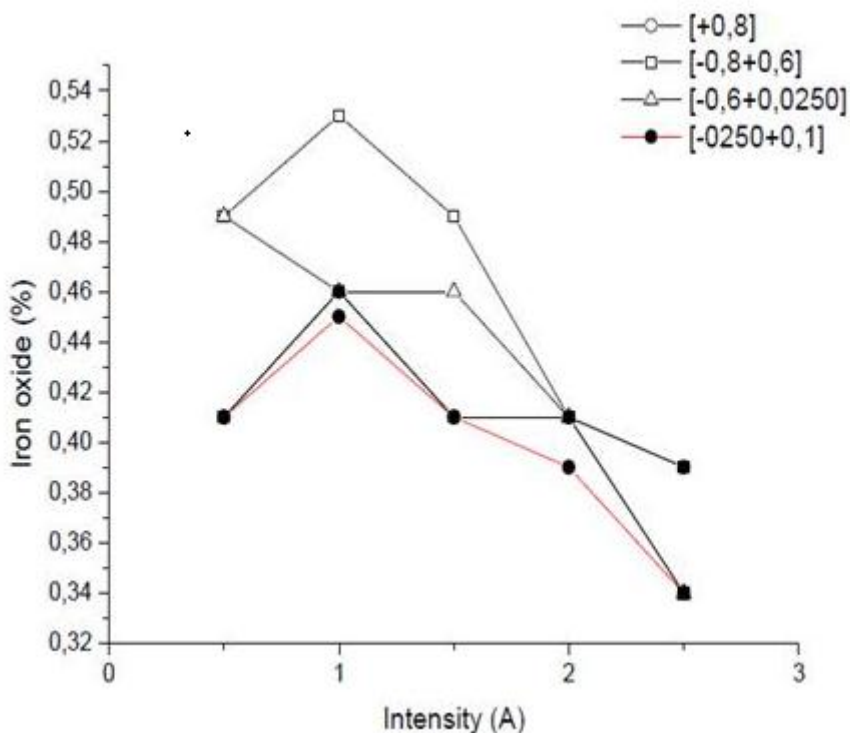

Fig.4. Iron oxides versus high intensity magnetic highlight the iron oxides rates as function of different slices
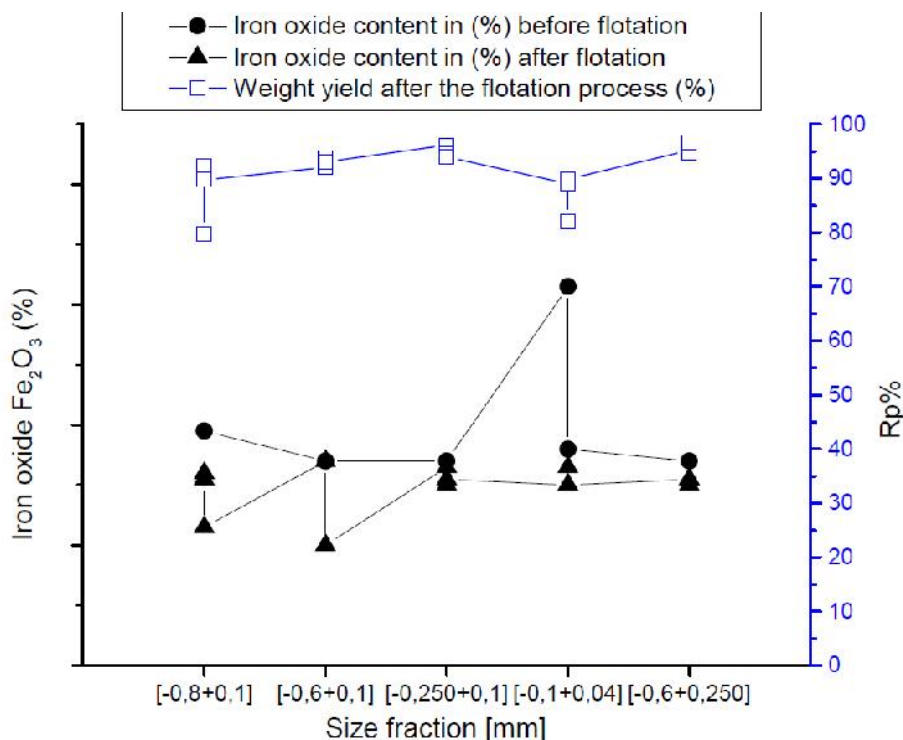
2.3 Flotation purification

On the basis of the results obtained during the magnetic separation process, a part of the size fractions treated by magnetic separation will be treated by flotation followed by leaching, the other part being treated directly by leaching. In this section, flotation requires extensive grinding up to 80 micron as described above. The weight average yield is 60.49 % for the group [-0.8 + 0.1] mm and 64.80 % for the fraction [-0.6 + 0.1] mm. The seresults are very close to those of the BRGM study. Throughout the obtained results, whatever the slices fractions treated by magnetic separation, the purification is limited to 0.34% of iron oxide, a value already encountered in the BRGM study, whose recovery rates exceed 99%. The flotation process applied for the size fractions treated with high intensity magnetic separation improves the purification by lowering Fe_2O_3 content for about one third for a collector consumption 0.05 kg/t to 0,200Kg/t respectively. The fine particles have been removed from upper size fractions [-0.8+ 0.1] mm and [-0.6 + 0.1] mm, where iron oxides are most concentrated, so that they are less rich in iron oxides. The contents of these oxides in the rough particles are released by grinding at [0.250] mm and removed by flotation. The final iron oxides (Fe_2O_3) content of the concentrates is lower than in the case of thinner fractions flotation [-0.6 +0.25] mm, [-0.25 + 0.1] mm   and [0.04 +-0.1] mm respectively (Figure 5 and Figure 6).The final content of iron oxide removed by magnetic purification and flotation under optimum conditions will be as follows : with grinding of feldspar up to [0.8] mm, where fractions less than [-0.1] mm are extracted, gives a yield of 54.85% with iron oxide rate of 0.23%. For fractions size [-0.6 + 0.1] mm subtracted from all comers milled to [0.8] mm, give yields in terms of weight of 38.56% with an iron oxide content of 0, 20%, shows an iron oxide content decrease from 40% to 50%. The uncrushed size fractions to [0.250] mm and purified by magnetic separation, flotation gave weight yields very similar to those given in the two previous cases with iron oxides content ranging between 0.31% and 0.34%. The lower level of this impurity is only 0.18%. This result is explained by the fact that the non-grinding does not release encrusted oxides (mixed grains) in the feldspar grains. From flotation practice, it is known that the working conditions especially collectors and froth depends significantly on the solids it contains. The presence of particles affects substantially the foam stability by reducing coalescence bubbles. The larger particles are caused to break the thin films easily because of

their greater mass, causing the falling-in and destabilization of reagent [28,29], which results in a low removal of iron oxides impurities. For other thinner size fractions, the final content of iron oxide after flotation does not go lower than 30% with a drop   about 11% for [-0.6 + 0.25] mm and [-0.25 +0.1] mm fractions and 50% for [-0.1 + 0.04]mm fractions. We note that the content of iron oxides is least important in the upper size fractions, the more go down to the fines were the content more increases ; this is due to the oxides physical characteristics, in particular oxides friability and nature connection which is associated with mineral feldspar, where it is finely disseminated. The size fraction [-0.6+ 0.1] mm contains more fines than [-0.6 0250]mm, hence the explanation that the Fe_2O_3 content of [-0.6 + 0.1] mm is higher (0.51%) than [-0.6 0250] mm (0.49%), then the iron oxides are concentrates in the fines [-0250 +0.1] mm. The levels content of Fe_2O_3 between 0.2% and 0.23% with rweight yields of 38.56% and 54.85% respectively is a commercial feldspar intended for the ceramics industry for high consumption.
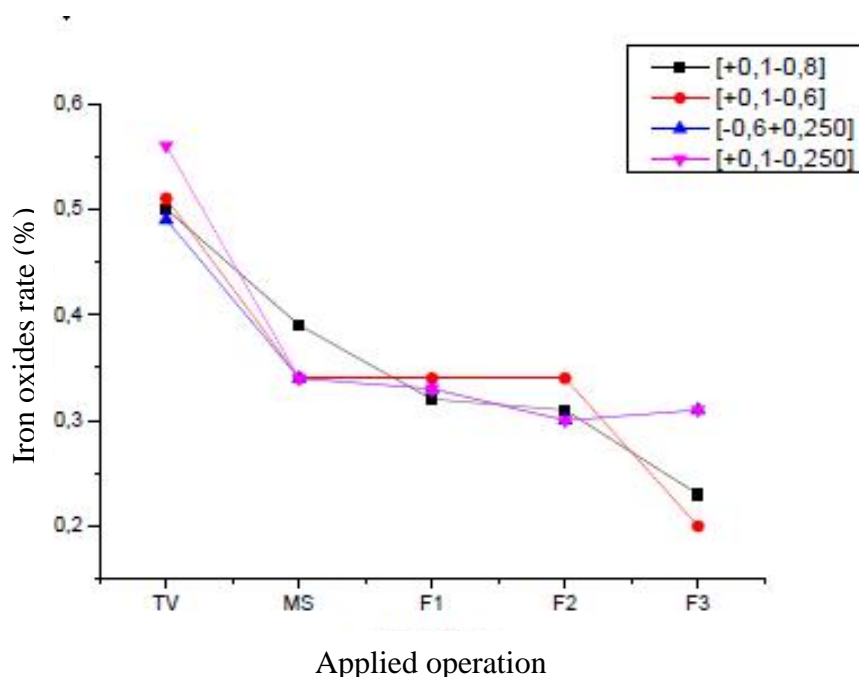

Fig.5. Iron oxides rate vs applied operation for different slices (TV: All comers, MS: magnetic separation, F: flotation process)

Fig.6. Iron oxides Fe$_2$O$_3$ rate vs size fraction before and after flotation process for different slices and obtained weight yield for applied process

2.4. Leaching purification

In order to further eliminate iron oxide particles, a hydro-metallurgical process is applied (the leaching process). It consists of the selective dissolution of the metals present in the feldspar ore by a suitable reagent in aqueous solution. In our case, ferric iron oxides are reduced to ferrous iron. The experimental protocol consists of a beaker equipped with a heat-insulating lid and limiting the evaporation of the etching solution. The lid has three holes, one for the acid injection, the second for the thermometer attachment for the temperature measuring of the pulp and the third one for the refrigerant tube location of the cooling gas which emerges under the evaporation of the etching solution. The etching solution stirring and heating and the ore mixture carried out using magnetic bars and a heating plate as shown on the Figure 7.
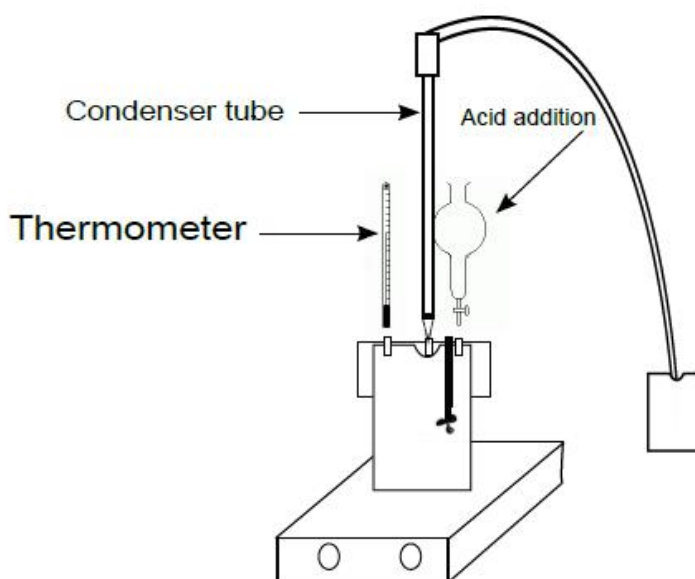
Fig.7. Experimental protocol of the leaching purification scheme for feldspar ore

2.4.1 Effect of temperature on the iron oxide reduction

In order to study the temperature effect on the iron oxides removal, the temperature varies from 40 °C to 90 °C, The temperature not exceeding 90 °C, due to intensive evaporation beyond this temperature, the nitric acid (HNO_3) amount is fixed at 5Kg/t, 7Kg/t and 10 Kg/t respectively. The initial rate of iron oxide was 0.5%, higher rate obtained by subsequent processes (magnetic separation and flotation). The solid/liquid ratio is fixed at 50%, the stirring time is 30 minutes and a quantity of ammonium bifluoride is 3 kg/ton. The sample weight is 50g. Through the obtained results, it should be noted that the higher the temperature, the greater the effciency of iron oxides Fe_2O_3 removal as shown on the Figure 8, which implies that the high temperature (90 °C) has a considerable effect on leaching performance. This is because the high recovery of oxides at higher temperatures is due to the increase in reaction and diffusion rates of reactants and products.[30–33]. The iron oxide content is lowered from 0.51% to 0.36% for a nitric acid consumption of 10kg/t. According to the test results, we noticed that the chemical leaching color varies little, especially with the nakedeye. The change in temperature evolves well in the sense of iron oxide content reduction, provides no significant change in the whiteness, however it should be noted that the last attempt give a more pronounced result but low though. Referring to these guidance tests, it appears that we

should increase the nitric acid consumption per ton of ore processed. The reasons that prevail for this measure are two fold : a temperature not exceeding 90 °C, due to intensive evaporation beyond this temperature and optimal pulp density to promote good homogenization of solid particles by magnetic stirring.
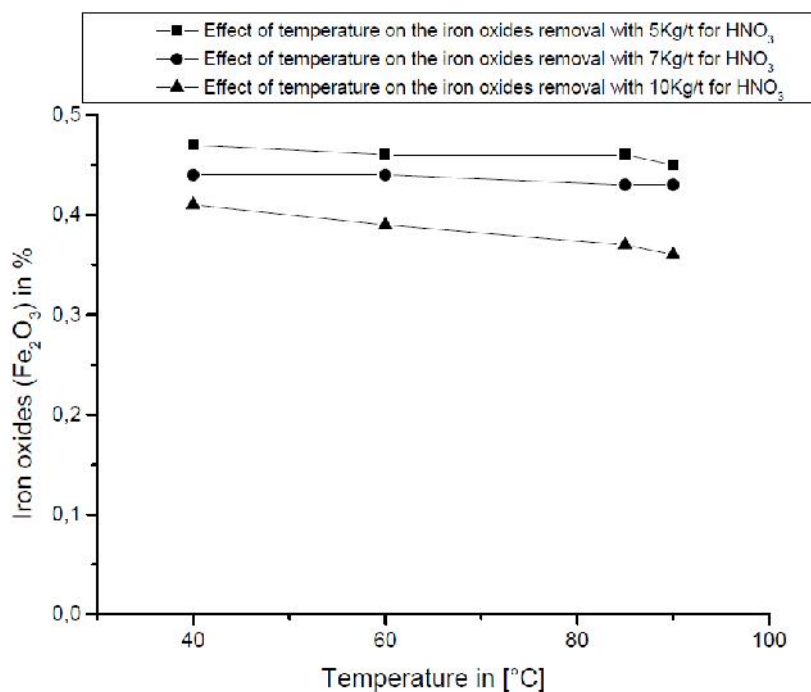

Fig.8. Iron oxides rate vs temperature highlighting the e_ect of nitric acid concentration.

2.4.2. Effect of nitric acid (HNO₃) amount

To study the influence of the nitric acid (HNO$_3$) concentration on the iron oxides removal, the operating conditions were fixed as follows : a solid/liquid mass ratio (50/50) in the presence of ammonium chloride (NH$_4$HF) at atemperature of 90 °C and the time stirring is fixed at 45 min. As shown on the Figure 9, the removal rate of iron oxides increases with increasing nitric acid concentration. However, the first part of the curve (accelerated slope) corresponds to the dissolution of the fine and free oxides, then a less rapid part corresponding to the dissolution of the oxides for mixed grains. This can be explained by the fact that the most dilute acid has the greatest activity, so that the reaction rate is faster at the initial stage, and then the acid activity decreases progressively with increasing concentrations, so that the slope becomes less accelerated [32–34]. According to these leaching tests, the trend towards a gradual reduction of the iron oxide content is maintained with the remark that the iron oxides

contents of the samples are very close and follow a descending order. It should also be noted that the content of 0.06% has been reached in iron oxide and the color change of feldspars is remarkable.
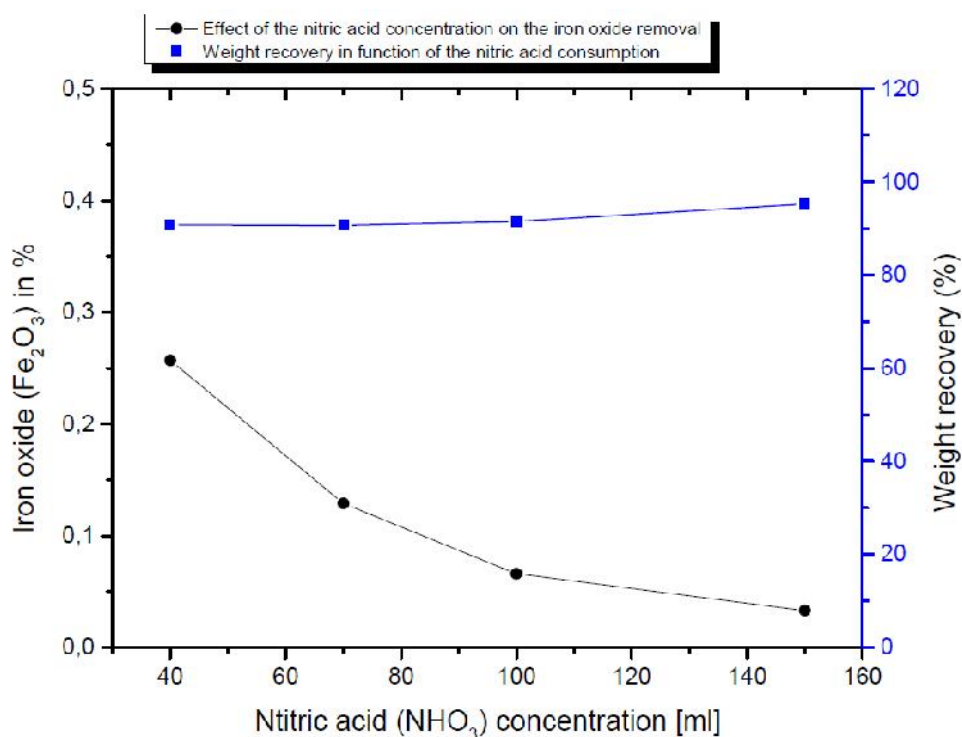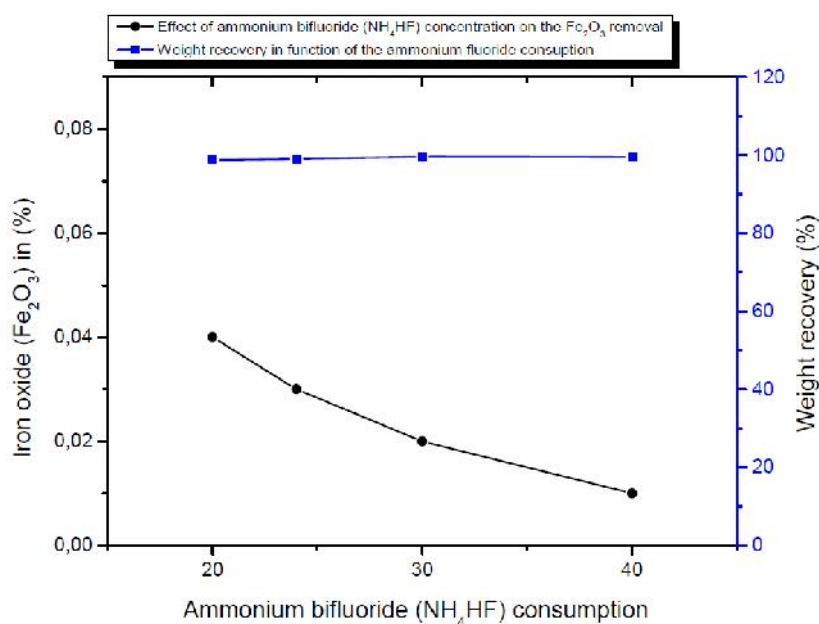

Fig.9. Iron oxides rate removal vs nitric acid concentration.

2.4.3. Effect of the consumption of reactant (NH₄HF)

In order to study the effect of ammonium bicarbonate concentration (NH_4HF) on the iron oxide particles removal, the nitric acid concentration (HNO_3) is fixed at 10kg, a 50/50 solid/liquid mass ratio and a stirring time of 45min in the presence of bifluorure ammonium (NH4HF) at 90 °C. As shown on the Figure 10, the rate of particle oxides(Fe2O3 removal increases with the increase in bifluorure ammonium concentration. However, the result of 0.04% of iron oxides (Fe_2O_3) which characterizes a good industrial feldspar does not require any additional purification, except in very special cases such as high enamelling, quality or manufacture of crystals. The variation in the amount of ammonium bifluoride results in a continuous decrease in the Fe_2O_3 content, reaching a value corresponding to a high quality feldspar sought by enameling. To reach a level of 0.06% of iron oxide, it was necessary to use 40 kg of ammonium bifluoride and 280 kg/t of nitric acid per tonne of feldspar without prior

treatment, whereas for an already treated feldspar with high intensity magnetic purification yields an iron oxide residue equivalent to 0.066%. This shows that, by gradually adding the two reagents, the amount of nitric acid could be reduced to 100 kg/t whereas that of ammonium bifluoride could bereduced to 20 kg. It appears that nitric acid can not be used alone as a leaching and that it should be mixed with another reagent in order to improve the effciency of the iron oxides removal.


Fig.10. Iron oxides rate removal vs the Bifluorure ammunium concentration as reactant.

2.4.4. Effect of particle size on the iron oxides removal

In order to investigate, the effects of particle sizes on iron oxides removal, the nitric acid (HNO_3) and bifluoride ammonium (NH_4HF) concentration are fixed at 10 kg/t and 20 kg/t respectively, solid/liquid ratio of 50/50 %, stirring time of 40 min and a solution temperature of 90 °C. The results are shown on the Figure 11. The resulting iron levels either by magnetic separation or flotation for different size fractions remains low for coarse fractions ; however the iron oxides recovery content is more important for coarse fractions and decrease as the dimensional particles become finer. The iron level decreases for size fractions greater than 0.1 mm. The lower slices <0.1 mm containing significant iron content levels. It is recognized that the increase of the particle size lead to a decrease of the reaction surface and the interfaces of the liquid-solid reactants for the fine particles are much larger than that for the coarse particles

and with time contact [32,33,34]. This is probably related to the physical characteristics of feldspar particles, including fine pure iron oxide grains or associated with other minerals that are coated with fine particles making it difficult contact between the acid and this iron oxide. In the following, we defined the subsequent parameters for removing iron oxide particles of different sizes for industrial glassware as well as for potters. For this purpose, the liquid/solid ratio is fixed at 50%, the stirring time is 30 minutes, the amount of (NH_4HF) is 20 kg /t, the nitric acid amount is 100 kg/t and the temperature is 90 °C. The obtained results are given on the Figure 11. To reach a level of 0.06% iron oxide, it took 40 kg of aluminum bifluoride and 280 kg of acid per tonne of feldspar treated without prior purification, whereas for an already ironed feldspar and for an iron oxide residual equivalent content of 0.044%, the consumption of the same reagents is reduced to 100 kg ofnitric acid and 20 kg/t of ammonium bifluoride.
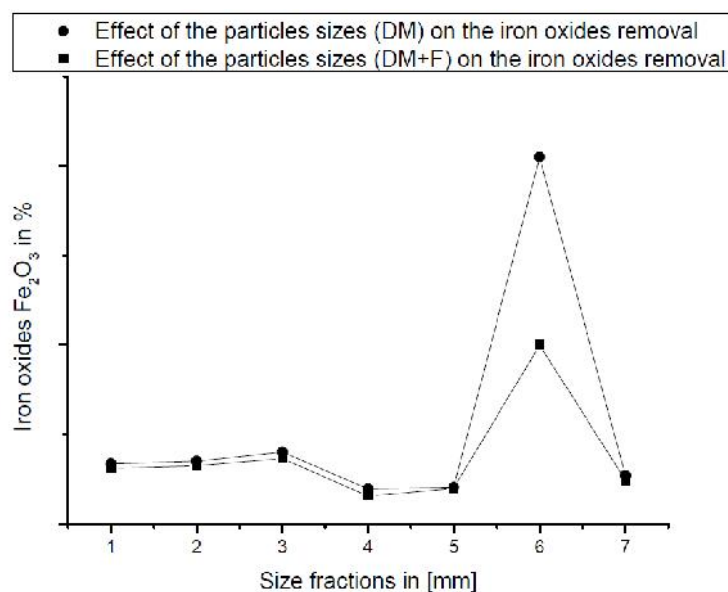
Fig.11. Iron oxides rate vs particle size fraction indicating the effect of magnetic (DM) and Flotation process (DM+F)

3. CONCLUSION

In this work, to achieve the optimal conditions for the chemical purification of Ain Barbar felspar, using nitric acid in the presence of ammonium bifluoride as a reducing agent, we preferred to examine all the characterization and recovery results achieved in the past. This allowed us to compare a number of results and extricate conclusions for the mining project

development. All chemical analyzes on all-comers or size fractions represent a panoply of iron oxides content, they give of a certain homogeneity in the sense that the majority are between 0.6-7%. The origin of the iron oxide impurity comes largely from surface oxidation and water seeping into cracks veins. Feldspar is very homogeneous in terms alkaline oxide, silica and free silica. This feldspar is mainly composed by potassium feldsparwith 9% of K_2O and 0.25% of Na_2O, 12% Al_2O_3, 75% SiO_2 and 0.59 to 0.7% of Fe_2O_3. Improved quality requires high intensity magnetic epuration and free silica reduction achieved only by flotation. Purification of size fractions [-0.6 + 0.1] mm [-0.6 + 0.250] mm [-0250 + 0.1] mm by high intensity magnetic separation, lowers the amount of iron oxide Fe_2O_3 until 0.34% with weight yields compared to all comers 41.26%, 19.30% and 23.06% respectively. As against the size fraction [-0.8 + 0.1] mm used by the glass industry, the rate of reduced iron is about 0.39% with yields in terms of weight of 60.89%. This purification yielded unsatisfactory results, the use of flotation purification of these same size fractions decreased the iron oxides rate from 0.39% to 0.23% for [-0.8+ 0.1] mm and 0.34% to 0.20% for [-0.6 + 0.1] mm with 54.63% 38.40% with yields in terms of weight respectively compared to all comers. These levels 0.20% and 0.23% iron oxide are appreciated by the glass and ceramics industry. For size fractions [- 0.6 + 0.250] mm,[-0250 +0.1] mm and [-0.1] mm, the purification by flotation, pushes downward the iron oxide content from 0.34% to 0.30% only, does not meet the requirements of consumers. The application of hydrometallurgical process, excluding the effect of pressure in an autoclave and using sulfuric acid yielded poor results in the experimental conditions. The process using nitric acid in a reducing medium (HF, NH_4F) is effective and pushes the degree of removal of Fe_2O_3 around 0.01%. This process applied to size fractions [-0.8 + 0.1] mm intended for the glass industry previously ironed by a high intensity magnetic field reduces iron levels up to 0.054 % with a weight of 58.32% compared to those of allcomers.

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