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ORIGINAL ARTICLE

Beneficiation of feldspar ore for application in the ceramic industry: Influence of composition on the physical characteristics

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Abstract In this study, physical and physicochemical experiments were carried out to improve the quality of feldspar ore in Sidi Aïch massive, located in the Gafsa region of south-western Tunisia. After determination of the mineralogical and the chemical composition, flotation methods were applied. In this study, non-hydrofluoric acid flotation methods used in feldspar-quartz separation were compared with each other and with the conventional HF/amine method. The results showed that conventional HF/amine method is the most effective and selective method.

Experimental studies indicate that an acceptable concentrate for industrial application can be obtained from these rocks. The feldspar yield was used to evaluate the process efficiency. Besides, the cone shrinkage, water absorption, degree of vitrification, mechanical properties (flexural strength) and dielectric behaviour were used to monitor the quality of the recovered feldspars.

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1. Introduction

The demand for feldspar as a raw material for the ceramic and glass industries is continuously increasing. In Tunisia, the traditional sources of feldspar, pegmatite and nepheline syenite ores do not exist. This implies a great importation of this raw material (26 millions dinars, at 2007). It became, therefore, imperative to look at other types of deposits, such as arkosic sandstone. In south and central Tunisia, extensive deposits of arkosic sandstone (Sidi Aïch Formation) are present. In this formation, there are two major industrial minerals potash feldspar (microcline) and quartz.

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 (Rabone, 1957; Shimoiizaka et al.,1976; Manser, 1975; Thom, 1962; Crozier, 1990; Demir et al., 2003).

The use of HF is no longer acceptable due to environmental restrictions and health hazards. Several research investigations

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Table 1 Chemical analysis of the different size fractions of Sidi Aich sand.

Size fraction (μm)	Mass (%)	CaO	MgO	SiO_2	Fe_2O_3	Al_2O_3	Na_2O	K_2O
Feed (raw material)	100	0.12	0.06	89.85	0.28	4.07	0.15	2.94
−500 + 315μ	2.85	0.62	0.19	82.22	0.57	7.95	0.18	4.77
−315 + 200μ	9.63	0.21	0.07	86.64	0.28	6.57	0.16	4.86
−200 + 125μ	75.7	0.46	0.05	83.56	0.31	8.16	1.83	2.21
−125 + 80μ	8.57	0.16	0.09	92	0.39	3.4	0.23	1.97
−80 + 63μ	1.1	0.45	0.19	87.71	0.85	4.91	0.54	2.12
−63μ	1	1.23	0.38	79.08	1.37	8.81	0.59	2.74

and experimental studies have been successful in developing a non-HF flotation process for the separation of quartz and feldspar (Katayanagi, 1974; Malghan, 1976; Malghan, 1981). El Salmawy et al. (1993a) floated quartz from feldspar using an anionic surfactant in the presence of metal hydroxy complexes or by using nonionic surfactants (El-Salmavy et al., 1993b). A combined cationic–anionic collector, N-tallow-1,3-propylene diamine/dioleate, was also applied on feldspar ores (Vidyadhar et al., 2002).

This paper describes the application of non-hydrofluoric acid flotation methods used in feldspar-quartz separation and the comparison with conventional HF/amine method. In order to enhance the feldspar beneficiation, this study seeks to determine the mechanical and dielectric behaviour of ceramic based on floated feldspar.

2. Materials and methods

The representative ore sample is obtained from Gafsa Province (Kanguet El Ouara), Tunisia (Table 1). For the size analysis, standard sieves from the "AFNOR" series were used. Minus 63 μm particles were separated by wet sieving after attrition in Wemco, Unitec machine, equipped with a 9 l cell. Attrition was carried out at 1400 rpm impeller speed for 5 min and 70 percent solids by weight. The oversized particles were reground to −250 μm in a ceramic ball mill in order to minimize the iron contamination of the sample. Flotation experiments were conducted with −250 + 63 μm test sample in a self-aerated Denver D12 flotation machine, equipped with a 3-l cell. Both conditioning and flotation were carried out at the same impeller speed (1200 rpm) and percent solids by weight (20–30%). A conditioning period of 5 min. for the first-stage conditioning and 3 min. for the subsequent stages of collector addition was utilized.

In order to reduce the TiO_2 and Fe_2O_3 content of the ore, an anionic collector, namely E 526 supplied by ArrMaz Florida, United States, which is based on sodium petroleum sulfonates and known to have selectivity for these minerals, was used.

The feldspar minerals and quartz were separated from each other by flotation of feldspar minerals and depression of quartz minerals. Both feldspar and quartz in amine flotation and in the absence of HF need not be floated at pH less than 4. However, in the presence of HF feldspar is floated at pH 2–3 while quartz does not float. Feldspar was floated using tallow amine acetate at a pH of 2–3 (Abdel-Khalek et al., 1994; Sekulic et al., 2004), with a pulp density of 25%. Hydrofluoric acid (HF) was used as quartz depressant and feldspar activator. This is accomplished by adding about 2000 g/t of 40% concentrated HF. No frother was added and the collector dosage was optimized at 600 g/t of Aero 3030C, collector supplied by CYTEC United states and composed of Amines, tallow alkyl acetates and Ethyl hexanole. Non-hdrofluoric acid flotation method was applied using anionic–cationic collector combination (Cutusamine 9002 + E 526 supplied by ArrMaz Florida, United States).

The floated fraction (the feldspar concentrate) and unfloated fraction (the quartz concentrate) were analysed for total balance by an Atomic Absorption Spectrophotometer (AAS) "Perkin Elmer" for the following oxides: SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, Na_2O, K_2O, TiO_2.

This feldspar was dry milled in a ball mill for 30 min to homogenize the mixture. The samples were dried for 48 h in air and then at 110 °C for 24 h in an electric furnace. After drying, they were formed into a cylinder with 16 mm in diameter and 5 mm in thickness using uniaxial moulding and then pressed at 100 MPa. The formed sample was sintered at 1230 °C for 24 h (condition used by the industrial).

The densification behaviour was described in terms of firing shrinkage, water absorption, and diametral compressive strength. Firing shrinkage values upon drying and sintering were evaluated from the variation of the length of the bodies. Water absorption values were determined from the weight differences between the as-sintered and the water saturated samples (immersed for 2 h in boiling water), according to the French standard (AFNOR EN 99, 1982).

The bending strength values were determined by using an universal testing machine (model NETSZH). We calculated average values of bending strength using the equation: $R\ (\mathrm{N/mm^2}) = 3FL/bh^2$, where F is the breaking load in kilograms, $L = 29.67$ mm the distance between supports, b the sample width in millimetres and h the sample thickness in millimetres. This test is carried out according to the French standard (AFNOR EN 100, 1982).

3. Sample characterization

Sidi Aïch sand is composed of weekly cemented grains. Grain size distribution of sample is shown in Table 1, about 90% of the sample is more than 125 μm. Examination of thin and polished sections was supplemented by SEM-EDX and X-ray diffraction analysis showed that the ore contains mainly quartz and microcline that display cross-hatch twinning and fresh cleavage (Fig. 1) with minor amounts of albite, kaolinite and illite (Fig. 2). Microscopic examination of heavy minerals, after separation in dense liquid (bromoforme), reveals accessorily the presence of zircon, rutile tourmaline and staurolite. The amount of this fraction does not exceed 1% (Gallala et al., 2009a,b).

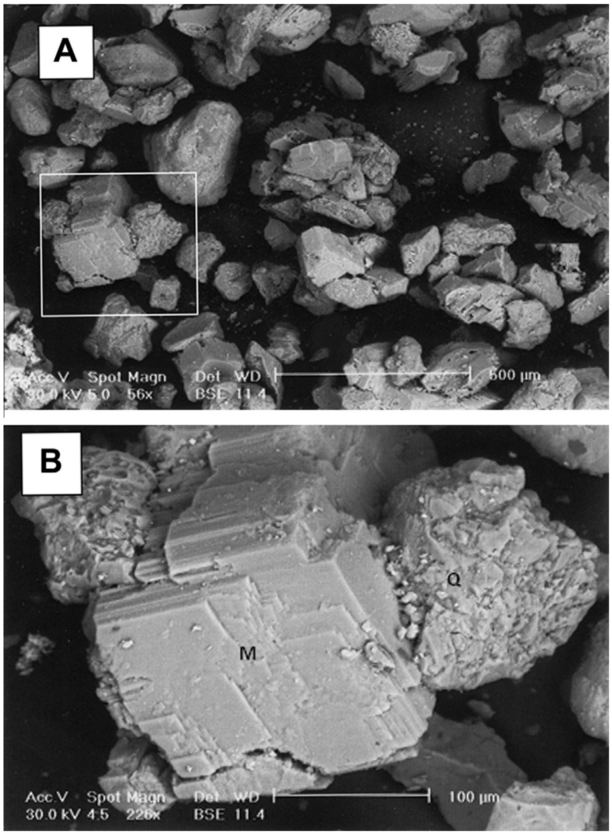

Figure 1 (A) SEM micrographs of Sidi Aïch sand; (B) detailed view of A (M, microcline; Q, quartz).

The chemical analysis of representative samples divided into six relevant size fractions, is presented in Table 1. The grain size analysis showed that 87% of the arkosic sandstone is less than 200 µm in diameter. From the chemical standpoint, the + 125–500 µm size fractions constitute about 75.75% of total weight.

4. Results and discussion

4.1. Flotation tests

Desliming is necessary to increase the percent floated (Burat et al., 2007). So, after attrition the desliming decreased the iron contents of the feed. The results obtained with attrition are summarized in Table 2.

Ferrous and others coloured minerals are floated in the first stage. The flotation of iron-bearing minerals was efficiently achieved with petroleum sulfonate collectors E526. The collector was added in a dosage of 200 g/t. Fe_2O_3 content (0.14%) which was found to be relatively low. Feldspar-quartz minerals separation was performed at the second stage of flotation. The separation can be achieved with cationic collectors in acid circuit, at a pH value in the vicinity between 2 and 3.

Ccomparison of the concentrates obtained shows that the choice of the CYTEC cationic collectors, Aero 3030C when HF is the pH regulator and the combination of cationic–anionic collectors (10% Cutusamine 9007/10%E526) when H_2SO_4 was the pH regulators, led to a reasonably selective feldspar flotation, using the following procedure.

Table 3 clearly shows that the conventional flotation with HF is still the most selective and effective method, although

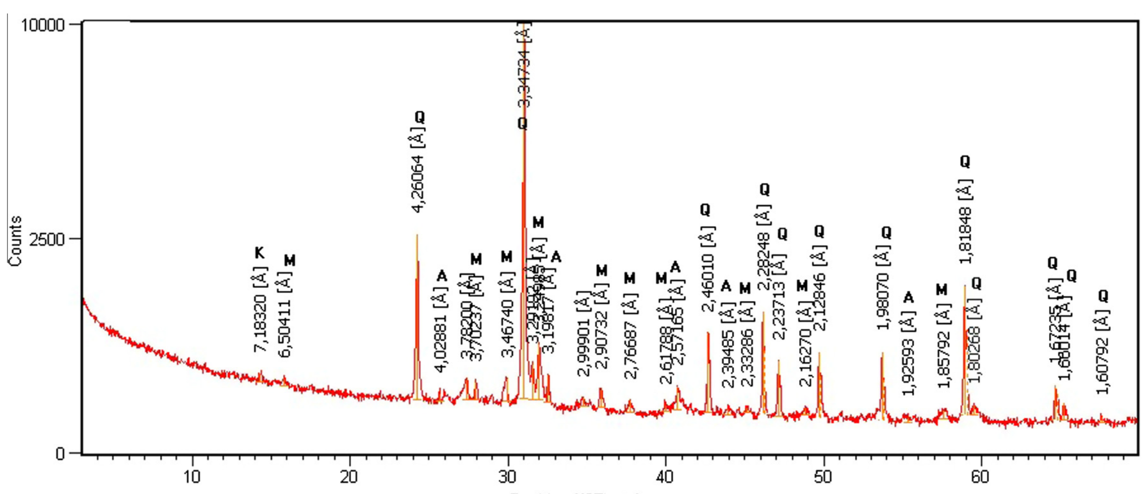

Figure 2 X-ray diffraction pattern of representative sample (A: albite; K: kaolinte; M: microcline and Q: quartz).

Table 2 Recovery and chemical composition of the ferrous products concentration.

			Mass (%)	Chemical composition							
				Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SiO_2
Raw material			100	0.28	4.07	0.06	0.12	0.06	0.15	2.94	89.85
First stage (Attrition)	After agitation of 10 min and sieving	Slime (fraction < 63 µm)	10.36	1.7349	–	–	–	–	–	–	–
Second stage	E526	Impurities (float)	2.62	2.559	15.87	–	0.64	–	3.77	3.79	73.57

Table 3 Recovery and chemical composition of the products of flotation concentration.

pH regulator	Collectors	Flotation products	Mass (%)	Chemical composition							
				SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2
HF	Aero 3030C	Feldspar (float)	28.22	59.73	17.47	0.14	< 0.01	4.97	0.53	15.92	0.04
		Quartz (sink)	67.22	95.32	0.60	0.19	0.05	2.47	0.03	0.67	0.03
H_2SO_4	Custamine 9007 + E 526	Feldspar (float)	26.07	65.63	17.68	0.14	< 0.01	0.02	3.83	12.7	< 0.01
		Quartz (sink)	60.92	98.52	0.70	0.09	0.051	0.012	0.015	0.51	0.10

Table 4 International standard limits for shrinkage, water absorption and bending strength (Konta, 1979).

Characters	Ceramic wall	Ceramic floor
Shrinkage (Shr.)	0–0.3%	5–6.5%
Water absorption (E)	14–17%	Less than 3%
Bending strength (BS)	Over 17 Nuten cm^2	Over 27.5 Nuten cm^2

it has harmful impact on the environment and health. The SiO_2 grade of quartz concentrate is reasonably high for some applications. On the other hand, feldspar concentrate with 0.53% Na_2O and 15.9% K_2O is considered to be sufficient for alkali content; particularly the ratio of $K_2O/Na_2O > 3$ makes this separation scheme attractive.

4.2. Suitability of Sidi Aïch feldspars for ceramic industries

The studied feldspars, of Sidi Aïch Formation, are exposed to four tests that are physical tests: shrinkage, water absorption, bending strength and determination of dielectric behaviour.

4.2.1. Technical tests

The quality of ceramic tiles is controlled by many tests. These tests were applied for feldspar in order to adapt the suitability of feldspars for ceramic industry. These tests include shrinkage, water absorption and bending strength according to international standard limits Table 4 (Konta, 1979).

Several samples from the Gafsa region were chosen for physical tests for feldspars. The results were illustrated by water absorption, shrinkage and bending strength (Table 5). Accordingly, these results indicate that feldspars are suited for the manufacture of wall and floor ceramic.

4.2.2. Dielectric tests

In this study, the trapping mechanisms of electric charges are explained using induced measured current (ICM). This well known method has been used in several papers (Berroug et al., 1997; Temga et al., 2006). Experiments aimed at testing the reproducibility of the absorbed current curves versus the injection time. In this way, a statistical study has been conducted on two samples (Fig. 3). The condition for electron injection at room temperature is a beam current of 1 nA during

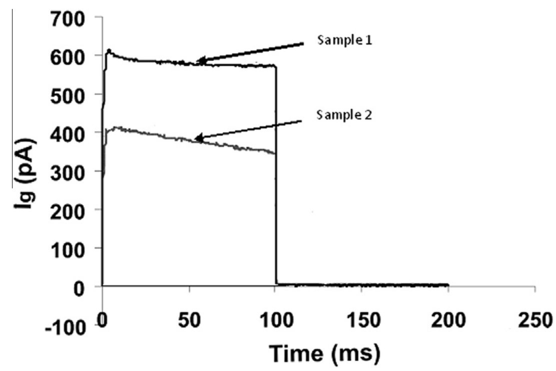

Figure 3 ICM curves. Variation of $I_g = f$ (time) for $t_{inj} = 100 =$ ms.

100 ms which determines a quantity of injected charges Q_i equivalent to 100 pC and we plotted the curves Ig = f (injection time). Samples admitted a very weak slope, hence it is a conductive material. There is a charge flow pronounced in such types of material and we do not expect any draping phenomena and our measured curve remains very stable. The capacity to trap charges increases with a reduction in the feldspar concentration, which is caused by the change in the composition of the glass phase of the ceramic (Gallala et al., 2010, 2011).

5. Conclusion

The studied alkali feldspars are petrographically classified into microcline with a minor amount of albite. The total alkali content (sum of $Na_2O + K_2O$) in Sidi Aïch feldspars is about 4%.

Experimental studies indicate that produced concentrates acceptable for the ceramic industry, can be obtained from this sand. Flotation tests with HF or H2SO4 as the pH regulators and Aero 3030C, Cutusamine 9002, amine acetate collectors led to yields of 95.44% and 86.99%, respectively, and provided feldspar concentrates assaying 17%Al2O3. These concentrates contained 0.14% total iron oxides,

The results of the physical tests illustrated by water absorption, shrinkage and bending strength beside their good resistance for electric charges suggest that the feldspars of the studied area can also satisfy the requirements of the ceramic industry.

Table 5 Water absorption % shrinkage and bending strength of Sidi Aïch feldspars.

Parameters	Drying shrinkage (%)	Firing shrinkage (%)	Total shrinkage (%)	Bending strength (N/mm^2)	Water absorption (%)
	3	8	11	30	0.4

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References

Abdel-Khalek, N.A., Yehia, A., Ibrahim, S.S., 1994. Beneficiation of Egyptian feldspar for application in the glass and ceramics industries. Miner. Eng. 7, 1193–1201.

AFNOR EN 99. Carreaux et dalles céramiques. Détermination de l'absorption d'eau; 1982. p. 6.

AFNOR EN 100. Carreaux et dalles céramiques. Détermination de la résistance à la flexion; 1982. p. 7.

Berroug, A., Bigarré, J. Fayeulle, S., Tréheux, D., 1997. Charging effects under electron beam injection on sapphire implanted with zirconium ions Conference on Electrical Insulation and Dielectric Phenomena, 1997 IEEE Annual Report. p.97.

Burat, F., Kokkilic, O., Kangal, O., Gurkan, V., Celik, M.S., 2007. Quartz–feldspar separation for the glass and ceramics industries. Miner. Metal. Process. 24, 75–80.

Crozier, R.D., 1990. Non-metallic mineral flotation, reagent technology. Ind. Miner., 55–65.

Demir, C., Bentli, İ.., Gülgönül, İ., Çelik, M.S., 2003. Differential separation of albite from microcline by monovalent salts in HF medium. Miner. Metal. Process. 20, 120–124.

El Salmawy, M.S., Nakahiro, Y., Wakamatsu, T., 1993a. The role of alkaline earth cations in flotation separation of quartz from feldspar. Miner. Eng. 6, 1231–1243.

El-Salmavy, M.S., Nakahiro, Y., Wakamatsu, T., 1993b. The role of surface silanol groups in flotation separation of quartz from feldspar using nonionic surfactants. XVIII International Mineral Processing Congress, 23–28 May, Sydney, Australia, pp. 553–559.

Gallala, W., Gaied, M.E., Montacer, M., 2009a. Concentration of potassium feldspar from low-grade Sidi Aïch sand in tunisia for industrial applications. Silicates Ind. 74, 125–130.

Gallala, W., Gaied, M.E., Montacer, M., 2009b. Detrital mode, mineralogy and geochemistry of the Sidi Aïch Formation (Early Cretaceous) in central and southwestern Tunisia: Implications for provenance, tectonic setting and paleoenvironment. J. African Earth Sci. 53 (4-5), 159–170.

Gallala, W. Les sables quartzo-feldspathiques de la Tunisie centro-méridionale: sédimentologie, minéralogie, minéralurgie et applications industrielles, PhD Thesis, Faculty of Sciences Sfax, Tunisia (2010).

Gallala, W., Gaied, M.E., Kchaou, B., 2011. Mineralogical and physico-chemical characterization of sintered Tunisian feldspathic sand. Annales de Chimie Science des Matériaux 35 (5), 261–267.

Katayanagi, A., 1974. Flotation separation of feldspar. United States Patent. Patent No: 3.844.939.

Konta, J., 1979. Ceramic Monographs, Handbook of Ceramics, Verlag Schmid Gmb, H Freiburg, pp: 800.

Malghan, S.G., 1976. Selective flotation of feldspar in non-fluoride medium. SME Preprint 76-1-1-331, Presented at the 1976 SME Annual Meeting. Denver, Colorado.

Malghan, S.G., 1981. Effect of process variables in feldspar flotation using non-hydrofluoric acid system. Mining Eng., 1616–1623.

Manser, R.M., 1975. Handbook of silicate flotation. Warren Spring Lab., 207.

Rabone, P., 1957. Flotation plant practice. Chapter VII, Flotation Methods. Mining Publication Ltd., pp. 208–209, revised and enlarged fourth edition.

Sekulic, Z., Canic, N., Bartulovic, Z., Dakovic, A., 2004. Application of different collectors in the flotation concentration of feldspar, mica and quartz sand. Miner. Eng. 17, 77–80.

Shimoiizaka, J., Nakatsuka, K., Katayanagi, T., 1976. Separation offeldspar from quartz by a new flotation process. In: Weiss, A. (Ed.), World Mining and Metals Technology. Port City Press, Baltimore, pp. 428–438.

Temga, T., Juvé, D., Tréheux, D., Guerret-Piécourt, C., Jardin, C., 2006. Conduction and trapping of electric charges in an anisotropic material after irradiation with an electron beam Application to TiO2 single-crystal. Nucl. Instr. Meth. B 245, 519–527.

Thom, C., 1962. In: Fuerstenau, D.W. (Ed.), Standard Flotation Separation, Froth Flotation, 50 th Anniversary Volume. American Institute of Mining and Metallurgical Engineers, New York, pp. 328–346 (Chapter 13).

Vidyadhar, A., Hanumantha Rao, K., Forssberg, K.S.E., 2002. Adsorpsiton of N-Tallow 1,3-Propanediamine-Dioleate collector on albite and quartz minerals, and selective flotation of albite from Greek Stefania Feldspar Ore. J. Colloid Interf. Sci. 248, 19–29.