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Surface vs. bulk analyses of various feldspars and their significance to flotation

I. Gülgönül [a,*], C. Karagüzel [b], M.S. Çelik [c]

[a] Balikesir University, Balikesir Technical High School, Balikesir, 10001, Turkey
[b] Department of Mining Engineering, Dumlupinar University, Kutahya, Turkey
[c] Department of Mining Engineering, Istanbul Technical University, Istanbul, Turkey

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Abstract

Cationic separation of Na-feldspar (albite) and K-feldspar (microcline) was earlier reported to be possible in the presence of monovalent salts. However, contrary to this result, the floatability of a series of K-feldspar minerals indicated that each microcline mineral exhibited different floatability and zeta potential patterns which in turn disputed the earlier results reported by our group. Comprehensive studies conducted on eight feldspar samples using ESCA and SEM/EDS probe analysis revealed the presence of nano spots on the surface of microcline; these nano spots with a dimension ranging anywhere from several nanometers to about 1000 nm not only distort the surface but also control the flotation behavior of the feldspar minerals. Interestingly, these spots shelter elemental impurities which could not be detected in the bulk analysis but assay several percents of Mn, Cu, Ba, Cr, Fe and Ni in the depth of 20 °A from the surface. These impurities are believed to be exposed upon preferential breakage of particles along the weak boundaries and modify the surface of microcline proportional with their numbers.
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1. Introduction

Significant amounts of commercial feldspar minerals, albite and microcline; exist in granite, siyenite and pegmatite rocks. A major amount of Na-feldspar and K-feldspar is used in glass and ceramics industry, respectively. The ratio of K_2O/Na_2O and the presence of coloring impurities such as Fe and Ti usually dictate the quality of these minerals. Feldspar deposits containing particularly only K-feldspars are diminishing. Feldspar ores or rocks that embody these two minerals in different proportions are naturally gaining an industrial importance. Therefore, there is an upsurge of interest to develop strategies to selectively separate albite and microcline or orthoclase (Demir et al. 2001, 2003a, 2003b and 2004; Karaguzel et al., 2006)

Similarities in the mineralogical, chemical and surface properties of feldspar minerals, however, make this separation challenging. Previous theoretical and experimental studies have mainly concentrated on the separation mechanism of quartz and feldspar (Klyachin

* Corresponding author. Tel.: +90 266 612 1212; fax: +90 266 612 1164.
E-mail address: gulgonul@balikesir.edu.tr (I. Gülgönül).

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Table 1
Summary of the literature on separation of Na and K feldspars

Researchers	Content
Shapolov and Polkin, 1958	Activation of microcline at high pH with Ca^{+2}
Joy et al., 1966	Depression of microcline at low pH with dodecylamine hydrochloride (DAH) in the absence of HF
Kovalenko, 1967	Flotation of K-feldspar and depression of Na-feldspar in the presence of $MgCl_2$ and $CaCI_2$
Yanis, 1968	Cationic flotation of Na–K feldspar using HF (patent)
Starikova, 1968	Increase of K_2O content in the presence of NaCI by fluoride activation
Revnivtzev et al., 1968; Revnivtzev and Putrin 1969	Depression of K feldspar with K^+, Rb^+, Cs^+ ve Ba^{+2} ions and that of Na–Ca-feldspar with Ca^{+2}, Na^+, Sr^{+2} ve Mg^{+2}; selectivity of albite significantly increased with KCI and that of microcline with NaCl
Klyachin et al., 1969	Cationic flotation of feldspars minerals using HCl or H_2SO_4 instead of HF (patent)
Marius and Laura, 1970	Cationic (Flotigam PA) flotation of individual feldspar minerals from a pegmatite are using NaCI; Na-feldspar was depressed
Sheiko, 1972	Selective adsorption of DAH on albite and microcline in the presence of NaCl and KCl
Yanis and Gorelik, 1973	Effect of Na^+, K^+ and Ca^{+2} ions against amine concentration; microcline floated selectively compared to albite. Depression of K-feldspars with K ion and that of Na-feldspars with Na^+ and Ca^{+2} ions, respectively.
Klunker et al., 1974	Floatability of feldspars was related to Na and K content of minerals, pulp, collector, HF concentration and crystal structure of feldspar minerals
Manser, 1975	Activation of albite with HF; K-feldspar was concentrated in scanvening stage
Severin et al., 1978	Floatability differences was shown to depend on crystal lattice and Na and K content of feldspar minerals
Uhlig, 1985	Floatability of different feldspar ores was tested
Ociepa, 1994	Surface charge of microcline was shown to be more negative than albite and oligoclase in amine medium indicating better floatability of microcline at pH 5.8
Bayraktar et al., 1999	Selective separation of alkali feldspars from pegmatite in the presence of HF, NaCl and amine at pH 2.5 the concentrate assayed 3.3% Na_2O and 13.1% K_2O
Demir et al., 2001, 2003a, 2003b and 2004	Separation of alkali feldspars using G-TAP with NaCl and $CaCl_2$ at both natural and low pH using HF; albite was depressed and microcline floated
Gulgonul, 2004	Attributing floatability pattern of various feldspar minerals to their surface impurities
Karaguzel et al., 2006	A new process flowsheet was proposed for commercial utilization of alkali feldspars from pegmatite. K content was raised to 10.51 K_2O while Na content at 3.02% Na_2O

et al., 1969; Manser, 1975; Fuerstenau and Raghavan, 1977; Rao and Forssberg, 1985; El-Salmavy et al., 1990, 1993a and 1993b; Vidyadhar et al., 2002). On the other hand, there are very few studies on the separation of Na–K feldspars but with contradictory results as shown in Table 1. There are two Russian (Yanis, 1968; Klyachin et al., 1969) and one American (Katayanagi, 1974) patents.

Demir et al. (2001 and 2003a) succeded to depress albite and float microcline with NaCl and $CaCl_2$ in the presence of a cationic reagent G-TAP either at natural pH or low pH values including HF. They indicated that selective separation of microcline and albite in the presence NaCl as an activating agent for microcline is dictated by the ability of inorganic cations to adsorb in the electrical double layer through either ion adsorption or ion

Table 2
Surface and bulk chemical analysis of albite and microcline samples

	Analysis	A1	M1	M2	M3	M4	M5	M6*
Si2p SiO_2	Surface,% Chemical, %	6.00 68.25	6.37 65.04	5.18 64.82	−66.26	−66.89	4.84 67.64	6.39 65.30
Al2p Al_2O_3	Surface, % Chemical, %	2.90 19.81	3.97 18.58	1.46 19.32	−18.59	−18.53	5.70 17.61	2.73 18.72
Fe2p3 Fe_2O_3	Surface, % Chemical, %	8.12 0.08	18.14 0.08	21.16 0.05	−0.08	−0.07	17.60 0.13	13.63 0.05
Na1s Na_2O	Surface, % Chemical, %	23.51 10.51	−1.23	−2.94	−1.64	−3.13	−1.39	−2.84
K2p3 K_2O	Surface, % Chemical, %	1.22 0.17	2.53 13.24	1,76 11.39	−12.79	−11.11	1.31 12.52	2.6 11.81
– CaO	Surface, % Chemical, %	−0.53	−0.05	−0.26	−0.18	−0.15	−0.13	−0.24
– MgO	Surface, % Chemical, %	−0.02	−0.01	−0.02	−0.05	−0.02	−0.03	−0.01
Cr2p3 Cr_2O_3	Surface, % Chemical, %	3.74 0.006	6.75 0.003	8.97 0.06	−0.006	−<0.001	8.17 0.003	7.46 0.024
– P_2O_5	Surface, % Chemical, %	−0.12	−<0.01	−0.57	−<0.01	−0.02	−<0.01	−0.32
– TiO_2	Surface, % Chemical, %	−0.10	−<0.01	−<0.01	−0.01	−<0.01	−<0.01	−0.01
– MnO	Surface, % Chemical, %	−<0.01	−<0.01	−<0.01	−<0.01	−<0.01	−<0.01	−<0.01
Ba3d5 Ba	Surface, % Chemical, ppm	5.46 15	4.49 3255	6.58 333	−63	−33	10.59 3102	8.43 228
Ni2p3 Ni	Surface, % Chemical, ppm	4.95 <20	3.78 <20	12.31 <20	−<20	−<20	9.44 <20	6.78 <20
– Sr	Surface, % Chemical, ppm	−125	−267	−110	−65	−25	−264	−70

∗M6 was used in studies of Demir, 2001.

exchange. The aim of this study is to show if these earlier findings are universal, i.e. applicable to all type of feldspar minerals. In order to test this hypothesis, a number of microcline samples from different localities have been subjected to a series of systematic, microflotation, zeta potential, SEM/EDS and ESCA measurements.

2. Experimental

2.1. Materials

Six microcline (M) and one albite (A) samples were used in the experiments. Albite and five of the microcline samples were obtained from Aydin–Cine region of Turkey and the other microcline sample was from Utah—USA. All samples were in the form of crystals and their bulk chemical analyses were performed in ACME laboratories of Canada using ICP (Inductively Coupled Plazma). ESCA analysis was done at the Materials Science Laboratories of University of Florida. SEM/EDS analysis was performed in Marmara Research Center of Turkey. The Scanning Electrone Microscope (SEM) with a brand name of Jeol JSM-6335F equipped with EDS (Energy Dispersive X-Ray Spectrometer) attachment was used for both image and probe analysis.

The samples were handground in an agate mortar to a size of −150+53 μm which were used for ESCA, SEM/EDS analyses and microflotation tests whereas the minus 53 μm fraction was used for zeta potential measurements. Genamin-TAP (faty alkyl peropylene diamine) is a cationic reagent used in flotation studies. The acidity was adjusted by HCl.

2.2. Methods

Electrokinetics measurements were performed using Zeta Meter 3.0 instrument which uses the microelectrophoresis method. Zeta potential was automatically calculated on the basis of applied voltage and velocity of the particles. A sample of 0.4 g feldspar in 100 ml of solution was conditioned for 10 min. The suspension was kept for 5 min to let the coarser particles settle. The measurements were performed at room temperature (25±2 °C).

Microflotation tests were carried out in a 150-ml column cell (25×220 mm) with a 15 μm frit and magnetic stirrer. The sample of 1 g was conditioned in 150 ml of solution containing the desired collector for 10 min. and then floated for 1 min. with nitrogen gas at a flow rate of 50 cm^3/min. The float and unfloat fractions were dried and weighed to calculate the percent floated.

The surface of each sample was analyzed by X-ray photoelectron spectroscopy known as XPS or ESCA (Electron Spectroscopy for Chemical Analysis); the results of both chemical and ESCA are given in Table 2.

3. Results and discussion

Na and K feldspars are typically found in the same matrix of various feldspar containing rocks such as pegmatites, granites and nefeline syenites. Interestingly, these minerals which exhibit similar physicochemical properties are not amenable to gravity separation techniques. But the addition of mono and multivalent ions, which undergo ion exchange or ion adsorption with the cations in the crystal lattice, induces charge differentation between Na and K feldspars and in turn causes changes in amine adsorption and in hydrophobicity as well. In an earlier study Demir et al. (2001) reported a floatability difference of 75% between albite and microcline in the presence of 0.267 mg/l G-TAP and $5 \cdot 10^{-2}$ M NaCl.

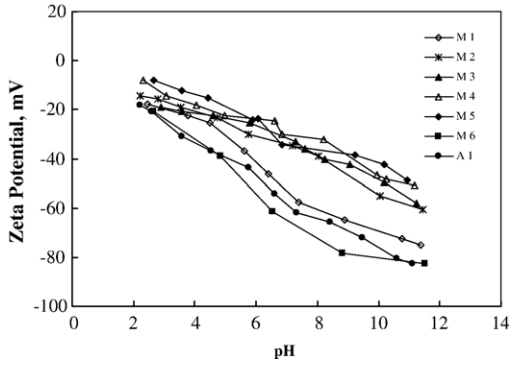

Fig. 1. Zeta potential profiles of different microcline samples (22± 1 °C), (A refers to albite and M to different kind of microclines).

Feldspar is negatively charged under most pH conditions; the negativity increases with increasing pH (Fig. 1). The isoelectric point (iep) of the samples is found by extrapolation at around pH 1.5, which is in agreement with the previous studies (Fuerstenau and Fuerstenau, 1982; Rao and Forsberg, 1993). The iep values are very low due to the broken bonds of Si–O and Al–O in the crystal structure during grinding process of feldspar. Various ions such as Na^+, K^+ and Ca^{+2}, which exist on the surface are released into the solution and impart the surface negative charges (Fuerstenau and Raghavan, 1977; Rao and Forssberg, 1985).

The zeta potential profiles of microcline and albite samples in Fig. 1 indicate that different chemical and mineralogical compositions resulted in different curves. The % K_2O and % Na_2O contents of the microcline samples extracted from Table 2 are as follows;

$$M1_{13.24} > M3_{12.79} > M5_{12.52} > M6_{11.81} > M2_{11.39} > M4_{11.11}\ \%\ K_2O\ \text{Contents} \quad \text{(i)}$$

$$M4_{3.13} > M2_{2.94} > M6_{2.84} > M3_{1.61} > M5_{1.39} > M1_{1.23}\ \%\ Na_2O\ \text{Contents} \quad \text{(ii)}$$

However, the zeta potential curves given in Fig. 1 did not follow the above order. For example, the zeta potential curve of M6 is most negative among others. Apparently, A1 sample is above M6 while the other curves, i.e. M1, M2, M3, M4, and M5, lie above A1 in the order of their negativity, respectively.

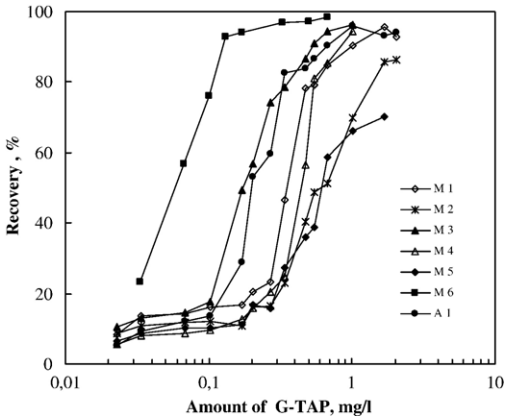

Fig. 2. Floatability of different microcline samples versus amine concentration (22±1 °C), (A: albite and M: different kinds of microclines).

Bulk analysis of this sample (M6), compared to the others, exhibits high levels of CaO and P_2O_5 and low levels of Ba (Table 2). On the other hand, K_2O/Na_2O ratio of this sample is 4.16. Theoretically, pure microcline contains 16.9% K_2O. However, the K_2O contents of microcline samples used in this study are lower than the theoretical values due to the replacement of Na, Ca and Ba with K. The deficiency in the K_2O values varies in the range of 3.66–5.51%; this so called perthitic structure is rather common among microcline and orthoclase occurrences.

Floatability of microcline samples with different chemical and mineralogical contents was determined to identify the extent of variation in different samples. The results of microflotation tests against the concentration of G-TAP are given in Fig. 2. At low amine concentrations, the recovery of the samples is almost the same. Above 0.1 mg/l G-TAP concentration, the floation recoveries exhibit different trends. Unfortunately, the role of Na_2O and K_2O contents of microcline could not be realized clearly, thus a meaningful order could not be obtained according to Na_2O and K_2O contents. For instance, the recovery curve of the microcline sample (M3) received from Utah (1.61% Na_2O and 12.79% K_2O) is the closest to that of albite. The M2 (2.94%Na_2O and 11.39% K_2O) and M5 (1.39% Na_2O and 12.59% K_2O) microcline samples, received from Aydin–Cine, have the least floatability properties.

The surface charge measurements given in Fig. 1 support the microflotation results in that no definitive order could be obtained. Therefore, variations of Na_2O and K_2O contents of microcline have different effects on their surface charges. Furthermore, the inherent cations such as Na, Ca and Ba change

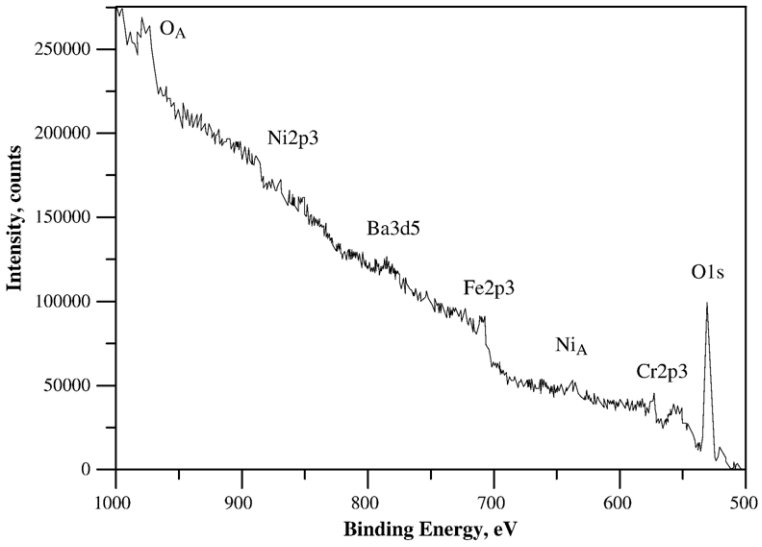

Fig. 3. A typical ESCA spectrum for microcline 1.

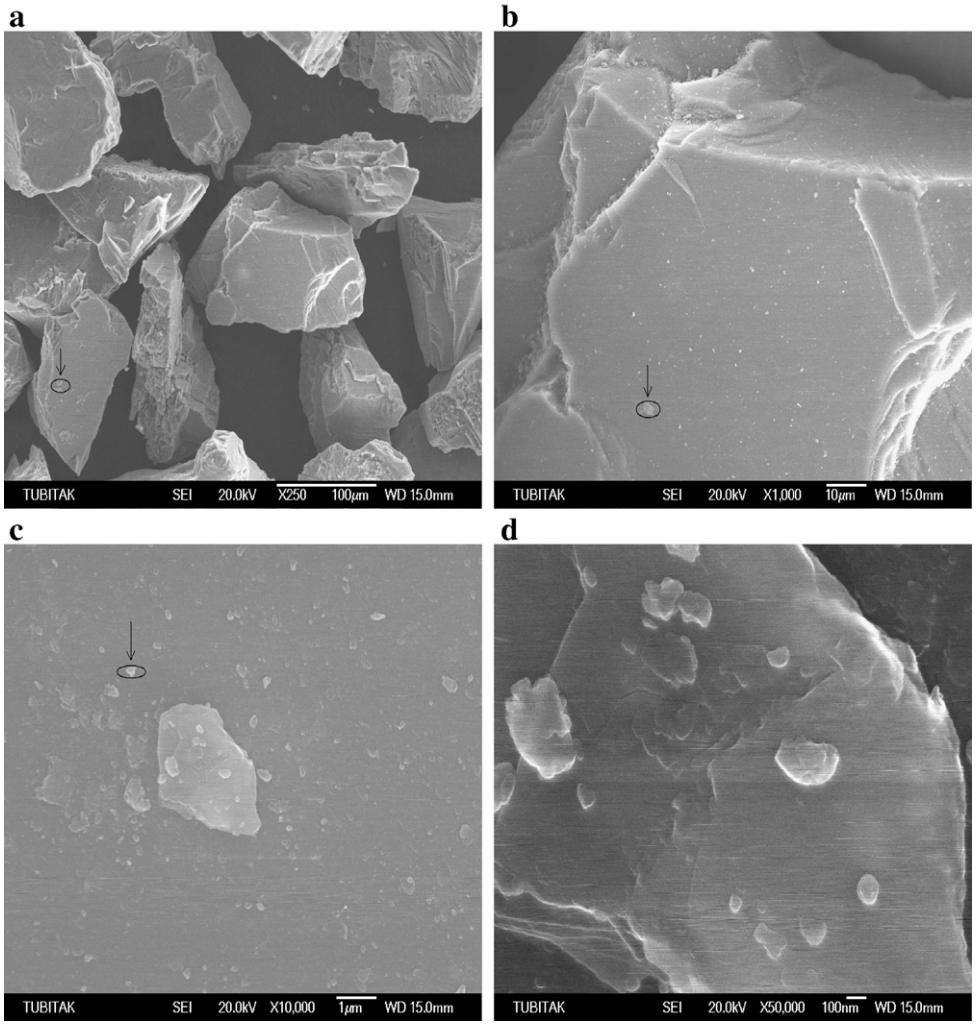

Fig. 4. SEM image of microcline1, a) 250× enlargement, b) 1000× enlargement, c) 10,000× enlargement, d) 50,000× enlargement; ; the circles in Pictures a, b, and c indicate the position of the subsequent enlargement.

in smaller quantities whereas K ion remains as dominant ion in the lattice. Due to this reason, accumulation of different ions on microcline surface plays an important role in the process. Thus, ESCA analyses were thus performed to determine the extent of accumulated ions on various surfaces.

The results of ESCA analyses of microcline reveal that impurities such as Fe, Ni and Cr are found at high levels (13.20–21.16% Fe, 3.78–12.31% Ni and 5.23–8.97% Cr), while that of chemical analyses indicates much lower quantities of maximum 0.13% Fe_2O_3, Ni<20 ppm and 0.024% Cr_2O_3. Similarly, the Ba contents in wet chemical analyses assayed at ppm levels (33–3255 ppm), however, in ESCA analyses Ba levels varried between 4.59 and 10.59% (Table 2). These differences in the analyses indicate that such impurities could not stem from the grinding process but rather present in the cyristal lattice or on the particle surface. ESCA is known to scan approximately the first 8 layers (20 °A in thickness) during the surface analysis. A typical ESCA spectrum for microcline1 given in Fig. 3 shows the peaks of prominent elements, i.e. Ni, Ba, Fe and Cr.

In order to test the reliability of ESCA results, a set of SEM/EDS probe analysis were concomitantly performed to find out the type and distribution of these elements and/or their compounds on the feldspar surface. Triclinic and massive structure of microcline particles can be seen clearly from the SEM images of M1 sample, in Fig. 4a (250 enlargements). However, the existence of the impurities could not be easily seen from these images. For more detailed images, SEM analysis were performed at larger magnifications of 1000, 10,000 and 50,000 (Fig. 4b, c and d), respectively. Each enlargement was performed on the previously selected area. The spots indicated with arrows shown in Fig. 4a to c were magnified in each consecutive figure by 1000×, 10,000× and 50,000× enlargements, respectively. These SEM views reveal

Table 3
SEM/EDS probe analysis of albite and microcline

Analysis	A1		M1		M2		M5		M6	
	1	2	1	2	1	2	1	2	1	2
Na	7.58	6.64	0.34	0.69	0.66	0.32	0.78	4.11	0.85	3.57
Al	9.03	9.45	9.25	8.65	9.73	9.26	9.04	9.12	9.53	8.70
Si	29.40	30.50	29.46	25.39	30.63	30.61	31.03	29.75	30.79	27.32
K	0.08	0.12	13.44	9.09	10.73	11.52	13.31	7.90	16.97	6.68
Ca	0.29	0.48	–	0.15	0.04	0.12	–	–	0.18	–
Cr	–	–	0.11	–	–	–	0.06	–	–	–
Mn	0.12	–	–	–	0.11	–	–	–	–	–
Fe	–	–	0.15	0.10	0.01	0.15	0.14	–	–	–
Ni	–	–	–	0.03	0.27	0.08	–	–	–	–
Cu	1.12	0.54	–	–	–	0.37	–	0.71	1.02	0.48
Sr	–	–	–	–	1.08	1.32	–	–	–	–
P	–	–	0.12	0.36	–	–	–	–	–	0.27
Ba	0.31	–	0.26	0.12	0.40	0.21	0.42	0.44	0.04	–
Zn	0.29	0.66	–	–	–	–	–	–	–	0.39
O	51.77	51.60	46.87	55.43	46.34	46.04	45.21	47.98	40.62	52.59

that some impurities in the form of spots coat the particle surface and appear to be well dispersed on the particle surface. The sizes of these spots are expected to range anywhere from several nanometers to 1000 nm with a thickness of around 100 nm or less.

To understand the nature and composition of the spots on the microcline surfaces better, EDS elemental probe analyses were performed and their results are given in Table 3. The existence of Ca, Mn, Cu, P, Zn and Sr elements were detected in these analyses (EDS) in addition to Fe, Ni and Cr, which were detected in ESCA analysis before. The results are an evidence of the existence of very small dispersed impurities on the surfaces of microcline and albite particles of −150 micron in size; these samples were considered as rather pure samples. Because the amount of existing elements in each sample differs, two probe analyses on two separate spots of each sample were performed. The results are presented in Table 3.

The differences in the results of three kinds of analyses (ICP, ESCA ve EDS) clearly reveal the existence of some spots containing Cu, Mn, Sr, Ba, Cr, Fe and Ni on the surfaces of microcline particles. These nano spots are believed to occur during the breakage action where particles were broken through their weak boundaries. Such preferential breakage is expected to create nano impurities on the surface of feldspar particles. The nature of spots in different shapes should be envisaged to be various forms of metal silicates sheltering ions like Ni, Cr, Cu or mica type impurities which again contain these elemental impurities. Characterization of the exact composition of the mineral itself requires meticulous ESCA studies on well known of such rare reference materials.

4. Conclusions

Flotation data of relatively pure 6 microcline samples with K_2O contents ranging from 11.11 to 13.24% show that they float in a wide range of amine concentration. Zeta potential data also show a considerable variation among the microcline samples. Neither flotation nor zeta potential data as a function of amine concentration correlates with their K_2O contents. This has clearly shown that the bulk chemical composition does not always dictate the extent of flotation.

Characterization tests on the surface of feldspar particles involving ESCA, SEM and EDS results clearly reveal the presence of nano impurities which shelter significant amounts of Ni, Cr, Mn, Fe, Ba, and Cu. The existence of such elements except Ba, interestingly, was not identified in the bulk analysis, but was independently detected inside the microspots using ESCA and SEM/EDS analysis.

These nano spots sizing several nanometers to 1000 nm are believed to be exposed upon preferential breakage of particles along the weak boundaries. The nano spots are presumed to modify the surface of microcline proportional to their numbers. The spots are not acid soluble and thus envisaged as some kind of silicate minerals, most probably mica. Their exact identification requires more careful and systematic studies.

It is proposed that the selectivity of Na–K feldspar strongly depends on the existence of such impurities on the surfaces. These impurities alter the hydrophobicity of the particles proportional with their distribution. In this regard, not only bulk chemical analysis but also surface analysis techniques such as ESCA and SEM/EDS probe analysis must be utilized to identify the mechanisms responsible in the flotation of feldspar minerals in general but more specifically with other minerals as well.

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