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Analysis of the Chemical Composition of Raw Materials Before and After Purification for Catalyst Preparation

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Abstract. This study is devoted to the investigation of the mineralogical and chemical characteristics of feldspar minerals, as well as to the role of surface phenomena in the selective separation of quartz–feldspar systems. Feldspars are among the most abundant tectosilicate minerals in the Earth's crust and are commonly classified into alkali feldspar and plagioclase series. The paper provides a comprehensive analysis of the influence of pH conditions, ζ -potential, collector adsorption, and the mechanisms of ion exchange and ion adsorption in flotation processes. Within the framework of the research, the chemical composition of feldspar samples was determined using X-ray fluorescence (XRF) analysis. Based on the obtained data, the major oxide components (SiO_2 , Al_2O_3 , K_2O , Na_2O , CaO) as well as the contents of minor and impurity elements were quantitatively evaluated. In addition, modern approaches aimed at quartz–feldspar separation under neutral and mildly alkaline conditions were reviewed, with particular emphasis on the selective adsorption mechanisms of cationic collectors, including amine- and imidazoline-based reagents, supported by literature analysis. The obtained results demonstrate significant scientific and practical relevance for improving the efficiency of feldspar flotation, optimizing reagent selection, and expanding the utilization of Si/Al resources present in feldspar raw materials for the synthesis of zeolites and molecular sieves. Furthermore, the findings provide a reliable basis for assessing the suitability of purified raw materials for catalyst preparation.

INTRODUCTION

Feldspars are among the most widely distributed tectosilicate minerals in the Earth's crust and are generally classified into two major series: alkali feldspars, $\text{KAlSi}_3\text{O}_8 \leftrightarrow \text{NaAlSi}_3\text{O}_8$ (orthoclase/microcline \leftrightarrow albite), and plagioclases, $\text{NaAlSi}_3\text{O}_8 \leftrightarrow \text{CaAl}_2\text{Si}_2\text{O}_8$ (albite \leftrightarrow anorthite) [1–3]. In mineral beneficiation and flotation processes, the selective separation of quartz from feldspar and other associated minerals plays a crucial role. This selectivity is strongly governed by surface-related factors such as ζ -potential, collector adsorption behavior, and the pH of the medium [4]. Alkali activation combined with hydrothermal synthesis is widely employed for the production of zeolites and molecular sieves, as this approach enables the transformation of Si/Al resources contained in feldspar into a reactive form. The surface charge characteristics of quartz and feldspar, the structure of the hydration layer, and the presence of active oxygen sites ($\equiv\text{Si}=\text{O}^-$, $\equiv\text{Al}=\text{O}^-$) critically determine reagent adsorption mechanisms. In particular, it has been extensively reported that in systems involving amine-type cationic collectors and Na^+ ions, two mechanisms—ion exchange and ion adsorption—may operate simultaneously [5–11]. In practical applications, quartz–feldspar separation is commonly conducted under acidic conditions, where both reagent adsorption and mineral surface charge characteristics are significantly altered. Pretreatment with HF has been shown to increase the negative surface charge of feldspar, thereby enhancing the adsorption of cationic collectors (e.g., amines) and improving flotation recovery [12–13]. In addition, several studies have demonstrated that dissolved Al(III) and Fe(III) ions can influence feldspar flotation through the formation of surface complexes and hydroxo-complexes. More recently, alternative approaches under neutral to mildly alkaline conditions have been proposed as viable substitutes for conventional acidic flotation schemes. These strategies aim to facilitate quartz–feldspar separation through the development of novel combined reagents and adsorption models operating in neutral or weakly alkaline environments [16–21]. It has been reported that in the presence of the cationic collector DDAC (bisdecyldimethylammonium

chloride) at $\text{pH} \approx 3$, potassium feldspar exhibits high flotation recovery, while fluorapatite remains poorly floated. The underlying mechanism has been elucidated using ζ -potential measurements, FTIR, and XPS analyses, demonstrating selective interactions between the collector molecules and oxygen sites on the K-feldspar surface. Similarly, in 2025, selective adsorption of an imidazoline-based collector (MIS) on K-feldspar—driven by a combination of electrostatic attraction and hydrogen bonding—was confirmed using FTIR and XPS techniques [28–31].

MATERIALS AND METHODS

The determination of the mineralogical structure, phase composition, and morphological characteristics of feldspar represents a fundamental step in ensuring the scientific reliability of this study. Accordingly, the structural features of the mineral samples were investigated using a combination of complementary physicochemical methods. The chemical composition of the mineral samples, including the major and minor elemental constituents of feldspar, was determined by X-ray fluorescence (XRF) analysis. Measurements were carried out using a high-performance energy-dispersive XRF spectrometer (Rigaku NEX CG). XRF analysis is based on the detection of characteristic fluorescent radiation generated by atoms in the outer electron shells when excited by incident X-rays. Since each element exhibits unique spectral lines with characteristic energies, this technique enables both qualitative and quantitative determination of elemental composition. The Rigaku NEX CG spectrometer employs polarized monochromatic secondary excitation technology combined with 3D Cartesian geometry X-ray optics, significantly enhancing the signal-to-noise ratio and allowing reliable detection of elements present at low concentrations. Sample preparation involved preliminary crushing and grinding of feldspar, quartz, and other accessory phases to obtain a homogeneous powder. The powdered samples were placed in standardized sample cups. One of the key advantages of this method is that the analysis can be performed without dissolution and without causing destructive effects on the samples. During measurements, samples were analyzed either in air or in a helium atmosphere to improve the sensitivity for light elements such as Na and Mg. The measurement conditions and analytical parameters were as follows. The X-ray source was a Pd-anode X-ray tube with a maximum power of 50 W. The detectable elemental range extended from sodium (Na, $Z = 11$) to uranium (U, $Z = 92$). The system was equipped with a high-resolution detector and an automatic sample changer, ensuring high repeatability and analytical precision.

TABLE 1. Mineralogical Composition of Feldspar Raw Material

No.	Mineral constituents of feldspar	Content (%)
1	Plagioclase feldspar	39
2	Orthoclase feldspar	12
3	Quartz	12
4	Pyroxene	11
5	Amphibole	5
6	Clay minerals	5
7	Mica	5
8	Olivine	3
9	Other minerals	8

The acquired spectra were processed using RPF-SQX software. Quantitative analysis was performed using the fundamental parameters (FP) method, which minimizes the number of required reference standards and effectively accounts for matrix effects. This approach is particularly efficient for mineral samples with unknown or variable compositions. Overlapping spectral lines were automatically deconvoluted, resulting in improved analytical accuracy. Based on the XRF results, the major oxide composition of feldspar minerals (SiO_2 , Al_2O_3 , K_2O , Na_2O , CaO) as well as the contents of minor impurity oxides (Fe_2O_3 , TiO_2 , etc.) were quantitatively determined. These data served as a basis for identifying feldspar types (alkali feldspar or plagioclase series) and for evaluating their suitability for flotation processes and zeolite synthesis. According to literature data, the chemical composition of feldspars primarily includes aluminum, silicon, and oxygen, along with potassium, sodium, or calcium depending on the specific mineral type. For example, orthoclase has the composition KAlSi_3O_8 , albite corresponds to $\text{NaAlSi}_3\text{O}_8$, and anorthite to $\text{CaAl}_2\text{Si}_2\text{O}_8$. Mineralogically, feldspars belong to the silicate group, specifically to the class of tectosilicates. Feldspar-bearing raw materials commonly contain associated minerals such as quartz, pyroxene, mica, amphibole, clay minerals, olivine,

and others (Table 1). Overall, feldspar and quartz-rich compositions account for approximately 50% of the total mass of the Earth's crust, reflecting their significant geological and industrial importance.

RESULTS AND DISCUSSION

Investigation of the mineralogical composition of feldspar is essential for understanding its chemical composition and for identifying valuable components that can be selectively extracted through chemical activation processes [5]. In the present study, the chemical composition of feldspar was examined by determining the contents of individual elements and their corresponding oxides.

TABLE 2. Chemical Composition of the Initial Feldspar Sample (wt% on a Dry Basis)

№	Sample No.	Oxide composition (%)						
		SiO ₂	Fe ₂ O ₃	FeO	TiO ₂	MnO	Al ₂ O ₃	CaO
1	№1	77,42	2,38	2,16	0,080	0,024	8,50	1,96
2	№2	77,20	2,70	2,43	0,10	0,028	8,50	2,24
3	№3	77,54	2,46	2,05	0,080	0,032	7,08	2,24
4	№4	77,84	2,54	2,09	0,10	0,032	8,03	2,52
5	№5	76,82	1,84	1,73	0,040	0,060	10,12	1,96
6	№6	76,12	1,71	1,30	0,040	0,028	12,12	1,12
7	№7	74,74	1,75	1,69	0,060	0,030	14,17	1,17
8	№8	76,46	1,91	1,66	0,060	0,028	13,12	1,12
9	№9	77,02	1,91	1,62	0,10	0,032	13,17	1,12
10	№10	76,68	1,83	1,43	0,080	0,040	12,12	0,84
№	Sample No.	Oxide composition (%)						
		MgO	K ₂ O	Na ₂ O	P.P.P	SO ₂	S _{overall}	P ₂ O ₅
1	№1	0,80	6,66	2,70	1,37	0,84	0,020	0,09
2	№2	0,20	6,05	2,70	1,07	1,06	0,030	0,11
3	№3	<0,5	8,25	2,70	1,04	0,88	0,039	0,11
4	№4	0,60	6,93	2,63	1,23	0,84	0,060	0,12
5	№5	<0,5	6,68	2,08	1,68	1,01	0,016	0,13
6	№6	<0,5	5,45	2,30	1,32	1,54	0,040	0,11
7	№7	<0,5	5,81	2,23	1,32	1,28	0,030	0,11
8	№8	0,40	5,69	1,96	1,19	1,10	0,010	0,12
9	№9	<0,5	5,32	1,86	1,48	0,93	0,030	0,11
10	№10	0,20	6,35	2,30	1,80	1,15	0,040	0,11

As part of the experimental procedure, feldspar samples with different particle sizes and degrees of dispersion were subjected to purification in order to remove associated impurity phases. Specifically, the raw material was fractionated into particle size ranges of: (a) 5.00 mm, (b) 2.50 mm, (c) 1.25 mm, and (d) 0.63 mm using standard sieves. In addition, representative samples were collected after passing through each corresponding sieve size. The purified feldspar samples were subsequently ground using a ball mill (VEB-4600 Wittenberg BT). The crushed materials of different particle sizes, as illustrated in Figure 2, were sieved again within the 5.00–0.63 mm size range. After further removal of accessory mineral phases, the samples were finely ground in a ball mill to a particle size of $d \leq 0.071$ mm. These finely dispersed samples (0.071 mm) were selected for subsequent chemical activation experiments. For acid activation, 100 g of feldspar sample was treated with 20 g of hydrochloric acid (HCl, 32 wt%), corresponding to the selected solid-to-acid ratio. The primary objective of HCl washing was to remove impurity phases and thereby increase the relative silicon content of the feldspar. According to the selected proportion, 500 g of feldspar was mixed with 100 g of 32 wt% HCl, and distilled water was added gradually until a slurry-like consistency was achieved. The resulting mixture was then heated in a muffle furnace at temperatures ranging from +50°C to +200°C until boiling commenced. After the onset of boiling, the reaction was maintained for a duration of 30 minutes to 1

hour. After approximately 30 minutes, the initial sample transformed into a viscous mass. Upon completion of a total boiling time of 1 hour, the material was thoroughly washed with distilled water until complete removal of residual acid was achieved. The solid phase was then separated using vacuum filtration. The filtered product was dried in a laboratory drying oven (SHS-80-01 SPU) at a temperature of 105–110°C. During the course of the study, the chemical composition of the first 20 samples was analyzed and each sample was assigned a numerical identifier for convenience. The chemical composition of the samples was determined on a dry basis and expressed in weight percent (wt%). The corresponding results of the chemical composition analysis are presented in Tables 2 and 3.

TABLE 3. Result of analysis of the initial feldspar by dry matter %

№	Sample No.	Oxide composition (%)					
		SiO ₂	Fe ₂ O ₃	FeO	TiO ₂	MnO	Al ₂ O ₃
1	№11	76,56	2,16	1,66	0,14	0,030	13,92
2	№12	76,18	2,16	1,58	0,20	0,030	13,40
3	№13	76,98	2,38	1,62	0,050	0,024	11,12
4	№14	77,36	1,98	1,58	0,12	0,040	12,10
5	№15	76,46	2,78	1,51	0,14	0,032	9,45
6	№16	73,88	1,43	1,00	0,12	0,040	14,65
7	№17	77,56	2,27	1,80	0,12	0,032	8,97
8	№18	77,78	1,75	1,43	0,30	0,028	9,45
9	№19	72,14	1,83	1,15	0,12	0,028	6,62
10	№20	80,56	1,98	1,50	0,36	0,024	11,00
№	Sample No.	Oxide composition (%)					
		MgO	K ₂ O	Na ₂ O	P.P.P	SO ₂	S overall.
1	№11	<0,5	4,66	1,49	1,90	1,23	0,030
2	№12	<0,5	5,45	1,55	1,69	1,01	0,030
3	№13	<0,5	5,45	1,96	1,86	1,23	0,016
4	№14	<0,5	5,32	1,86	1,70	1,06	0,040
5	№15	0,81	4,66	1,78	1,92	1,10	0,043
6	№16	<0,5	4,30	1,70	2,11	1,19	0,020
7	№17	1,41	5,45	1,96	1,37	0,88	0,020
8	№18	1,01	3,19	2,57	1,42	1,10	0,020
9	№19	0,81	2,80	1,89	1,64	0,88	0,020
10	№20	2,02	2,18	1,96	1,42	0,97	0,020
							0,10

Based on the results obtained from samples numbered 1 to 10 collected from the Chiyali site, the content of silicon dioxide (SiO₂) was analyzed in ascending order. The observed sequence was as follows: SiO₂ content of sample No.1 – 77.42%, No.2 – 77.20%, No.3 – 77.54%, No.4 – 77.84%, No.5 – 76.82%, No.6 – 76.12%, No.7 – 74.74%, No.8 – 76.46%, No.9 – 77.02%, and No.10 – 76.68%. The maximum concentration was observed in sample No.10. The largest decrease in SiO₂ content occurred between samples No.6 and No.7 (-1.38%), whereas the most significant increase was observed between samples No.7 and No.8 (+1.72%). Overall, the SiO₂ content initially decreased and then increased. The highest value was recorded for sample No.4 at 77.84%, while the lowest value was observed in sample No.7 at 74.74%. These variations ranged from 0.1% to 3.0%. For samples No.11 to No.20, the SiO₂ content was determined as follows: No.11 – 76.56%, No.12 – 76.18%, No.13 – 76.98%, No.14 – 77.36%, No.15 – 76.46%, No.16 – 73.88%, No.17 – 77.56%, No.18 – 77.78%, No.19 – 72.14%, and No.20 – 80.56%. In these samples, the SiO₂ content varied between 0.1% and 8.0%, with the largest change (8.42%) observed between samples No.19 and No.20. The variations in SiO₂ content at the Chiyali site reflect the wide distribution of feldspar in the region. Quantitative analyses of the oxides (SiO₂, Al₂O₃, Fe₂O₃, TiO₂, and other associated compounds) in the samples are presented in Tables 2 and 3. These results indicate the suitability of the Chiyali feldspar samples for subsequent experimental studies and scientific analysis. It was found that SiO₂ is the predominant component in the feldspar samples, providing a comprehensive indicator of the overall chemical quality. Additionally, the quantitative contents of Al₂O₃ and K₂O

were also notably high. Chemical treatments with HCl were performed to remove excess salts and metal compounds, and the resulting oxide compositions of the treated samples are presented in Table 4.

TABLE 4. Quantitative Composition of Oxides in Feldspar

Nº	Oxides	Content, %	Unit
1	Si	0.0187	mass%
2	Na ₂ O	3.28	mass%
3	Al ₂ O ₃	13.9	mass%
4	SiO ₂	75.6	mass%
5	SO ₃	0.0296	mass%
6	K ₂ O	5.90	mass%
7	CaO	0.584	mass%
8	TiO ₂	0.102	mass%
9	V ₂ O ₅	(0.0056)	mass%
10	MnO	0.0090	mass%
11	Fe ₂ O ₃	0.125	mass%
12	Co ₂ O ₃	(0.0014)	mass%
13	NiO	0.0009	mass%
14	CuO	(0.0008)	mass%
15	ZnO	0.0008	mass%
16	Ga ₂ O ₃	0.0013	mass%
17	Rb ₂ O	0.0285	mass%
18	SrO	0.0140	mass%
19	Y ₂ O ₃	0.0009	mass%
20	ZrO ₂	0.134	mass%
21	SnO ₂	0.0018	mass%
22	TeO ₂	(0.0007)	mass%
23	BaO	0.0529	mass%
24	HfO ₂	(0.0017)	mass%
25	Ta ₂ O ₅	0.0034	mass%
26	ReO ₂	0.0013	mass%
27	Ir ₂ O ₃	(0.0010)	mass%
28	Tl ₂ O ₃	0.0009	mass%
29	PbO	0.0041	mass%
30	Dy ₂ O ₃	0.0057	mass%
31	U ₃ O ₈	0.0017	mass%

Based on the analysis results, the increasing order of silicon dioxide (SiO₂) and other oxides in the feldspar samples was determined as follows: SiO₂ – 75.6% > Al₂O₃ – 13.9% > K₂O – 5.90% > Na₂O – 3.28% > CaO – 0.584% > ZrO₂ – 0.134% > Fe₂O₃ – 0.125% > TiO₂ – 0.102%. The high content of silicon dioxide (SiO₂ – 75.6%) indicates that the acid treatment with hydrochloric acid was effective, confirming that SiO₂, as the main component of feldspar, can be significantly enriched through chemical processing. The content of other important components, such as Al₂O₃, was found to be 13.9%, highlighting its significance in the composition. Minor oxides, including TiO₂, MnO, and Fe₂O₃, were present in relatively low amounts. The quantitative analysis of oxides after washing and neutralization with HCl allowed for accurate determination of the elemental composition of feldspar. The results are illustrated in Figure 1. Based on experimental analyses, the quantitative distribution of elements in feldspar, consistent with their corresponding oxides, was determined. The results indicate that the most abundant elements occur in the following order, Si – 38.8% > Al – 8.07% > K – 5.36% > Na – 2.99% > Ca – 0.460% > Zr – 0.110% > Fe – 0.103%. In addition, trace amounts of Cl, Ti, Mn, Fe, and Zr were detected, while relatively rare elements such as U, Co, and Ir were identified at very low concentrations (mass %). The obtained results are illustrated in Figure 1 as a diagram representing the elemental quantitative composition [6; 21-29].

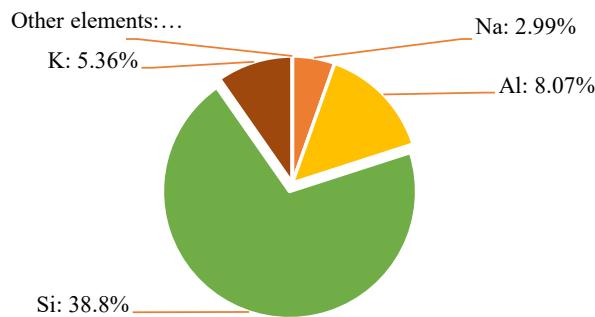


FIGURE 1. Quantitative indicators of the elemental composition in feldspar

Silicon dioxide (SiO_2) is the dominant component of feldspar. Treatment with hydrochloric acid (HCl) enabled the removal of excess associated impurities, including soluble salts, carbonates, and iron oxides. This process resulted in an increase in the relative content of silica within the feldspar matrix. The chemical composition analysis confirms the validity of the scientific conclusions derived from the experimental data.

CONCLUSION

Under the selected experimental conditions, feldspar was activated using hydrochloric acid and subsequently washed with distilled water until complete removal of residual acid. This procedure allowed for the precise determination of the concentrations of constituent oxides and elements. According to the analytical results, the oxide composition of feldspar decreases in the following order: $\text{SiO}_2 - 75.6\% > \text{Al}_2\text{O}_3 - 13.9\% > \text{K}_2\text{O} - 5.90\% > \text{Na}_2\text{O} - 3.28\% > \text{CaO} - 0.584\% > \text{ZrO}_2 - 0.134\% > \text{Fe}_2\text{O}_3 - 0.125\% > \text{TiO}_2 - 0.102\%$. The results demonstrate that SiO_2 and Al_2O_3 are the predominant oxides in the feldspar composition, while the remaining oxides are present in significantly lower amounts. Following oxidation and subsequent neutralization through HCl washing, Si, Al, K, Na, Ca, Zr, and Fe were identified as the most abundant elements in the treated feldspar sample.

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