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Elimination of Ferric Ion Effect on Separation between Kyanite and Quartz Using Citric Acid as Regulator

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Abstract: Ferric ions produced during grinding influence the flotation separation between kyanite and quartz adversely. In this study, citric acid was used as a regulator to eliminate the effect of ferric ions on the separation of kyanite from quartz with sodium oleate (NaOL) as a collector. The microflotation test results indicated that the quartz was selectively activated by $FeCl_3$ and maintained significant quartz recovery. However, the citric acid could selectively eliminate the effect of ferric ions on the quartz and minimally influenced the kyanite. Contact angle tests demonstrated that $FeCl_3$ significantly increased the interaction between NaOL and quartz, resulting in the high hydrophobicity of quartz, and the addition of citric acid made the quartz surface hydrophilic again but slightly influenced the kyanite. Fourier-transform infrared spectroscopy showed that $FeCl_3$ facilitated NaOL adsorption onto the quartz surface, and the addition of citric acid eliminated the activation of $FeCl_3$ on the quartz, resulting in the nonadsorption of NaOL onto the quartz surface. However, the $FeCl_3$ and citric acid exhibited a negligible effect on NaOL adsorption onto the kyanite surface. X-ray photoelectron spectroscopy analysis indicated that the citric acid eliminated $FeCl_3$ activation on the quartz.

Keywords: kyanite; quartz; citric acid; ferric ion; selective elimination

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1. Introduction

Kyanite is an essential metamorphic nesosilicate with the chemical formula Al_2SiO_5, and it is widely used in metallurgy, building materials, ceramics, and other industrial sectors [1,2]. It can also be used for preparing silicon–aluminum alloys and metal fibers [3]. However, with the extensive exploitation of kyanite resources, low-grade kyanite, rich in quartz and muscovite impurities, has become relatively more abundant [4]. Thus, reducing the impurity contents (for example, quartz) is required to improve the quality of kyanite concentration powder.

Kyanite is produced in commercial grades using several mineral processing methods, such as hand sorting, gravity separation, magnetic separation, flotation, and roasting [5–8]. Because kyanite properties are becoming increasingly complex, flotation is widely applied for kyanite beneficiation. Many studies on the separation of kyanite and quartz were focused on the development of collectors, such as anionic sodium hexadecanesulfonate [9], dodecylamine hydrochloride [10], sodium dodecyl sulfate [11], and potassium oleate [12]. Nonetheless, despite the several novel collectors used in these experimental studies, sodium oleate (NaOL) remains the most widely used collector for the direct flotation separation between kyanite and quartz [13,14].

The minerals are crushed and ground before the flotation process is started, and ferric ions released from the steel-grinding medium may activate gangue minerals [15]. Therefore,

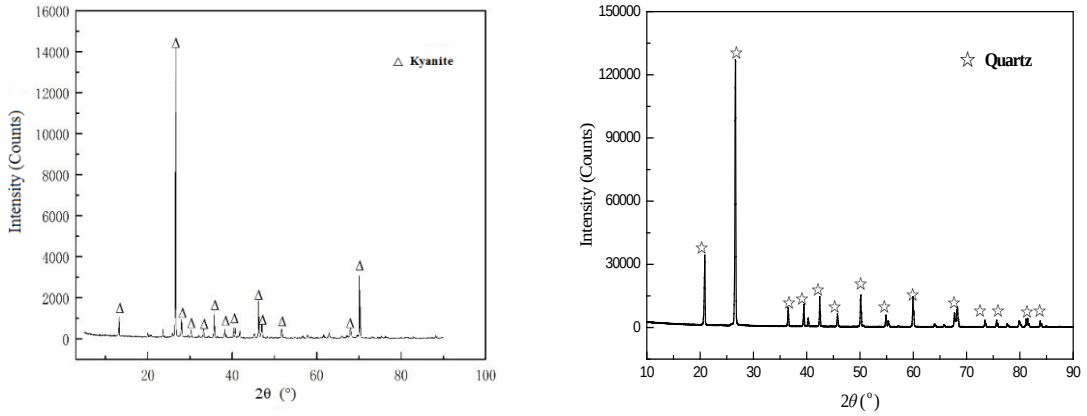
the flotation separation of kyanite from quartz in the presence of NaOL as a collector can cause a large amount of quartz to float together with kyanite, reducing the grade of kyanite in the concentrate [16–18]. Thus, the activation of quartz by ferric ion should be eliminated to enhance flotation separation between kyanite and quartz efficiently.

Citric acid is an environmentally friendly organic chelating agent, which has been widely used in recent years. Many research studies have shown that citric acid can form a stable complex with iron, prevent iron precipitation, increase the concentration of soluble iron in water, and promote the production of more active oxygen free radicals [19–21]. Therefore, in this study, a citric acid regulator was employed to achieve efficient separation through flotation and increase the difference in the flotation performance of kyanite and quartz using NaOL as a collector in the presence of ferric ions. The effects of the citric acid and ferric ions on the flotation behavior of these two minerals were investigated through experimental microflotation tests. Furthermore, the effects of the different adsorption behaviors and selective reaction mechanisms of the citric acid and ferric ion on the flotation separation between the two minerals were determined through contact angle measurements, Fourier-transform infrared (FTIR) analysis, and X-ray photoelectron spectroscopy (XPS) measurements.

2. Materials and Methods

2.1. Materials and Reagents

Separate kyanite and quartz minerals were obtained from Henan and Hebei Provinces in China, respectively [22]. The X-ray powder diffraction patterns of the samples are shown in Figure 1, and the chemical analysis results are listed in Table 1. The kyanite and quartz purities were 98.60% and 99.31%, respectively.



(**a**) (**b**)

Figure 1. X-ray powder diffraction patterns of (**a**) kyanite and (**b**) quartz.

Table 1. Chemical composition analysis of mineral samples (wt%).

Minerals	SiO_2	Al_2O_3	TFe	MgO	CaO	TiO_2	Mn	Loss on Ignition
Kyanite	36.52	60.40	0.81	0.18	—	0.060	—	0.51
Quartz	99.31	—	0.08	—	—	—	—	—

The −0.074 + 0.045 mm fraction was used for the experimental flotation tests and contact angle analyses. Analytical grades of citric acid, NaOL, and $FeCl_3$ were obtained from Aladdin Industrial Corporation, Shanghai, China, NaOH and HCl solutions were used to adjust the pH, and deionized water was used for all experimental tests.

2.2. Microflotation Tests

Microflotation tests were performed using an XFG flotation machine manufactured with a 30 mL plexiglass cell [22]. Deionized water (20 mL) and the sample (2.0 g) were placed in the microflotation cell and mixed at a stirring speed of 1920 rpm. Figure 2 shows the flotation test procedure for the microflotation test (a mixture of kyanite and quartz in a mass ratio of 1:1). First, the slurry was stirred for 2 min to ensure that the minerals were mixed thoroughly. Subsequently, NaOH or HCl was then added to adjust the slurry pH for 2 min. In addition, $FeCl_3$, citric acid, and NaOL were then added sequentially, with each addition accompanied by pulp stirring for 3 min. Next, the froths were scraped manually with a plastic blade for 3 min. The froth products and scraping, drying, and weighing to calculate recovery. Furthermore, X-ray fluorescence analysis was performed to determine the chemical compositions of the artificially mixed mineral concentrations.

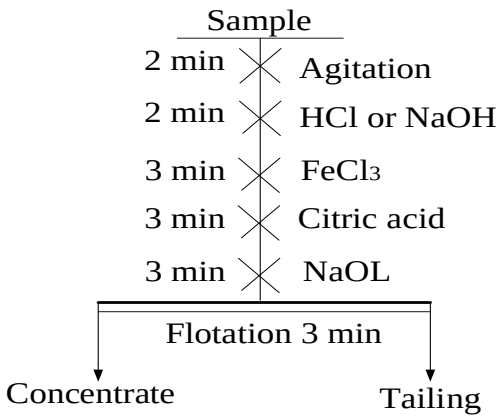


Figure 2. Procedure for flotation tests.

2.3. Contact Angle Tests

Contact angle measurements were detected using a OCA25 analyzer (Dataphysics). Then, 2 g of the sample were added to 20 mL of deionized water, and the pH of the pulp was adjusted. Subsequently, regulators were added during stirring on a magnetic stirrer, and the solution was stirred before 5 min. The pulp was adjusted based on the flotation process. Finally, the solid fraction was washed three times with distilled water and dried at 313 K for detect.

2.4. FTIR Spectroscopy Measurement

FTIR was tested using a FTIR-650S spectrometer at 298 ± 2 K within 4000–400 cm^{-1} [23]. The mineral samples were ground to approximately 5 μm. The solution was prepared using 1 g of the sample and 20 mL of distilled water. The pulp was adjusted based on the flotation process. Finally, the solid fraction was washed three times with distilled water and dried at 313 K. For the measurements, a 1 mg sample was mixed with 100 mg of spectrally pure KBr and pulverized using an agate mortar [24,25]. The resulting mixture was then pressed into a thin specimen for analysis.

2.5. XPS Measurement

XPS spectra were obtained using an Axis Ultra DLD Kratos AXIS SUPRA spectrometer. The binding energy scale was corrected using a C1s peak at approximately 284.80 eV as the internal binding energy standard [26,27]. A 2 g sample was added to 20 mL of distilled water and centrifuged at 2000 rpm, and the pulp was adjusted based on the flotation process. Finally, the centrifuged solid mineral was washed three times with deionized water and then dried at 313 K for analysis.

3. Results

3.1. Microflotation Tests

Flotation tests were performed using NaOL as the collector to observe the floatability of the untreated minerals. Several research studies have illustrated that kyanite and quartz can be efficiently separated in a weakly alkaline environment, and the NaOL concentration is approximately 140–160 mg/L [2,3,9,22]. The flotation test results at a pH of 7.5 and 150 mg/L of NaOL at increasing the $FeCl_3$ concentration are presented in Figure 3.

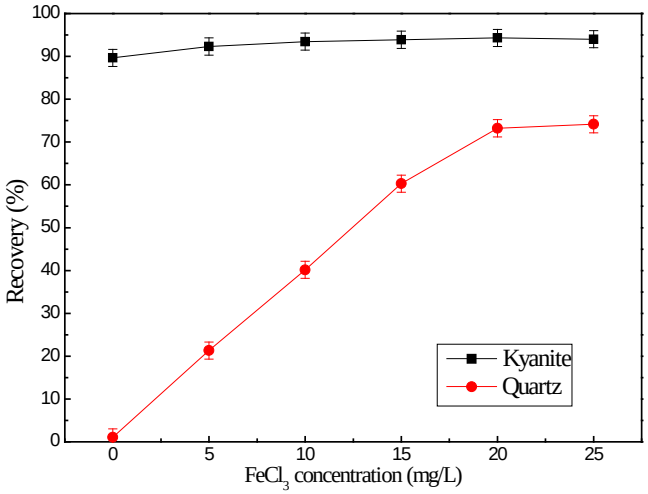


Figure 3. Effect of $FeCl_3$ concentration on flotation behavior of single minerals.

Within the evaluated range of the $FeCl_3$ concentration (Figure 3), the kyanite recovery fluctuated between 90% and 94%, illustrating that the kyanite exhibited good floatability. However, when the $FeCl_3$ content was increased from 0 to 20 mg/L, the quartz recovery increased from 1.06% to 73.22%, demonstrating that a certain amount of quartz could be floated [22]. With an increase in the $FeCl_3$ concentration, the difference in floatability between the kyanite and quartz decreased gradually, and the difficulty in separating kyanite from quartz increased progressively. These trends shown that $FeCl_3$ had a selective activation impact on quartz flotation, consistent with the results reported in the literature [3,22].

Flotation tests were performed using citric acid as a regulator to reduce the activation effect of ferric ions on the quartz during flotation. The flotation test results for the increasing citric acid concentration at a pH of 7.5, 40 mg/L NaOL, and 20 mg/L $FeCl_3$ are shown in Figure 4.

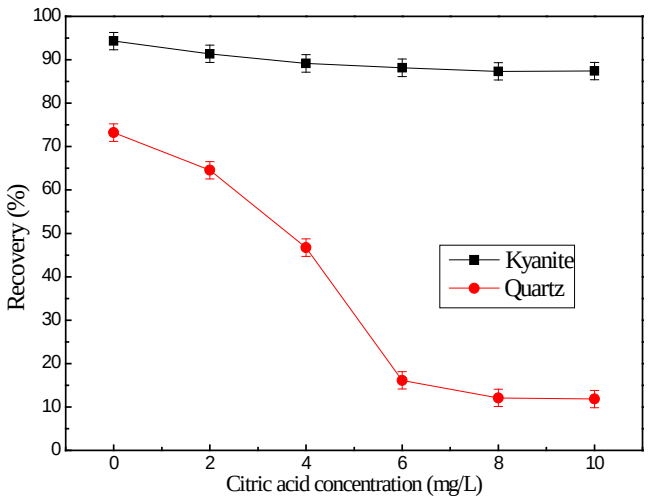


Figure 4. Effect of citric acid concentration on flotation behavior of single minerals.

Figure 4 shows that within the range of the citric acid concentration investigated in this study, the kyanite recovery decreased from 94.32 to 87.41 mg/L. Furthermore, the quartz recovery trend was similar to the kyanite recovery trend; when the citric acid was increased from 0 to 10 mg/L, the quartz recovery rate decreased from 73.22% to 11.83%. This result indicated that the citric acid could cause the depression of both the kyanite and quartz. However, the decrease in the quartz recovery was approximately 8.9 times that of the kyanite, and the recovery difference between the kyanite and quartz was the maximum when an 8 mg/L citric acid was added. Therefore, the citric acid could weaken the ferric ion effect on quartz flotation and minimally influenced the kyanite.

To further certificate the maximum flotation difference between the kyanite and quartz was measured at 8 mg/L citric acid content, the artificially mixed minerals (the mass ratio of kyanite and quartz is 1:1) was observed. The recoveries and grades of the minerals are listed in Table 2.

Table 2. Mixed-mineral flotation test results.

Flotation Conditions	Product	Yield (%)	Grade (%)		Recovery (wt%)	
			Al_2O_3	SiO_2	Kyanite	Quartz
NaOL: 150 mg/L; pH: 7.5; $FeCl_3$: 20 mg/L.	Concentrate	81.50	35.50	64.46	92.00	71.00
	Tailings	18.50	13.60	86.39	8.00	29.00
	Feed	100.00	31.45	59.26	100.00	100.00
NaOL: 150 mg/L; pH: 7.5; $FeCl_3$: 20 mg/L; Citric acid: 8 mg/L.	Concentrate	51.62	52.51	47.43	86.11	16.89
	Tailings	48.38	9.08	90.91	13.89	83.11
	Feed	100.00	31.45	59.26	100.00	100.00

When citric acid was added, the yield of the concentrate decreased from 81.50% to 51.62%, the Al_2O_3 grade increased from 35.50% to 52.51%, and the grade of SiO_2 decreased from 64.46% to 47.43% (Table 2). Moreover, the recovery of kyanite had little change, but the recovery of quartz decreased from 71.00% to 16.89% compared to NaOL + $FeCl_3$, confirming that the citric acid significantly weakened the effect of ferric ions on quartz flotation and enhanced the separation efficiency between the kyanite and quartz.

3.2. Contact Angle Tests

After the flotation agents were adsorbed onto the mineral surfaces, their surface potentials changed, significantly influencing mineral floatability [23,28]. Under conditions similar to those of the flotation tests, contact angle measurements were conducted (Table 3).

Table 3. Contact angles of mineral samples under different test conditions.

Test Conditions	Contact Angles of Minerals (°)	
	Kyanite	Quartz
Natural minerals	23	14
NaOL: 150 mg/L	92	17
$FeCl_3$: 20 mg/L; NaOL: 150 mg/L	97	72
$FeCl_3$: 20 mg/L; Citric acid: 8 mg/L; NaOL: 150 mg/L	91	22

In previous studies [22,29,30], the contact angles of natural kyanite and quartz were 23° and 14°, respectively, indicating that the minerals are hydrophilic. With only NaOL treatment, the contact angles of the kyanite and quartz obtained in this study were 92° and 17°, respectively, indicating that NaOL could significantly enhance kyanite hydrophobicity [3,19]. When $FeCl_3$ and NaOL were added, the contact angles of quartz increased from 17° to 72°, demonstrating that $FeCl_3$ significantly improved the interaction between NaOL and quartz, resulting in high hydrophobicity of quartz [22]. When $FeCl_3$, citric acid, and

NaOL coexisted, the contact angles of kyanite and quartz were 91° and 22°, respectively, suggesting that citric acid addition made the quartz surface hydrophilic; however, a slight influence on kyanite was observed, and the significant recovery of kyanite was maintained.

3.3. FTIR Measurements

The effects of the regulator on the NaOL adsorption onto the kyanite and quartz were investigated through FTIR measurements at a pH of 7.5 using 20 mg/L $FeCl_3$, 8 mg/L citric acid, and 150 mg/L NaOL. The FTIR spectra of the reagents and samples are shown in Figures 5 and 6, respectively.

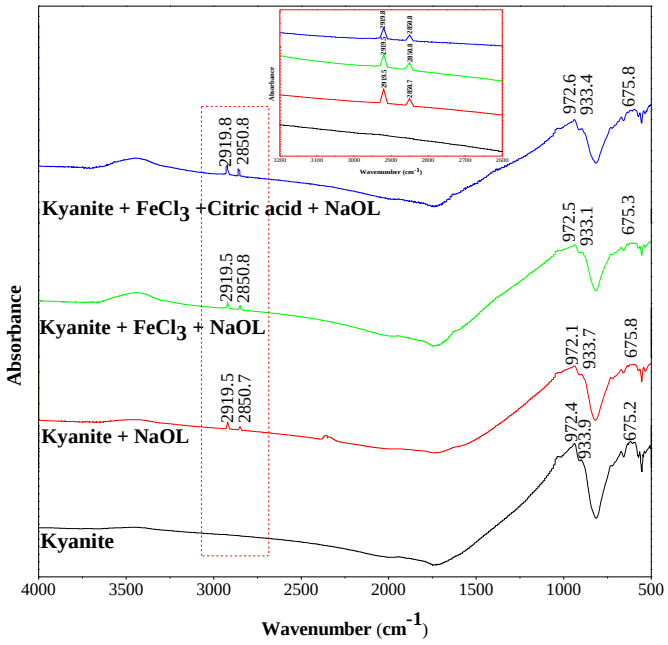


Figure 5. FTIR analysis of kyanite.

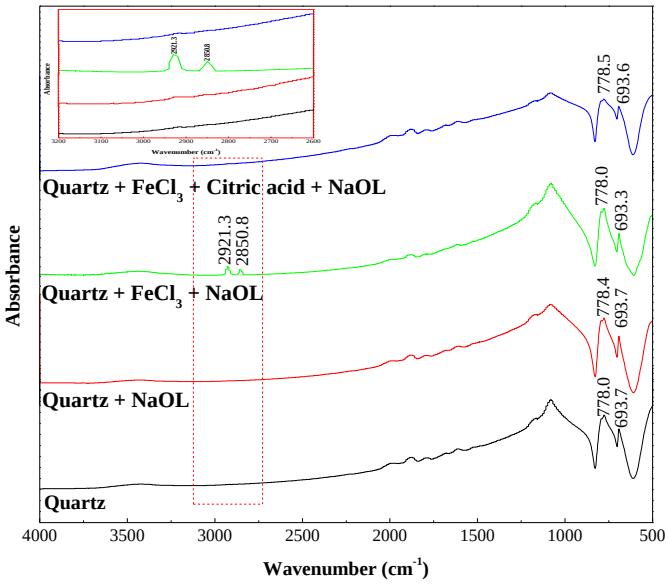


Figure 6. FTIR analysis of quartz.

The kyanite peak at approximately 675 cm^{-1} corresponded to the Si–O tetrahedron symmetrical bending vibration peak; however, the kyanite peaks at approximately 972 and 933 cm^{-1} were attributed to the Si–O asymmetric stretching vibration of the tetrahedron

peaks [22] (Figure 5). Figure 6 shows that the characteristic peaks of quartz at approximately 778 and 693 cm^{-1} correspond to Si–O symmetrical bending vibrations and Si–O–Si asymmetrical bending vibrations, respectively [23,31].

When NaOL and kyanite coexist, the new peaks appearing at 2918.5 and 2850.7 cm^{-1} corresponded to the symmetrical of $–CH_3$ and asymmetric stretching vibrations of $–CH_2$ in NaOL, respectively [28,32] (Figure 5). These results suggested that the NaOL was adsorbed onto the kyanite surface. For the kyanite treated with $FeCl_3$ and NaOL, symmetrical vibrations of methyl $–CH_3$ and asymmetric stretching vibrations of methylene $–CH_2$ were detected, and the same peaks were detected in the kyanite treated with $FeCl_3$, citric acid, and NaOL. These results indicated that the $FeCl_3$ and citric acid regulators had negligible effects on the adsorption of NaOL on the kyanite surface.

The FTIR spectra of natural quartz and quartz + NaOL were similar, indicating that NaOL was not adsorbed onto the quartz surface (Figure 6). With the addition of $FeCl_3$ and NaOL, the new peaks appearing at 2921.3 and 2850.8 cm^{-1} corresponded to the symmetrical of $–CH_3$ and asymmetric stretching vibrations of $–CH_2$ in NaOL, respectively [28,32]. Hence, $FeCl_3$ facilitated NaOL adsorption onto the quartz surface. For the quartz treated with $FeCl_3$, citric acid, and NaOL, the characteristic peaks of quartz were somewhat equal to those of the quartz treated with only NaOL. This result indicated that the addition of citric acid eliminated the activation of $FeCl_3$ on the quartz, resulting in nonadsorption of NaOL onto the quartz surface.

3.4. XPS Measurement

XPS measurements were performed for kyanite and quartz at a pH of 7.5, 20 mg/L $FeCl_3$, and 8 mg/L citric acid to identify the reaction mechanisms between the regulator and the minerals. The binding energy results for the kyanite and quartz are shown in Figures 7 and 8, respectively.

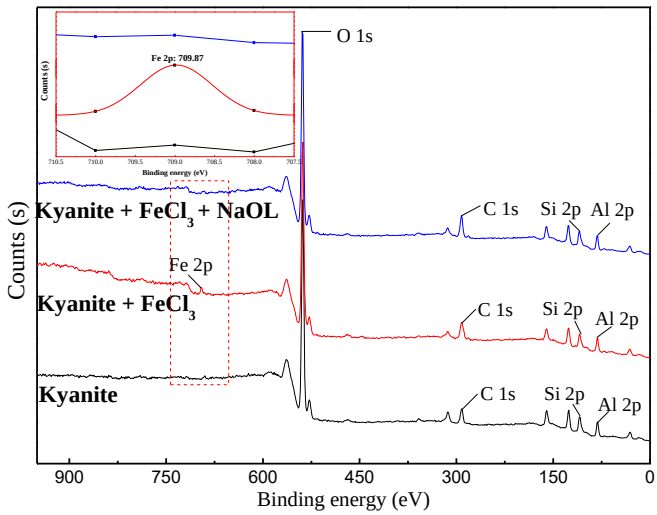


Figure 7. Binding energy of kyanite with and without regulator treatment.

The XPS measurements could not detect Fe on the natural kyanite and quartz surfaces (Figures 7 and 8). In the presence of $FeCl_3$, Fe 2p peaks appeared on the kyanite and quartz surfaces at 709.87 and 710.64 eV, respectively, suggesting that Fe species could precipitate on the surfaces [22]. For the kyanite and quartz treated with $FeCl_3$ + citric acid, the characteristic peaks of Fe 2p disappeared, indicating that the citric acid prevented the adsorption of Fe species onto the kyanite and quartz surfaces.

The relative contents of surface elements of minerals with and without regulator treatment were analyzed (Table 4) to evaluate the effect of the citric acid on the mineral adsorption of ferric ions.

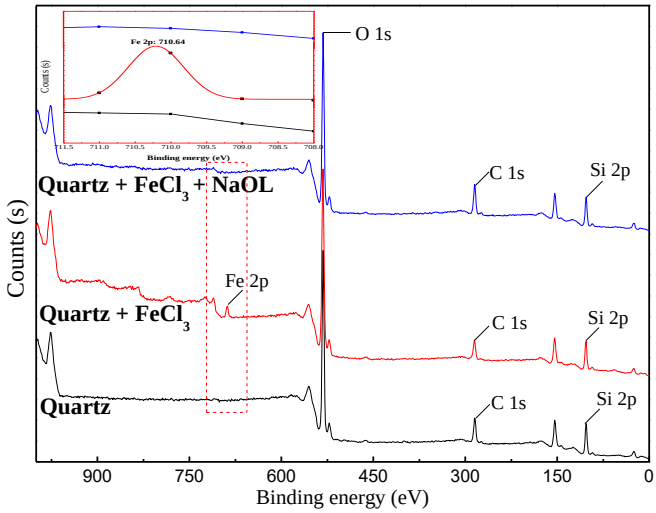


Figure 8. Binding energy of quartz with and without regulator treatment.

Table 4. Content of element of samples surface with and without regulator treatment.

Minerals	Reagent	Content of Element (%)				
		C 1s	O 1s	Si 2p	Al 2p	Fe 2p
Kyanite	None	14.51	56.34	12.92	16.24	—
	$FeCl_3$	17.20	53.48	12.01	15.73	1.57
	$FeCl_3$ + Citric acid	17.94	53.14	12.23	16.57	0.12
Quartz	None	12.18	55.35	32.48		—
	$FeCl_3$	18.34	52.67	26.97		2.02
	$FeCl_3$ + Citric acid	22.13	49.28	28.50		0.09

The atomic percentages of Fe 2p in the kyanite and quartz surfaces when they were treated with only $FeCl_3$ were 1.57% and 2.02%, respectively (Table 4). However, when citric acid was added after $FeCl_3$, the atomic percentages of Fe 2p on the kyanite and quartz surfaces were 0.12% and 0.09%, respectively. These results showed that ferric ions in the solution were adsorbed onto the kyanite and quartz surfaces, and the citric acid could decrease the ferric ion content adsorbed onto the kyanite and quartz surfaces.

4. Conclusions

Based on the above results, the following conclusions are drawn.

1. The addition of citric acid weakened the effect of ferric ions on quartz flotation and had minimal influence on kyanite.
2. In the presence of NaOL and $FeCl_3$, the addition of citric acid made the quartz surface hydrophilic again but also slightly deceased the hydrophobicity of the kyanite. Thus, the citric acid maintained the significant recovery of kyanite.
3. Citric acid could eliminate the activation of $FeCl_3$ on the quartz, resulting in the nonadsorption of NaOL onto the quartz surface. However, the $FeCl_3$ and citric acid had a negligible effect on NaOL adsorption onto the kyanite surface.
4. Citric acid decreased the ferric ion content adsorbed onto the kyanite and quartz surfaces.

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