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Flotation separation of quartz from phosphorite using an imidazole ionic liquid collector and its adsorption mechanism

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Abstract: In this study, an imidazole ionic liquid (dodecyl-tri-methylimidazolium chloride) was employed as a collector to separate quartz from phosphorite. The micro-flotation experiments of a single mineral found that it had selective collecting ability for quartz than phosphorite. Mixed mineral flotation experiments confirmed that efficient separation results could be obtained using the imidazole ionic liquid as the collector. A concentrate with a 31.44% grade of phosphorite could be obtained with a 0.285 kg/Mg collector dosage at neutral pH, which was much better than the traditional collector dodecylamine. The adsorption mechanism of the imidazole ionic liquid on the surface of phosphorite and quartz was investigated by contact angle and zeta potential measurements, Fourier transform infrared and X-ray photoelectron spectroscopy analyses. These results showed that the adsorption of imidazole ionic liquid at the quartz surface was stronger than that of phosphorite, and the collector adsorbability difference between quartz and phosphorite resulted in the efficient flotation separation. Consequently, the dodecyl-tri-methylimidazolium chloride salt is an effective collector for reverse flotation of quartz from phosphorite.

Keywords: reverse flotation, imidazole ionic liquid, desilication, phosphorite, quartz

1. Introduction

With the development of society, high-grade collophanite is becoming less and less. At present, low-grade collophanite is mainly used in China (Li et al., 2016; Li et al., 2021). To realize the sustainable development and utilization of phosphorite, the focus of current research must be shifted from rich ore to low-grade phosphorite (Li et al., 2021). Because quartz is the main gangues existing in phosphorite, removing silica minerals is the key factor for developing and utilizing phosphorite.

At present, the more advanced process is the double reverse flotation process, which usually takes off magnesium first and then desilicates. The process of magnesium removal in dual reverse flotation is relatively advanced, but the variety of cationic desilication reagents in phosphorite research is limited (Yang et al., 2021). After the removal of magnesium from collophanite, dodecyl trimethylsilyl ammonium chloride was employed for desilication, which presented a better sorting ability and defoamation ability than dodecylamine and ether amine (Liu et al., 2018). It was reported from the results of Liu et al. (2018) that a cationic reverse flotation desilication collector was used in a coarse separation experiment of a low-grade calcination colophite in Hubei province. The grade and recovery of phosphorite concentrate can reach 31.55% and 79.73%, respectively (Liu et al., 2018; Rao et al., 2020). The combination of kerosene and quaternary ammonium cationic as the collector can selectively adsorb on the surface of silicon-containing minerals from phosphorite with a particle size of 0.6~1.2 mm (Snow

et al., 1988; Xu et al., 2019). However, the traditional agents normally cause environmental pollution with high dosage (Ai et al., 2021; Liu et al., 2021), so the researchers still need to find a new type of environmentally friendly and economic collector. An imidazolium ionic liquid was selected in this study, which was a new type of soft functional material widely used in various applications due to its excellent physical and chemical properties, such as the broad liquid flow, low volatilization, and vapor pressure, high thermal stability, and non-toxic (Winterton et al., 2006).

Ionic liquids are salts made of anions and cations that present a liquid state at room temperature or below 100°C (Qi et al., 2014). It is also widely used in mineral flotation fields. For example, the ionic liquids of Aliquat-336 (C₂₅H₅₄ClN) and TOMAS (Tricaprylmethyl ammonium salicylate) have been utilized as collectors in the froth flotation of quartz. They are quaternary ammonium-based ionic liquids, where the ammonium head can easily adsorb onto the quartz surface through electrostatic interaction, and the alkyl groups cause surface hydrophobicity (Sahoo et al., 2014; 2015). The quaternary ammonium salt cationic collector ionic liquid can easily dissolve in water, which still has a good collecting ability in the presence of depressants (Li et al., 2019). The development of ionic liquid promoted the development of oxidized ore flotation desilication. It will become one of the important research directions of oxidized ore in the future. At present, the ionic liquid collector is mainly used in reverse flotation of silicate minerals from iron ore and magnesite, but its application in phosphorite has not been studied yet.

In the reverse flotation of phosphorite, the selection of inhibitors is also important (Xie et al., 2010; Zhou et al., 2013; Derhy et al., 2020). Sulfuric acid, phosphoric acid, and their derivatives are commonly used for phosphate minerals. But the traditional regulator-Na₂CO₃ will have a neutralization reaction with the acid inhibitor, the agent may be wasted and cannot have a strong inhibitory effect (Luo et al., 2020). Therefore, the inhibitor selected in this experiment is sodium tripolyphosphate, which is alkaline and does not react with acid regulators.

Therefore, this work selected an imidazole ionic liquid (dodecyl-tri-methylimidazolium chloride salt) as a collector for the reverse flotation of quartz from phosphorite. The interaction mechanism of the collector onto mineral surfaces was revealed by contact angle test, zeta potential, Fourier transform infrared, and X-ray photoelectron spectroscopy.

2. Materials and methods

2.1. Materials

The high purity samples of quartz and phosphorite were obtained from Guangzhou Mingfa Mineral Specimen Manufacturing Co., Ltd, China. The chemical compositions are shown in Tables 1 and 2.

Table 1. Chemical compositions of quartz (wt%)

Minerals	P ₂ O ₅	SiO ₂	K ₂ O	CaO	Al ₂ O ₃	CO ₂
Quartz	0.23	98.23	0.03	0.31	0.11	1.02

Table 2. Chemical compositions of phosphorite (wt%)

Minerals	P ₂ O ₅	SiO ₂	F	CO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃
Phosphorite	36.67	1.06	3.85	1.15	55.43	0.03	0.04

According to the chemical analysis result (Tables 1 and 2), P₂O₅ in phosphorite was 36.67%, while SiO₂ in quartz was 98.23%. Quartz mineral has less impurity content, and its high purity could eliminate the interference of impurity on the experimental results (Liu et al., 2021).

In this work, the collector of dodecyl tri-methyl-imidazolium chloride salt has the advantage of simple preparation conditions for the dissolution point of 32~34°C, which was obtained from Lanzhou chemical physics research institute of Chinese Academy of Sciences, China (the structural formula is shown as Fig. 1). The phosphate inhibitor of sodium tripolyphosphate was obtained from Tianjin Hengxing Chemical Reagent Manufacturing Co., Ltd, China. The dodecylamine used in the comparative experiment was purchased from Shanghai Macklin Biochemical Co., Ltd, China. The inhibitor is soluble starch from Sinopharm Chemical Reagent Co., Ltd, China. Sodium hydroxide and

hydrochloric acid were used to adjust the pulp pH.

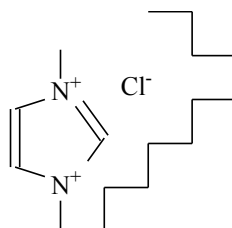


Fig. 1. The structural formula of dodecyl-tri-methylimidazolium chloride salt

2.2. Methods

2.2.1. Micro-flotation experiments

The flotation tests of each pure mineral and artificially mixed minerals were carried out in an XFG-type flotation machine in 30 cm³ and 60 cm³ cells at 10% pulp density. In each test, 2 g mineral samples were dispersed in the 30 cm³ cell with 20 cm³ deionized water. The pulp pH was adjusted to the appropriate value and stirred for 2 min with 1300 rpm. Then, an ionic liquid collector or inhibitor was added to the pulp and stirred for 2 min. The aeration flotation was conducted for 4 min. Finally, the froth products and the tailings were collected and weighed after drying for calculating the recovery and grade. As for the flotation of mixed minerals, quartz and phosphorite were mixed at a certain ratio (1:2) as the feed mineral samples using a 60 cm³ cell. Flotation procedures were followed in the same way.

2.2.2. Contact angle experiments

The contact angle of quartz and phosphorite was measured using the KRUSS-DSA100 droplet analyzer. Experiments were conducted in a cubic glass chamber and the humidity of the chamber was controlled by an automatic humidity controller to 70±2%. The mineral sample was first interacted with the reagent solution and then suppressed to a flat surface for measurement. Each test was performed three times, and the average result was reported as the final value.

2.2.3. Zeta potential measurements

Malvern Zetasizer Zeta-Nano was used to measure the zeta potentials of the mineral. The pure mineral sample was ground to < 5 μm and weighed 10 mg in a beaker for preparation. The NaOH and HCl solutions with certain concentrations were used to adjust a series of pH, which were added to the beaker of pure minerals. The required concentrations of collector and inhibitor were added afterward. After magnetic stir for 10 min, the mineral suspension was filtered, dried, and sent for measurement. Each condition was conducted for three independent tests and averaged as the final result.

2.2.4. Fourier transform infrared (FTIR) analysis

Fourier transform infrared (FTIR) spectra were used to analyze the minerals samples by the Nicolet6700. 2 g fine phosphorite was ground in an agate mortar under 5 μm, then put in a beaker with 50 cm³ distilled water. Afterward, the ultrafine mineral powders were put into a solution with a certain concentration of collector/inhibitor, and magnetic stirrer for 30 min. Then, the mineral suspension was filtered and washed with distilled water more than three times and put in the vacuum drying oven under 40°C overnight. Finally, it can be sent for the FTIR analysis.

2.2.5. X-ray photoelectron spectroscopy (XPS)

X-ray photoelectron spectroscopy was used to analyze the element distributions at mineral surfaces before and after collector adsorption using the Escalab 250XI. The mineral samples were ground to < 5 μm and then added to the beaker with the required concentration of ionic liquid collector. Afterward, it was stirred for 30 min and then filtered, washed, and dried in the vacuum drying oven for tests. Note that the XPS results have been calibrated with C1s (284.80 eV).

3. Results and discussion

3.1. Characterization of collector

The FTIR spectra for the imidazole ionic liquid collector is seen in Fig. 2. 3063, 3084, and 3152 cm^{-1} bands are related to the stretch vibration absorption of =C-H from aromatic hydrocarbon. The symmetric, antisymmetric, and flexural vibrations of CH_3 , CH_2 , and CH are in 2919 cm^{-1} , 2852 cm^{-1} , 1473 cm^{-1} , and 729 cm^{-1} . The sharp bands in 1573 cm^{-1} , 1473 cm^{-1} were attributed to the C=C stretch vibration from aromatic hydrocarbon. The bands between 1317 cm^{-1} were related to the in-plane bending vibration of =CH from aromatic hydrocarbon. The bands at 862 cm^{-1} were attributed to out-plane bending vibration of =CH from an aromatic hydrocarbon. The band in 3477 cm^{-1} was related to the stretching vibration absorption of N-H, and the stretching vibration absorption peak of the imidazole ring was located at 1179 cm^{-1} . All peak patterns in different regions represented the ionic liquid.

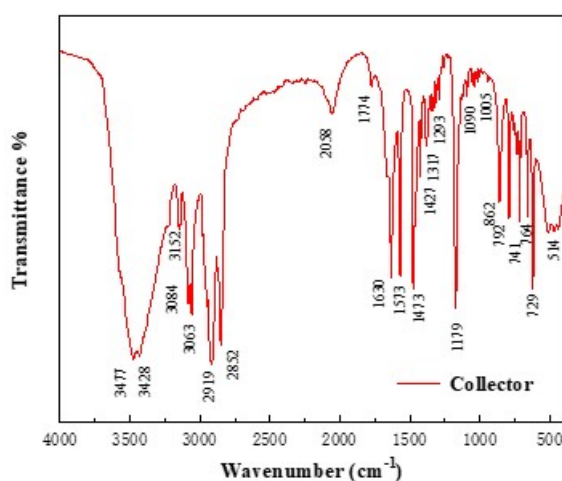


Fig. 2. Infrared transmission spectrum of ionic liquid (dodecyl-tri-methylimidazolium chloride salt)

3.2. Micro-flotation experiments

3.2.1. Effects of collector concentration and pulp pH on the mineral flotation

The addition of agents can effectively improve the hydrophobic degree on mineral surfaces, hence increasing the floatability of minerals (Zhang, 2020). Figure 3 found that the recovery of quartz and phosphorite increased with the increasing collector dosage. Quartz recovery increases slowly after 0.7125 kg/Mg, while phosphate recovery rate increases slowly after 1.425 kg/Mg. When the collector concentration reached 0.285 kg/Mg, the recoveries of quartz and phosphorite were 92% and 22%, respectively. At this time, the recovery difference between quartz and phosphorite reaches the maximum, which is conducive to the embodiment of mineral selectivity of the collector.

Figure 4 shows the flotation recovery of quartz and phosphorite as a function of pH. The recovery difference between quartz and phosphorite was the largest at pH 7. As a result, a better separation between phosphorite and quartz using the ionic liquid can be achieved at a neutral pH.

3.2.2. Mixed mineral flotation experiments

Based on the results of the single-factor flotation experiment, the optimal flotation conditions of the reagents were determined. The flotation process was carried out under the optimal flotation conditions of dodecylamine, ensuring the same amount of collector and the best inhibition effect (Smith et al., 1990; Wu, 2017). Thus, the mixed minerals flotation tests in the 1:2 mass ratio of quartz: phosphorite were also conducted in this study, and the results are shown in Table 3. It can be seen from Table 3 that the grade of P_2O_5 in the concentrate increased from 24.31% to 31.44%, and the grade of quartz decreased from 32.74% to 20.02%, showing that the results were better than dodecylamine (The grade of P_2O_5 in the concentrate is 26.51%, and quartz is 32.44%). The experimental results of artificially mixed ore flotation confirmed that the ionic liquid collector has an excellent flotation performance and can effectively

separate phosphorite and quartz. The beneficiation efficiency of dodecyl-tri-methylimidazolium chloride salt is better than that of traditional collector dodecylamine.

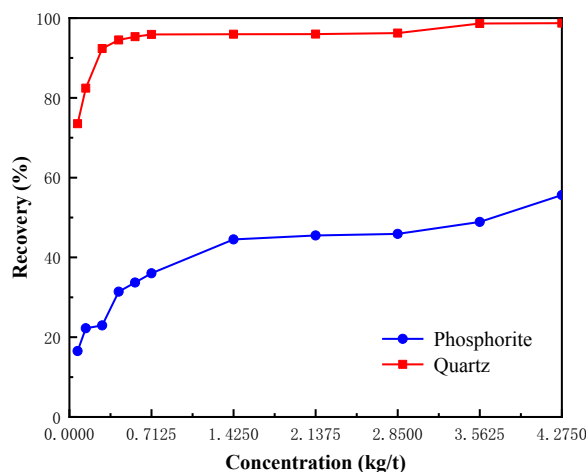


Fig. 3. Effects of imidazole ionic liquid concentration on the flotation at neutral condition

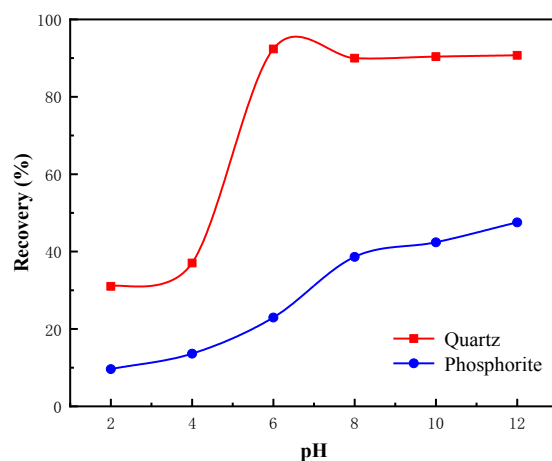


Fig. 4. Effects of pulp pH on the flotation with 0.285 kg/Mg imidazole ionic liquid

Table 3. Comparison between artificial mixed ore reverse flotation results and traditional reagent

	Product	Yield (wt.%)	Grade (%)		Recovery (%)		Efficiency (%)
			P ₂ O ₅	SiO ₂	P ₂ O ₅	SiO ₂	
Imidazole ionic liquid collector (0.285 kg/Mg)	Concentrate	76.00	31.44	20.02	93.15	11.36	66.08
	Tailings	24.00	1.73	73.02	6.85	88.64	
	Feed	100.00	24.31	32.74	100.00	100.00	
Dodecylamine (0.285 kg/Mg)	Concentrate	79.88	25.81	32.44	93.35	20.87	48.79
	Tailings	20.12	18.35	33.93	6.65	79.13	
	Feed	100.00	24.31	32.74	100.00	100.00	

3.3. Contact angle of minerals before and after treating with imidazole ionic liquid

According to the relevant research (Li et al., 2015), the critical micelle concentration (CMC) of the imidazole ionic liquid (dodecyl-tri-methylimidazolium chloride) is about 20 mmol/dm³. In this study, the collector dosage was only about 0.02 mmol/dm³, which is far less than the CMC value. Thus, the contact angle measurements were conducted using the corresponding collector dosage as mixed mineral flotation experiments.

Quartz and phosphorite were separately treated in the absence and presence of imidazole ionic liquid. As shown in Fig. 5(a), the bare quartz substrate showed 45° contact angle. After the quartz interacted with the collector, the contact angle increased to 100° (Fig. 5(b)). Figure 5(c) revealed a 21.5° contact angle for the bare phosphorite substrate. After treating with the collector, the contact angle improved to 56.50° (Fig. 5(d)). Thus, the addition of imidazole ionic liquid could selectively make quartz hydrophobic. These results also indicated that the adsorption density of imidazole ionic liquid at the quartz surface was higher than that on the phosphorite surface.

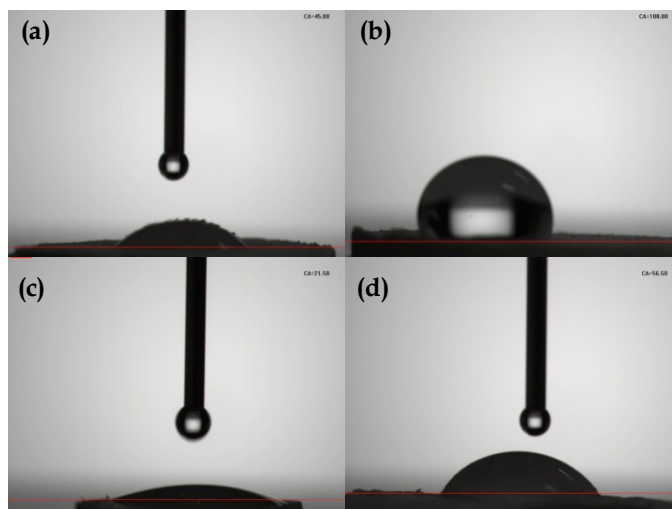


Fig. 5. The contact angle of quartz (a) before and (b) after the treating with imidazole ionic liquid; the contact angle of phosphorite (c) before and (d) after interacting with imidazole ionic liquid

3.4. Adsorption mechanism

The zeta potentials of quartz and phosphorite before and after interacting with the imidazole ionic liquid are shown in Fig. 6. In the studied pH range, the zeta potential of quartz increases much more than that of phosphorite. Especially at pH 6, the zeta potential of quartz was increased from -43 mV to 5 mV, while the zeta potential of phosphorite only reduced from -4 mV to -2 mV. The results showed that the imidazole ionic liquid collector has a more obvious adsorption effect on the quartz surface.

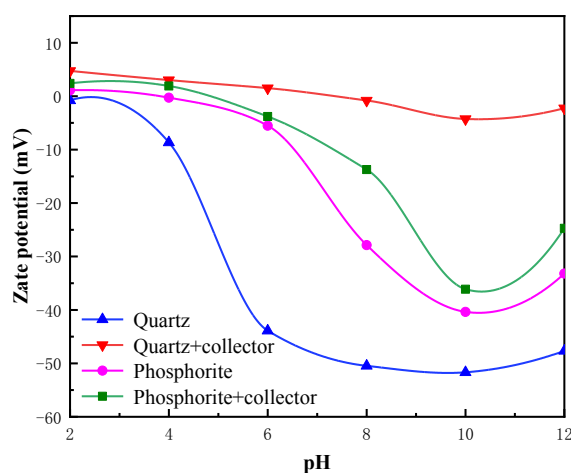


Fig. 6. Zeta potentials of quartz and phosphorite as a function of pH with or without collector

Figs. 7 and 8 showed the FTIR spectra of quartz and phosphorite before and after interacting with the imidazole ionic liquid collector. The bands found at 3436 cm^{-1} and 3446 cm^{-1} for quartz were related to the adsorbed water. The peaks at 1618 cm^{-1} were attributed to the stretching vibration of $\text{C}=\text{C}$ from aromatic hydrocarbon. In the infrared spectrum of quartz, two new absorption peaks formed at 2924 cm^{-1} and 2848 cm^{-1} after the collector adsorption (Ming et al., 2004; Nayak et al., 2007). The two new

peaks were attributed to adsorbed imidazole ionic liquid collector and primarily corresponded to vibrations of methyl and methylene of dodecyl-tri-methylimidazolium chloride salts. It indicated that the collector on the quartz surface was mainly caused by electrostatic physical adsorption (Zhou, 2016).

In the infrared spectrum of phosphorite, most of the peaks were nearly not changed. In addition, new characteristic peaks were found at 1789 cm^{-1} (C=O) after interacting with collector and inhibitor, indicating that the chemical adsorption of reagents onto the phosphorite surface (Weng, 2014). From the offset degree of inhibitor characteristic peak, the strong interaction between inhibitor and phosphorite can be inferred, leading to selective adsorption of inhibitor onto the phosphorite surface, preventing the collector adsorption. This behavior was consistent with the mixed mineral flotation.

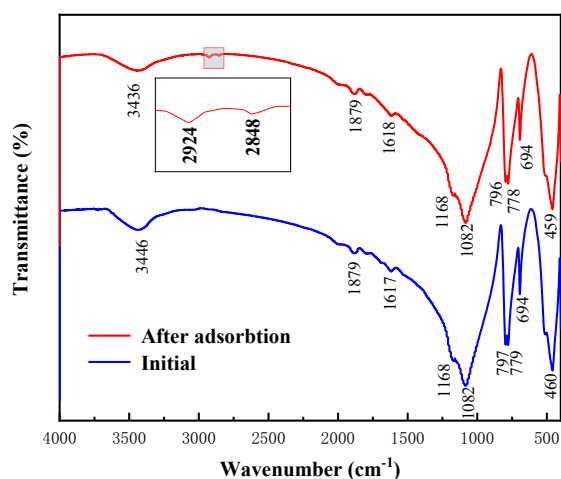


Fig. 7. FTIR of quartz before and after adsorption with ionic liquid

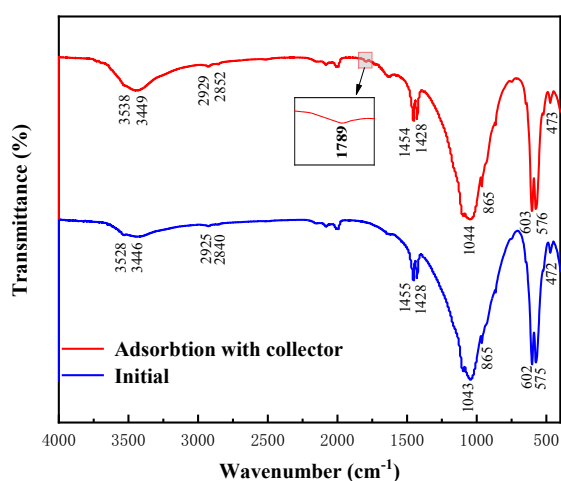


Fig. 8. FTIR of phosphorite before and after adsorption with ionic liquid

XPS analysis was carried out on quartz and phosphate ore after treating with the collector. As collectors contained the characteristic element N, a detailed analysis was mainly carried out on the N1s, and the results were shown in Figs. 9(a) and (b). Figure 9(a) shows the N1s spectrum of quartz after interacting with the imidazole ionic liquid collector. Several peaks could be observed in this spectrogram, mainly because the two nitrogens on the imidazole ring were in different states after collector adsorption. It was also found that the peak was located at about 402.28 eV binding energy, which was consistent with the characteristic peak of typical amino N1s reported in the literature (Aydin et al., 2013). In Figure 9(b), a weak N signal was detected on the surface of phosphorite after the interaction with the imidazole ionic liquid collector. It indicated that the collector adsorption on the phosphorite surface was much smaller than the quartz surface. Thus, XPS results revealed the different adsorption behavior of the collector at phosphorite and quartz surface.

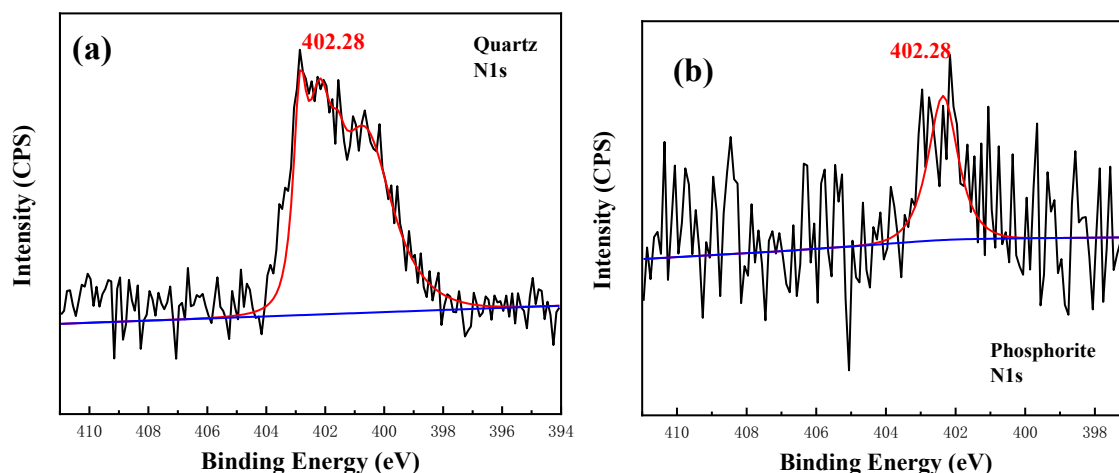


Fig. 9. XPS N1s of (a) quartz and (b) phosphorite after adsorption by the imidazole ionic liquid

4. Conclusions

In this study, a novel imidazole ionic liquid (dodecyl-tri-methylimidazolium chloride) was first applied in the reverse flotation of quartz from phosphorite. The micro-flotation tests of single minerals showed that imidazole ionic liquid had selectively collecting ability for quartz. Thus, the efficient flotation separation of quartz from phosphorite might be achieved using this imidazole ionic liquid as the collector. Mixed mineral flotation tests found that 31.44% P_2O_5 grade and 93.15% flotation recovery of phosphorite concentrate could be achieved using 0.285 kg/Mg imidazole ionic liquid as the collector, the flotation performance of which is much better than the collector dodecylamine. Contact angle measurement revealed that quartz became more hydrophobic than phosphorite after treating with the collector. Zeta potential measurements showed that the addition of imidazole ionic liquid could positively change the zeta potentials of minerals, while the quartz curve was moved more obvious than phosphorite, indicating the stronger adsorption of collector onto quartz surface. Fourier transform infrared and XPS analysis showed that this imidazole ionic liquid could be selectively adsorbed onto quartz surface via chemisorption. Based on the works mentioned above, it could be claimed that dodecyl-tri-methylimidazolium chloride is an efficient collector for the flotation separation of quartz from phosphorite.

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