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The mechanism study on aryl-substituted aromatic acid ionic liquid as the collector for quartz flotation

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Abstract: An aryl-substituted aromatic acid ionic liquid (M-X) was synthesized through hexadecyl trimethyl ammonium bromide and sodium salicylate, and it was employed as a flotation collector for the separation of quartz from magnetite. Laboratory flotation studies of magnetite and quartz were conducted using the M-X and dodecylamine (DDA) as collector. The results showed that the M-X has a stronger collecting ability and selectivity for quartz without starch. The adsorption mechanism of M-X on quartz and magnetite surfaces was studied by Fourier transform infrared spectroscopy (FTIR), zeta potential and X-ray photoelectron spectroscopy (XPS) analysis. Results indicated that the adsorption of M-X on the quartz surface was more efficient than that on the magnetite surface. And the salicylate anion in M-X was found to have depression effect to magnetite.

Keywords: ionic liquid, flotation collector, quartz, magnetite

1. Introduction

Quartz is one of the most common gangues existed in a large amount of natural mineral groups. It needs to be suitably separated by froth flotation for achieving desirable concentrate of valuable mineral from gangue. For it's usually found that the lower grade ores have sophisticated mineralogical textures. In addition, the mineral particle values are often liberated at finer size. Thus, flotation can be one of the best processes for the selective separation of target minerals that are crushed at finer sizes.

There are mainly two types of magnetite ore collectors used in flotation process. One is anionic collectors which contain a hydrocarbon and alkali salts of straight chain fatty acids, alkyl aryl sulfonates or cationic collectors containing a hydrocarbon group, and the salts of bases, usually chlorides or acetates (Sahoo et al., 2015). Another is cationic collectors mostly contain primary aliphatic amines, diamines, and quaternary ammonium salts (Smith and Scott, 1990). Reverse cationic flotation of silica is so far the most efficient method for the separation of magnetite ore with silica gangue and widely used in the separation of quartz bearing impurities (Hedberg, 1970). And it is often effective for the separation of magnetite ore and silica gangue by feeding finer particles in reverse flotation (Englert et al., 2009). The quartz is floated using aliphatic amines, monoamines, diamines and quaternary ammonium salts such as dodecylamine (DDA), cetyl trimethylammonium bromide (CTAB), quaternary ammonium salts, ether or ester amines (Papini et al., 2001). Filippov and Severov have widely reported the type and molecular structure of the collectors as well as depressants on reverse cationic flotation of quartz (Filippov et al., 2014). The most utilized fatty amines are no longer employed for its low solubility. And so they have been replaced by ether amines. The ether amines are much more soluble with the presence of extra hydrophobic group, and more effective in comparison with DDA (Araujo et al., 2005; Lima et al., 2005). Some other studies relating to amines and diamines have also achieved better performance in the flotation of quartz (Liu et al., 2009; Vieira and Peres, 2007). It was reported that a novel quaternary

ammonium surfactants containing ester bonds demonstrated stronger selectivity and collectability than DDA for the flotation of quartz (Weng et al., 2013; Wang and Ren, 2005). A Gemini surfactant, ethane-1,2-bis(dimethyl-dodecyl-ammonium-bromine) presented stronger collecting power and superior selectivity than the dodecylammonium chloride for quartz (Huang et al., 2014). However, these collectors still have the disadvantage of large dosage, environmental pollution and other problems (Xia et al., 2009). So, it's necessary to find new collectors with less dosage and less pollution.

The ionic liquid Aliquat-336 (Sahoo et al., 2015) and TOMAS (Sahoo et al., 2014) have been utilized and studied in the froth flotation as collectors for quartz in relating papers. Aliquat-336 and TOMAS are quaternary ammonium based ionic liquids, where the ammonium head is responsible for getting attached to the quartz surface electrostatically and the bulky alkyl groups cause the hydrophobicity (Sahoo et al., 2015). Because of their extremely low vapour pressure, wide temperature range and chemical stability (Qi et al., 2014; Neves et al., 2014). Ionic liquids showed a significant potential as phase transfer catalysts or extractants, and they were also considered to be able to replace the conventional organic solvents (Ferreira et al., 2014; Li et al., 2014).

In this context, we synthesized a novel quaternary ammonium based ionic liquid with a long alkyl chain and the collecting capabilities for quartz was studied. Characterization studies have been performed to support the flotation test. Furthermore, results of flotation tests on a mixture of quartz and magnetite was reported in the paper. And the results of flotation tests using ionic liquid in comparison with dodecylamine have also been investigated.

2. Experimental

2.1. Materials

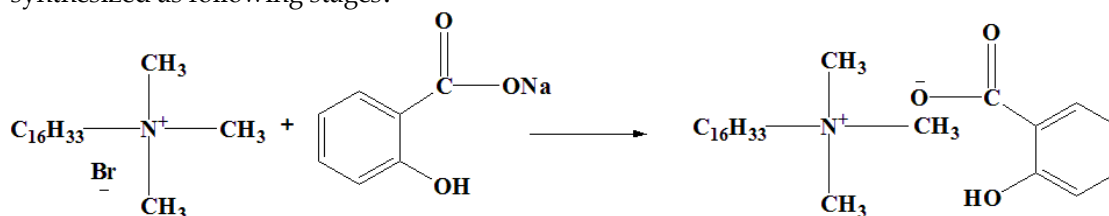
The high purity quartz samples obtained from The Julius Kruttschnitt Mineral Research Center of university of Queensland in Australia, were ground to 50% of which finer than 200 μ m, assaying 99.9% SiO₂ were used in the flotation experiment. Pure refined magnetite ore obtained from the NCS Testing Technology Co., Ltd of China. The samples were below 88 μ m and used for flotation experiment. The chemical analysis results of the refined magnetite ore were listed in Table1.

Table 1. Chemical analysis results of magnetit (mass fraction, %)

Element	TFe	FeO	SiO ₂	CaO	MgO	V ₂ O ₅	MnO
Content%	70.69	27.98	0.293	0.066	0.045	0.258	0.28
Element	P	S	K ₂ O	Na ₂ O	Al ₂ O ₃	TiO ₂	Zn
Content%	0.012	0.0031	0.012	0.010	0.32	0.535	0.030

2.2. Reagents

The quaternary ammonium based ionic liquid (M-X) used in the flotation studies as collector was synthesized as following stages:



This compound was synthesized by the reaction between the hexadecyl trimethyl ammonium bromide and sodium salicylate. The mixture was stirred in the trichloromethane for 12h with suitable temperature until the liquid was transparent. Resulting product was washed with deionized water. The rest liquid was heated in the rotary evaporator until the solvent evaporated. And then the product was obtained after cooling under room temperature.

The dodecylamine (DDA) was mixed with acetic acid in a certain ratio, then configured into collector with deionized water. The concentration of M-X and DDA was ranged from 0.0025mmol/L to

0.06mmol/L. The pH regulator was made from NaOH and HCl solutions (5% aqueous solution). The depressant was made through mixing anionic starch and deionized water.

2.3. Methods

2.3.1 Procedure for flotation test

The flotation tests of pure quartz and magnetite were carried out in a XFG-type laboratory flotation machine in a 40cm³ cell with a 10% pulp density. The agitation time was 2 min with 2000rpm. And then the pulp pH was conducted at desired value by NaOH or HCl solutions. The collectors were added into the cell and the conditioning time was extra 2 min. The air was introduced into the pulp to form froth. The flotation time was maintained at 4min. The concentrate and tailings were collected severally, dried, weighed and then they were analysed and calculated to determine the grade or recovery. The flotation for artificial mixed ore of quartz and iron was made at a certain ratio (1:3) and it was conducted in the same way as mentioned above.

2.3.2. Zeta potential measurement

All the zeta potential measurements were conducted by a Brookhaven Zeta Plus Zeta-potential analyzer (USA). The samples used for zeta potential measurement were ground to -5 μ m. A mineral suspension containing 0.1g pure quartz or magnetite ore was prepared in a NaCl solution at 10⁻³ mol/L with 100cm³ beaker. And all the pH was conducted at desired value. After the zeta potential measurement was completed, the treated samples at a certain concentration have been measured. The results shown in this research are the average of three independent measurements with a typical variation of ± 2 mV.

2.3.3. FTIR spectroscopy

Fourier transform infrared (FTIR) spectroscopy was performed to characterize the synthetic collector and study the adsorption mechanism of ionic liquid. The characterization of the synthetic collector was achieved with KBr pellets using the Fourier transform infrared spectrometer Nicolet (Thermo Fisher Scientific, USA). The samples used for FTIR were ground to -5 μ m and prepared with KBr pellets using the Fourier transform infrared spectrometer.

2.3.4. X-ray photoelectron spectroscopy (XPS)

The surface analyses of quartz and iron samples were carried out using the equipment ESCALAB 250Xi (Thermo Fisher Scientific, USA). The binding energies were determined from the spectra taken with Al K α ($h\nu=1486.6\text{eV}$) radiation and a hemispherical energy analyzer. A pressure of 6×10^{-10} mbar was maintained during the experiment. All the samples were ground to -5 μ m, and then the ionic liquid were added into a 100cm³ beaker with the samples, and stirred for 30min. Then the particles were separated by filtration, thoroughly washed with deionized water and dried at room temperature. Poly vinyl alcohol (PVA) was used for as a binder in making the pellets.

3. Results and Discussion

3.1. Characterization of collector

For the analysis of the synthetic collector, the infrared transmission spectrum is given in Fig.1. As can be seen in Fig. 1. The bands found in 3070cm⁻¹, 3052cm⁻¹, 3030cm⁻¹ and 3017cm⁻¹ are related to the stretch vibration absorption of =C-H from aromatic hydrocarbon. The asymmetric and symmetric stretch vibration of CH₃ and CH₂ can be observed in 2920cm⁻¹, 2870cm⁻¹, 2850cm⁻¹. The sharp bands in 1593cm⁻¹, 1486cm⁻¹, 1462cm⁻¹ are due to C=C stretch vibration from aromatic hydrocarbon. The 1378cm⁻¹ band is ascribed to symmetric deformation vibration of CH₃ and 1389cm⁻¹ band is due to CH₃ asymmetric deformation vibration. The bands between 1318cm⁻¹ to 1029cm⁻¹ are related to in-plane bending vibration of =CH from aromatic hydrocarbon. The bands between 860cm⁻¹ and 669cm⁻¹ are attributed to out-plane bending vibration of =CH from aromatic hydrocarbon. The band found in 719cm⁻¹ arose from the in-plane bending vibration of CH₂ in the long chain of the synthetic collector.

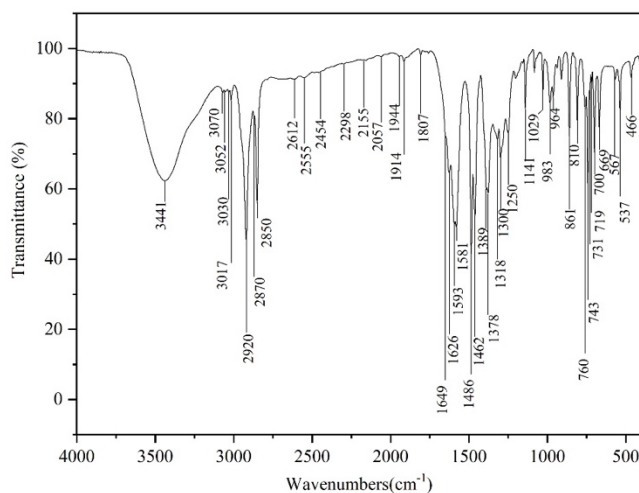


Fig. 1. Infrared transmission spectrum of ionic liquid (M-X)

3.2. Froth flotation test

The collecting capability of ionic M-X and DDA was studied without adjusting pH, the results were shown in Fig.2 and Fig.3, respectively. The effects of temperature on the flotation of quartz by using M-X and DDA were shown in Fig. 4. The optimum pulp pH was studied with M-X as collector in Fig. 5. The effects of starch on the flotation of quartz by using M-X as collector were in Fig. 6.

3.2.1. Effect of collector concentration on the flotation

The effect of collector concentration on the recovery of quartz and magnetite using M-X as collector was shown in Fig. 2. The experiments were conducted at 25°C without the starch at the neutral pH. When the concentration of M-X increased from 0.005mmol/L to 0.02mmol/L, the recovery of quartz increased dramatically from 25% to 96%. Continue adding M-X, the recovery of quartz almost not changed. As for the magnetite, the M-X performed almost equally. When the concentration of M-X was less than 0.015mmol/L, none of the magnetite was collected. With a dosage of M-X increased to 0.06mmol/L, the maximum recovery of magnetite reached only 10%. These results indicated that the M-X has a stronger collecting capability to quartz than to magnetite. The effect of collector concentration on the recovery of quartz and magnetite using DDA can be seen in Fig. 3. When the concentration of DDA was 0.02mmol/L, the recovery of quartz and magnetite was 77% and 40%, respectively. Continue adding the DDA the recovery of quartz and magnetite were both rising. When the concentration of DDA reached 0.06mmol/L, 97% quartz and 60% magnetite were recovered. This demonstrated that DDA has a weaker selectivity and collectability compared to M-X.

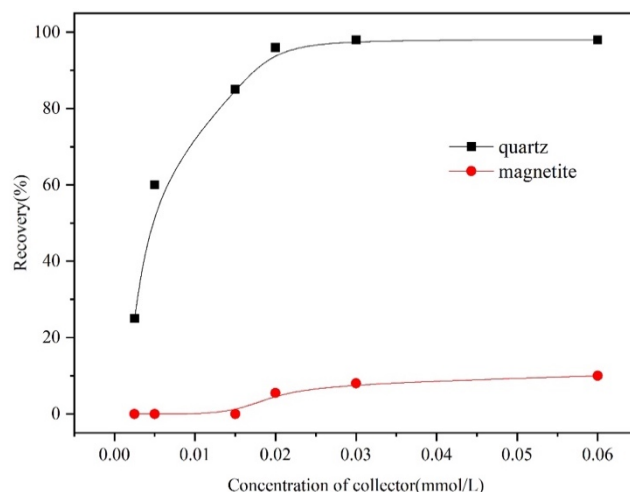


Fig. 2. Effect of ionic liquid (M-X) concentration on the flotation

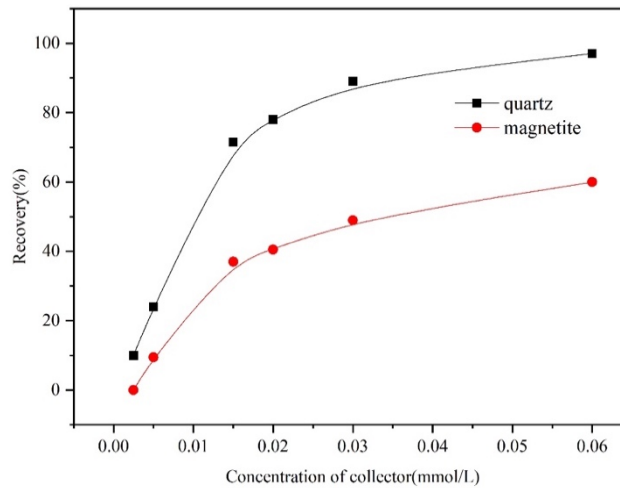


Fig. 3. Effect of DDA concentration on the flotation

3.2.2. Effect of temperature on the flotation

The effect of temperature on the recovery of quartz by using M-X and DDA as collector is shown at Fig. 4. All experiments were conducted at the concentration of 0.02mmol/L without starch, at neutral pH. As results presented in Fig. 4, among the range of 5°C-35°C, the recovery of quartz increased steadily as the temperature rising to 25°C for both collectors. Continue to increasing temperature, recovery of quartz declined. But the M-X apparently appeared to a better flotation performance in all ranges of temperature than DDA.

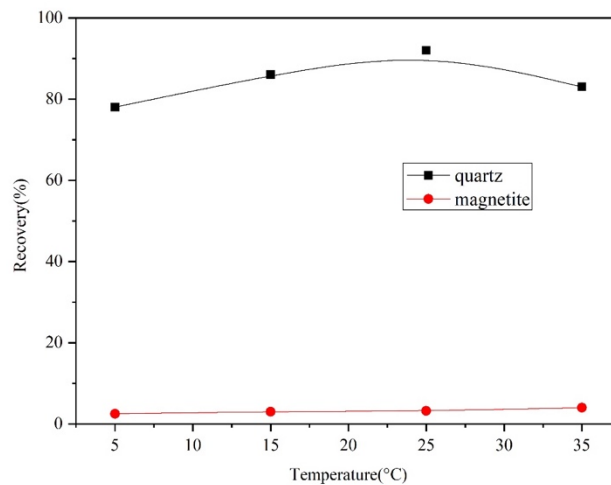


Fig. 4. Effect of temperature on quartz recovery

3.2.3. Effect of pulp pH on the flotation

The flotation behavior of quartz and magnetite with the M-X as a collector from pH 3 to 11 was shown in Fig. 5. The experiments were conducted at 25°C without starch, the concentration of M-X was maintained at 0.02mmol/L, and the pulp pH were adjusted to certain value by using NaOH and HCl. When the experiments were conducted in acidic areas (pH<7) the recovery of quartz increased from 75% to 94%, the magnetite recovery almost were not changed (about 5%). At the alkali pHs, while the recoveries of quartz decreased to 60%, the magnetite recoveries slightly increased to 9%. The results shown that the collectability of M-X would be influenced either in the acidic and alkaline pulp. As a result, the M-X has a better floatability at neutral pH.

3.2.4. Effect of starch on the flotation

The effect of starch on the recovery of quartz or magnetite by using M-X as collector is showed in Fig.

6. All experiments were conducted at 25°C, the concentration of M-X 0.02mmol/L, and at neutral pH. As we can see in Fig. 6, with the concentration of starch rising from 0.1mg/L to 0.6mg/L, the recovery of quartz declined from 90% to 80%. When the concentration of starch reached 0.8mg/L, the recovered quartz and magnetite are 70% and 2% respectively. These results demonstrated that the anionic starch had negative impact on flotation.

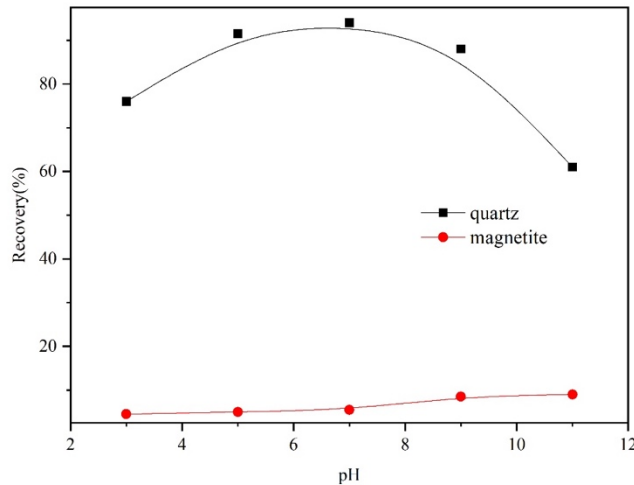


Fig. 5. Effect of pH on the flotation by using ionic liquid(M-X)

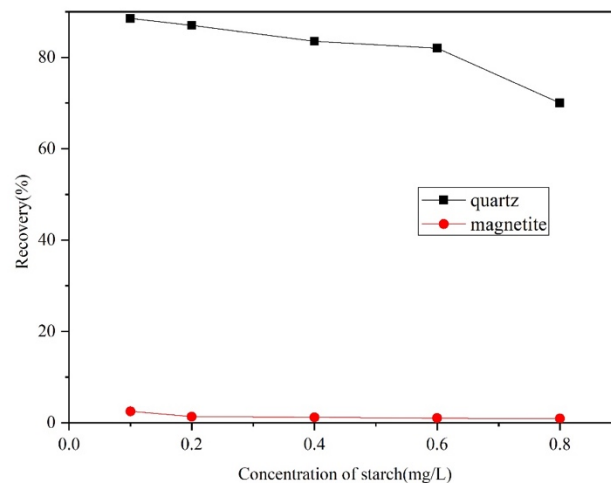


Fig. 6. Effect of starch on the flotation by using ionic liquid(M-X)

3.2.5. Reverse flotation results of the mixture of quartz and magnetite

The flotation results of the mixture of quartz and magnetite using M-X as collector were shown in Table 2. The experiments were performed at 25°C without starch at neutral pH, and the concentrations of M-X and DDA at 0.02mmol/L. As can be observed in Table 2, the total Fe content of the feed was 51.95%. Concentrate obtained using M-X has a recovery of Fe above 98.58% with 66.68% grade. In comparison, concentrate obtained using DDA achieved a recovery of Fe only 65.34% with a grade of 67.03%. And a 36.47% grade of Fe was obtained in the tailing using DDA, whereas there was only 3.20% grade of Fe in the tailing from M-X treated sample. The experiment was also performed with hexadecyl trimethyl ammonium bromide as collector under the same conditions, and there was no froth produced during the flotation.

3.3. Zeta potential measurements

The zeta-potential of quartz or magnetite in the presence of M-X(0.02mmol/L) was shown in Fig. 7. As

can be seen, the addition of M-X made the zeta-potentials more positive, which demonstrated that there is electrostatic force in this flotation (Weng et al., 2013).

At Fig. 7, at the pH 7, while the zeta-potential of quartz increased from -59.7mV to -33.9mV the zeta-potential of magnetite from -15.2mV to -7.3mV. It was clear that the surface charge shifted on the quartz surface were more significant than that on the magnetite surface. These results indicated that the adsorption of M-X on the quartz surface was more efficient than that on the magnetite.

Table 2. Results of reverse flotation of the mixture of quartz and magnetite with M-X

Collector	Products	Productivity (wt.%)	Grade(TFe)/%	Recovery(TFe)/%
M-X	Concentrate(magnetite)	76.80	66.68	98.58
	Tailings(quartz)	23.20	3.20	1.42
	Feed	100.00	51.95	100.00

Table 3. Results of reverse flotation of the mixture of quartz and magnetite with DDA.

Collector	Products	Productivity (wt.%)	Grade(TFe)/%	Recovery(TFe)/%
DDA	Concentrate(magnetite)	50.66	67.03	65.34
	Tailings(quartz)	49.34	36.47	34.66
	Feed	100.00	51.95	100.00

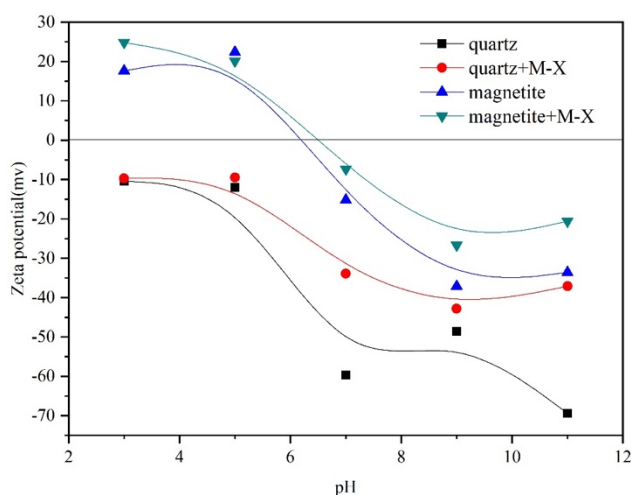


Fig. 7. Zeta potential of minerals vs. pH in deionized water and M-X solution

3.4. Adsorption mechanism

The FTIR was performed to study the possible inter-action of the M-X with quartz and magnetite. The FTIR spectra of quartz and magnetite interacted with M-X were analyzed to confirm the adsorption mechanism and they were shown in Fig. 8 and 9. The bands were found at 3435cm^{-1} and 3454cm^{-1} for quartz and magnetite related to adsorbed water. The peak appeared at 3037cm^{-1} is represented the stretch vibration absorption of =C-H from aromatic hydrocarbon. The peaks were found at 2952cm^{-1} , 2921cm^{-1} , and 2852cm^{-1} suggesting the asymmetric stretching vibration of CH_3 and CH_2 group. The peaks at 1625cm^{-1} , 1592cm^{-1} , 1495cm^{-1} , 1484cm^{-1} , and 1456cm^{-1} were due to the stretching vibration of C=C from aromatic hydrocarbon. The band found at 1379cm^{-1} indicated that the special symmetric deformation vibration of CH_3 in the long alkyl chain. And the peak at 1389cm^{-1} is represented the asymmetric deformation vibration of CH_3 . The peaks at 1318cm^{-1} and 1299cm^{-1} were assigned to in-plane bending vibration of =C-H from aromatic hydrocarbon. FTIR results were showed that the structure of M-X and confirmation the adsorption of M-X on the surface of quartz.

There were two characteristic peaks of magnetite at 572 cm^{-1} , 469 cm^{-1} which were almost unaltered at the Fig. 9. The peaks found at 2954 cm^{-1} , 2924 cm^{-1} , and 2854 cm^{-1} were corresponded to the stretching vibration of CH_3 and CH_2 group. Other peaks at 1484 cm^{-1} and 1102 cm^{-1} were nearly not changed. These adsorption results formed that the adsorption of M-X on magnetite surface was much weaker than that on quartz.

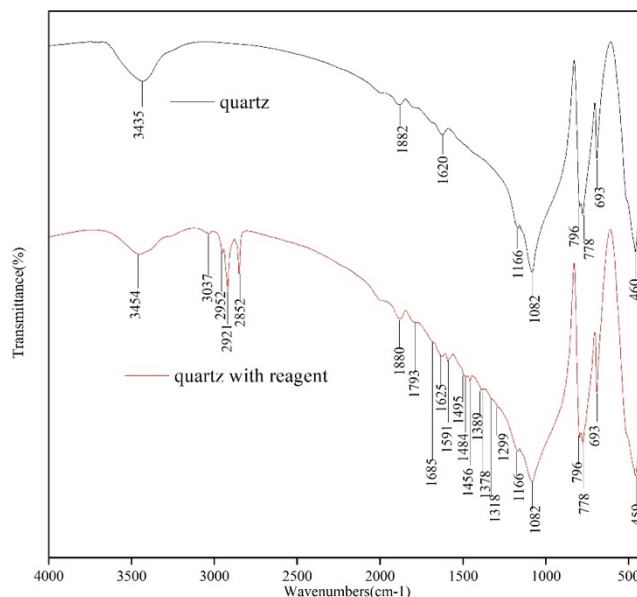


Fig. 8. FTIR spectra of quartz with synthetic ionic liquid(M-X) treated samples

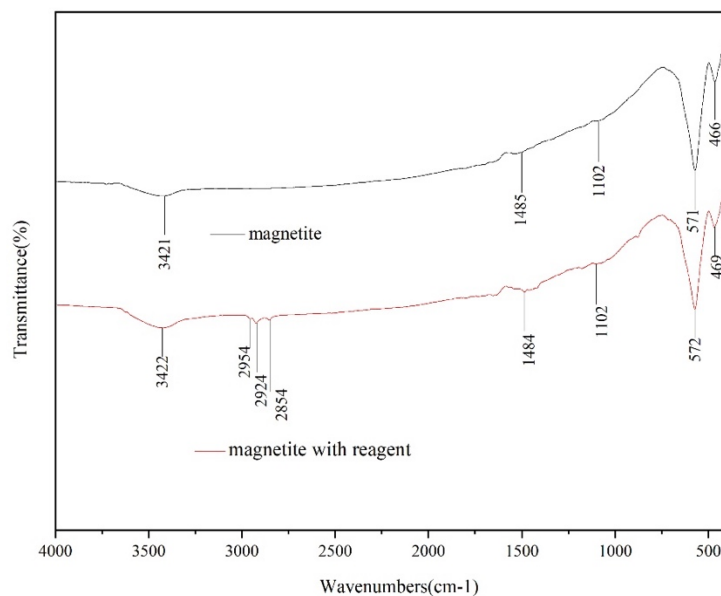


Fig. 9. FTIR spectra of magnetite with synthetic ionic liquid(M-X) treated samples

3.5. XPS studies

The XPS spectra of quartz and quartz treated with M-X were shown in Fig. 10. A peak at 284.03 eV was observed as the standard $\text{C}(1s)$. At the quartz spectra, two peaks were found in 102.77 eV and 153.45 eV related to $\text{Si}(2p)$ and $\text{Si}(2s)$, respectively. After the treatment of quartz with M-X, the binding energy of $\text{Si}(2p)$ and $\text{Si}(2s)$ were nearly not changed. These results showed there was no chemical adsorption of M-X on quartz surface. In spectra of M-X treated quartz a small new peak can be observed at 400.66 eV which related to $\text{N}(1s)$. The new peak of $\text{N}(1s)$ revealed that the presence of nitrogen and the adsorption of M-X onto the quartz surface.

C(1s) peak of the spectra treated with M-X was subjected to curve fitting and the results can be seen in Fig. 11. The peaks presented at 284.68eV and 286.46eV were related to the C-H and C-O linkage respectively. The proportion of the peak area corresponded the proportion of the C-H and C-O in the structure of M-X. And this indicated the adsorption of M-X on the surface of quartz.

The XPS spectra of magnetite and magnetite treated with sodium salicylate were analysed to see whether the salicylate anion have any influence on flotation. the results were shown in Fig. 12. The peak at 284.8eV was the standard C(1s). The peak at 711.15eV was related to the Fe(2p) at the magnetite spectra. After treatment with sodium salicylate the binding energy of Fe(2p) was decreased by 0.6eV to 710.55eV. This indicated that the chemisorption of sodium salicylate on the magnetite surface. According to these results, the conclusion can be inferred that the salicylate anion worked as depressant.

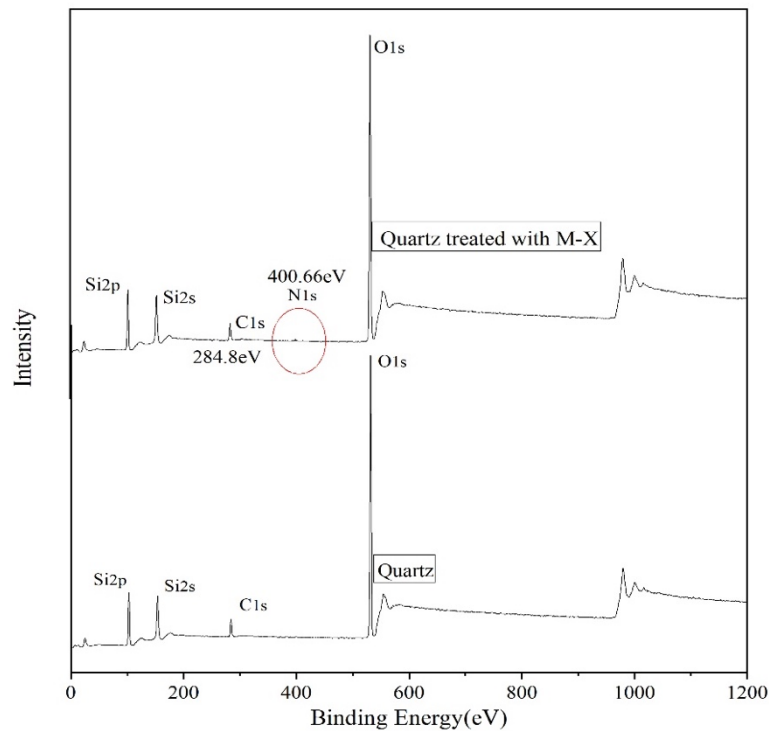


Fig. 10. XPS spectra of quartz and quartz treated with M-X

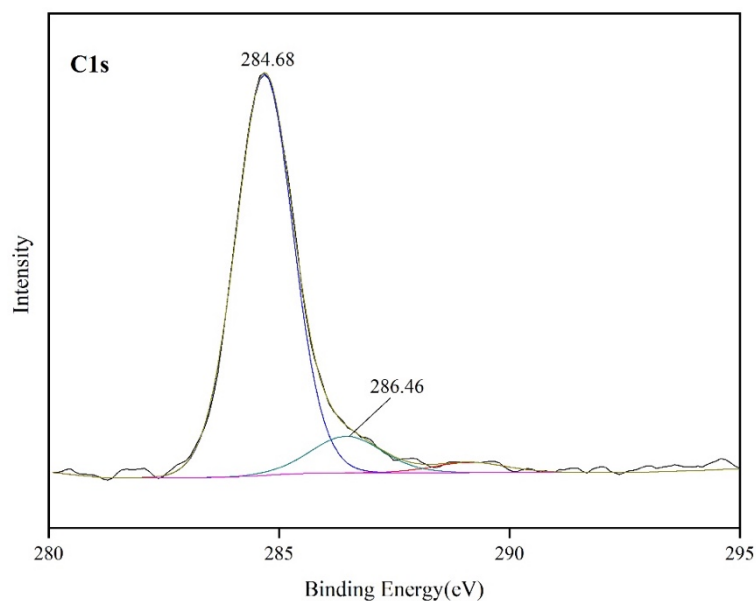


Fig. 11. Curve fitting of C(1s) from the spectra treated with M-X

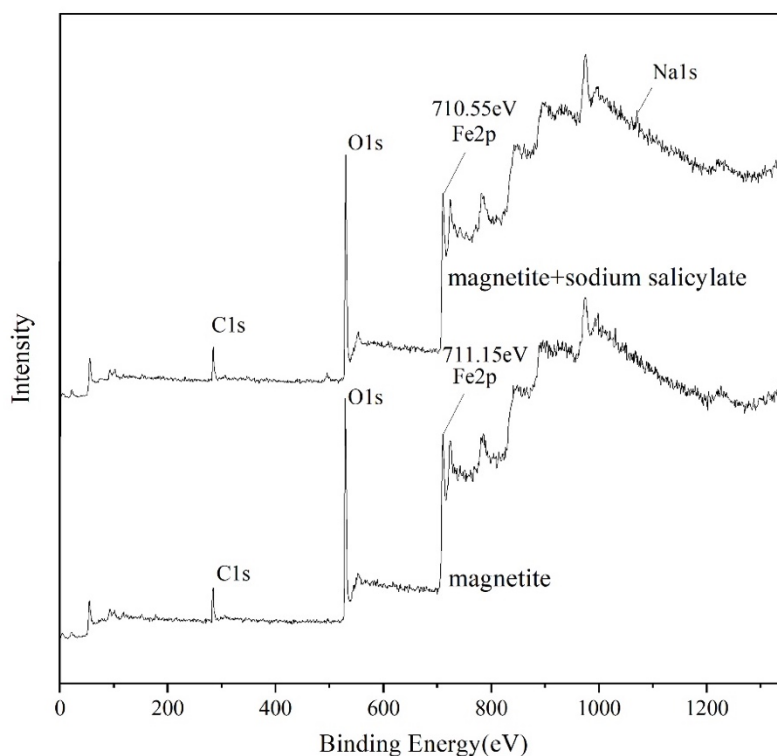


Fig. 12. XPS spectra of magnetite and magnetite treated with sodium salicylate

4. Conclusions

An aryl-substituted aromatic acid ionic liquid M-X was synthesized and applied as flotation collector in this paper. Flotation studies indicated that the anionic starch is not necessary when using the M-X as collector. M-X appeared a stronger collecting capability and selectivity than DDA at the pure quartz flotation. The M-X also had a better performance in a wide range of temperature than DDA. The maximum recovery of quartz was obtained at 0.02mmol/L M-X as collector, 25°C, and at neutral pH. In the flotation of mixture of quartz and magnetite, 98.58% of Fe was recovered and the grade was increased from 51.95% to 66.68. The spectra of FTIR and XPS confirmed the adsorption between M-X and quartz. The results of zeta-potential demonstrated the adsorption between M-X and quartz was more efficient than that of magnetite. Which corresponded to the flotation performance that M-X had better collecting ability to quartz than to magnetite. According to the XPS spectra of magnetite. Sodium salicylate was absorbed on the magnetite surface chemically and so had a depression effect on the magnetite. The zeta potential, XPS and FTIR results confirmed that the interaction between M-X and quartz occurred, and the salicylate in the M-X have a depression effect on magnetite, which explains why the starch is not necessary in the flotation.

Acknowledgements

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