

Effect and mechanism of siderite on reverse anionic flotation of quartz from hematite

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Abstract: Reverse flotation technology is one of the most efficient ways to improve the quality and reduce impurity of iron concentrate. Mineral processors dealing with hematite face a challenge that the flotation results of reverse flotation of hematite are poor in presence of siderite using fatty acid as collector, starch as depressant of iron minerals and calcium ion as activator of quartz at strong alkaline pH. In this work, the effect of siderite on reverse anionic flotation of quartz from hematite was investigated. The effect mechanism of siderite on reverse flotation of hematite was studied by solution chemistry, ultraviolet spectrophotometry (UV) and Fourier transform infrared spectroscopy (FTIR). It was observed that siderite had strong depressive effect on quartz in flotation using sodium oleate as collector, corn starch as depressant of iron minerals and calcium chloride as activator of quartz at strong alkaline pH. The starch was adsorbed onto calcium carbonate by chemical reaction which was formed by CO_3^{2-} from siderite dissolution and Ca^{2+} from calcium chloride as activator of quartz and precipitated on the surface of quartz, which resulted in improving the hydrophilic ability of quartz.

Key words: siderite; quartz; hematite; mineral dissolution; starch; calcium carbonate; sodium oleate; froth flotation

1 Introduction

Existing studies on the flotation of iron ores have shown that iron ores can be floated with both anionic and cationic collectors [1–7]. However, the flotabilities of iron ore minerals such as hematite and siderite are always lower than that of quartz under similar conditions [8–12].

Reverse cationic/anionic flotation technology is one of the most efficient ways to improve the quality and reduce impurity of iron concentrate [13–14]. During the flotation process, quartz is attached to air bubble and floats upward into a froth layer leaving hematite as the non-floated fraction. Iron minerals are generally depressed with starch [15–19]. In most iron ore beneficiation plants, amine is generally used as collector of quartz and starch is generally used as depressant of iron minerals [7, 20]. In China, fatty acid is often employed as collector, calcium ion is generally employed

as activator of quartz and starch is often employed as depressant of iron minerals according to iron ore characteristic and technological conditions [21–23].

In recent years, mineral processors dealing with hematite have faced a challenge that the flotation results of reverse flotation of hematite are poor in presence of siderite using fatty acid as collector, starch as depressant of iron minerals and calcium ion as activator of quartz at strong alkaline pH [24–25]. The Fe grade in non-floated product decreases with increasing the siderite content in iron ores. However, researchers have not been able to find the exact reasons for the phenomenon. In this work, the pure mineral flotation tests were conducted using sodium oleate as collector, corn starch as depressant of iron minerals and calcium chloride as activator of quartz to discuss the effect of siderite on reverse flotation of hematite. This work aimed to investigate the effect mechanism of siderite on reverse flotation of hematite by solution chemistry, ultraviolet spectrophotometry (UV) and Fourier transform infrared spectroscopy (FTIR).

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2 Materials and methods

2.1 Materials and reagents

The siderite sample and hematite sample were obtained from Anshan City, Liaoning Province, China. The quartz sample was purchased from Yingkou City, Liaoning Province, China. The three samples were carefully ground in a ball mill, processed by gravity concentration and sieved to (−106+45) μm size fractions. X-ray diffraction analysis and chemical analysis of three samples showed that the siderite, hematite and quartz samples were of high purity. The siderite sample contained about 44.65% Fe (mass fraction), the hematite sample contained about 67.45% Fe (mass fraction), and the quartz sample had a purity of 99.77% SiO₂ (mass fraction).

Sodium oleate with 98% purity was used as the anionic collector. Calcium chloride with 99% purity was used as the activator of quartz. Corn starch was employed as the depressant of siderite and hematite, which was dissolved in distilled water by adding 20% NaOH by weight at 50 °C on a hot plate. Analytical grade NaOH was used for pH adjustment to keep the pH at about 11.4. Distilled water was used in all experiments.

2.2 Method

Flotation experiments were carried out in a 30 mL flotation cell with impeller speed of 1500 r/min. A 2.0 g (2.7 g) sample of particle size (−106+45) μm composed of quartz, hematite and siderite in different proportions by mass according to test design was mixed with 25 mL distilled water in flotation cell for 1 min. Then, NaOH was added to keep the pH at about 11.4 and the pulp was conditioned for 2 min. And then, calcium chloride, starch, sodium oleate were added separately and the pulp was conditioned for 2 min with each reagent, respectively. The flotation time was fixed for 3 min at room temperature (25 °C). After flotation, the floated product and non-floated product were collected, filtered, dried, weighed and analyzed, respectively.

The sodium oleate adsorption capacity adsorbed onto minerals was measured by ultraviolet spectrophotometer with the type of Spectro Flex 6600. The steps that have been taken are similar to those of Ref. [26].

Perkin Elmer Spectrum One FT-IR Spectrometer had been used to understand the reagent-mineral interaction with the type of Nicolet380 FT-IR.

3 Results and discussion

3.1 Effect of siderite on flotation separation of hematite and quartz

Figure 1 shows the quartz recovery and Fe grade in

non-floated product as a function of percentage of siderite in mixed minerals using sodium oleate as collector, corn starch as depressant of iron minerals and calcium chloride as activator of quartz at pH 11.4. As can be seen, the Fe grade in non-floated product strongly decreases, from 54.15% to below 40%. Meanwhile, the quartz recovery in non-floated product significantly increases, from 19.50% to 98.00%. That means that siderite has strong negative impact on reverse flotation of hematite.

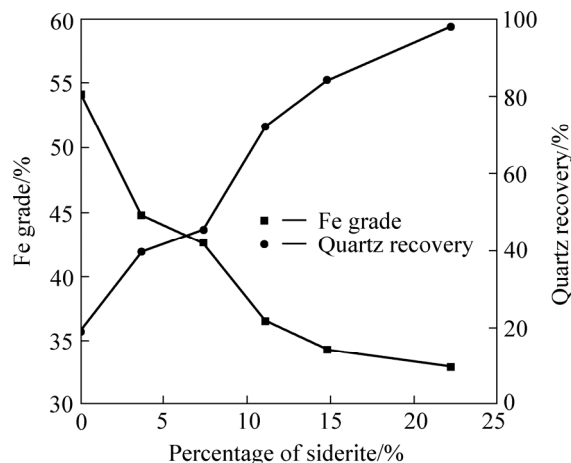


Fig. 1 Quartz recovery and Fe grade in non-floated product as a function of percentage of siderite (sodium oleate dosage: 160 mg/L; corn starch dosage: 60 mg/L; calcium chloride dosage: 100 mg/L; pH:11.40; a 2.7 g sample: composed of siderite, hematite and quartz, proportion by mass of hematite and quartz: 4:5)

3.2 Effect of siderite and hematite on quartz flotation

The results shown in Fig. 2 indicate that both the SiO₂ grade and quartz recovery in floated products slightly decreases with increasing the percentage of

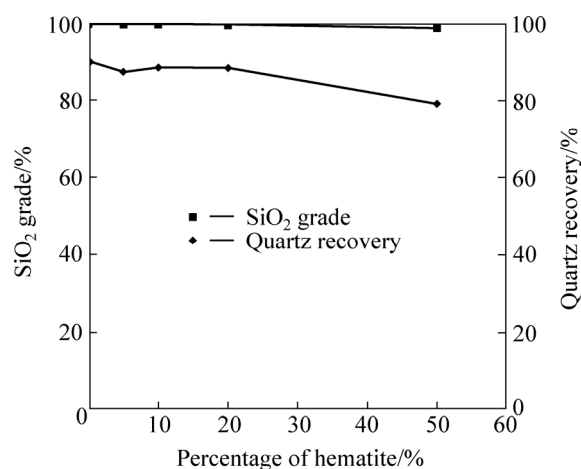


Fig. 2 SiO₂ grade and quartz recovery in floated product as a function of percentage of hematite (sodium oleate dosage: 160 mg/L; corn starch dosage: 60 mg/L; calcium chloride dosage: 100 mg/L; pH: 11.40; a 2.0 g sample: composed of hematite and quartz in different proportions by mass)

hematite. That means hematite may have little impact on quartz flotation using sodium oleate, corn starch and calcium chloride at strong alkaline pH.

Figure 3 shows SiO₂ grade and quartz recovery in floated product as a function of percentage of siderite by mass. It is very clearly seen that as far as the percentage of siderite increases, the quartz recovery in floated product decreases sharply. When the percentage of siderite is only 5%, the quartz recovery is reduced from 90.05% to 44.88%. The results are consistent with observations reported by ZHANG et al [24] and YANG [25].

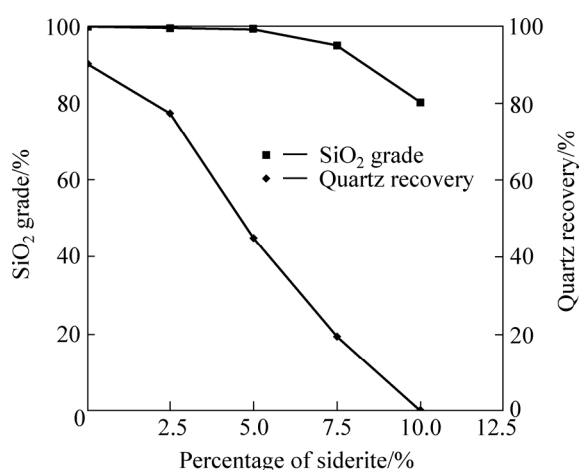


Fig. 3 SiO₂ grade and quartz recovery in floated product as a function of percentage of siderite (sodium oleate dosage: 160 mg/L; corn starch dosage: 60 mg/L; calcium chloride dosage: 100 mg/L; pH: 11.38; a 2.0 g sample: composed of siderite and quartz in different proportions by mass)

The results shown in Figs. 2 and 3 stress the role that the cause why siderite can deteriorate the reverse flotation of hematite is due to the passive impact of siderite on quartz flotation.

3.3 Effect of sodium oleate dosage on quartz flotation from quartz-siderite mixed minerals

From Table 1, it can be seen that the quartz recovery in floated product slightly increases and the sodium oleate adsorption capacity on minerals changes a little when sodium oleate dosage increases from 160 mg/L to 240 mg/L. That means that the main cause why siderite has strong negative impact on quartz flotation has little relationship to sodium oleate consumption by siderite.

3.4 Effect of reagent type on quartz flotation from quartz-siderite mixed minerals

The quartz recovery in floated product in presence and absence of corn starch is listed in Table 2. It can be seen clearly that in anionic flotation of quartz using sodium oleate, corn starch and calcium chloride at strong alkaline pH, when the percentage of siderite increases from 0% to 5%, the quartz recovery in floated product reduces from 93.80% to 62.20%. However, in absence of corn starch, the quartz recovery increases from 62.20% to 88.32%. That illustrates that siderite has little impact on quartz flotation in absence of corn starch, and has significant impact on quartz flotation in presence of corn starch. Therefore, it can be concluded that the cause why siderite has strong passive impact on quartz flotation is mainly due to corn starch.

It can be seen from Table 3 that the sodium oleate adsorption capacity on mixed minerals (siderite 0.2 g, quartz 1.8 g) in presence of sodium oleate, calcium chloride and starch significantly decreases from 1.63 mg/g to 0.26 mg/g in contrast with in absence of starch. That illustrates that when starch and siderite both exist, the adsorption of sodium oleate onto quartz is baffled. However, it can also be seen that when the percentage of siderite is 0%, the sodium oleate adsorption capacity on quartz in presence of sodium oleate, calcium chloride and starch only changes a little in contrast with in absence of starch. These results are consistent with the research reported by JIANG et al [26] and MA [27], who reported that starch had little impact on quartz activated by Ca²⁺ in anionic flotation. From the results above, it can be calculated that the performance that starch has depressing effect on quartz activated by Ca²⁺ in flotation is correlated with siderite. Only when siderite is present, starch can have depressing effect on quartz in flotation. Therefore, the passive impact of siderite on quartz in flotation is caused by both starch and siderite.

3.5 Effect of siderite dissolution on quartz flotation

The results in Table 4 show that siderite dissolution has an even greater impact on quartz flotation. The quartz recovery in floated product is greatly improved by stripping the siderite dissolution in flotation.

It is shown by the data [29–30] siderite is easily dissolved in water. The higher the pH is, the easier the siderite dissolves. In reverse anionic flotation of iron

Table 1 Quartz recovery and sodium oleate adsorption capacity on mineral as a function of sodium oleate dosage

pH	Mineral	Percentage of siderite/%	Reagent dosage/(mg·L ⁻¹)			Quartz recovery/%	Sodium oleate adsorption capacity/(mg·g ⁻¹)
			Corn starch	Calcium chloride	Sodium oleate		
11.4	Quartz	10	60	100	160	3.35	0.26
	and		60	100	200	3.89	0.26
	siderite		60	100	240	5.63	0.30

Table 2 SiO₂ grade and quartz recovery in floated product as a function of reagent types in presence and absence of siderite

pH	Mineral	Percentage of siderite/%	Reagent/(mg·L ⁻¹)			SiO ₂ grade/%	Quartz recovery/%
			Sodium oleate	Calcium chloride	Corn starch		
11.4	Quartz and siderite	0	160	100	60	99.95	93.80
		5	160	100	60	99.90	62.20
		5	160	100	0	99.92	88.32

Table 3 Sodium oleate adsorption capacity on quartz as a function of corn starch in presence and absence of siderite

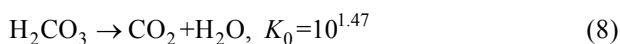
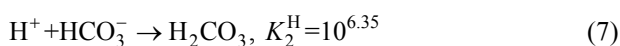
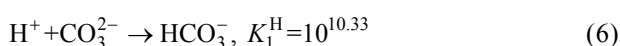
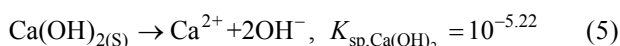
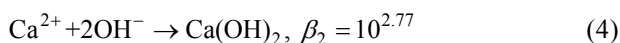
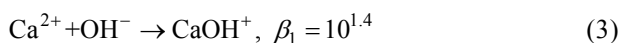
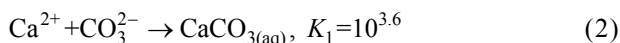
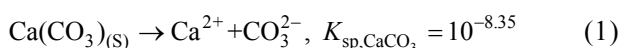
pH	Mineral	Reagent/(mg·L ⁻¹)			Sodium oleate adsorption capacity/(mg·g ⁻¹)
		Sodium oleate	Calcium chloride	Corn starch	
11.4	Quartz 1.8g and siderite 0.2 g	160	100	0	1.63
				60	0.26
11.4	Quartz 2.0 g	160	100	0	2.00
				60	1.89

Table 4 Influence of siderite dissolution on quartz flotation

pH	Mineral	Reagent (mg/L)			Method of flotation	Quartz recovery/%
		Sodium oleate	Calcium chloride	Corn starch		
11.40	Quartz and siderite (7.5%)	160	100	60	Direct flotation method *	22.63
					Flotation method by stripping siderite dissolution **	77.37

Notes: *The direct flotation method is as follows: a 2.0 g sample (quartz 1.85 g and siderite 0.15 g) was mixed in flotation cell for 1min. Then, NaOH was added to keep the pH at 11.4, and the pulp was conditioned for 2 min. And then, all kinds of reagents were added separately and conditioned for 2 min respectively. Finally, flotation was performed for 3 min. **The flotation method by stripping the siderite dissolution is as follows: Firstly, a 0.15 g siderite sample is dissolved in distilled water for 30 min; And then, the siderite sample was stripped from siderite dissolution, cleaned three times with distilled water and mixed with 1.85 g quartz as the test sample. Thirdly, the test sample was mixed in flotation cell for 1 min. And the rest of steps were the same as the method of direct flotation.

ores, calcium ion is often added to activate quartz, which is in favor of siderite dissolution. The dissolved components logarithm diagram of siderite shows that there is a lot of CO₃²⁻ ion in siderite dissolution at strong alkaline pH. Therefore, when there is a lot of CO₃²⁻, calcium ion composition is complicated. Calcium ion in sodium carbonate system may be the following reaction. The principle reaction of calcium ion in sodium carbonate system is as follows [31–32]:



Different composition concentrations of calcium ion in balance system of sodium carbonate as a function of

pH are shown in Fig.4 according to the Eqs. (1) to (8). Fig.4 shows that calcium ion exists in the form of Ca²⁺ when pH is below 9.98, and in the form of CaCO₃ precipitation when pH is above 9.98. Because there is much CO₃²⁻ from siderite dissolution at pH 11.4, when calcium ion is added as activator of quartz, it will exist on the surface of quartz in the form of CaCO₃ precipitation, which makes the surface of quartz have the same properties as calcium carbonate. In other words, the flotation characteristics of quartz are similar to calcite.

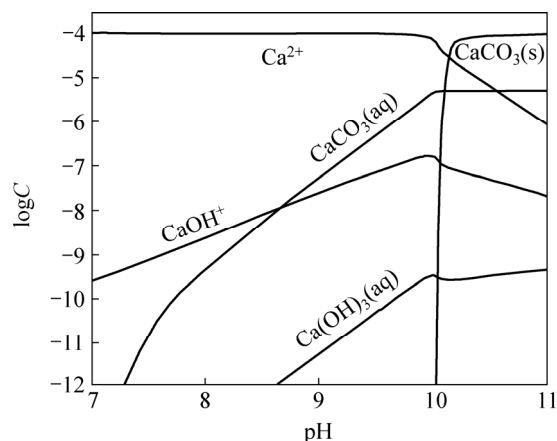


Fig. 4 Species distribution of Ca²⁺ in balance system of sodium carbonate (Concentration of Ca²⁺: 1.0×10⁻⁴ mol/L)

3.6 Effect of carbonate ion and starch on quartz flotation

In order to verify both CO_3^{2-} dissolved from siderite and starch lead to reduction of quartz recovery in quartz flotation, quartz recovery as a function of sodium carbonate dosage and starch dosage are shown in Figs. 5 and 6, respectively.

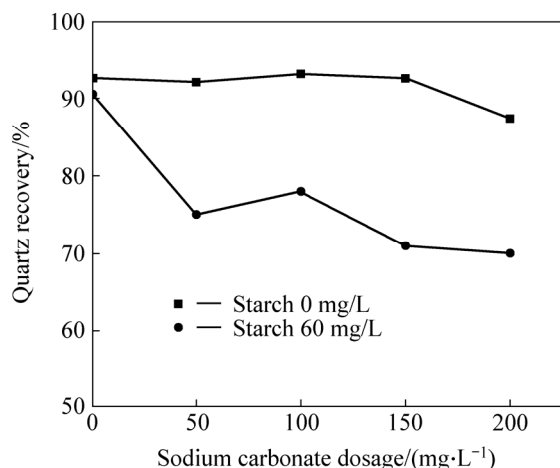


Fig. 5 Quartz recovery as a function of sodium carbonate dosage in presence and absence of starch (sodium oleate dosage: 160 mg/L; calcium chloride dosage: 100 mg/L; pH: 11.38; a 2.0 g sample composed of quartz)

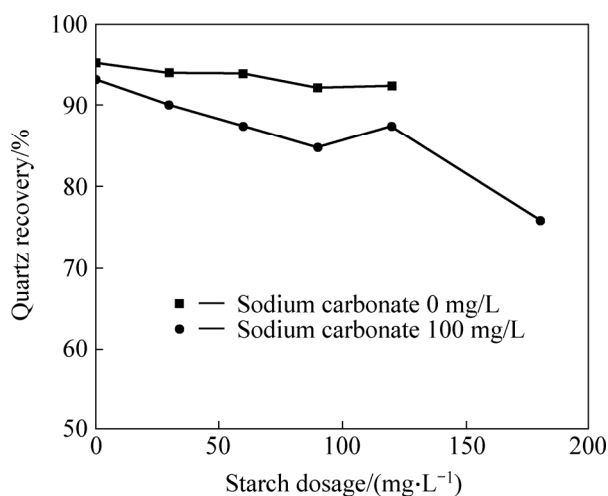


Fig. 6 Quartz recovery as a function of starch dosage in presence and absence of sodium carbonate (sodium oleate dosage: 160 mg/L; calcium chloride dosage: 100 mg/L; pH: 11.38; a 2.0 g sample composed of quartz)

The results from Figs. 5 and 6 indicate that starch has weak impact on quartz recovery in flotation in absence of CO_3^{2-} , and has strong negative impact on quartz recovery in presence of CO_3^{2-} . Therefore, it can be seen that both CO_3^{2-} dissolved from siderite and starch have strong passive impact on quartz recovery in flotation. And it can be speculated the reason of that is the starch has strong depressive effect on quartz covered by calcium carbonate in flotation.

3.7 FTIR studies of minerals before and after reaction with different reagents

3.7.1 FTIR spectra of calcium carbonate before and after reaction with starch

The FTIR spectra of calcium carbonate before and after reaction with starch are shown in Figs. 7(a) and (b). It can be seen clearly from Fig. 7(b) that a new peak at 1620.91 cm^{-1} appears due to interaction with starch. And the peak at 3445.56 cm^{-1} may be the result of the shift of the peak at 3431.44 cm^{-1} attributed to stretching vibrations of hydroxyl group owing to some chemical bonding with starch. The peak at 1408.09 cm^{-1} may be the result of the shift of the peak at 1421.53 cm^{-1} due to asymmetric stretching vibrations of C—O groups. On the basis of FTIR spectra, it is clear that chemical interaction occurs between calcium carbonate and starch. It is also clear from FTIR spectra that starch is specifically adsorbed onto calcium carbonate. That is accordance with the foregoing analysis. These results are consistent with the research reported by LI et al [33] and PINTO et al [34], who reported that starch had strong depressing effect on calcite (CaCO_3) in flotation owing to the chemical interaction between starch and calcite.

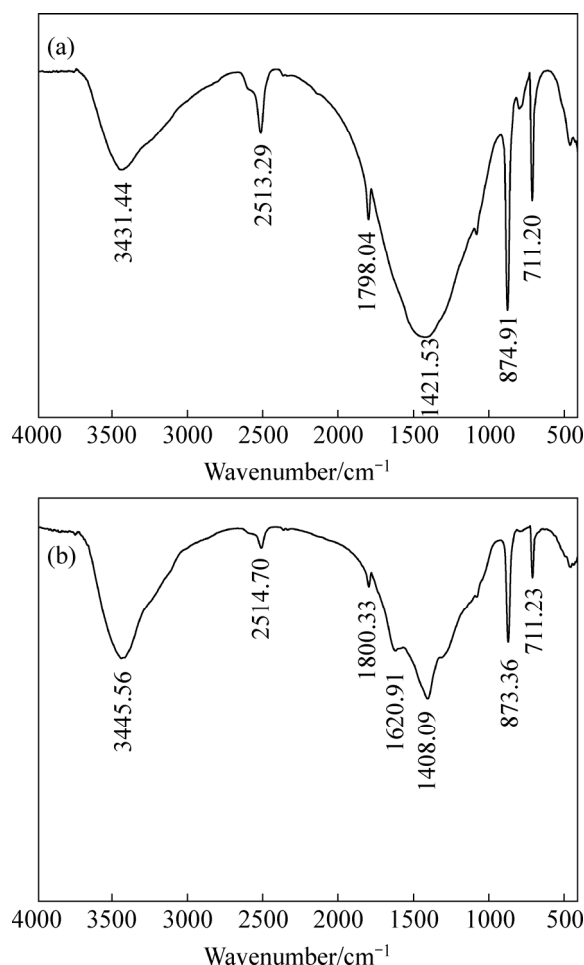


Fig. 7 Infrared spectra of calcium carbonate before (a) and after (b) reaction with starch

3.7.2 FTIR spectra of quartz before and after treatment with different reagents

In order to reaffirm that the starch is adsorbed onto quartz attributing to calcium carbonate on the surface of quartz, the FTIR spectra of quartz before and after treatment with calcium chloride and starch, and with sodium carbonate, calcium chloride and starch are shown in Fig. 8.

Figures 8(a) and (b) show that there is no new peak on quartz after treatment with calcium chloride and starch. That means the adsorption of starch onto quartz is

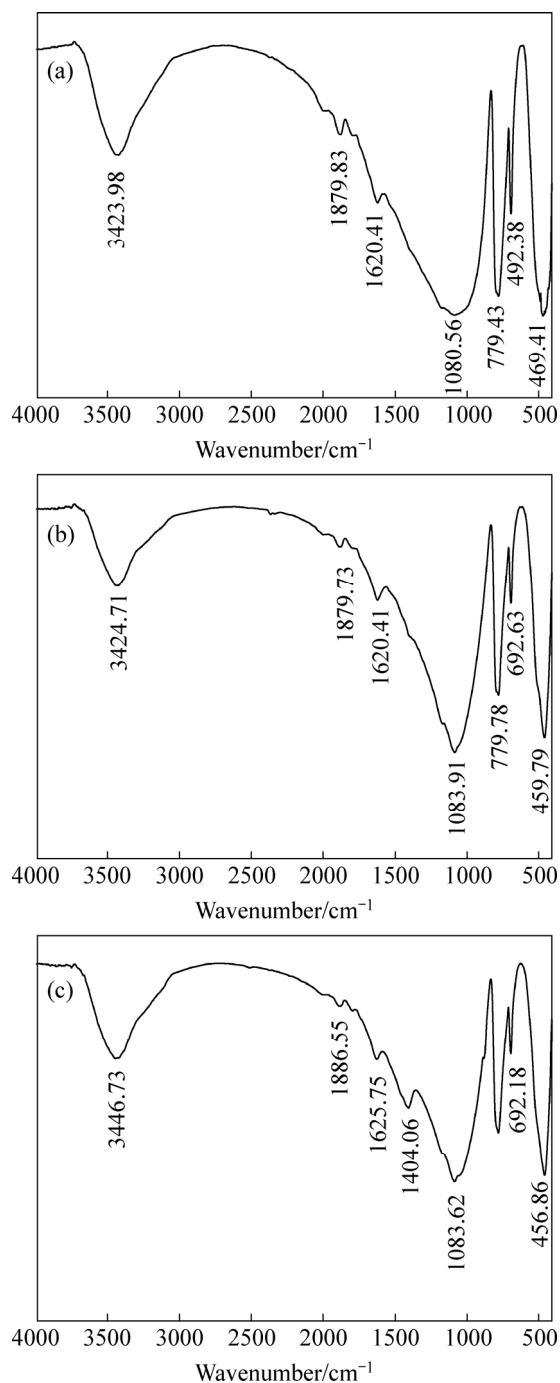


Fig. 8 Infrared spectra of quartz before treatment (a), after treatment with calcium chloride and starch (b) and with sodium carbonate, calcium chloride and starch (c)

very weak. A possible explanation is starch in absence of CO_3^{2-} is adsorbed onto quartz through CaOH^+ only by the electrostatic attraction and hydrogen-bond interaction, whose acting force is very weak [27].

As can be seen from Figs. 8(a) and (c), a new peak at 1404.06 cm^{-1} appears on the spectrum of quartz after treatment with sodium carbonate, calcium chloride and starch. The peak at 1404.06 cm^{-1} is due to bending vibrations of CH_2 group of starch. According to the results from Fig. 7(b) and Fig. 8(c), the new peak is just attributed to the chemical adsorption of starch onto calcium carbonate. Meanwhile, the peak at 3423.98 cm^{-1} shifts to the peak at 3446.73 cm^{-1} , which is due to stretching vibrations of hydroxyl group of starch. The results above indicate that chemical reaction between starch and quartz in presence of sodium carbonate and calcium chloride occurs.

In conclusion, it can be seen from the results above that the reason siderite has strong negative effect on quartz flotation at strong alkaline pH is that starch is adsorbed onto calcium carbonate by chemical reaction which is formed by CO_3^{2-} from siderite dissolution and Ca^{2+} from calcium chloride as activator of quartz and precipitates on the surface of quartz, which results in improving the hydrophilic ability of quartz.

4 Conclusions

1) Siderite has strong negative impact on flotation separation of hematite and quartz using sodium oleate as collector, corn starch as depressant of iron minerals and calcium chloride as activator of quartz at pH 11.4. The quartz recovery in non-floated product significantly increases along with the percentage of siderite. The reason of that is siderite has strong passive effect on quartz flotation.

2) That siderite has strong negative impact on quartz flotation using sodium oleate as collector, corn starch as depressant of iron minerals and calcium chloride as activator of quartz at strong alkaline pH is irrelevant to sodium oleate consumption by siderite and is concerned with starch and CO_3^{2-} from siderite dissolution. The starch is adsorbed onto calcium carbonate by chemical reaction which is formed by CO_3^{2-} from siderite dissolution and Ca^{2+} from calcium chloride as activator of quartz and precipitates on the surface of quartz, which results in improving the hydrophilic ability of quartz.

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