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# The flotation separation of barite-calcite using sodium silicate as depressant in the presence of sodium dodecyl sulfate

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**Abstract:** The flotation separation of barite from calcite using sodium dodecyl sulfate (SDS) as the collector and sodium silicate (SS) as the depressant has been concretely studied in this paper. The results show that SDS has high collecting ability for both barite and calcite and that the flotation separation of barite from calcite cannot be achieved by using SDS alone. The depressant sodium silicate has a selective depression effect on calcite by the control of the pulp pH at 9.0 in the presence of SDS. Through locked cycle tests, a concentrate containing 95.54% $BaSO_4$ can be obtained from an actual ore containing 24.5% $BaSO_4$, with a recovery of 86.11%. The zeta potential measurements, adsorption analysis and infrared spectrum studies reveal that the pre-adsorption of sodium silicate strongly favors the adsorption of SDS on the calcite surface and adversely affects SDS adsorption on the barite surface. This property is the reason for the selective depression of SS on calcite.

*Keywords:* sodium dodecyl sulfate; sodium silicate; barite; calcite; flotation

## 1. Introduction

Barite ($BaSO_4$) is a kind of important non-metallic mineral containing barium, which is widely used in industrial fields such as the petroleum and chemical industries, and as paint and filler (Gurpinar et al., 2004; Scott et al., 2010; Kecir and Kecir, 2016; Ren et al., 2017). Barite is mainly produced from primary barite deposits, where barite has been considered the primary mineral. However, there is a growing trend in processing the ores that contain barite in the form of gangue, which can be applied as the secondary mineral (Bulatovic, 2014).

In recent years, since the high-grade resources are being reduced, the processing of low-grade barite has become necessary not only for meeting the current demand but also for environmental considerations (Blackburn, 1988; Naseem et al., 2011). The resources of low-grade barite mainly include the following two types: a) barite exists in the tailings of sulfide ore, usually combined with fluorite, and many studies have researched this, with some results; and b) barite exists in the tailings of high-purity barite through gravity separation, usually associated with calcite and quartz, and few studies have been done on it. Consequently, experiments should be carried out on the selective separation of barite from calcite/quartz.

Due to the differences of composition, structure and properties between salt minerals and quartz, it is relatively easy to separate them by flotation. However, the flotation separation of barite from calcite is difficult to achieve because of the similar physical and chemical properties on the mineral surface, which has led to the reduced selectivity of conventional collectors such as fatty acids and fatty acid derivatives (Amakonah et al., 1985; Fuerstenau et al., 1992; Ozcan and Bulutcu, 1993; Hu et al., 2011; Hu et al., 2012; Shepeta et al., 2012; Zhang et al., 2012). Generally, the adsorption mechanism of these collectors is chemisorption onto the mineral surface (Atademir et al., 1981; Marinakis and Shergold, 1985). Hence, it is almost impossible to separate barite from calcite by fatty acids added alone (Feng et al., 2015). Unfortunately, depressants used for barite separation, such as quebracho, organic colloids,

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hydrosols and sodium phosphates, have also inhibited barite flotation to a large extent with conventional fatty acid collectors (Hernáinz and Calero, 1993; Hicyilmaz et al., 1993). Accordingly, many researchers have come to the consensus that selecting a proper depressant is crucial for the selective flotation in the presence of new efficient collectors.

Sodium dodecyl sulfate (SDS), as an anionic surfactant, has been widely used in the flotation separation of specific components, such as celestite, fluorite and calcite, gypsum and calcite, and apatite and calcite, with good results (Saitoh et al., 2007; Shariati et al., 2011); these results support the idea that the separation of the two minerals barite and calcite may be realized by applying SDS. Therefore, a selective depressant, sodium silicate（SS）, has been identified by some researchers; sodium silicate can be a bound complex with metal ions through its silicate groups to make the mineral surface hydrophilic. Zhang et al. reported via XPS analysis that sodium silicate can be selectively adsorbed on the surface of calcite and fluorite, while less so on a scheelite surface (Zhang et al., 2014). A previous study also found that SS could be adsorbed more strongly on the calcite surface than on scheelite (Gao et al., 2016). Thus, a new attempt to inhibit the gangue mineral calcite by applying sodium silicate will be performed in the presence of SDS. The objective of this study is to investigate the effects of SDS and SS on the flotation performance of barite and calcite. The mechanism of selective separation was investigated through dynamic potential, adsorption and infrared spectrum analysis. Sodium oleate (NaOL) was used as a collector for comparison.

## 2. Materials and methods

### 2.1. Pure minerals and reagents

The pure barite was taken from a Chinese mine in Shi Zhu Yuan (Chenzhou) in Hunan Province, and the pure calcite sample was purchased online. All the pure minerals were crushed by a hammer, hand-picked, and then ground in a porcelain mill and dry-sieved to -74 μm for single mineral flotation. A portion of this size fraction of particles was further ground in an agate mortar to obtain -5 μm particles for electrophoresis experiments and IR tests. XRD analysis and chemical composition tests were carried out before conducting the flotation experiments. The purity of barite and calcite was 98.8 % and 97.5 %, respectively. XRD results are shown in Fig. 1. The sample of actual barite ore was obtained from a deposit in Hunan. The sample was crushed, ground and screened into -2 mm particles. Chemical analysis of the sample is shown in Table 1.

The sample of sodium dodecyl sulfate (SDS) used in this study was obtained from the Tianjing Bodi Chemical Reagents Development Center, Tianjin, China. Sodium oleate (NaOL), used for comparison, was provided from the Tianyu Oleo Chemical Corporation (Sichuan, China). The sodium silicate (SS) used in this study was a kind of water-soluble silicate with a molecular formula of $Na_2SiO_3{\cdot}9H_2O$. Analytical-grade HCl and $Na_2CO_3$ were used as pH modifiers.

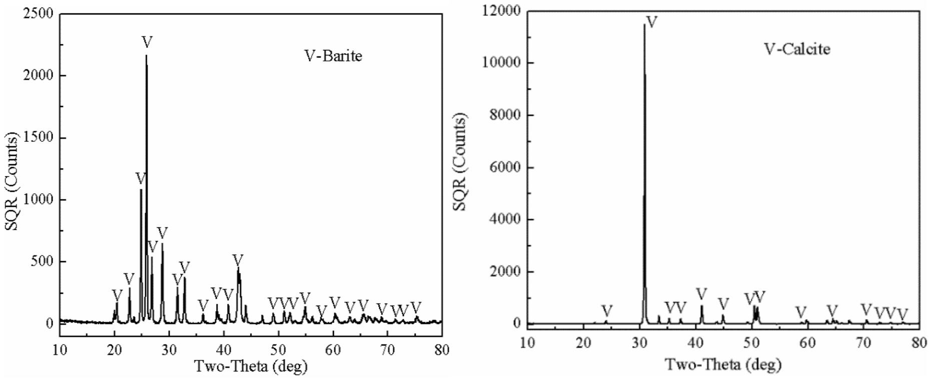

Fig. 1. XRD diagrams of barite and calcite

Table 1. Chemical analysis of the barite sample

| Element | BaO | $SiO_2$ | $Al_2O_3$ | CaO | MgO | Fe | Cu | S | Ag |
|---|---|---|---|---|---|---|---|---|---|
| Grade (mass fraction, %) | 15.9 | 46.01 | 10.45 | 13.68 | 9.38 | 1.15 | 0.044 | 3.31 | 0.004 |

### 2.2. Flotation experiment

Pure mineral flotation experiments were carried out in a mechanical agitation flotation machine (shown in Fig. 2) with a volume of 40 $cm^3$. The mineral suspension was prepared by adding 2 g of pure minerals to 40 $cm^3$ of solution. The pH of the mineral suspension was adjusted to the desired value by adding $Na_2CO_3$ or HCl stock solutions. The prepared sodium silicate and SDS solution were added at a desired concentration and conditioned for 3 min. Each flotation experiment was carried out for 4 min.

Fig. 2. XFG flotation machine used for the flotation tests

### 2.3. Zeta potential measurements

The zeta potential measurements were conducted by using a Malvern Zetasizer Nano ZS90 (England) equipped with a rectangular electrophoresis cell. The suspensions were prepared by adding 20 mg of purified minerals that were ground to -5 μm in an agate mortar and 40 $cm^3$ of ultrapure water. After magnetic stirring for 5 min and settling for 10 min, the supernatant of the dilute fine-particle suspension was obtained for the zeta potential measurement. During stirring, the suspension was conditioned by corresponding reagents at different pH values. Each sample was measured at least three times, and the averages were used as the final results.

### 2.4. Adsorption tests

For each test, 2 g of mineral sample was put into a conical flask with 40 $cm^3$ of distilled water; HCl or $Na_2CO_3$ was added to adjust pH after dispersing for 1 min in an ultrasonicator; and then, the appropriate concentration of reagents was added. The suspension of 2 g of minerals in a 40 $cm^3$ solution of the desired initial concentration and pH was shaken for 30 min to ensure adsorption equilibrium. After being centrifuged, the concentration of SDS in the supernatants was determined using TOC. The calculation formula of adsorption quantity is shown in Eq. 1:

$$\Gamma = \frac{(C_0 - C) \times v \times 10^3}{2} \tag{1}$$

where $\Gamma$ is the adsorption quantity (g/Mg), $C_0$ is the initial reagent concentration (mg/$dm^3$), $C$ is the tested concentration in the supernatant (mg/$dm^3$), $V$ is the pulp volume ($dm^3$).

### 2.5. Infrared spectroscopy measurement

The infrared spectra were performed with a Spectrum One (Version BM) FT-IR (USA) spectrometer at an environmental temperature of 25 °C. The samples were prepared by adding 1 g of pure minerals that were ground to less than 2 μm, and desired reagents were placed in a Plexiglas cell with 40 min of conditioning time. After washing three times using ultrapure water, the washed samples were vacuum-dried below 30 °C and used for the later FT-IR analysis. The spectra of the solids were taken with KBr pellets. The data were collected from 4000 to 400 $cm^{-1}$.

## 3. Results and discussion

### 3.1. Single mineral flotation

The effect of pH on the flotation recovery of barite and calcite using SDS and NaOL as collectors was studied by micro-flotation tests, as shown in Fig. 3. It could be seen from Fig. 3 that the flotation recovery of barite with SDS increased in the pH range from 8.0 to 10.0, and at an optimum pH of approximately 9.0, the best floatability of barite was obtained, with a recovery of approximately 91 %. As for calcite, the floatability was good with a recovery higher than 90 % over the same pH range tested for the barite flotation. When using NaOL as the collector, the optimum flotation pH of barite ranged from 6.0 to 8.0, and the max recovery of 79 % was obtained at pH 8.0, which was less than the max recovery with SDS as the collector. The flotation results of calcite with NaOL were similar to those in the presence of SDS. The results indicated that it was necessary to use a depressant for the selective separation of barite from the calcite.

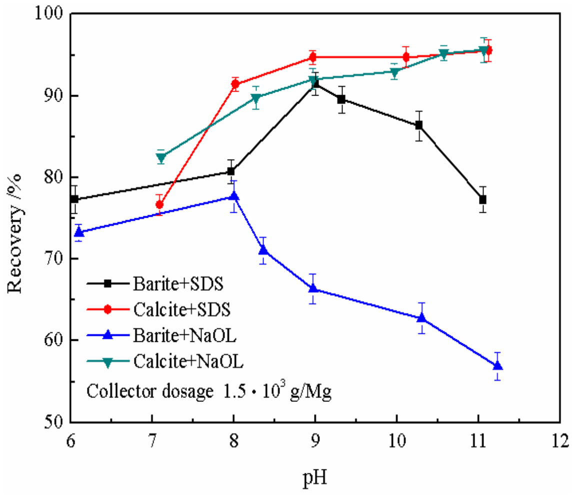

Fig. 3. Effect of pH on the flotation recovery of barite and calcite

The effect of collector SDS and NaOL dosage on barite and calcite at the best flotation pH is shown in Fig. 4. The results showed that the recovery of both barite and calcite increased with increasing concentration of the collector and then reached a maximum value. The max recovery of barite with SDS and NaOL as the collector was more than 90 % at a concentration of $2 \cdot 10^3$ g/Mg and $4 \cdot 10^3$ g/Mg, respectively, while the recovery of calcite reached max values of 94 % and 89 %. There was little difference in the floatability of barite and calcite over the studied collector dosage range. Therefore, it was unavoidable to use a depressant on calcite to realize the separation of barite from calcite.

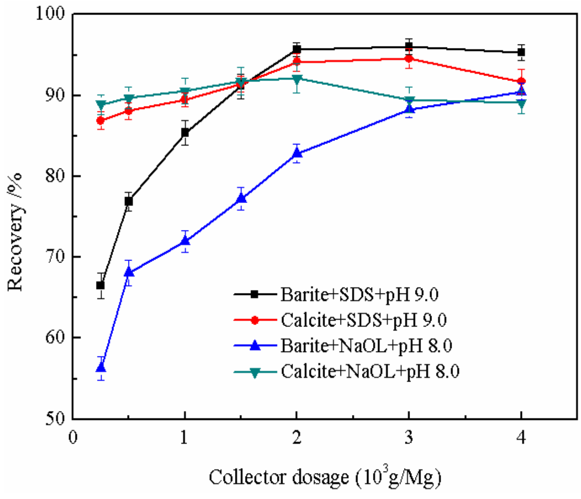

Fig. 4. Effect of collector dosage on barite and calcite flotation

The effects of sodium silicate dosage on the flotation of barite and calcite in the presence of SDS and NaOL are presented in Fig. 5. It could be seen from Fig. 5 that when using NaOL as the collector, the recovery of barite had a dramatic drop as the SS dosage increased, while calcite maintained a good floatability with a recovery over 70 %, indicating that SS had a stronger depressing effect on barite than on calcite. In contrast, when SDS was used as the collector, the recovery of calcite decreased rapidly with the increase in SS dosage, while that of barite changed slightly. It was clear that a certain selectivity could be obtained with the combination of SS and SDS.

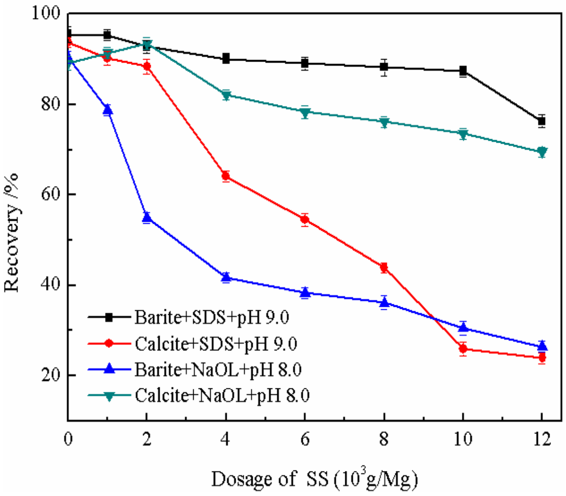

Fig. 5. Effect of SS dosage on barite and calcite flotation with SDS 2 ·$10^3$ g/Mg and NaOL 4 ·$10^3$ g/Mg

Fig. 6 shows the effect of pH on the flotation of barite and calcite using sodium silicate as the depressant. The micro-flotation indicated that the recovery of barite was higher than that of calcite over the pH range tested. The optimal pH range for the separation of barite from calcite was 8.0-10.0, and at a pH value of approximately 9.0, barite and calcite floated 87.6 % and 25.9 %, respectively. This implied that SS could be applied in the separation of barite from calcite.

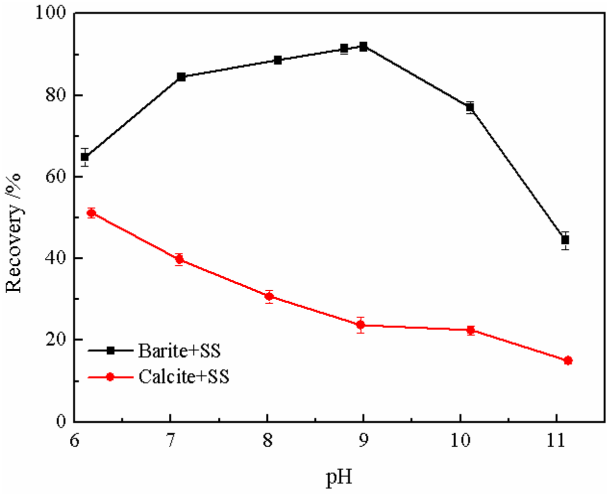

Fig. 6. Effect of SS on barite and calcite flotation under different pH value with SDS 2 ·$10^3$ g/Mg and SS 1 · $10^4$ g/Mg

### 3.2. Actual mineral flotation

The flotation results suggested that it might be possible to separate barite from calcite by depressing the latter with sodium silicate in the presence of SDS. As shown in Fig. 7, the locked cycle flotation process included one roughing, one scavenging and two cleaning steps, and the results are displayed in Table 2. The purity and recovery of $BaSO_4$ in the barite concentrate were 95.54 % and 86.11 %, respectively, implying the high efficiency of the barite flotation separation from calcite and quartz. This study has solved the problem of the flotation separation of barite and calcite, providing a technical support for the flotation recovery of quartz type barite.

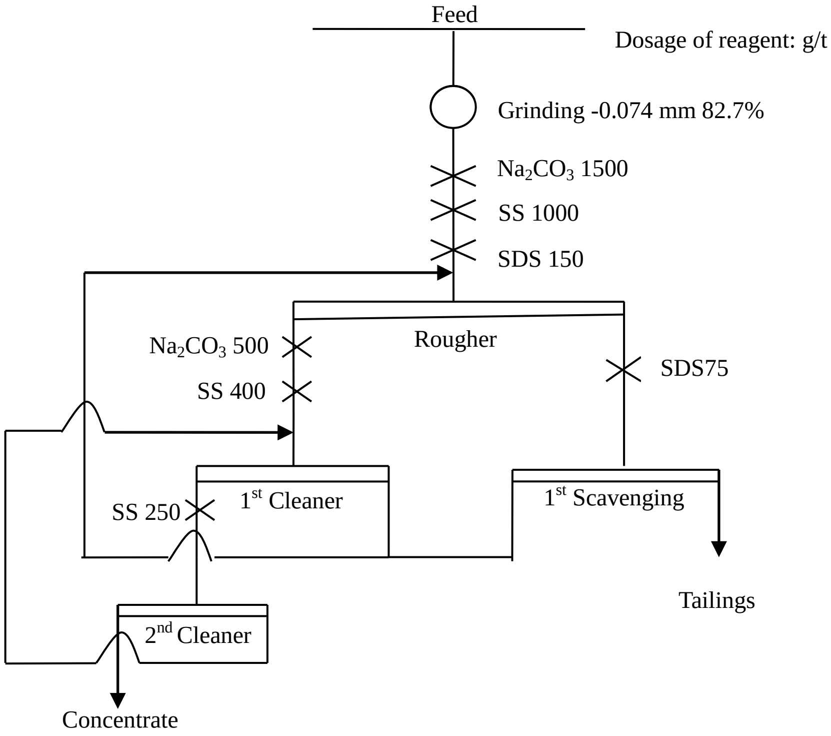

Fig. 7. Closed-circuit test flowsheet

Table 2. Results of closed-circuit experiments

| Product | Productive rate / % | Grade / % | Recovery / % |
|---|---|---|---|
| Barite concentrate | 22.09 | 95.54 | 86.11 |
| Tailing | 77.91 | 4.36 | 13.89 |
| Crude ore | 100.00 | 24.51 | 100.00 |

### 3.3. Zeta potentials

Zeta potential measurement was considered as a basic method for studying interactions of ionic species with minerals. Fig. 8 shows the effect of pre-treatments of sodium silicate on the calcite and barite surfaces in the presence of SDS.

It was clear from Fig. 8 that conditioning in $2 \cdot 10^3$ g/Mg SDS changed the zeta potential of barite from -51.2 mV to -49.1 mV. This result illustrated that the conditioning of barite with sodium silicate prior to SDS addition had not prevented the adsorption of SDS on barite. Furthermore, the conditioning of calcite in $2 \cdot 10^3$ g/Mg SDS with the pretreatment of sodium silicate gives a -35.0 mV zeta potential, which was approximately the same value obtained for calcite in sodium silicate alone (-22.0 mV), illustrating that the addition of sodium silicate prevented the adsorption of SDS on calcite, which was important for realizing the selective separation of calcite from barite in the presence of SDS.

### 3.4. Adsorption

To examine the effect of SDS and sodium silicate addition on mineral electrokinetics and micro-flotation, the quantity of SDS adsorbed on minerals was determined.

In a $2 \cdot 10^3$ g/Mg solution, the amount of SDS adsorbed at pH 9.0 on various minerals as a function of sodium silicate concentration is shown in Fig. 9. With the increase in sodium silicate, the amount of SDS adsorbed on calcite and barite both decreased. The drop in the adsorption quantity of SDS on calcite is more obvious than that for barite. When the addition of sodium silicate was 1·$10^4$ g/Mg, the maximum adsorption difference between barite and calcite was observed, which was consistent with the results shown in Fig. 5. This result illustrated that sodium silicate might be chemically absorbed on the calcite surface, while having a weak chemisorption on the barite surface. Conditioning of calcite with sodium silicate prior to SDS addition could significantly influence the adsorption of SDS on calcite but nearly did not prevent SDS being adsorbed on barite.

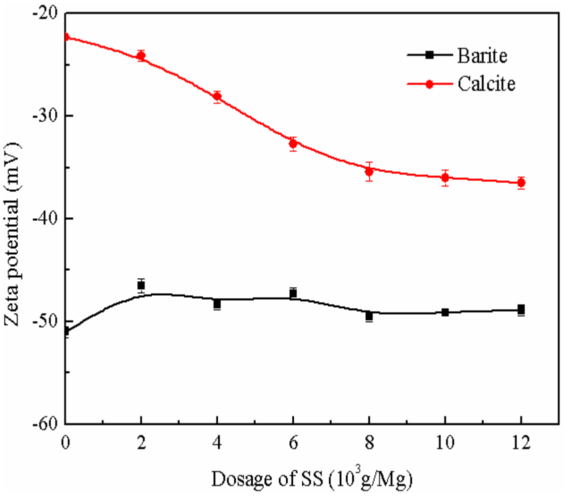

Fig. 8. Zeta potentials of barite and calcite versus SS dosage with SDS 2·$10^3$ g/Mg at pH 9.0

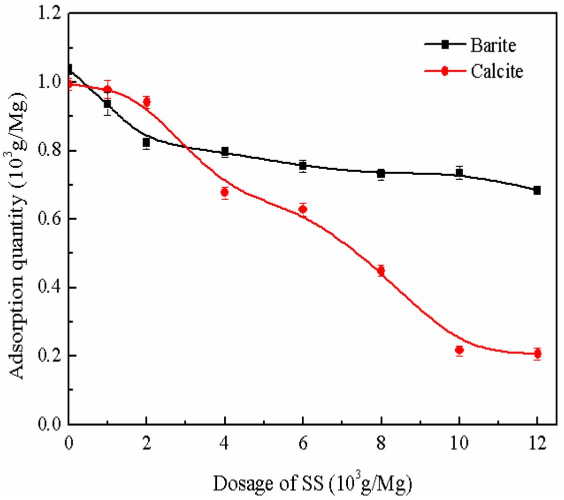

**Fig. 9.** Effect of SS dosage on the adsorption capacity of SDS with SDS 2·$10^3$ g/Mg at pH 9.0

### 3.5. Infrared spectroscopy measurements

Fig. 10 presents the FTIR spectra of the flotation reagents. In the FTIR spectrum of SDS, strong characteristic bands of C-H stretching vibrations were present at 2921.3 cm$^{-1}$ and 2853.7 cm$^{-1}$. Peaks at approximately 1251.7 cm$^{-1}$ and 1221.2 cm$^{-1}$ were due to the anti-symmetric stretching vibration of S=O, the peak at 1082.5 cm$^{-1}$ belonged to the symmetric vibration absorption of S=O, and the vibration of S-O in the sulfate appeared at 831.4 cm$^{-1}$ (Leitch et al., 2012; Viana et al., 2012). For the FTIR spectrum of $Na_2SiO_3 \cdot 9H_2O$, the peak at 3407.3 cm$^{-1}$ was assigned to the stretching vibration peak of associated O-H among sodium silicate, the peak at 1661.2 cm$^{-1}$ represented the O-H bending vibration, the peak at 1445.1 cm$^{-1}$ represented the stretching vibration of free O-H, and 995.4 cm$^{-1}$ and 461.8 cm$^{-1}$ represented the stretching vibration peak of Si-O-Si in $SiO_3^{2-}$ and the bending vibration peak of Si-O-Si in $SiO_4^{4-}$.

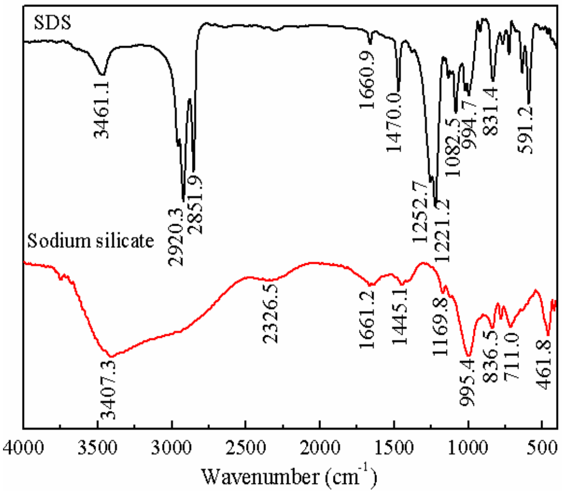

Fig. 10. Infrared spectra of reagents

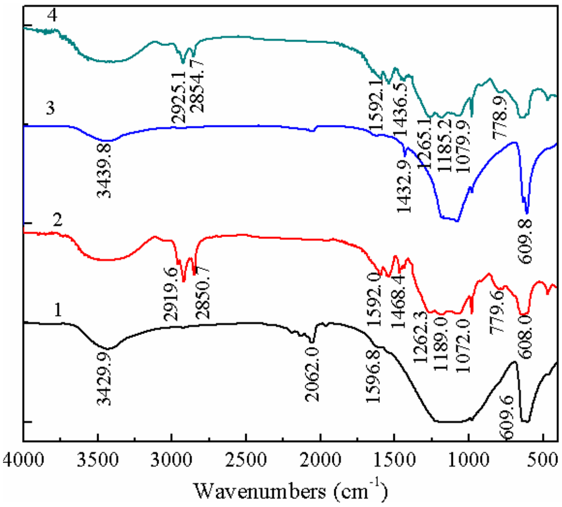

Fig. 11. The IR spectra of barite conditioned with different reagents: 1 - barite; 2 – barite + SDS; 3 – barite + sodium silicate and 4 – barite + sodium silicate + SDS

To determine the mechanism of reagents in the flotation separation of barite from calcite, the FTIR spectra of barite and calcite conditioned with SDS or SS at pH approximately 9.0 were obtained (Figs. 11 and 12).

The IR spectrum of barite after interacting with SDS is shown as line 2 in Fig. 11. Two new absorption peaks appeared at approximately 2919.6 cm$^{-1}$ and 2850.7 cm$^{-1}$ due to the stretching vibrations of C-H in the $CH_2$ and $CH_3$ groups. These signals indicated the presence of SDS adsorbed on barite. Additionally, the wavenumbers of the S=O signals decreased from 1252.7 to 1262.3 cm$^{-1}$, from 1221.2 to 1189.0 cm$^{-1}$ and from 1082.5 to 1072.0 cm$^{-1}$, changes that were the results of the chemical adsorption of SDS on barite. The IR spectrum of barite after interacting with sodium silicate is shown as line 3. There appeared two peaks at 3439.8 cm$^{-1}$ and 1432.9 cm$^{-1}$, which were attributed to the stretching vibration of associated O-H and free O-H, illustrating that sodium silicate occurred chemical adsorption on barite surface. The IR spectrum of barite that interacted with SDS with the pre-adsorption of sodium silicate is shown as line 4. The characteristic adsorption bands of SDS still appeared at 2925.1 cm$^{-1}$, 2854.7 cm$^{-1}$, 1436.5 cm$^{-1}$, 1265.1 cm$^{-1}$, 1185.2 cm$^{-1}$ and 1079.9 cm$^{-1}$. These results illustrated that sodium silicate could not interfere with the adsorption of SDS on the surface of barite.

The IR spectrum of calcite after interacting with SDS is shown as line 2 in Fig. 12. There existed not only a new band that appeared at 2922.2 cm$^{-1}$ in line 2 due to the stretching vibrations of the $CH_2$ groups but also a symmetric stretching band of S=O at 1076.1 cm$^{-1}$ that had shifted by 6.4 cm$^{-1}$; these changes indicated that the collector SDS was chemically adsorbed on the calcite surface (Zeng et al., 2017). The

IR spectrum of calcite after interacting with sodium silicate is shown as line 3. There were two new peaks at 1021.6 $cm^{-1}$ and 449.1 $cm^{-1}$ due to the stretching vibration of Si-O-Si, indicating that sodium silicate occurred chemical adsorption on calcite surface. The FTIR spectrum of calcite treated with SDS with the pre-adsorption of sodium silicate is shown as line 4. The spectrum showed that the characteristic adsorption bands of SDS disappeared and that the characteristic adsorption bands of sodium silicate appeared at 1019.3 and 451.3 $cm^{-1}$. These results illustrated that sodium silicate interfered with the adsorption of SDS on the calcite surface.

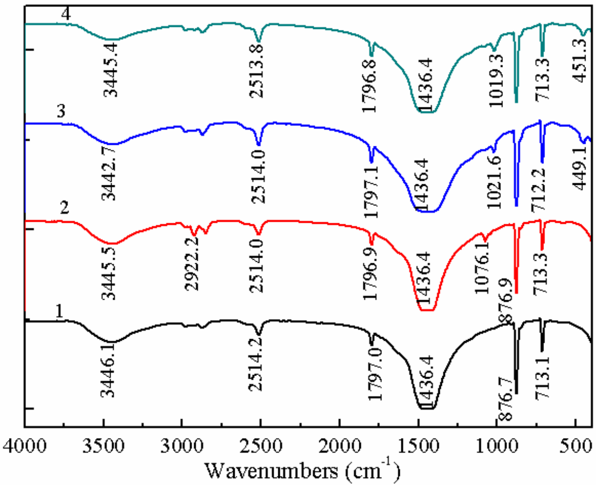

Fig. 12. The IR spectrum of calcite conditioned with different reagents: 1 - calcite; 2 – calcite + SDS, 3 – calcite + sodium silicate and 4 – calcite + sodium silicate + SDS

## 4. Conclusions

The anionic collector SDS exhibited a strong collecting capacity for both barite and calcite in micro-mineral flotation experiments, leading to the difficulty of separating them. Sodium silicate was an efficient depressant on calcite in the presence of SDS. The preferable flotation pH ranged from 8.0 to 10.0, and at approximately pH 9.0, the large floatability difference of barite and calcite was obtained with a variation of 61.7 %. In the actual mineral flotation experiments using an ore containing 24.5 % $BaSO_4$, when sodium carbonate and sodium silicate were associated as modified reagents with SDS as the collector, a concentrate grading of 95.54 % with a recovery rate of 86.11 % was obtained.

The dynamic potential measurement, adsorption and FTIR spectral analysis revealed that chemisorption was the main adsorption mechanism of SDS on barite and calcite. The pre-adsorption of sodium silicate interfered with the adsorption of SDS on the calcite surface while not interfering with its adsorption on the barite surface.

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