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Mechanism of calcium lignosulfonate in apatite and dolomite flotation system

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Abstract: Since the physical and chemical properties of apatite and dolomite can be similar, the separation of these two minerals is difficult. Therefore, when performing this separation using the flotation method, it is necessary to search for selective depressants. An experimental research was performed on the separation behavior of apatite and dolomite using calcium lignosulfonate as a depressant, and the mechanism by which this occurs was analyzed. The results show that calcium lignosulfonate has a depressant effect on both apatite and dolomite, but the depressant effect on dolomite is stronger at the same dosage. Mechanism analysis shows that the adsorptive capacity of calcium lignosulfonate on dolomite is higher than that of apatite, which is due to the strong reaction between calcium lignosulfonate and the Ca sites on dolomite. In addition, there is a hydrogen bond between calcium lignosulfonate and dolomite, which further prevents the adsorption of sodium oleate to dolomite, thus greatly inhibiting the flotation of dolomite.

Keywords: apatite; dolomite; calcium lignosulfonate; depression mechanism

1. Introduction

Phosphate-containing mineral is an important natural resource, and as one of the sources of fertilizer, are of great significance for food production [1–3]. Mined phosphate is also used in the medical, dye manufacturing, national defense, and other industries. Apatite is the main phosphate mineral for extracting phosphorus from igneous- and sedimentary-hosted ores [4]. There are many associated minerals in apatite ore, especially dolomite [5]. The physical and chemical properties of apatite and dolomite are similar, which is not conducive to the flotation separation of the two minerals [6–8].

Flotation is a widely used method of mineral enrichment, the separation of target minerals from gangue, and most of the phosphate ore in the world is processed by this method [9–10]. Fatty-acid collectors are widely used in apatite flotation [11], but high-grade apatite concentrates are difficult to isolate due to the poor selectivity of such collectors, making the selection of depressant a critical element of the process [12].

Depressants play a crucial role in both direct flotation and reverse flotation of minerals [13–16]. In order to separate apatite from carbonate minerals (dolomite or calcite) using the direct flotation method, carbonate minerals can be depressed by aromatic sulfonate polymers, hydrofluoric acid, citric acid, gum Arabic, sodium silicate and so on [12,15,17–18]. In the reverse flotation system, apatite can be depressed by a number of agents, such as dipotassium hydrogen phosphate, sodium pyrophosphate [12], or phosphoric acid [19], with carbonate floated using fatty acids as collectors. The above depressants have some problems, including complex production process and environmental pollution. Meanwhile, the separation of apatite from carbonate minerals remains a challenge to the phosphate flotation industry. The development of an environmentally acceptable and selective depressant is therefore of interest to the mineral industry.

Calcium lignosulfonate is an anionic polymer consisting of sulfonic acid, carboxylic acid, phenolic hydroxyl groups, and alkyl groups [20–21], and it shows the properties of dispersibility, wettability, and adsorption [22]. It can be used as dispersants, adhesives, depressor, and chelators of heavy metal ions [23]. Calcium lignosulfonate comes from a wider range of sources than other organic depressors (guar gum, locust bean gum) and is made from lignin, it has the advantages of non-toxic, renewable, and low cost [24–26]. There are many reports about calcium lignosulfonate in mineral flotation. Calcium lignosulfonate as depressor can selectively depress calcite in scheelite flotation [27–28]. Lignosulfonate is also used as depressant for the flotation of muscovite [29]. Moreover, calcium lignosulfonate also has depressant effect on chalcopyrite [30], talc [31].

Although calcium lignosulfonate has many applications in flotation, its selective depression of dolomite in apatite flotation system is rarely studied. The purpose of this study is to

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confirm if calcium lignosulfonate is effective in the separation of apatite and dolomite.

2. Experimental

2.1. Materials

Apatite (hydroxyapatite) and dolomite purchased from Ye's mineral specimen store in Huadu District, Guangzhou were manually crushed and mechanically pulverized, and particles in the –74+38 μm were selected for flotation. Fig. 1 shows the X-ray diffraction analysis of apatite and dolomite. Table 1 represents the samples' multi-element analysis. The purity of apatite and dolomite is 93.60wt% and 98.34wt%, respectively.

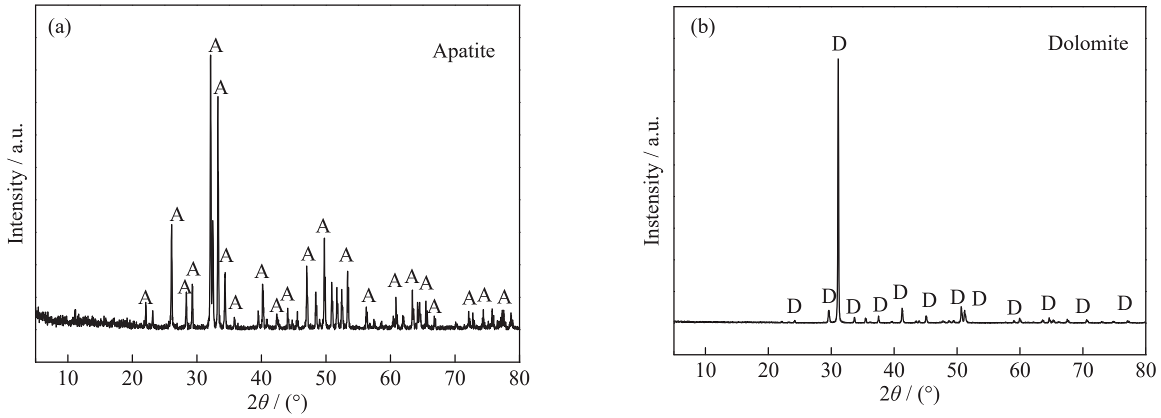

Fig. 1. X-ray diffraction diagrams of (a) apatite and (b) dolomite.

Table 1. Multi-element analysis results of minerals

Mineral	P_2O_5 / wt%	MgO / wt%	Purify / wt%
Apatite	39.37	—	93.60
Dolomite	—	21.34	98.34

The agents used in the experiment were sodium oleate (NaOl) as collector. Calcium lignosulfonate was obtained from Shanghai Macklin Biochemical Co., Ltd., China. The structure of calcium lignosulfonate is showed in Fig. 2. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) as pH regulator were purchased from Xilong Scientific Co., Ltd., China. Distilled water was used in the experiments.

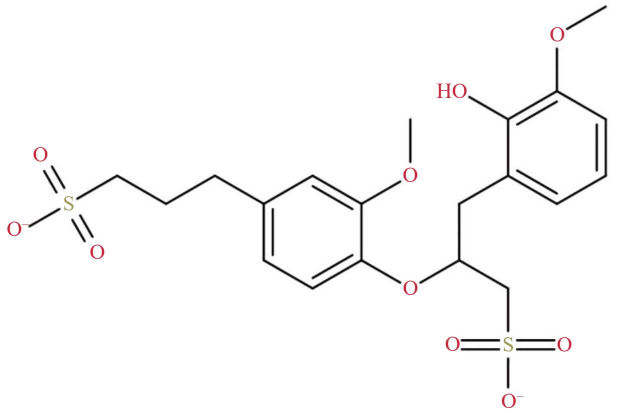

Fig. 2. Structure of calcium lignosulfonate.

2.2. Micro-flotation tests

Micro-flotation tests were carried out using XFG flotation machine [32]. For each flotation test, 2 g of sample and 40 mL distilled water were added to the flotation cell and mixed for 2 min while the pH of pulp was conditioned using pH regulator. Depressant and collectors were then added in turn and conducted for 3 min respectively. Scrape the foam manually for 3 min, the collected product will be dried and weighed to calculate the flotation recovery. Each flotation test was carried out three times, and the average value was calculated to plot.

2.3. Contact angle measurements

The contact angle was measured by JY-82C contact angle meter of Chengde Dingsheng Tester Co., Ltd., Chengde, China. After polishing the sample, it was immersed in the prepared reagent solution, and then the processed samples were dried at low temperature in a vacuum drying oven for contact angle measurement. Each sample was measured 3 times at different positions.

2.4. Adsorption quantity measurements

Adsorption amount of agent on the samples surface was determined indirectly by a total organic carbon analyzer (made by ELEMENTAR, Germany). 1 g of sample and 50 mL distilled water was added to a beaker and the pH of suspension was adjusted. After adding calcium lignosulfonate, the suspension was conditioned in a shaking incubator for 60 min at 25°C. The minerals in suspension were then filtered and the filtrate was collected for analysis.

2.5. Fourier transform infrared spectroscopy (FTIR) measurements

FTIR was performed by an ALPHA FTIR spectrometer of Bruker, Germany. 1 g of mineral and 50 mL distilled water was added to a beaker, add 20 $mg \cdot L^{-1}$ of calcium lignosulfonate if necessary, and stirred for 10 min at the pH value of 9. After adding calcium lignosulfonate solution, the suspension was conditioned in a shaking incubator. The minerals in suspension were then filtered and dried. 1 mg sample was mixed with 100 mg of spectrally pure KBr [33] and the mixture was pressed using a Carver® hydraulic press to form the pellet. The spectrum was scanned over 4000–450 cm^{-1} range on the transmission mode of the instrument [34].

2.6. XPS measurements

The chemical composition of minerals before and after interacted with the depressant was determined by using X-ray photoelectron spectrometry (XPS). 2 g of sample (particle size <10 μm) was mixed with 40 mL distilled water. Then 20 $mg\cdot L^{-1}$ of calcium lignosulfonate was added and the pH value of suspension was conditioned to 9. The suspension was conditioned in a shaking incubator for 30 min at 25ºC. Clean and dry the minerals to prepare for XPS measurements.

3. Results and discussion

3.1. Micro-flotation test results

As shown in Fig. 3, recoveries of dolomite and apatite improve greatly during the NaOl dosage experiment when the NaOl concentration increases from 0 to 1×10^{-4} $mol\cdot L^{-1}$. Beyond that dosage, the recoveries of dolomite and apatite only change slightly. These results show that apatite and dolomite cannot be separated only by adjusting the dosage of NaOl.

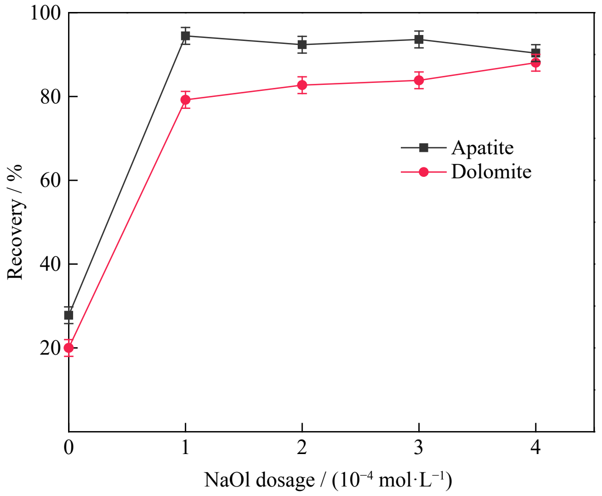

Fig. 3. Effect of NaOl dosage on the flotation of dolomite and apatite (pH value = 9).

When the dosage of NaOl is 1×10^{-4} $mol\cdot L^{-1}$, the effect of pH value on the flotation of dolomite and apatite is investigated with and without calcium lignosulfonate. The results are shown in Fig. 4. Without calcium lignosulfonate, the recovery of apatite increases with pH value from 6 to 9, and reaches the highest at a pH value of 9. For dolomite, the recovery slightly increases as pH value increases from 6 to 10, with a maximum value at a pH value of 10. The flotation behavior of the two minerals is similar. Therefore, the separation of dolomite and apatite cannot be achieved by simply adjusting pH without adding depressant. When calcium lignosulfonate is present, its ability to depress apatite gradually lessens as pH value increases from 6 to 10. Unlike its effect on apatite, calcium lignosulfonate has an obvious depressant effect on dolomite at pH value 6–10, with recovery below 10%. The greatest difference in recovery between the two minerals is observed at a pH value of 9. These results indicate that the addition of calcium lignosulfonate makes it possible to separate the two minerals.

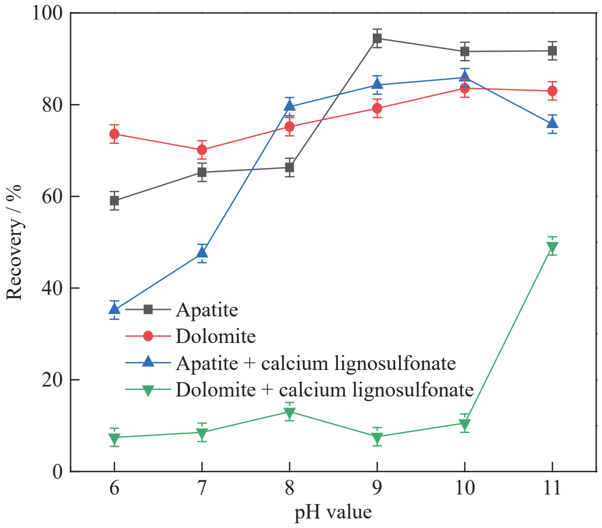

Fig. 4. Effect of pH value on the flotation of dolomite and apatite. Calcium lignosulfonate dosage: 20 $mg\cdot L^{-1}$, NaOl dosage: 1×10^{-4} $mol\cdot L^{-1}$.

When the NaOl dosage is 1×10^{-4} $mol\cdot L^{-1}$, the effect of calcium lignosulfonate dosage on the flotation of apatite and dolomite is investigated. Fig. 5 shows that calcium lignosulfonate has depressant effect on both apatite and dolomite, but the depressant effect on dolomite is stronger. When the dosage of calcium lignosulfonate is 20 $mg\cdot L^{-1}$, the huge difference in recovery between dolomite and apatite indicates that calcium lignosulfonate can separate dolomite and apatite.

Flotation separation test of artificial mixed ore of dolomite and apatite is carried out. Flotation results of mixed ore before and after the use of calcium lignosulfonate are shown

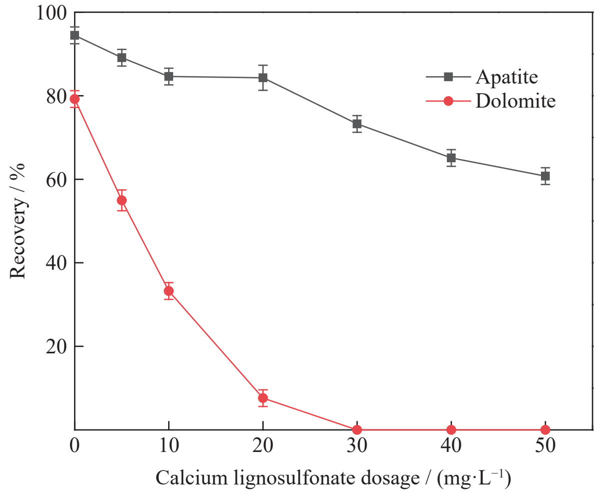

Fig. 5. Effect of calcium lignosulfonate dosage on the flotation of dolomite and apatite (pH value = 9).

in Fig. 6. Fig. 6 shows that after adding calcium lignosulfonate, the recovery and grade of apatite increase from 65.20% and 18.16% to 68.32% and 23.56%, indicating that calcium lignosulfonate can effectively depress dolomite in the process of apatite flotation.

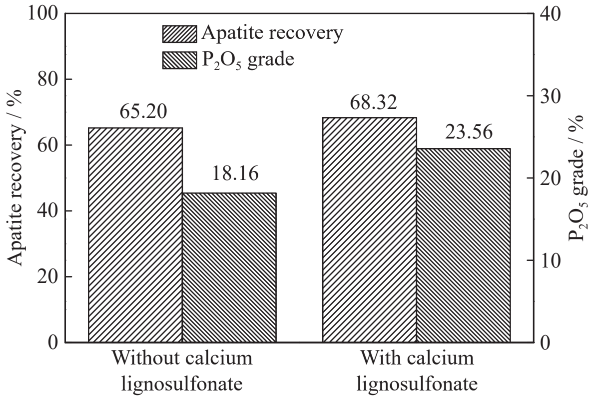

Fig. 6. Effect of calcium lignosulfonate on flotation results of artificial mixed ores of dolomite and apatite. Calcium lignosulfonate dosage: 20 mg·L^{-1}, NaOl dosage: 1 × 10^{-4} mol·L^{-1}, pH value = 9.

3.2. Adsorption quantity analyses

The adsorption behaviors of calcium lignosulfonate on dolomite and apatite are investigated. Fig. 7 illustrates that as the calcium lignosulfonate dosage increases from 0 to 50 mg·L^{-1}, its adsorption density on both minerals increases. However, the absolute adsorption density of calcium lignosulfonate on apatite is much lower than that on dolomite. Specifically, when the concentration of calcium lignosulfonate is 20 mg·L^{-1}, the adsorption density of calcium lignosulfonate on dolomite is 7.7 mg·m^{-2}, and on apatite it is 3.3 mg·m^{-2}, half that of dolomite.

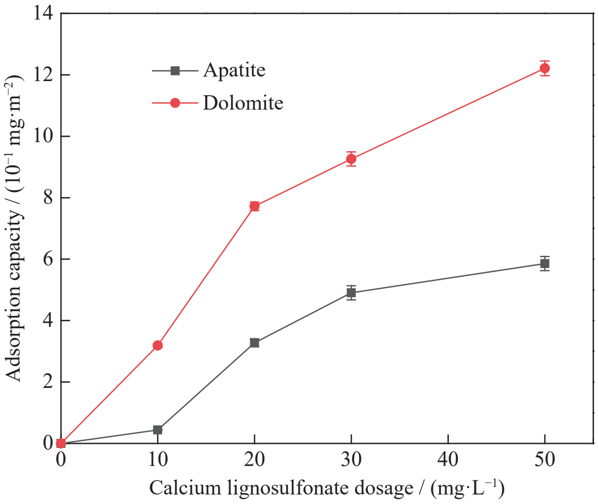

Fig. 7. Adsorption capacity of calcium lignosulfonate on apatite and dolomite (pH value = 9).

3.3. Contact angle analysis

The effect of calcium lignosulfonate on apatite and dolomite surface wettability is studied and the result is shown in Figs. 8 and 9. Figs. 8 and 9 show that the average contact angles of apatite and dolomite surfaces without any reagent are 74.1° and 68.2°, respectively. The average contact angles of these two minerals increase by 34.7° and 34.6°, respectively, when they react with NaOl. After the treatment of sodium oleate and calcium lignosulfonate, the average contact angles of apatite is 87.0° and that of dolomite 59.3°, and the dolomite is more affected by calcium lignosulfonate, and its hydrophobicity decreases more. Combined with the adsorption test, calcium lignosulfonate is adsorbed more on dolomite surface, which increases the hydrophilicity of dolomite to a greater extent.

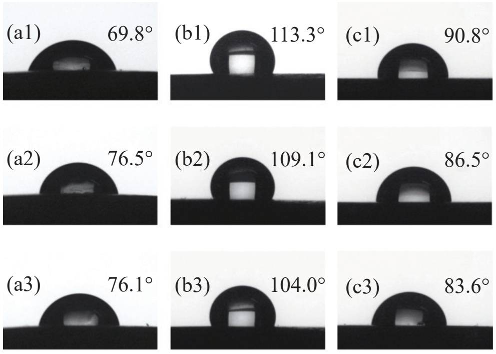

Fig. 8. Effect of agents on mineral contact angle: (a1–a3) apatite; (b1–b3) apatite + NaOl; (c1–c3) apatite + calcium lignosulfonate + NaOl.

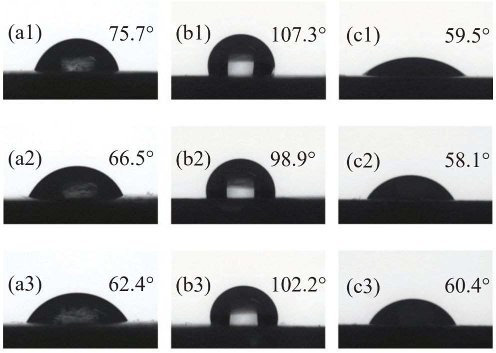

Fig. 9. Effect of agents on mineral contact angle: (a1–a3) dolomite; (b1–b3) dolomite + NaOl; (c1–c3) dolomite + calcium lignosulfonate + NaOl.

3.4. FTIR analysis

The FTIR of dolomite and apatite with and without calcium lignosulfonate are compared to estimate the reaction mechanism of calcium lignosulfonate on the surface of dolomite and apatite. The infrared spectrums of calcium lignosulfonate and apatite are shown in Fig. 10(a). For calcium lignosulfonate, the peaks at 3422, 1600, 1217, and 1115 cm^{-1} are associated with hydroxyl, benzene ring, phenolic hydroxyl, and $-SO_3$. The broad band at 1044cm^{-1} is attributed to mixed peak of $-SO_3$ and C–O–C [35–37].

For apatite, the peak at 3429 cm^{-1} represents the stretching vibration of –OH, which confirms that the apatite used in

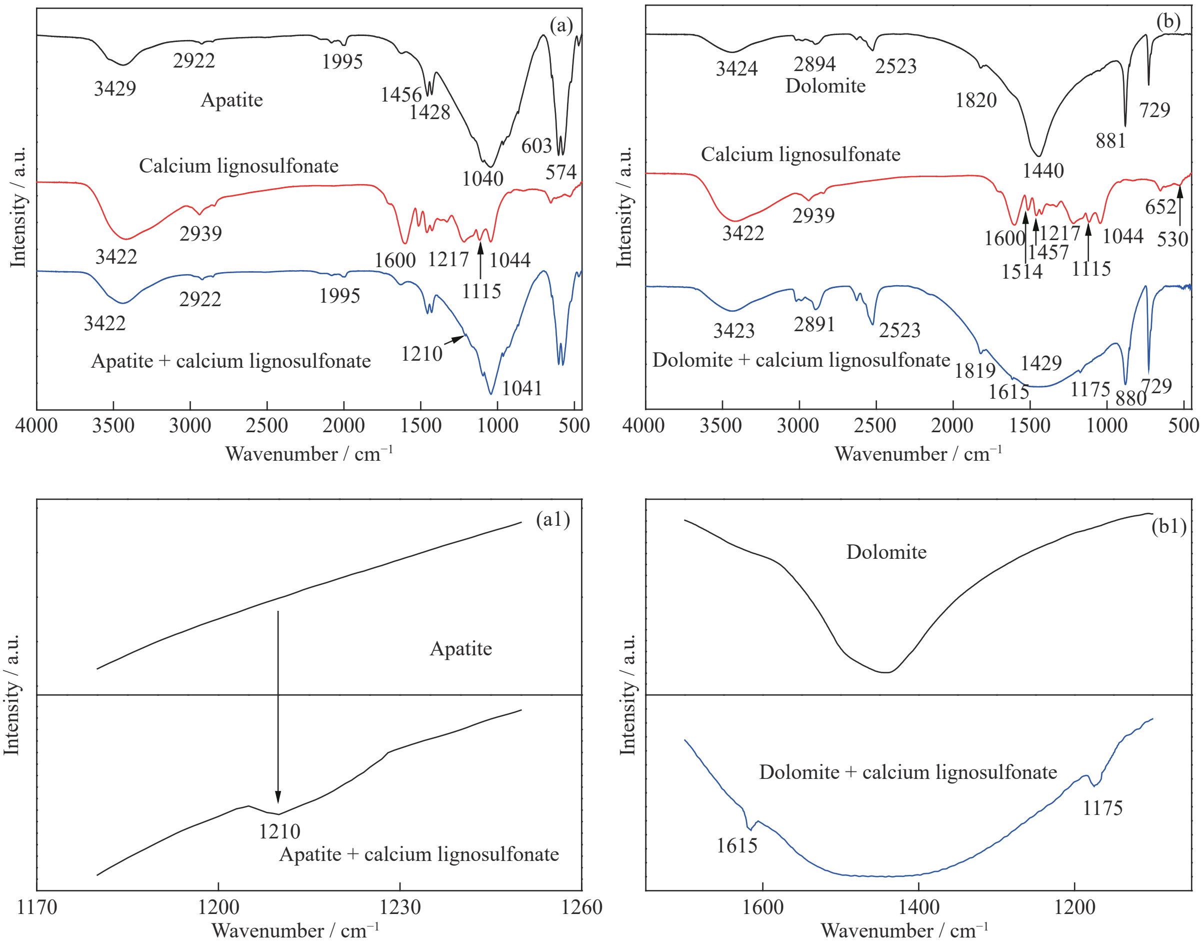

Fig. 10. Infrared spectra of (a, a1) apatite and (b, b1) dolomite with and without calcium lignosulfonate.

the experiment is hydroxyapatite. The peaks at 574 and 603 cm^{-1} are P–O bending bands of apatite, the peak at 1040 cm^{-1} is P–O asymmetrical stretching band [38–39]. After adding calcium lignosulfonate, a new weak phenolic hydroxyl peak appeared at 1210 cm^{-1} (Fig. 10(a1)), and P–O asymmetrical stretching band shifted from 1040 to 1041 cm^{-1}, indicates that there was a weak adsorption between calcium lignosulfonate and apatite.

Fig. 10(b) describes the infrared spectrum of dolomite with and without calcium lignosulfonate. For dolomite untreated by the depressant, the adsorption band at 1440 cm^{-1} represents C–O asymmetry band of dolomite, the bands at 880 and 729 cm^{-1} are attributed to C–O bending bands of dolomite [40]. After interaction with calcium lignosulfonate, the dolomite has new peaks at 1615 and 1175 cm^{-1}, which are derived from the vibration peaks of the benzene ring and $-SO_3$ of calcium lignosulfonate, respectively (Fig. 10(b1)). The C–O asymmetry band of dolomite changed from 1440 to 1429 cm^{-1} and its peak pattern became wider. These results indicate that calcium lignosulfonate is adsorbed on dolomite surface by strong chemical reaction.

3.5. XPS analysis

The interaction mode of apatite and dolomite with calcium lignosulfonate was investigated by XPS. The spectra of Ca 2p and O 1s of these two minerals before and after the reaction with calcium lignosulfonate were studied. Fig. 11 depicts the Ca 2p of apatite and dolomite. The Ca 2p peaks on apatite surface are shown in Fig. 11(a). Before reaction with calcium lignosulfonate, the Ca $2p_{1/2}$ and Ca $2p_{3/2}$ fitting peaks of apatite appear at 350.77 and 347.21 eV, respectively [41–42]. After the addition of calcium lignosulfonate, Ca 2p peaks of apatite are observed at 350.90 and 347.33 eV, respectively, without too much binding energy shift, illustrating that the adsorption between calcium lignosulfonate and apatite is weak. Fig. 11(b) depicts the peaks of Ca 2p on dolomite surface. After interaction with calcium lignosulfonate, the peak of Ca $2p_{1/2}$ and Ca $2p_{3/2}$ shifts from 350.64 and 347.04 eV to 350.90 and 347.30 eV, respectively. The shift of 0.26 eV binding energy of the Ca 2p (significant for measurements with a resolution of 0.2 eV) indicates that calcium lignosulfonate is adsorbed to the surface of dolomite through a strong chemical reaction at calcium sites on the dolomite surface.

Fig. 12 depicts the O 1s spectra of apatite and dolomite with and without calcium lignosulfonate. As shown in Fig. 12(a), before reaction between apatite and calcium lignosulfonate, the peaks locate at 532.93 and 531.06 eV, corresponding to the terminal hydroxyl group on surface of apatite and the PO_4^{3-} of apatite itself [43]. After the addition of calcium lignosulfonate, the position of –OH remains unchanged, but the peak area increases, indicating that calcium lignosulfonate is adsorbed on apatite surface by means other than hydrogen-bond interaction. Combined with the Ca 2p peak shift of

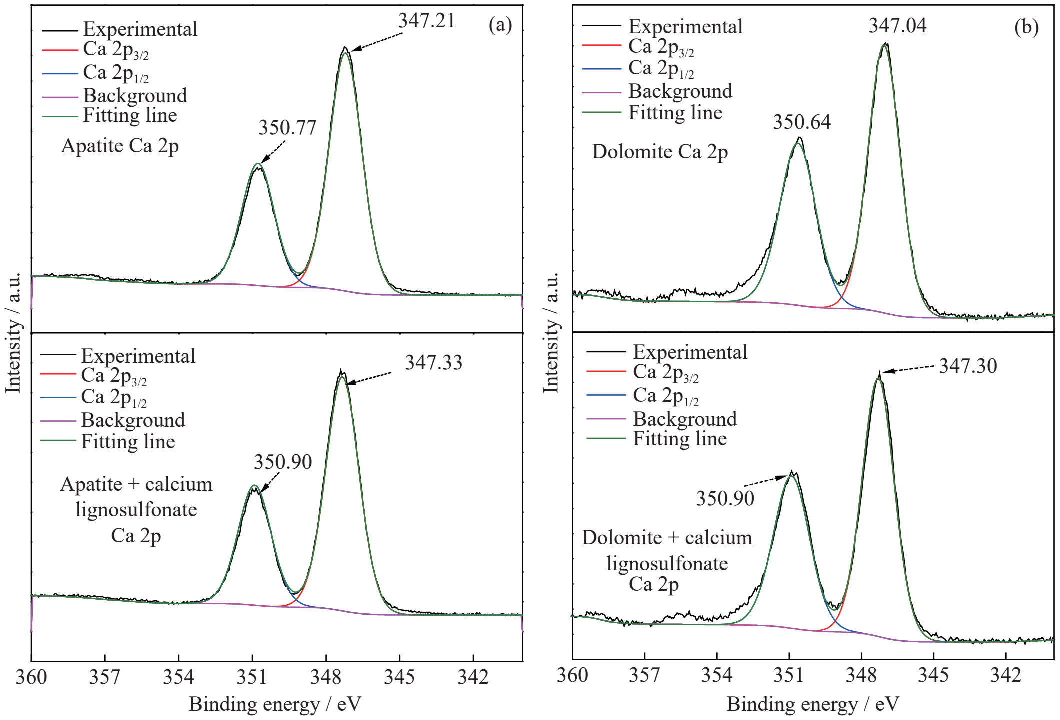

Fig. 11. Ca 2p spectrum of (a) apatite and (b) dolomite with and without calcium lignosulfonate.

apatite, it can be concluded that calcium lignosulfonate increases the –OH content on the surface of apatite through chemical reaction with the Ca site on apatite surface. In Fig. 12(b), the peaks at binding energies of 531.92 and 531.49 eV belong to –OH and CO_3^{2-}, respectively. After interaction with calcium lignosulfonate, the position of –OH shifts from 531.92 to 532.94 eV, indicating that there is a hydrogen bond between calcium lignosulfonate and dolomite. The peak shown at 533.67 eV is attributed to the $-SO_3$ of calcium lignosulfonate, which also indicates that calcium lignosulfonate has been adsorbed on the dolomite surface. Combined with the binding energy shift of dolomite Ca 2p, it can be speculated that $-SO_3$ of calcium lignosulfonate reacts with Ca site on dolomite surface and adsorbed on the dolomite.

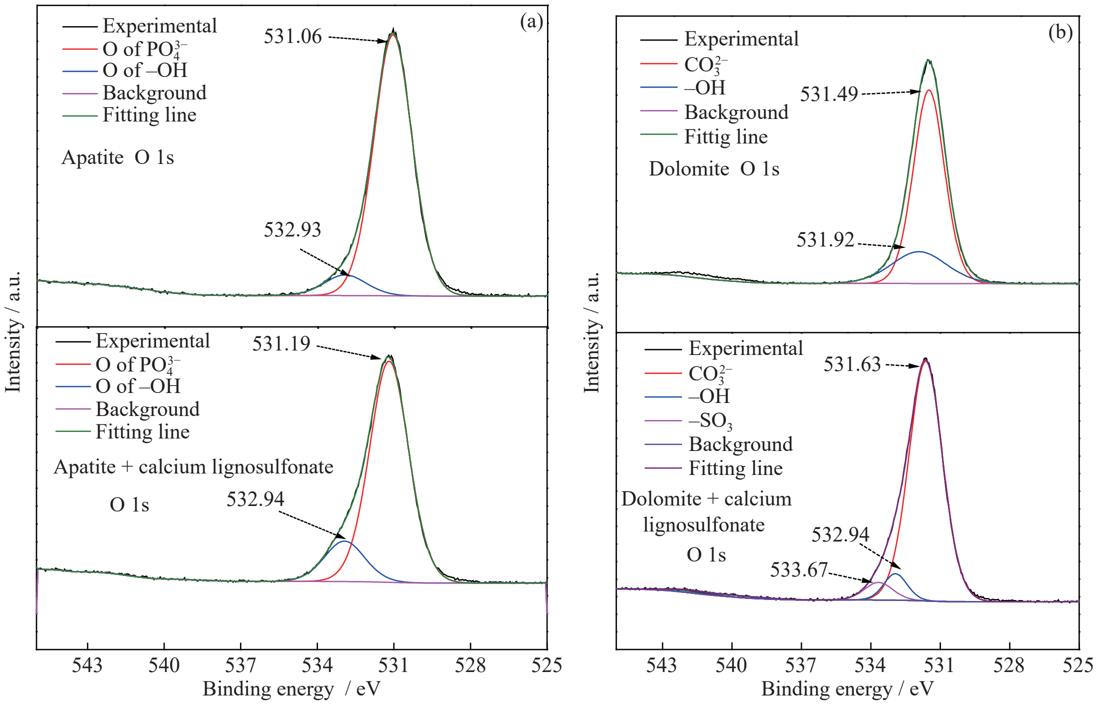

Fig. 12. O 1s spectrum of (a) apatite and (b) dolomite with and without calcium lignosulfonate.

4. Conclusions

Calcium lignosulfonate has a depressant effect on both apatite and dolomite, but has a stronger depressant effect on dolomite. With a calcium lignosulfonate dosage of 20 mg·L^{-1} and a pH value of 9, the recoveries of the two minerals are significantly different.

(1) Contact angle tests show that the hydrophobicity of dolomite decreases significantly with calcium lignosulfonate. Adsorption tests show that the adsorption density of dolomite is higher than that of apatite at the same concentration of calcium lignosulfonate. The difference of adsorption density on the surface of dolomite and apatite also explains the difference of hydrophobicity on the surface of mineral.

(2) Infrared spectrum analysis and XPS analysis show that calcium lignosulfonate reacts with Ca sites on dolomite and apatite, and calcium lignosulfonate is also adsorbed on dolomite by hydrogen bonds, making the adsorption density of calcium lignosulfonate on apatite and dolomite surface different. Specifically, calcium lignosulfonate reacts more strongly with dolomite, resulting in higher adsorption density than apatite, further impeding the adsorption of sodium oleate to dolomite, thereby increasing the difference of flotation behavior between the two minerals.

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Conflict of Interest

The authors declare no potential conflict of interest.

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