

The removal of dolomite from collophane using reverse flotation process enhanced by compound collector

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Abstract: To improve the magnesia removal effect of collophane and increase the P₂O₅ grade of phosphorus concentrate, the fatty acid collector mixed with sodium dodecyl sulfate (SDS) was used as the compound collector in reverse flotation for magnesia removal in acid system. Solution surface tension measurement, Zeta potential test and infrared spectrum analysis were conducted to analyze the intensified effect of compound collector for dolomite removal. The results showed that under the conditions of grinding fineness -0.074 mm accounted for 89.8%, sulfuric acid dosage 12.0 kg/t, phosphoric acid dosage 3.0 kg/t, SDS compound proportion 3%, collector dosage 1.0 kg/t, the P₂O₅ grade of concentrate is increased from 25.7% to 29.94%. SDS can reduce the surface tension of slurry, which is conducive to the formation and stability of flotation foam. Zeta potential became negative due to the compound collector adsorbed on the mineral surface. Moreover, the -CH₂ antisymmetric stretching vibration peak originated from fatty acid and SDS were detected after the interaction of collectors with dolomite. The compound collector was effectively adsorbed on the surface of dolomite, enhancing the magnesium removal effect of collophane.

Keywords: collophane, dolomite removal, reverse flotation, compound collector, SDS

1. Introduction

Phosphate rock is a non-renewable strategic mineral resource, which plays an important role in food production and phosphorus chemical industry (Li et al., 2017). Phosphorus is widely used in agriculture, chemical industry, food, glass, ceramics, medicine and other fields. And 66% of the phosphate ore is used for the production of phosphate fertilizer (DAP, MAP, TSP), 6% for the production of animal feed, 9% for the food industry. The rest for the production of other phosphorus fine chemical products, such as detergent, phosphorus extraction agent and flame retardant, etc. Due to the continuous development of agriculture and high-tech industry, the demand for phosphorus fertilizer and phosphorus fine chemical products is increasing, while as an important raw material of phosphorus chemical industry (Liu et al., 2022). With the exhaustion of highly grade phosphorus resources, more and more attention has been paid to the development and utilization of low-grade phosphate ore.

Hubei province is relatively rich in phosphate resources, but most of them are middle and low grade collophane, which can't be directly used for wet acid production (Peng, 2017). It is necessary to preconcentrate and remove harmful impurities, mainly including dolomite, quartz, feldspar and other silicone-calcareous gangue (Yu et al., 2016; Li et al., 2022). In the production process of wet-process phosphoric acid, excessive magnesium content in collophane will increase acid consumption and phosphoric acid viscosity. It is easy to form gelling substances to reduce the filtration speed of wet-process phosphoric acid. Therefore, it is necessary to remove magnesium priorly to obtain qualified phosphorus concentrate. Flotation is the most effective method used in collophane separation

process (Liu et al., 2020). However, the apatite in collophane mainly exist in amorphous or cryptocrystalline form. And the floatability of apatite and dolomite is quite similar, which greatly improves the difficulty of separation (Zhang et al., 2021; Xie et al., 2022). The reverse flotation is commonly used for separating high magnesium collophane. The process is simple, which can be carried out at low ambient temperature to reduce energy consumption. Its product particle size is relatively coarse, which is conducive to the subsequent thickening treatment. And it conforms to the flotation process principle of "less flotation, more inhibition" (Du et al., 2016).

Flotation reagent plays an important role in collophane flotation. According to the recent report (Zhao et al., 2016), using a single reagent is difficult to achieve efficient separation of apatite. The magnesium gangue minerals in collophane can be removed by anionic flotation process with high drug consumption and production cost. Due to the synergistic effect between reagents, different combinations of reagents have obvious advantages in the selective separation of collophane, and better separation effect can be obtained than that of using single reagent (Xu et al., 2017). For example, the presence of non-ionic surfactants can enhance the adsorption of ionic surfactants on mineral surfaces (Wu et al., 2022). Therefore, the combination of different types of collectors is an important direction of reagent research and development for collophane separation. Nonylphenol poly-oxyethylene ether can increase the contact angle of sodium oleate droplets on the surface of apatite. And the surface tension test further confirmed that the addition of non-ionic surfactant could improve the activity of the agent significantly, because it had a synergistic effect on the formation of mixed micelles in solution and the decrease of surface tension (Sis and Chander, 2003). Mixing anionic collectors with non-ionic surfactants also improves the selectivity of the flotation process. For example, alkyl hydroxamic acid has a higher selectivity for collophane flotation than conventional fatty acid/fuel oil. When it was further applied to fine crystal apatite flotation using alcohol as collector, the recovery was significantly improved (Wang and Nguyen, 2006).

In this paper, a self-made anionic collector mixed with SDS were used to separate dolomite mineral from collophane. The flotation behaviour of apatite and dolomite with compound collector was researched, and the adsorption characteristics of compound collectors on the surface of different minerals were identified. The aim of this paper is to provide technical and theoretical support for efficient separation of dolomite collophane using reverse flotation process.

2. Materials and methods

2.1. Reagents and materials

2.1.1. Collophane sample

The ore sample used in the test was collected from Yichang, Hubei province. The chemical multi-elemental analysis is shown in Table 1. The collophane is mainly composed of CaO (40.85%) and P₂O₅ (25.70%), and the impurities are mainly composed of SiO₂ and MgO with the content of 15.8% and 5.17%, respectively. The X-ray diffraction spectra (XRD) results of collophane, dolomite and apatite were shown in Fig. 1. The occurrence of phosphate minerals of collophane mainly existed in the form of hydroxyapatite, and the gangue minerals mainly include dolomite, larnite and quartz.

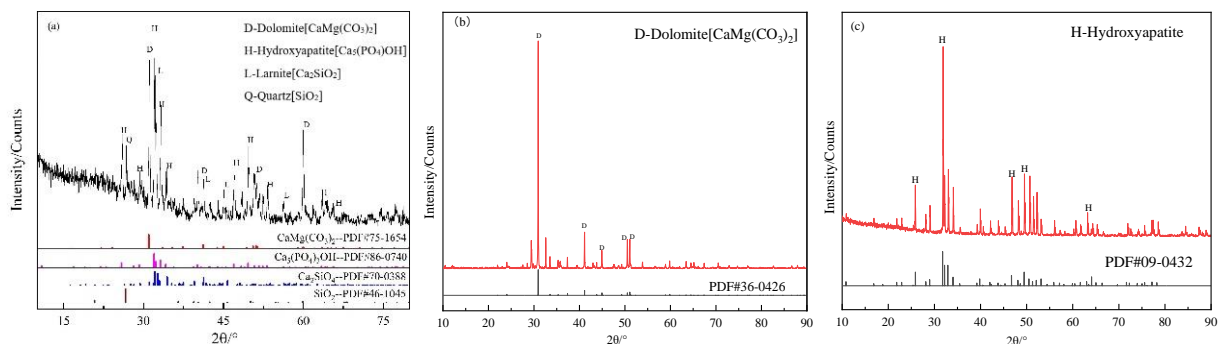


Fig. 1. XRD spectra of collophane (a), dolomite (b) and apatite(c)

Table 1. Chemical analysis of the collophane (wt.%)

Element composition	CaO	P ₂ O ₅	SiO ₂	Al ₂ O ₃	MgO	SO ₃	K ₂ O	Fe ₂ O ₃
wt.%	40.85	25.70	15.83	5.62	5.17	1.94	1.71	1.39
Element composition	BaO	TiO ₂	MnO	SrO	Cr ₂ O ₃	CuO	ZrO ₂	
wt.%	0.29	0.26	0.13	0.07	0.02	0.01	0.005	

2.1.2. Reagents

The fatty acid was prepared by saponification with waste oil generated from catering industry, which was used as collector for dolomite. The reagents such as sulfuric acid, phosphoric acid, sodium dodecyl sulfonate (SDS) used in this work were of analytical reagent grade, which were purchased from Aladdin. Both collector and depressant were diluted in water to a concentration of 2.0%.

2.2. Flotation experiments

The RK-III flotation machine with a volume of 0.5 L was used for the single reverse flotation. 166.7 g collophane was used for each reverse flotation. Then the regulators and collector were successively added into flotation cell. And 2 min stirring was conducted after the addition of each reagent without air inflation. Subsequently, the pulp was aerated with a flowrate of 0.20 L/min, and the sample was floated for 6 min with impeller speed of 2000 r/min. The concentration and tailing were collected for the measurement of P₂O₅ grade and calculation of recovery.

2.3. Surface tension

The surface tension of collector was measured by platinum plate method. In the preparation process, the glassware was cleaned with distilled water, followed by washing with prepared solution several times. Then the platinum plate was flame treated to remove any organic contamination after the cleaning with distilled water. Subsequently, a 20 mL sample of solution was taken to measure the surface tension after the stabilization. The pH was controlled at a certain value with NaOH, phosphoric acid and sulfuric acid.

2.4. FTIR spectra

The Fourier transform infrared (FTIR) spectra of the dolomite before and after collector adsorption were obtained in the range from 400 to 4000 cm⁻¹ with an Avatar 300 from the Thermo Electron Co. Particularly, 0.20 g of dolomite and apatite single minerals were equilibrated with 100 mL of the collector solution at a concentration of 100 mg/L and at a pH of 5.0. The solution was stirred for 2.0 h. Then the samples were washed with distilled water several times, followed by drying at 40°C for 6.0 h. The single minerals were mixed with KBr powder and then pressed into a thin plate for FTIR spectra analysis.

2.5. Zeta potential

Zeta potential analysis was performed with a Zetasizer instrument from Malvern (Nano-ZS90, Malvern, UK). 0.1 g of -38 μm apatite or dolomite was firstly conditioned with 100 mL of solution for 20 min stirring at 250 rpm. And the pH was adjusted to a certain value with NaOH, phosphoric acid and sulfuric acid. Then a certain amount of compound collector was added into the solution. After conditioning for another 10 min without stirring. 5.0 mL of supernatant liquid was transferred into cuvettes to measure zeta potential at room temperature.

3. Results and discussion

3.1. Flotation behaviors of collophane using single collector

3.1.1. Grinding fineness

Grinding fineness has significant influence on flotation performance. The apatite is finely disseminated and intimately associated with the dolomite. It is necessary to liberate them by grinding to certain fineness (Zeng and Yang, 2022). The effect of grinding fineness on reverse flotation of collophane was shown in Fig.2. With the grinding time increasing from 6 min to 10 min, the mass fraction less than 0.074 mm gradually increased from 64% to 89.8%. And the P_2O_5 grade of concentrate rises to the maximum value of 26.28% with a slight P_2O_5 recovery decrease. Then the P_2O_5 grade and P_2O_5 recovery of concentrate both decrease with the increase of grinding fineness. The valuable minerals and gangue minerals are not dissociated enough with a small grinding fineness, resulting in a noneffective flotation separation. However, when the grinding is too fine, the minerals can interact with agents without selectivity, making the separation more difficult. As shown in Fig.2, the P_2O_5 grade and P_2O_5 recovery of tailing both increase with the increase of grinding time.

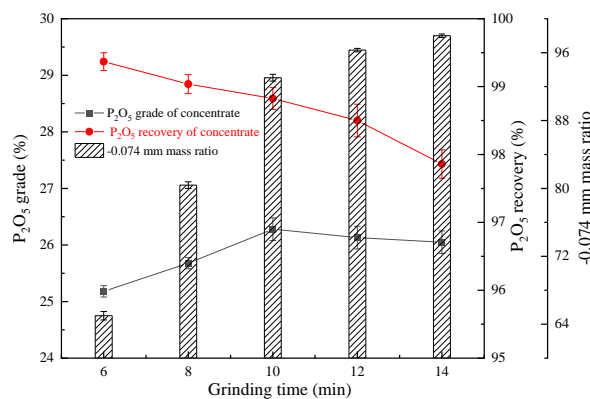


Fig. 2. Effect of grinding fineness on reverse flotation of collophane

3.1.3. Dosage of H_2SO_4

Sulfuric acid is the commonly used as pH regulator in reverse flotation of collophane (He et al., 2018). The floatability of apatite was selectively depressed in acidic system, resulting in the flotation of dolomite for the separation of apatite and dolomite. Under the conditions of grinding fineness of -0.074 mm mass fraction accounted for 89.8%, collector dosage of 1.0 kg/t and flotation time of 6 min, the influence of sulfuric acid dosage on reverse flotation performance of collophane was presented in Fig. 3.

As shown in Fig 3, the P_2O_5 grade of concentrate gradually increases with increasing sulfuric acid dosage, and the P_2O_5 recovery slightly decreases. When the dosage of sulfuric acid is below 6.0 kg/t, the depression effect of apatite is not obvious at a higher pH. And the flotation performance of dolomite is poor, resulting in the lower grade of concentrate. The pH of slurry decreases with the increase of sulfuric acid dosage, and the CO_3^{2-} on the surface of carbonate minerals is preferentially dissolved. In particular, a large amount of Ca^{2+} is dissolved from dolomite at pH=5.0, and it can increase the activated cation (Mg^{2+}) density on the mineral surface (Han and Zhong, 1998). And the flotation performance of dolomite can be increased, thus improving the grade of concentrate. When the dosage of sulfuric acid is 12.0 kg/t, the P_2O_5 grade increases to 26.25%, and the recovery is 98.85%. With the increasing dosage of sulfuric acid, the P_2O_5 grade and recovery of concentrate can't be improved. Too much sulfuric acid will make phosphate rock dissolve and scale, which will affect the flotation efficiency (Li et al., 2022). Therefore, the dosage of sulfuric acid was chosen to be 12.0 kg/t.

3.1.4. Dosage of H_3PO_4

Phosphate is the localized ion of apatite, and phosphoric acid is often used as the depressant in collophane reverse flotation (Wei and Zhang, 2022). It can be adsorbed on the exposed Ca^{2+} site on the surface of apatite to hinder the adsorption of collector on the surface of apatite (Liu et al., 2017).

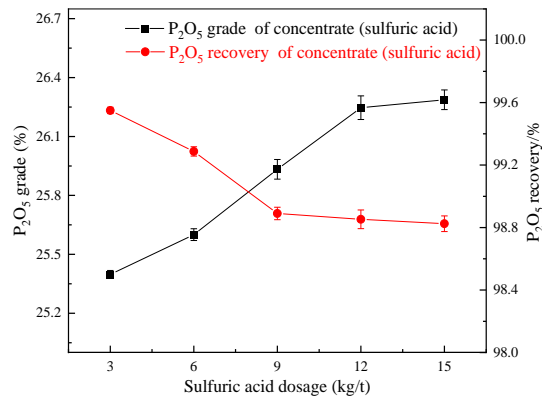


Fig. 3. Effect of sulfuric acid dosage on collophane flotation

Phosphoric acid is often mixed with sulfuric acid as the depressant in reverse flotation of collophane (Al-fariss and El-nagdy, 2013). Under the conditions of grinding fineness of -0.074 mm mass fraction accounted for 89.8%, sodium stearate dosage of 1.0 kg/t, sulfuric acid dosage of 12.0 kg/t and flotation time of 6 min, the effect of phosphoric acid dosage on reverse flotation performance of collophane was shown in Fig. 4.

It can be seen from Fig. 4 that the P₂O₅ grade of concentrate is significantly improved after the addition of phosphoric acid. With the increase of phosphoric acid dosage, the P₂O₅ grade increases and the recovery decreases, indicating that phosphoric acid can effectively depress the flotation of apatite. When the dosage of phosphoric acid is 3.0 kg/t, the highest P₂O₅ grade of concentrate is 27.14% with the recovery of 97.02%. Compared with the dosage of 4.0 kg/t, the recovery has no significant improvement. Therefore, the dosage of phosphoric acid was determined as 3.0 kg/t.

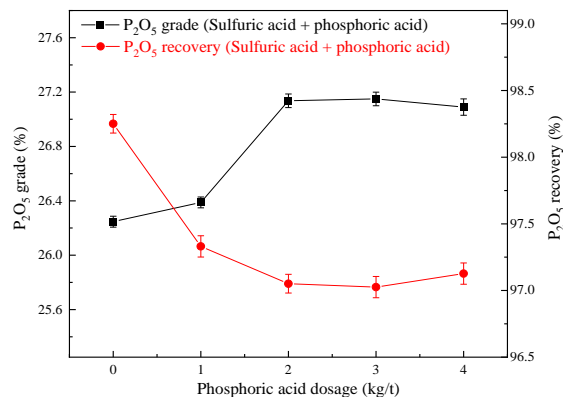


Fig. 4. Effect of phosphoric acid dosage on collophane flotation

3.2. Flotation behaviors of collophane using compound collector

3.2.1. Compound collector reagents

The reverse flotation effect of collophane was not obvious only using fatty acid as collector. However, the flotation performance can be significantly improved under the effect of compound collector (Huang and Liu, 2021). SDS has a good solubilization and foaming ability, and it can be used as an effective collector for carbonate minerals mainly containing dolomite. However, under conditions of large air charge and fast stirring speed, the flotation foam is easy to be viscous, which affects the flotation effect (Zhao et al., 2016; Sun and Liu, 2017; Xie et al., 2021). The combination of fatty acid and SDS can enhance the separation of dolomite from collophane.

Under the conditions of grinding fineness of -0.074 mm mass fraction accounted for 89.8%, sulfuric acid dosage of 12.0 kg/t, phosphoric acid dosage of 3.0 kg/t, collector dosage of 1.0 kg/t and flotation time of 6 min, the effect of the compound collector on reverse flotation performance was shown in Fig. 5.

As shown in Fig. 5, the P₂O₅ grade of concentrate is significantly increased with the addition of SDS, indicating that the dolomite can be effectively removed by the compound collector. With the increase

of the compound proportion of SDS, the P_2O_5 grade of concentrate increases first and then decreases. And the P_2O_5 recovery of concentrate decreases first and then increases. After the combination of collector, the surface tension of solution decreases, the interaction between dolomite and compound collector is promoted by the synergistic effect between molecules. With the further increase of SDS, the interaction probability between fatty acid and SDS decreases, leading to dolomite remaining in phosphorus concentrate. Moreover, apatite is easily entrained into the foam product with the increase of SDS, resulting in the decrease of the P_2O_5 grade and the recovery. When the proportion of SDS is 3.0%, the P_2O_5 grade of concentrate of single reverse flotation is 29.94%, and the recovery is 90.85%.

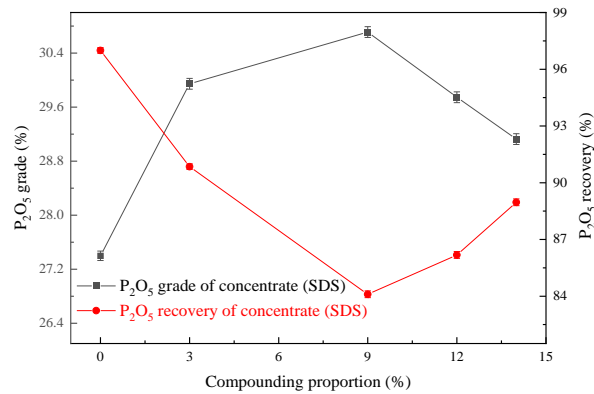


Fig. 5. Effect of compound collector on reverse flotation

3.2.2. Collector dosage

Under the conditions of grinding fineness of -0.074 mm mass fraction account for 89.8%, sulfuric acid dosage of 12.0 kg/t, phosphoric acid dosage of 3.0 kg/t, proportion of SDS is 3.0%, flotation time of 6 min, the influence of compound collector dosage on colophane single reverse flotation was shown in Fig. 6. When the collector dosage is below 0.75 kg/t, due to the low collector concentration in the flotation system, the interaction between collector and dolomite is weak. The collector adsorbed to dolomite minerals are not enough to adhering to the bubbles, so the grade of phosphorus concentrate is maintained at a low level. When the collector dosage increased to 1.0 kg/t, the P_2O_5 grade of concentrate increased significantly to 29.94% with the recovery of 90.85%. When the collector dosage is above 1.0 kg/t, the selectivity of the collector reduced, resulting in the gradual decline of the P_2O_5 grade and a slight increase of P_2O_5 recovery. Therefore, the best flotation effect was achieved at the compound collector dosage of 1.0 kg/t.

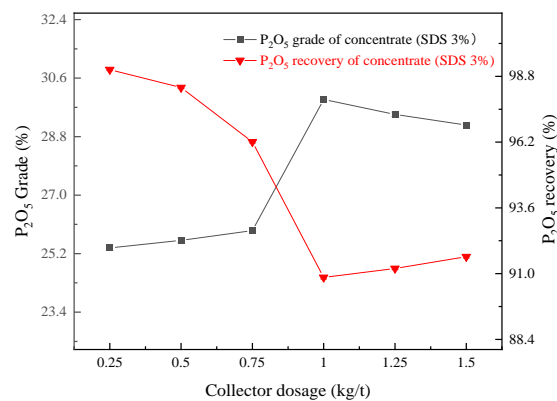


Fig. 6. Effect of collector dosage on reverse flotation

3.3. Intensified flotation mechanism of compound collector

3.3.1. Surface tension

The automatic surface tension tester was conducted to measure the surface tension of single collector solution and compound collector solution at different concentrations. The results were shown in Fig. 7.

With the increase of collector concentration, the surface tension of the solution gradually decreases. And the surface tension of the compound collector solution is significantly lower than that of the single collector solution, indicating that the compound collector is more conducive to improving the stability of flotation foam and improving the reverse flotation index of colophane. The results were consistent with the experimental results of flotation with compound collector in Fig.6. In addition, the surface tension of two collector solutions did not decrease significantly when the concentration was higher than 125 mg/L. Because the concentration has exceeded the critical micelle concentration, and the increasing concentration has little effect on the surface tension of the solution (Li et al., 2022).

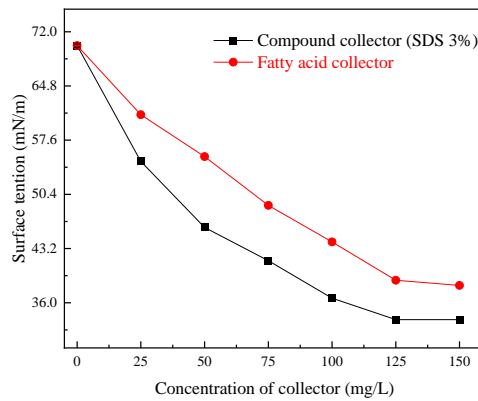


Fig. 7. Relationship between surface tension and concentration of pharmaceutical solution

3.3.2 Zeta potential analysis

The Zeta potentials of apatite and dolomite in pure water, single collector solution and compound collector solution were shown in Fig. 8 (a) and (b). With the increase of pH, the adsorption of OH^- on exposed Ca^{2+} and Mg^{2+} sites on mineral surface increases. The Zeta potential of dolomite and apatite gradually shifts to negative values with the increase of pH. Fig. 8 (a) showed that the isoelectric point of apatite in pure water is about 3.35. And the Zeta potential decreases after the addition of collector due to the co-adsorption of H_2PO_4^- hydrolyzed by phosphoric acid and collectors. (Zhang and Xu, 2020; Zhang and Zhang, 2022). Fig. 8 (b) showed that the isoelectric point of dolomite in distilled water is about 3.00, and the Zeta potential of the compound collector is significantly lower than that of the single collector. It indicates that the compound collector is easier to be adsorbed on the surface of dolomite, which enhances the separation effect of dolomite in the reverse flotation. According to Fig. 8, the surfaces of apatite and dolomite are both negatively charged at $\text{pH}=5.0$. And the anionic collector can not be adsorbed on the mineral surface by electrostatic attraction (Cao et al., 2015). The adsorption of the fatty acid collector on mineral surface is physical adsorption and chemical adsorption under the conditions of weak alkaline or acid (Gong et al., 2017). Therefore, it can be speculated that the fatty acid collector and the compound collector are adsorbed on the mineral surface by chemical or physical adsorption.

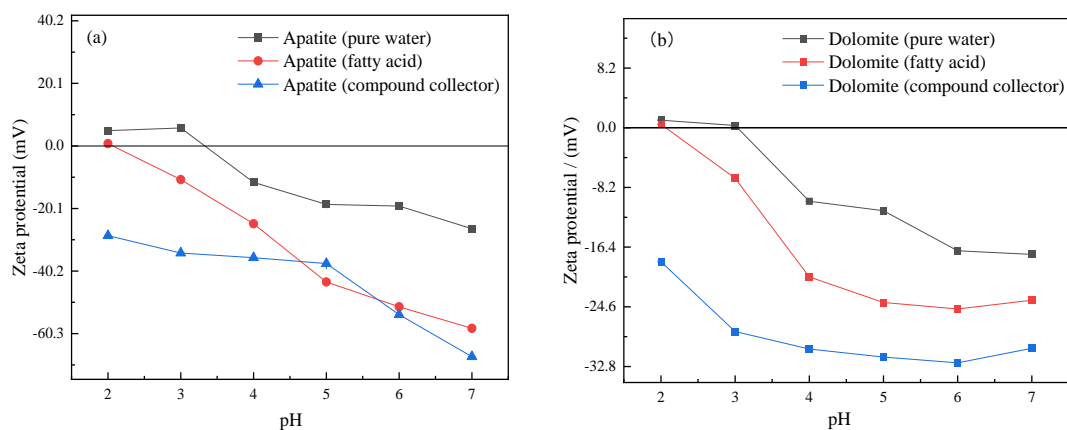


Fig. 8. Effect of pH on Zeta potential of (a) apatite, and (b) dolomite in various reagents

3.3.3. FTIR spectra analysis

Fourier transform infrared spectroscopy (FTIR) was carried out to analyze the changes of surface functional groups of apatite and dolomite before and after interaction with reagents, and the FTIR spectra were shown in Fig. 9. As shown in Fig.9(a), the 3455, 2920, 2850 and 1556 cm^{-1} are the -OH stretching vibration absorption peak, -CH₂ antisymmetric stretching vibration absorption peak, -CH₂ symmetric stretching vibration peak, and C=O stretching vibration absorption peak, respectively (Yun and Zhu, 2022). In Fig.9(b), the curves marked 1 to 4 presented the FTIR spectra of apatite before and after the interaction with single collector and compound collector. 3450.62, 1042.91 and 603.27 cm^{-1} are the -OH stretching vibration absorption peak, PO₄³⁻ antisymmetric stretching vibration absorption peak and PO₄³⁻ asymmetric variable angle vibration absorption peak of apatite, respectively. 1640 cm^{-1} is the absorption peak of -OH in water molecule, it may be caused by water absorption by the sample powder in contact with air (Liu et al., 2022). Due to the sample preparation, the absorption intensity of apatite after the interaction with single collector becomes weak and increase slightly with the compound collector. The other characteristic absorption peaks do not shift significantly after the interaction between the compound collector and apatite. It indicates that the compound collector only weakly adsorbed on the surface of apatite. According to Fig.9(c), the curves marked 1 to 4 presented the FTIR spectra of dolomite before and after the interaction with single collector and compound collector. The absorption peak around 2919 cm^{-1} appeared after the interaction of dolomite with fatty acid and compound collector, which are corresponded to the anti-symmetric stretching vibration peak of -CH₂. It demonstrates that the fatty acid is effectively adsorbed on the surface of dolomite, which is conducive to the reverse flotation of dolomite. The curve 3 showed that only a slight -CH₂ stretching vibration peak at 2919.75 cm^{-1} was found after the interaction of dolomite and SDS. Therefore, the dolomite can be selectively separated by co-adsorption of the fatty acid and SDS in reverse flotation.

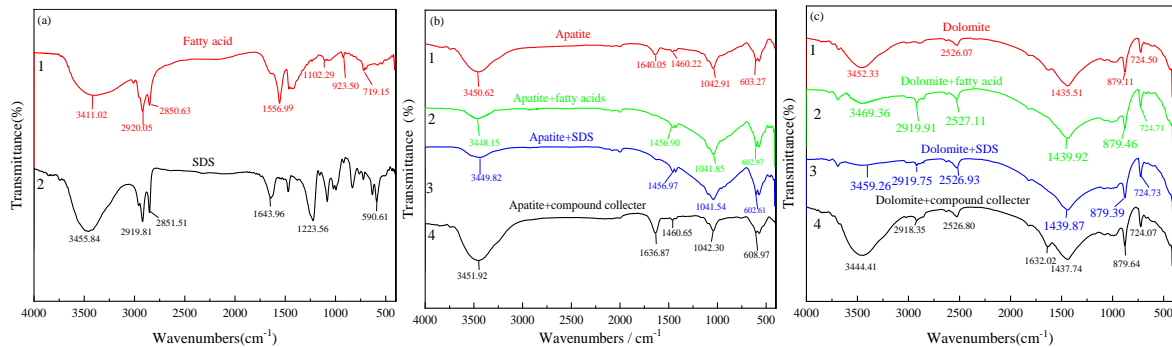


Fig. 9. FT-IR spectra of collectors(a), apatite (b) and dolomite (c) before and after the interaction with collectors

4. Conclusions

- (1) The removal of dolomite was not effective using single fatty acid collector. The P₂O₅ grade of collophane could not be improved significantly. Under the conditions of grinding fineness -0.074 mm accounted for 89.8%, sulfuric acid dosage 12.0 kg/t, phosphoric acid dosage 3.0 kg/t and fatty acid collector dosage 1.0 kg/t, the P₂O₅ grade of phosphorus concentrate of 27.14% and the recovery of 97.02% were obtained by reverse flotation using single fatty acid collector.
- (2) The flotation performance of dolomite was enhanced by the combination of SDS and fatty acid. Under the conditions of the compound proportion of SDS of 3.0% and the compound collector of 1.0 kg/t, the P₂O₅ grade of phosphorus concentrate was 29.94% and the recovery was 90.85%. The compound collector could improve the P₂O₅ grade of phosphorus concentrate, indicating that SDS and fatty acid chemical molecules have synergistic effect to improve the collecting performance of dolomite.
- (3) The surface tension of the solution under the action of compound collector is less than that of single collector, which is conducive to improve the stability of flotation foam. The Zeta potential of pure minerals shifted to negative value after the addition of collector, and the negative Zeta potential shift of compound collector was greater than that of single collector, revealing that the compound collector was more easily adsorbed on mineral surface. The FT-IR analysis results showed that the

characteristic peaks of compound collector were detected on dolomite, but not on apatite. It reveals that the compound collector can be selectively adsorbed on dolomite, enhancing the removal effect of dolomite.

Acknowledgments

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