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Study on the mechanism of action of various metal ions on the surface of monazite

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Abstract: By studying the various metal ions released from the grinding process, and conducting the flotation test of monazite and symbiotic ore, using zeta potential measurements and FTIR infrared spectrum analysis methods, the influence of metal ions on the surface of monazite and the mechanism of action are obtained. The results showed that the main metal ions in the flotation environment of monazite are Ca²⁺, Ba²⁺, and Fe³⁺. When the pulp pH value was 8, Ca²⁺, Fe³⁺, and Ba²⁺ concentration was 3×10⁻⁴ mol/dm³, 3×10⁻⁵ mol/dm³, and was 2×10⁻⁴ mol/dm³ respectively, and OHA collector dosage was 5×10⁻⁴ mol/dm³, while the flotation recoveries of monazite, calcite. And fluorite was above 95 %, 40.43 %, and below 45.32 %, and the recovery rate of barite and bastnaesite in the presence of Ca²⁺ and Fe³⁺ is below 54.32 % and 38.68 %, respectively and the effective separation of monazite and symbiotic ore was obtained. The zeta potential measurements showed that when only metal ions are added to monazite, monazite (IEP) shifts to the right, and the negative charge on the surface of monazite decreases, which may be due to the positive charge of metal ions adsorbed on the surface of monazite, increasing the positive charge on the surface of monazite. When Ca²⁺, Ba²⁺, Fe³⁺, and OHA were added to the monazite pulp, the IEP of monazite moved to the left, and the negative charge of monazite shifted to the positive direction as a whole, indicating that Ca²⁺, Ba²⁺, and Fe³⁺ were adsorbed on the surface of monazite and increase the positive charge on the surface of monazite. When the pH value was $7 \sim 10$, the surface of monazite was the same as that before and after the action of the agent, and both were negatively charged. At this time, OHA can still adsorb on the calcite, that was, the chemical affinity overcomes the same electric repulsion and adsorbs, so that the potential of the monazite after the action is reduced, which is chemical adsorption. The FTIR showed that when the metal ions Ca²⁺, Ba²⁺, and Fe³⁺ are added to activate the surface of monazite, the peak area of the organic functional group-CH₂-CH₃ at 3000-2800 cm⁻¹ was enhanced, and a new peak appears at 1382 cm⁻¹. The reason is that the N-O-H functional group of OHA collector was adsorbed on the surface of monazite, and a red shift (44 cm⁻¹) occurs, and a new peak appears at 1454 cm⁻¹. The reason is that the C-N functional group of OHA collector is adsorbed on the surface of monazite. The lone electron pair of the nitrogen atom of the amide group in the OHA forms chelates with Ca²⁺, Ba²⁺, and Fe³⁺ atoms, which further increases the adsorption amount of OHA. This showed that the adsorption of monazite surface by metal ions provides active sites for the adsorption of OHA collectors. These phenomena indicate that the adsorption capacity of OHA on the surface of monazite activated by Ca2+, Ba2+, and Fe3+ is enhanced and a stable five-membered ring metal chelate is formed. Combined with flotation results and zeta potential data, infrared spectroscopy analysis, the adsorption capacity of Ca²⁺, Ba²⁺, and Fe³⁺ activated monazite surface: Fe³⁺>Ca²⁺>Ba²⁺.

Keywords: flotation, monazite, octyl hydroxamic acid, associated minerals, metal ions

1. Introduction

Common symbiotic minerals include fluorite, calcite, iron minerals, barite, dolomite, feldspar, apatite, bastnaesite, etc. (Qin et al., 2021), and these minerals often affect the flotation behaviour results of monazite.

At present, some researchers believe that metal ions are not conducive to the flotation of monazite, and some believe that metal ions are beneficial to the flotation of monazite. For example, Chelgani et al. (2015) and Cheng et al. (1993) studied the flotation of bastnaesite using sodium oleate to study the effect of dissolved ions in gangue minerals such as barite and fluorite. It was shown that Ca^{2+} , and F^- dissolved Ba2+ from gangue minerals reduce the recovery of bastnaesite, which may be due to the formation of insoluble oleate products with metal ions. In the presence of different ions dissolved in calcite, the flotation performance of monazite was studied by using hydroxamic acid collector (Zhang and Honaker, 2018; Zhang et al., 2017). It was found that due to the preferential adsorption of calcium hydroxide on the surface of monazite, Ca²⁺ in the solution was not conducive to the recovery of monazite, resulting in a low flotation recovery. Espiritu et al. (2019) also used DFT simulation to study the effect of dissolved ions in dolomite on the flotation of monazite, and found that Ca2+ and Mg2+ ions reduced the floatability of monazite due to the adsorption of calcium carbonate or magnesium hydroxide on monazite and the precipitation of calcium or magnesium oleate. Zheng et al. (2021) and Zeng et al. (2022) found that adding a small amount of Fe³⁺ to the pulp before SOH can significantly improve the flotation of fine monazite. This is because the addition of Fe³⁺ promotes the adsorption of SOH and greatly improves the hydrophobicity of the surface of monazite.

The composition of metal ions mentioned in the literature affects the flotation process (Ai et al., 2017; Gao et al., 2017, Jin et al., 2015). In the flotation process of monazite, Ce³⁺ will not release into the flotation environment because monazite is an insoluble mineral, while fluorite, calcite, barite, iron mineral, dolomite, feldspar, and apatite are semi-soluble minerals. Symbiotic minerals will release metal ions in the solution, so Fe³⁺, Ca²⁺, Mg²⁺, Ba²⁺, Na⁺, and so on are the most important in the flotation solution environment of monazite (Tiam et al., 2018; Tang et al., 2023; Yu et al., 2015). These ions will affect the change of flotation chemical environment (Cao et al., 2014). Therefore, it is necessary to analyze the activation or inhibition of these ions on the flotation of monazite and symbiotic minerals, and to obtain the available metal ions, and it is necessary to maintain the appropriate proportion of metal ion concentration. The effect of the concentration and proportion of metal ions on the flotation process is very important (Dong et al., 2019; Wang, 2022).

In the flotation process of monazite, the collector with good collecting and selectivity is selected as the main flotation reagent, which can improve the flotation effect. Therefore, in this study octyl hydroxamic acid (OHA) (Chen et al., 2017) was chosen as a collector to investigate the release behavior of metal ions in monazite and symbiotic minerals, as well as the influence of metal ions on the flotation of monazite and symbiotic minerals. The mechanism of metal ions on the surface of monazite was analyzed by zeta potential measurements and FTIR infrared spectroscopy analysis. In the flotation of monazite and symbiotic minerals, reasonable flotation reagents and conditions were obtained to ensure the appropriate proportion of metal ion concentration, and control and adjust it, hence so as to improve the separation and flotation effect of monazite.

2. Materials and methods

2.1. Materials

The raw ore of monazite was selected from Baotou Bayan Obo (China) rare earth ore with a particle size of -3mm. The main rare earth mineral in the rare earth ore was monazite, and the symbiotic minerals included bastnaesite, fluorite, calcite, barite, magnetite, potassium slate, and albite. XRD of raw ore sample is shown in Fig. 1. Pure mineral extraction: monazite is derived from Baotou Bayan Obo ore (China) (further purification of mineral samples by shaking table separation, and then hand-selected purification under electron microscope, with a purity of 95%), bastnaesite is derived from Weishan ore (China) (flotation concentrate is removed by ultrasonic water washing, with a purity of 95%), fluorite (hand-selected, with a purity of 99%), barite (hand-selected, with a purity of 98.8%), calcite (hand-selected, with a purity of 99.5 %) is derived from Baotou Bayan Obo ore (China). XRD of the pure mineral sample is shown in Fig. 2 to Fig. 6. The XRD of pure minerals showed that the diffraction peak was single and there is no impurity peak, which meets the test requirements.

Pharmaceutical sources: The pH regulator for the analysis of pure HCl, NaOH, from the State Pharmaceutical Group (China). Analysis of metal ions selected pure FeCl₃, CaCl₂, BaCl₂, purity 99.9 %,

from Lianyungang (China). Octyl hydroxamic acid (OHA) was used as a collector in the flotation test, with a purity of 99.9 %, from Shanghai Yuanye Biotechnology Co., Ltd. (China). Methyl isobutyl methanol (MIBC) was used as a frother, with a purity of 99 %, from Shanghai Yuanye Biotechnology Co., Ltd. (China). Type I deionized DI water prepared by mil-9 water system was used in all experiments, and the resistivity was $18.2 \text{ M}\Omega \cdot \text{m}$. The manufacturer was Sigma Odrich (Shanghai) Trading Co., Ltd.



Fig. 2. XRD patterns of pure minerals (a: Monazite, b: Bastnaesite, c: Fluorite, d: Calcite, e: Barite)

2.2. Methods

2.2.1. ICP-OES test method

Avio 200 inductively coupled plasma spectrometer (ICP-OES, PE, USA) was used to determine the concentration of metal ions in the filtrate after the grinding. The wavelength of Ca²⁺ is 393.37 λ / nm, the wavelength of Fe³⁺ is 238.20 λ / nm, the wavelength of Ba²⁺ is 214.44 λ / nm, the wavelength of Al³⁺ is 396.15 λ / nm, the wavelength of Mg²⁺ is 279.55 λ / nm, the wavelength of La³⁺ is 333.74 λ / nm, and the wavelength of Ce³⁺ is 367.47 λ / nm. Before the measurement, the calibration curve of the metal element to be measured is 0.99999, and the filtrate is tested. The analysis was repeated 3 times, and the data were analyzed by mean value.

2.2.2. Flotation experiments

Flotation experiments were carried out in the flotation machine of the Wuhan Lock hanging tank, and the impeller speed was rpm 1g pure monazite and symbiotic ore were mixed with 40 cm³ DI water at 2000 rpm. The pH value of the slurry was adjusted by 2 % NaOH and 2 % HCl solutions. Metal ions, collector OHA (5×10⁻⁴ mol/dm³) and methyl isobutyl methanol (30 ppm) were added in turn, adjusted for 5min, 5min, and 1min, respectively, and floated for 5min.Subsequently, the concentrate and tailings samples were dried and weighed. The flotation recovery rate was calculated according to Eq (1), and three repeated tests were carried out under the same conditions.

$$\varepsilon = \frac{m_1}{m_1 + m_2} \tag{1}$$

where: ϵ is the flotation recovery %; m_1 and m_2 are the amounts of the concentrate; and the tailings, respectively, in the tank.

2.2.3. Zeta potential measurements

The zeta potential measurements for the samples were carried out using a zeta potential analyzer (ZetaPlus, Brookhaven Instruments, USA). All the measured background electrolytes were (KCI), (Purity 99.9 %, made by Tianjin Zhiyuan Chemical Reagent Co., Ltd., China) with a concentration of 0.1 mol/dm³. The particle size of the tested mineral sample was less than 5 µm. The effect of metal ions on the surface potential of mineral suspension under different pH values was studied by suspending 50 mg of pure monazite and symbiotic mineral particles in 50 cm³ OHA solution. After adjusting the pH value, stirring for 5 min and precipitating for 3 min. After the precipitation was completed, the supernatant was collected for determination. Each measurement was repeated three times and the average value was reported.

2.2.4. FTIR spectroscopy analysis

The Fourier transform infrared spectrometer FTIR was used to analyze the infrared spectra of minerals before and after the action of the agent. The KBr tablet was used with a measurement range of $4000 \sim 400$ cm⁻¹. Preparation of infrared analysis samples: 1g ore sample was ground to less than 5µm by agate, and 40 cm³ DI water was added to the flotation tank. According to the flotation test conditions, the pH value was stirred, added, and adjusted to 8. After full action, the solid-liquid separation was carried out, and the DI water with the same pH value was used to wash 3 times. The obtained ore sample was dried at 40 °C for infrared spectroscopy.

3. Results and discussion

3.1. Determination of metal ion components in pulp

The RK / ZQM type 150×50 ball mill was selected as the mill, the inner lining was the steel liner, the steel ball medium was selected, the grinding concentration is 65 %, the rotation speed was 1500 rpm, and the 500 g monazite ore ball mill with a particle size of -3 mm was used for the grinding for 8 min. And the grinding water is deionized water. After the solid-liquid separation of the slurry obtained by the grinding with a centrifuge, the type and concentration of metal ions in the supernatant were tested by ICP-OES, and the results are shown presented in table 1.

As seen from table 1 that the main metals in the raw ore were Ca²⁺, Ba²⁺, Mg²⁺, Al³⁺, Fe³⁺, K⁺, and Na⁺. The content of Ca²⁺ was the highest, reaching 60.37 mg/dm³. This was because calcite, dolomite, and fluorite released Ca²⁺ ions, while the content of Mg²⁺ was 6.13 mg/dm³, which released from dolomite; the content of Ba²⁺ was higher, which mainly released from barite during the grinding process, and the content was 27.15 mg/dm³. The contents of Fe³⁺, K⁺, and Na⁺ were low, which are released from magnetite, potassium and albite, which were 2.31 mg/dm³, 2.12 mg/dm³, and 2.08 mg/dm³, respectively. The contents of rare earth ions La³⁺, Ce³⁺, and Nd³⁺ were extremely low, all of which were 0.01 mg/dm³.

Ion species	Ca ²⁺	Mg ²⁺	Fe ³⁺	Ba ²⁺	Al ³⁺
Content (mg/dm ³)	60.37	6.13	2.31	27.15	0.34
Ion species	K+	Na ⁺	La ³⁺	Ce ³⁺	Nd ³⁺
Content (mg/dm ³)	2.12	2.08	< 0.01	< 0.01	< 0.01

Table 1. Types and contents of metal ions in raw ore pulp

It can be concluded that there are mainly Ca²⁺, Ba²⁺, Mg²⁺, Al³⁺, and Fe³⁺ ions in the pulp. Therefore, the metal ions released from monazite, fluorite, calcite, barite, magnetite, and bastnaesite were mainly investigated, and the influence of metal ions on the flotation of monazite and symbiotic ore was also investigated.

3.2. Release behavior of metal ions in grinding process

The steel ball medium was selected, the grinding concentration was 65 %, and the rotation speed was 1500 rpm. Pure monazite, fluorite, calcite, barite, and bastnaesite samples with a particle size of -0.074 mm were taken and added to the ball mill grinding. After the solid-liquid separation of the slurry obtained by filtering and grinding with a filter, the type and concentration of metal ions in the filtrate were tested by ICP-OES. The release behavior of metal ions in the lattice was studied with the increase of grinding time.



Fig. 3. Effect of grinding time on the release of metal ions from single minerals

It can be analyzed from Fig. 3 that the content of Ca^{2+} released from calcite increased rapidly to 24.3 mg/dm³ at 5min of grinding. With the increase of grinding time, the content of Ca^{2+} increased slowly, reaching 43.25 mg/dm³ at 35 min. When the monazite and symbiotic ore in the grinding fluorite with the increase in time, Ca^{2+} content increased slowly, when 35 min reached 25.67 mg/dm³, compared with calcite release of Ca^{2+} content, fluorite release of Ca^{2+} content was low. This was the fracture of metal ion bonds, the size of lattice energy, and the hydration energy of ions in the grinding process of lattice ions on the surface of minerals (Zheng, 2020). Therefore, in the aqueous solution during the grinding process, Ca^{2+} in calcite was easier to integrate into the aqueous solution than calcite and fluorite.

With the increase of grinding time, the content of Ba2+ increased gradually. At 10 min, the content of

Ba²⁺ was 30.31 mg/dm³. After that, the content of Ba²⁺ in the filtrate reached 38.9 mg/dm³ with the increase of grinding time. With the decrease of grinding particle size, the dissolution of Ba²⁺ on the surface of the lattice also depends on the size of the lattice energy, that is, Ba-O (the oxygen atom inSO²⁻₄), and the electronegativity of O in SO²⁻₄ group is further reduced by the common electron pair of S atoms, resulting in the relatively weak attraction of O atoms in SO²⁻₄ of barite to the electrons in Ba atoms, so in the aqueous solution during the grinding process, Ba²⁺ in barite is easier to integrate into the aqueous solution (Nie, 2022).

It can be analyzed from Fig. 3 that the content of Fe^{3+} increased with the increase of grinding time, and reached 10.24 mg/dm³ at 35 min. The release of Fe^{3+} content is due to the exposure of iron atoms in the aqueous solution during the grinding process of magnetite, which is released. Another reason for the increase of Fe^{3+} content is that in the grinding process, the medium iron ball may pollute the filtrate, and there is a part of Fe^{3+} content, which is inevitable in mineral processing. It can be seen from Fig. 3 that the release of Ce^{3+} increases with the increase of grinding time, but the increase is extremely slow. Whether it is bastnaesite or monazite, the release of Ce^{3+} content is less than 1 mg/dm³ at 35 min, indicating that monazite and bastnaesite are insoluble, and Ce^{3+} is also difficult to release into an aqueous solution.

In summary, during the grinding process, the metal ions released from monazite and symbiotic ore increase with time, but the content of Ca^{2+} released from calcite is the most, followed by fluorite, and the content of Ba^{2+} released from barite is also larger. Magnetite can also release Fe^{3+} into the aqueous solution, and these ions change the flotation environment. However, the Ce^{3+} in monazite and bastnaesite is difficult to release into the solution. Therefore, in the flotation environment of monazite, there are mainly Ca^{2+} , Ba^{2+} , and Fe^{3+} .

3.3. Effect of metal ions on the flotation of monazite and paragenetic ore

3.3.1. Effect of Ca²⁺ on flotation of monazite and associated minerals

The experimental conditions of Fig. 4 are as follows: OHA concentration was $5 \times 10^4 \text{mol/dm}^3$, Ca²⁺ dosage was $1 \times 10^4 \text{mol/dm}^3$, MIBC dosage is 30 ppm, pH value of the suspension is adjusted by 2 % NaOH and 2 % HCl. It shows that when pH < 8, the flotation recovery of monazite increases with the increase of pH, and the recovery rate reaches the maximum at about pH = 8, which is 87.29 %. After that, the recovery decreased rapidly with the increase of pH, and the recovery rate reaches the maximum at about pH = 8, which is 87.29 %. After that, the recovery decreased rapidly with the increase of pH, and the recovery rate reaches the maximum at about pH = 9, which is 35.27 %. After that, the recovery rate decreased rapidly with the increase of pH, and the recovery of calcite increases of pH, and the recovery rate was only 15.28 % at pH = 12. The flotation recovery of calcite increases with the increase of pH value, and the recovery rate reaches the maximum at about pH = 10, which is 61.21 %. After that, the recovery rate decreased rapidly with the increase of pH, and the recovery rate was only 32.14 % at pH = 12. The flotation recovery of barite increases with the increase of pH value, and the recovery rate reaches the maximum at about pH = 8, which is 62.43 %. After that,



Fig. 4. Effect of Ca²⁺ on the flotation of monazite and gangue minerals as a function of pH

the recovery rate decreased rapidly with the increase of pH, and the recovery rate was only 20.17 % at pH = 12. The flotation recovery rate of bastnaesite increases with the increase of pH value, and the recovery rate reaches the maximum at pH = 8, which is 61.23 %. After that, the recovery rate decreased rapidly with the increase of pH value, and the recovery rate was only 32.74 % at pH = 12. It shows that Ca²⁺ has different effects on different minerals, and the effects of different pH values are also different. This may be related to the fact that Ca²⁺ hydrolyzes different dominant components at different pH values, and different dominant components have adsorption effects on different minerals, thus affecting OHA adsorption minerals. Under the same conditions, when the pH is equal to 8, the recovery rate of monazite is 87.29 %, while the recovery rates of fluorite, calcite, barite, and bastnaesite are 31.78 %, 52.73 %, 62.43 %, and 61.23 %, respectively. The dominant component of hydrolysis of Ca²⁺ at pH = 8 activates the surface of monazite, which is beneficial to the flotation of monazite by OHA.

The test conditions of Fig 5: OHA concentration was 5×10^{-4} mol/dm³, pulp pH value is 8, MIBC dosage is 30 ppm, adding different concentrations of Ca²⁺ concentration to investigate the flotation effect of monazite and gangue minerals. It can be seen from the diagram that with the increase of Ca²⁺ concentration, the flotation of monazite has a certain activation effect. When the concentration of Ca²⁺ was 3×10^{-4} mol/dm³, the recovery rate of monazite is 96.48 %; the recovery of fluorite, calcite, barite and bastnaesite decreased obviously, which were 8.08, 11.94, 18.82, and 16.57 percentage points, respectively. When the concentration of Ca²⁺ was 4×10^{-4} mol/dm³, it decreased by 13.1 percentage points. It can be concluded from the above data that a certain concentration of Ca²⁺ will activate monazite, and excessive Ca²⁺ may consume the amount of OHA, thus reducing the recovery rate of monazite.



Fig. 5. Effect of Ca²⁺ concentration on flotation of monazite and gangue minerals

The concentration of Ca^{2+} has a great influence on the flotation of fluorite, calcite, barite, and bastnaesite. For the flotation of fluorite, high concentration of Ca^{2+} will inhibit its flotation effect, because the hydroxyl group on the surface of fluorite can form a chemical bond with Ca^{2+} , forming a calciumfluorite complex that is not easy to float (Wang et al., 2022; Wang et al., 2023), and low concentration of Ca^{2+} will not affect the flotation effect. For the flotation of calcite, Ca^{2+} concentration has little effect on its flotation performance. For the flotation of barite, low concentration of Ca^{2+} has little effect, but high concentration of Ca^{2+} will inhibit its flotation effect. This is because the high concentration of Ca^{2+} will compete with the barium oxide ions on the active center, reducing its surface activity and inhibiting the flotation effect (Dai et al., 2020). For the flotation of bastnaesite, high concentration of Ca^{2+} will inhibit its flotation effect, the reason is similar to fluorite.

3.3.2. Effect of Fe³⁺ on flotation of monazite and paragenetic ore

The effects of different pH conditions and Fe³⁺ concentration on flotation minerals were studied, as shown in Figs. 6 and 7. The experimental conditions in Fig. 6 are as follows: the concentration of OHA was 5×10^{-4} mol/dm³, the amount of Fe³⁺ was 1×10^{-5} mol/dm³, the amount of MIBC foaming agent was 30 ppm, and the pH value of the pulp is adjusted with 2 % NaOH and 2 % HCl.

Fig. 6 shows that when pH < 8, the flotation recovery of monazite, barite and bastnaesite increases with the increase of pH, and the recovery rate reaches the maximum at pH = 8, and the recovery rates were 87.29 %, 60.23 % and 55.38 %, respectively. After that, the recovery rate decreased rapidly with the increase of pH value. At pH = 12, the recovery rate of monazite was only 41.27 %, while the flotation recovery rates of barite and bastnaesite were 17.27 % and 30.15 %. The flotation recovery of fluorite increases with the increase of pH value, and the recovery rate reaches the maximum of 60.28 % at pH = 10. After that, the recovery rate decreased rapidly with the increase of pH, and the recovery rate was only 18.23 % at pH = 12. The flotation recovery of calcite increases with the increase of pH, and the recovery rate reaches the maximum at about pH = 10, which is 50.11 %. After that, the recovery rate decreased rapidly with the increase of pH, and the recovery rate was only 35.21 % at pH = 12. It shows that Fe³⁺ has different effects on different minerals, and the effects of different pH values are also different. This may be related to the fact that Fe³⁺ hydrolyzes different dominant components at different pH, and different dominant components have adsorption effects on different minerals, thus affecting OHA adsorption minerals. Under the same conditions, when the pH was 8, the recovery of monazite was 89.23 %, while the recovery of fluorite, calcite, barite and bastnaesite were 53.01 %, 40.73 %, 60.23 %, and 55.38 %, respectively. The dominant component of Fe³⁺ hydrolysis at pH = 8 activates the surface of monazite, which is beneficial to the flotation of monazite by OHA.



Fig. 6. Effect of Fe^{3+} on the flotation of monazite and gangue minerals as a function of pH

Under different pH conditions, the effect of Fe^{3+} on the flotation of monazite and symbiotic minerals may be different. Under acidic conditions, iron ions can form complexes with metal oxide minerals such as monazite, thereby weakening their surface charge and making it difficult to be adsorbed by flotation agents (Zheng et al., 2021). Under neutral conditions, the effect of Fe^{3+} is relatively small, and the flotation performance of monazite will be improved. Under alkaline conditions, the flotation effect of Fe^{3+} will be enhanced. Therefore, in the presence of Fe^{3+} , it is necessary to adjust the pH value to 8 to ensure the effective separation of monazite and symbiotic minerals.

The experimental conditions in Fig. 7 are as follows: the concentration of OHA was 5×10^{4} mol/dm³, the pH value of pulp is 8, the dosage of MIBC foaming agent was 30 ppm, and different concentrations of Fe³⁺ are added to investigate the flotation effect of monazite and gangue minerals. It can be seen from the figure that with the increase of Fe³⁺ concentration, the flotation of monazite has a certain activation effect. When the concentration of Fe³⁺ was 4×10^{-5} mol/dm³, the recovery rate of monazite is 98.34 %. The barite increases with the increase of Fe³⁺ concentration. When the concentration of Fe³⁺ was 1×10^{-5} mol/dm³, the recovery rate of barite is 78.12 %. Other gangue minerals have different degrees of inhibition; when the concentration of Fe³⁺ was 3×10^{-5} mol/dm³, the recovery of fluorite, calcite and bastnaesite decreased significantly, which were 36.73%, 22.45%, and 59.97% percentage points, respectively. It can be concluded from Fig. 7 that a certain concentration of Fe³⁺ will activate monazite. The reason is that when the concentration of Fe³⁺ increases, Fe³⁺ will form a complex with the surface oxide of monazite, which will increase the amount of positive charge on its surface, thus enhancing its adsorption performance for collectors. The surface charge of symbiotic minerals decreases with the

increase of Fe³⁺ concentration, which leads to the decrease of adsorption capacity of flotation agent and affects its flotation performance.



Fig. 7. Effect of Fe³⁺ concentration on flotation of monazite and gangue minerals

3.3.3. Effect of Ba²⁺ on flotation of monazite and associated minerals

The test conditions of Fig 8: OHA concentration was 5×10^4 mol/dm³, Ba²⁺ dosage was 1×10^4 mol/dm³, MIBC dosage was 30 ppm, with 2 % NaOH, 2 % HCl to adjust the pH value of the pulp.



Fig. 8. Effect of Ba2+ on the flotation of monazite and gangue minerals as a function of pH

Fig. 8 shows that the effect of Ba^{2+} on monazite is different under different pH conditions. Under weak acidic conditions (pH = 5~7), Ba^{2+} will form an adsorbate with hydroxyl or oxide on the surface of monazite, increasing its surface polarity, thereby enhancing the flotation performance of monazite. Under weak alkaline conditions (pH = 8~9), the adsorption of hydroxide on the surface of monazite will occur. At this time, Ba^{2+} will form an electrically neutral $Ba(OH)_2$ with hydroxide, which will alleviate the surface electrical properties and improve the flotation rate of monazite. When pH = 8, the recovery rate of monazite is 85.29 %. At higher pH (pH>10), oxides appear on the surface of monazite. At the same time, $Ba(OH)_2$ formed by Ba^{2+} and hydroxide is too much, and the corresponding potential on the surface is too positive, resulting in a decrease in the surface polarity of monazite, thus weakening its flotation performance (Liu et al., 2021). In summary, under acidic or neutral conditions, Ba^{2+} is beneficial to the flotation of monazite, while under higher alkaline conditions, it is not conducive to the flotation of monazite. Therefore, in actual production, it is necessary to control and adjust Ba^{2+} under different conditions to achieve the best flotation effect.

Under different pH conditions, the effect of Ba²⁺ on the flotation of fluorite, calcite, barite and bastnaesite is also different: Flotation of fluorite: Under weak acidic and weak alkaline conditions, Ba²⁺

is beneficial to the flotation of fluorite, because Ba²⁺ can form an adsorbate with the hydroxyl or oxide on the surface of fluorite, increase the surface polarity, thereby enhancing its flotation performance. When pH = 8, the recovery rate of fluorite is up to 45.27%, however, under high pH conditions, Ba²⁺ will form a chemical bond with F-on the surface of fluorite, which makes the surface wettability of fluorite worse, thus weakening its flotation performance (Chen and Zhu, 2021). Flotation of calcite: Under acidic or neutral conditions, Ba2+ will form a Ca-calcite complex with carbonate ions on the surface of calcite, which increases its flotation performance. When pH = 6, the recovery rate of calcite is up to 45.27 %. However, at higher pH conditions, Ba²⁺ will form an electrically neutral Ba(OH)₂ with hydroxide, resulting in a decrease in surface electrical properties, thereby weakening the flotation performance (Liu et al., 2021). Flotation of barite: Under weak acidic conditions, low concentration of Ba²⁺ can improve the flotation rate of barite, because Ba²⁺ can form a hydrophilic active center on the surface and increase its flotation performance. The flotation of bastnaesite: The effect of Ba²⁺ on the flotation of bastnaesite is greatly affected by the pH value. When the pH value is 5~9, Ba2+ can significantly improve the flotation rate of bastnaesite, and with the increase of Ba²⁺ concentration, the recovery rate will gradually increase. When the pH value is less than 5 or greater than 9, the flotation effect of Ba2+ will be reduced. This is because at a lower pH value, the surface of bastnaesite will be affected by hydrogen ions (Xiang et al., 2022), and the charge on the surface of the mineral will become more cationic, which will reduce the amount of adsorption with positively charged Ba²⁺, thereby reducing the flotation effect; at higher pH values, the surface charge of bastnaesite will become more negatively charged (Chen and Zhu, 2021), and it is difficult to produce adsorption reaction with positively charged Ba²⁺, thus affecting the flotation effect.

The experimental conditions in Fig. 9 are as follows: the concentration of OHA is 5×10^{-4} mol/dm³, the amount of Ba²⁺ is a variable, the amount of MIBC foaming agent is 30 ppm, and the pH value of the slurry is adjusted to 8 with sodium hydroxide and hydrochloric acid.

The effect of Ba²⁺ concentration on monazite flotation is very significant. Monazite is a phosphate mineral, and it usually needs to add a large number of auxiliary collectors to achieve better flotation results. However, by reasonably controlling the concentration of Ba²⁺, the flotation rate of monazite can be significantly improved, thereby reducing the dependence on auxiliary collectors. In the presence of low concentration of Ba²⁺, Ba²⁺ can form hydrophilic functional groups on the surface of monazite to promote its flotation, thereby improving the flotation rate. At the same time, the hydroxide ions on the surface of monazite will form Ba(OH)₂ (Moonchul et al., 2022) with Ba²⁺, which will ease the surface electrical properties and further improve the flotation rate. When the concentration of Ba²⁺ is 2×10^{-4} mol/dm³, the recovery rate of monazite is 95.84 %. However, when the concentration of Ba²⁺ is too high (more than 2×10^{-4} mol/dm³), too much Ba²⁺ will form too much Ba(OH)₂ with hydroxide, and the surface electrical property is too positive, resulting in the decrease of the surface polarity of monazite, thus weakening its flotation performance (Chen and Zhu, 2021). Therefore, in the flotation process of monazite, it is necessary to reasonably control the concentration of Ba²⁺ to achieve the best flotation effect. In general, the appropriate concentration of Ba²⁺ is between $1 \sim 2 \times 10^{-4}$ mol/dm³.



Fig. 9. Effect of Ba2+ concentration on flotation of monazite and gangue minerals

The presence of Ba²⁺ has a certain influence on the flotation process of fluorite, calcite, barite and bastnaesite. When the concentration of Ba^{2+} is 2×10^{-4} mol/dm³, the recovery of fluorite, calcite, barite and bastnaesite is 45.32 %, 23.47 %, 72.78 %, and 84.83 %, respectively. For the flotation of monazite, the presence of Ba^{2+} is beneficial to inhibit the flotation recovery of fluorite; for the flotation of calcite, the presence of Ba2+ will also have a certain impact on its flotation effect. Low concentration of Ba2+ has a little effect on the flotation of calcite, but when the concentration of Ba²⁺ is too high, it will inhibit the decrease of calcite flotation recovery or unable to flotation. For barite, the presence of low concentration of Ba²⁺ can increase the surface charge density of barite, thereby enhancing its adsorption capacity with air bubbles and promoting its flotation effect. However, if the concentration of Ba²⁺ is too high, it will compete with Ba²⁺ in barite for adsorption sites, resulting in a decrease in the surface charge density of barite, thereby weakening its adsorption capacity with air bubbles and reducing its flotation effect (Wang et al., 2023). In the flotation of bastnaesite, Ba^{2+} also has a certain influence on its flotation effect. However, when the concentration of Ba²⁺ is high, the flotation of bastnaesite will be inhibited, because Ba²⁺ will form a Ba-fluoroceria complex with fluorine ions on the surface of bastnaesite, which will affect its flotation performance (Ran et al., 2017). Therefore, it is very important to control the concentration of Ba²⁺ in the flotation of monazite to ensure the maximum flotation effect and recovery rate of monazite.

3.4. Discussion

The flotation of monazite, calcite, fluorite, barite, and bastnaesite by metal ions was summarized in Table 2. The dosage of OHA collector was 5×10^4 mol/dm³, the pulp pH value was 8, Ca²⁺, Fe³⁺, and Ba²⁺ concentration were 3×10^4 mol/dm³, 3×10^{-5} mol/dm³, and was 2×10^{-4} mol/dm³ respectively.

Table 2. Flotation recovery of monazite and associated minerals/%

Test condition	Monazite	Calcite	Fluorite	Barite	Bastnaesite
OHA+Ca ²⁺	96.48	40.43	24.71	45.37	38.68
OHA+ Fe ³⁺	98.34	-	15.64	54.27	5.28
OHA+ Ba ²⁺	95.84	23.47	45.32	72.78	84.83

It can be seen from table 2 that a certain concentration of Ca^{2+} , Fe^{3+} and Ba^{2+} has an activation effect on monazite, while the re-concentration effect on calcite, fluorite and barite bastnaesite is general, and some symbiotic minerals are inhibited. In addition, a certain concentration of Ba^{2+} has an activation effect on barite and bastnaesite. Therefore, controlling the concentration of metal ions can change the flotation environment, so as to effectively separate monazite from symbiotic ore. On the whole, when the pulp pH value was 8, Ca^{2+} , Fe^{3+} , and Ba^{2+} concentration was $3 \times 10^{-4} \text{ mol/dm}^3$, $3 \times 10^{-5} \text{ mol/dm}^3$, and was $2 \times 10^{-4} \text{ mol/dm}^3$ respectively the effective separation of monazite and symbiotic ore.

3.5. Zeta potential analysis of metal ions on the surface of monazite

The change of zeta potential of mineral particles caused by the addition of flotation reagents may reflect the adsorption of flotation reagents on the mineral surface. Fig. (a) shows the zeta potential changes with pH before and after the interaction of Ca^{2+} (3×10⁻⁴ mol/dm³), Ba^{2+} (2×10⁻⁴ mol/dm³), Fe^{3+} (3×10⁻⁵ mol/dm³) and monazite. Fig. 10(b) shows the results for zeta potential of monazite Ca^{2+} , Ba^{2+} , and Fe^{3+} using OHA as a function of pH.

As shown in Fig. 10(a), the isoelectric point (IEP) of pure monazite occurs at pH = 6 when no reagent was added. When only metal ions were added to the monazite, the monazite (IEP) shifts to the right, and the negative charge on the surface of the monazite decreases. This may be due to the positive charge of the metal ions adsorbed on the surface of the monazite, increasing the positive charge on the surface of the monazite, followed by Ca²⁺, and Ba²⁺.

As shown in Fig. 10(b), After adding OHA only, the IEP of monazite decreased significantly, showing electronegativity, indicating that the negatively charged octyl hydroxamic acid ion adsorbed on the surface of monazite. After adding Ca²⁺, Ba²⁺, Fe³⁺, and OHA, the IEP of monazite moves to the left, and the zeta potential of monazite shifts to the positive direction as a whole, indicating that Ca²⁺,

Ba²⁺, and Fe³⁺ adsorbed on the surface of monazite and increase the positive electricity on the surface of monazite. From the diagram, it can be seen that the zeta potential of monazite surface shifts to the positive direction is most affected by Fe³⁺, followed by Ca²⁺, and finally Ba²⁺, indicating that in the flotation of monazite, the ability of metal ions to adsorb the surface of monazite is: Fe³⁺ > Ca²⁺ > Ba²⁺.



Fig. 10. Effects of Ca2+, Ba2+ and Fe3+ on zeta potential of monazite surface

Combined with the analysis of the flotation behavior of monazite in the presence of Ca^{2+} , Ba^{2+} , and Fe^{3+} , it can be seen that the adsorption of Ca^{2+} , Ba^{2+} , and Fe^{3+} on the surface of monazite enhances the electrostatic adsorption of negatively charged OHA ion collector on the surface of monazite, and strengthens the adsorption of OHA on the surface of monazite to achieve the effect of activation. Due to the interaction between metal ions and OHA, the adsorption characteristics of OHA are changed, which is beneficial to the flotation of monazite. when the pulp pH value was 7~10, the surface of monazite is the same as that before and after the action of the agent, and both are negatively charged. At this time, OHA can still adsorb on the monazite, that is, the chemical affinity overcomes the same-sex electric repulsion and adsorbs, so that the potential of the monazite after the action is reduced, which is chemical adsorption.

3.6. Infrared spectrum analysis of metal ions on the surface of monazite

Fig. 11 shows the infrared spectra of monazite before and after the action of OHA collector and OHA collector with Ca^{2+} (3×10-4 mol/dm³), Ba^{2+} (2×10-4 mol/dm³), and Fe^{3+} (3×10-5 mol/dm³) ions, at pH = 8.

It can be seen from Fig. 11 that in the infrared spectrum of monazite, PO_3^{2-} groups are shown at 616 cm⁻¹, 541cm⁻¹, PO_4^{3-} groups at 1052cm⁻¹, $H_2PO_4^{-}$ was the bending vibration peak of monazite at 1385 cm⁻¹, $H_2PO_4^{-}$ is the stretching vibration peak at 1628cm⁻¹ (Xue, 2003), and OH- group of water molecules in solid at 3452cm⁻¹. In the infrared spectrum of OHA, the N-O-H bending vibration peak is at 1424 cm⁻¹, the C-N stretching vibration peak was at 1468 cm⁻¹ (Shu et al., 2019), the -C=O stretching vibration peak was at 1664 cm⁻¹, the -N-H and -O-H superimposed stretching vibration peaks are at 3260 cm⁻¹, and the -CH₂-/-CH₃ stretching vibration peaks are at 2916 cm⁻¹ and 2847 cm⁻¹ (Zhang et al., 2020; Shu et al., 2020). The infrared spectrum of the interaction between monazite and OHA shows a small organic peak-CH₂-/-CH₃ at 2927 cm⁻¹ and 2852 cm⁻¹, indicating that OHA is weakly adsorbed on the surface of monazite and only an unstable quaternary ring complex is formed.

When OHA is activated on the surface of monazite by Ca²⁺, Ba²⁺, and Fe³⁺, a strong -CH₂-/-CH₃ stretching vibration peak appears at 3000cm⁻¹~2800cm⁻¹, and a large red-shift occurs. The reason is that the -CH₂-/-CH₃ functional group of OHA collector is adsorbed on the surface of monazite. A new peak appeared at 1382cm⁻¹, which was due to the adsorption of N-O-H functional group of OHA collector on the surface of monazite, and the red shift (44 cm⁻¹) occurred. A new peak appeared at 1454 cm⁻¹, which was due to the adsorption of OHA collector on the surface of monazite. The lone electron pair of the nitrogen atom of the amide group in OHA forms chelates with Ca²⁺, Ba²⁺, and Fe³⁺ atoms to further increase the adsorption capacity of OHA, which indicates that the adsorption of metal ions on the surface of monazite provides active sites for adsorbing OHA collectors. It shows that

the adsorption capacity of OHA on the surface of monazite activated by Ca²⁺, Ba²⁺, and Fe³⁺ is enhanced, and a stable five-membered ring metal chelate is formed, which is consistent with the literature report (Tian et al., 2018). It can be seen from the figure that the organic functional group -CH₂-/-CH₃ peak of OHA adsorbed on the surface of monazite activated by Fe³⁺ is stronger, while the organic peak -CH₂-/-CH₃ adsorbed on the surface of monazite activated by Ca²⁺ is slightly weaker, and the monazite activated by Ba²⁺ is weaker. It can be concluded that Ca²⁺, Ba²⁺, and Fe³⁺ activated monazite surface adsorption capacity: Fe³⁺>Ca²⁺>Ba²⁺.



Fig. 11. Infrared spectra of Ca²⁺, Ba²⁺ and Fe³⁺ on monazite in OHA

4. Conclusions

In view of the release behavior of metal ions from monazite symbiotic ore in OHA system and the influence of metal ions on the flotation and mechanism of monazite, the grinding and micro-flotation processes were adopted, and the ICP OES, zeta potential and infrared spectrum analysis were used. The following conclusions were drawn:

During the grinding process, the metal ions released from monazite and symbiotic minerals increase with the increase of grinding time, mainly including Ca²⁺, Ba²⁺, and Fe³⁺. These metal ions may change the flotation environment.

In the micro-flotation process, OHA collector was used. When the pulp pH value was 8, Ca^{2+} , Fe^{3+} , and Ba^{2+} concentration was 3×10^{-4} mol/dm³, 3×10^{-5} mol/dm³, and was 2×10^{-4} mol/dm³ respectively. while the flotation recoveries of monazite, calcite. And fluorite was above 95 %, 40.43 %, and below 45.32 %, and the recovery rate of barite and bastnaesite in the presence of Ca^{2+} and Fe^{3+} is below 54.32 % and 38.68 %, respectively and the effective separation of monazite and symbiotic ore was obtained. The favorable flotation ability of metal ions for monazite was $Fe^{3+} > Ca^{2+} > Ba^{2+}$. The metal ions Ca^{2+} , Ba^{2+} , and Fe^{3+} have a general effect on the flotation of calcite, fluorite, barite and bastnaesite. Among them, calcite and fluorite have an inhibitory effect. Only a certain concentration of Ba^{2+} has a certain activation effect on barite and bastnaesite. Therefore, controlling the concentration of metal ions can change the flotation environment, so as to effectively separate monazite from symbiotic ore.

Under the zeta potential analysis, when only Ca^{2+} , Ba^{2+} , and Fe^{3+} are added to monazite, the negative charge on the surface of monazite decreases, after adding Ca^{2+} , Ba^{2+} , Fe^{3+} , and OHA, the IEP of monazite moves to the left, and the dynamic potential of monazite shifts to the positive direction as a whole, indicating that Ca^{2+} , Ba^{2+} and Fe^{3+} are adsorbed on the surface of monazite and increase the positive electricity on the surface of monazite. Secondly, the ability of metal ions to adsorb monazite surface is: $Fe^{3+} > Ca^{2+} > Ba^{2+}$. The mechanism behavior is that the adsorption of Ca^{2+} , Ba^{2+} , and Fe^{3+} on the surface of monazite enhances the electrostatic adsorption force of negatively charged OHA ion collector on the surface of monazite, strengthens the adsorption of OHA on the surface of monazite, and achieves the effect of activation. When the pulp pH value was 7~10, the surface of monazite is the same as that before and after the action of the agent, and both are negatively charged. At this time, OHA can still adsorb on the monazite, that is, the chemical affinity overcomes the same-sex electric repulsion and adsorbs, so that the potential of the monazite after the action is reduced, which is chemical adsorption.

In the infrared spectrum analysis, it is concluded that after the action of OHA and monazite alone, a weak adsorption phenomenon occurs and an unstable four-membered ring complex is formed; when metal ions Ca²⁺, Ba²⁺, and Fe³⁺ were added to activate the surface of monazite, the organic functional group -CH₂- / -CH₃ peak of OHA adsorbed by monazite was enhanced. Through the flotation results, zeta potential data and infrared spectroscopy analysis, it is shown that the adsorption capacity of OHA on the surface of Ca²⁺, Ba²⁺, and Fe³⁺ activated monazite is enhanced, which adds new adsorption sites to the surface of monazite. The metal ions adsorbed on the surface of monazite form a stable five-membered ring metal chelate with OHA, which improves the adsorption capacity of OHA. The ability of Ca²⁺, Ba²⁺, and Fe³⁺ to adsorb OHA after activating the surface of monazite: Fe³⁺> Ca²⁺> Ba²⁺. This study is helpful to enrich the activation theory of metal ions in ore pulp, and also confirms that effective mineral flotation separation depends not only on the strength of collector-mineral interaction, but also on the chemistry of flotation solution to a large extent.

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