

## Study on the separation effects of the novel collector ODD on magnesite and quartz

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**Abstract:** Traditional magnesite desilication flotation collectors struggle to efficiently remove quartz from low-grade magnesite, prompting the exploration of new, highly selective flotation collectors. Addressing this need has become a focal point in mineral processing research. This study introduced heptadecylamine ethylimidazoline quaternary ammonium salt (ODD) as a quartz flotation collector for separating quartz from magnesite. Flotation experiments involving single minerals and artificially mixed minerals demonstrated that magnesite and quartz could be effectively separated under specific conditions: an ODD concentration of 40mg/L and pH=7.0. Zeta potential assessments revealed that the adsorption of ODD increased the potential of quartz by 4.4 times compared to magnesite. Furthermore, contact angle measurements illustrated that ODD selectively increased the hydrophobicity of the quartz surface while not affecting the contact angle of magnesite. X-ray photoelectron spectroscopy (XPS) analysis indicated that ODD's selective adsorption at the quartz surface through interaction with the O sites on quartz rather than magnesite. Drawing from these findings, a flotation separation model from magnesite and quartz under the influence of ODD was formulated.

**Keywords:** magnesite, quartz, ODD, selective adsorption, flotation separation

### 1. Introduction

Magnesium carbonate is the main chemical component of magnesite (Yang et al., 2017; Sun et al., 2021). As a vital raw material for magnesium metals and compounds, magnesite is a crucial resource with many uses in both agriculture and industry (Xie et al., 2023; Park et al., 2020). China has deposits of 3.6 billion tons of magnesite, the majority of which are located in Liaoning Province (86% of the whole country) (Chen et al., 2024; Haifler et al., 2024). The main hubs for the extraction of magnesite are found in places like Australia, China, Russia, Canada, and Brazil. Of these, Australia stands out as one of the world's top producers of magnesite, taking advantage of its plentiful resources and cutting-edge mining techniques to become a significant supplier to the magnesite market worldwide. Significant magnesite deposits are also found in China, but the country's mining techniques and manufacturing capabilities are not up to par, leaving it heavily dependent on imports to satisfy demand. Magnesite extraction and utilization will continue to be focus areas due to the steadily increasing demand for magnesium materials and compounds, which play a crucial role in the global resource landscape.

Both direct and reverse flotation methods are used in the magnesite flotation process. The reverse flotation process is mainly used for desilication. In the mineral and purification process of magnesite, reducing the SiO<sub>2</sub> content is the key to improving magnesite grade. In magnesite, the primary silica-bearing vein mineral is quartz (Xue et al., 2024; Wang et al., 2023; Declercq et al., 2023). In desilication processes, dodecylamine (DDA) was commonly used to separate magnesite from quartz. The analysis indicates that the main modes of action of DDA on the mineral surface were electrostatic attraction and hydrogen bonding. However, DDA has certain disadvantages, including poor resilience at low temperatures, high foaming tendencies, large electrostatic effects from polar groups, and weak steric hindrance. As a result, in the presence of DDA as a collector, reverse flotation separation of magnesite and quartz might result in the flotation of magnesite together with silica-bearing vein minerals,

decreasing the magnesite recovery in the concentrate. To maximize resource usage in intricate low-grade magnesite deposits, the search for a cationic collector with strong selectivity towards quartz becomes crucial.

Heptadecylamine Ethylimidazoline Quaternary Ammonium Salt (ODD) is a highly silicon-selective surfactant, and its unique structural features endow it with outstanding performance in various applications (Okoth et al., 2016). The long-chain alkyl provides hydrophobicity, the imidazoline ring offers polarity and hydrophilicity, and the quaternary ammonium salt group carries a positive charge (Rajendiran and El Rassi, 2023). These characteristics suggest that ODD may exhibit favorable effects in flotation processes. Therefore, ODD has the potential as a desilication collector for magnesite reverse flotation and has research value.

In this research, experiments on both single and mixed mineral flotation were conducted to investigate the selective trapping mechanism of ODD. Zeta potential, contact angle, and X-ray photoelectron spectroscopy (XPS) tests were utilized in further studies to clarify this process. This research aims to provide practical implementation references and insights into low-grade magnesite on-site desilication beneficiation procedures.

## 2. Materials and methods

### 2.1. Experimental materials

The quartz used in the experiment came from Dashiqiao, Liaoning, while the magnesite was purchased from Haicheng, Liaoning. Several preliminary processes were applied to the ore, including vibrating screening, crushing. Subsequently, the powder was carefully mixed to create ore samples with a particle size of  $-74+44\mu\text{m}$  for further experiments. The findings of the mineral X-ray diffraction study were presented in Fig. 1, while the results of the chemical multi-element analysis were provided in Table 1. Notably, the test results indicated that quartz and magnesite had purity levels of 99.11% and 99.28%, respectively, meeting the requirements for single-mineral flotation testing.

The chemically pure reagent ODD used for experiments was provided by the Shanghai McLean Biochemical Technology Co., Ltd.. The flotation test media used in the experiment was deionized water, and the pH was adjusted with sodium hydroxide and hydrochloric acid. Among all the chemicals used in this work, ODD was chemically pure and was prepared using dimethyl sulfoxide (DMSO) as a solvent and diluted to the corresponding volume with deionized water.

Table 1. The chemical composition of mineral samples

| Sample    | MgO(%) | CaO(%) | SiO <sub>2</sub> (%) | Al <sub>2</sub> O <sub>3</sub> (%) | Total Fe (%) | Purity (%) |
|-----------|--------|--------|----------------------|------------------------------------|--------------|------------|
| Magnesite | 47.47  | 0.57   | 0.50                 | 0.05                               | 0.15         | 99.28      |
| Quartz    | -      | -      | 99.11                | -                                  | 0.11         | 99.11      |

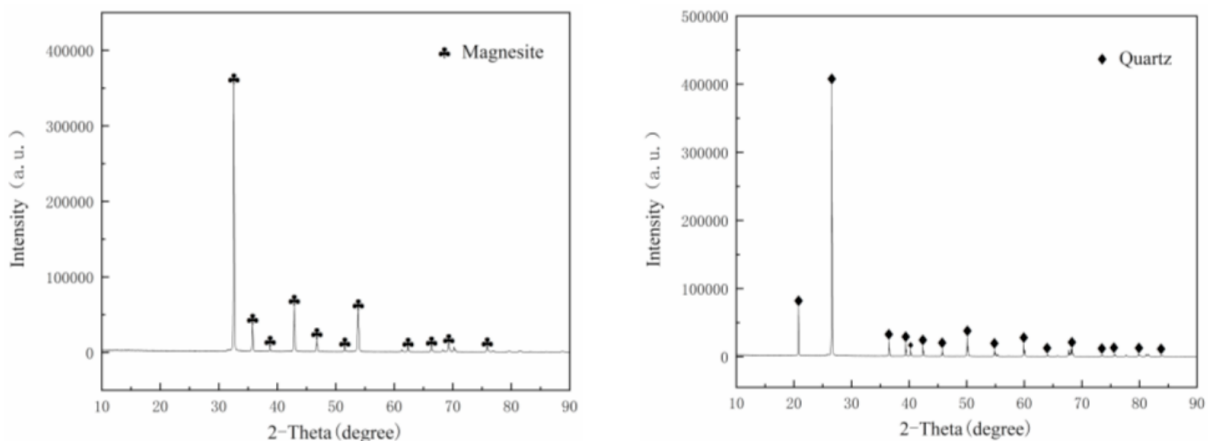


Fig. 1. The XRD analysis of mineral samples

## 2.2. Test methods

### 2.2.1. Flotation tests

A hanging tank flotation machine with a 30 mL flotation capacity was used to examine the flotation of 2g of each test ore sample for single and binary mixed ore samples. Firstly, stir the slurry for two minutes, then add the collector and pH regulator in sequence according to the flotation test procedure shown in Fig. 2, and finally perform a 3-minute scraping. Throughout the experiment, the flotation machine operated with 20mL of deionized water, rotating at a speed of 1800 r/min. Following the experiment, each concentrate was dried and weighed. And the mineral recoveries were determined by equations (1). where  $\epsilon$  expresses the mineral recovery (%),  $\beta$  refers to the grade (MgO or SiO<sub>2</sub>) of this mineral in concentrates, (%);  $\theta$  represents the grade (MgO or SiO<sub>2</sub>) of this mineral in tailings, (%),  $m_c$  and  $m_t$  separately denote the mass of the concentrate and tailing products, (g).

$$\epsilon = \frac{\beta m_c}{\beta m_c + \theta m_t} \times 100\% \quad (1)$$

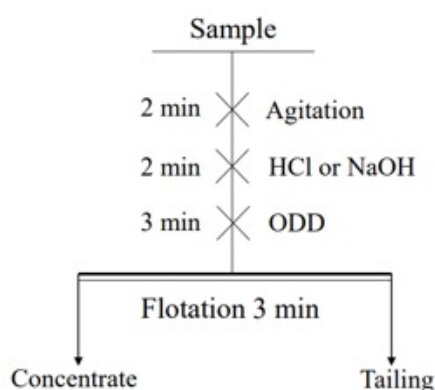


Fig. 2. Flotation experimental process

### 2.2.2. Zeta potential measurement

An American Zeta Finder analyzer was used to determine the samples' zeta potential during the test. To start, 20 mg of the ore samples were crushed very finely until the grains were around 5  $\mu\text{m}$  in size. After that, the samples were added to a 40 mL KCl electrolyte solution that had a concentration of  $1 \times 10^{-3}$  mmol/L. After agitating the mixture for ten minutes, chemicals were added sequentially, and the pH was adjusted according to the flotation test protocol. Lastly, the solution was given five minutes to settle before the supernatant was removed in order to quantify it.

### 2.2.3. Contact angle

Contact-angle measurements were conducted using a Theta Flex analyzer (Biolin). 20 mL of deionized water was added to a 2 g sample (particle size:  $-74 + 45 \mu\text{m}$ ), followed by stirring for 2 min. Thereafter, the pH of the solution was adjusted, and the stirring was continued. The collector ODD was then added, and the mixture was stirred for 3 min. The solution was then filtered and the solid fraction was washed with distilled water thrice. Finally, the solid fraction was dried at 313 K. In the process of contact-angle tests, the powdered samples were pressed into a cylindrical product with a size of  $\Phi 1.0 \text{ cm} \times 0.2 \text{ cm}$  by an electric table machine under a pressure of 490000 N. The contact angle was measured by dropping a water droplet with a diameter of approximately 2–3 mm on the surface of the sample using the microsyring of the contact angle measuring instrument.

### 2.2.4. X-ray Photoelectron Spectroscopy (XPS) measurements

The sample preparation for XPS was identical to that for FTIR spectroscopy. XPS spectra of samples treated with and without ODD at pH 7.0 were obtained using a Thermo Scientific VG ESCALAB 250Xi spectrometer. The binding energy scale was corrected using a C1s peak at approximately 284.80

eV as the internal binding energy standard. A 2 g sample was placed in 20 mL of deionized water, centrifuged at 3000 rpm for 10 min, and subjected to solid-liquid separation. The centrifuged solid mineral was washed three times with deionized water, and the solid fraction was dried and analyzed.

### 3. Results and discussion

#### 3.1. Single mineral floatability test

##### 3.1.1. Effect of pH on the floatability of single minerals

The ODD dosage was fixed at 40 mg/L to investigate the effect of slurry pH on the floatability of magnesite and quartz, and the results are depicted in Fig. 3.

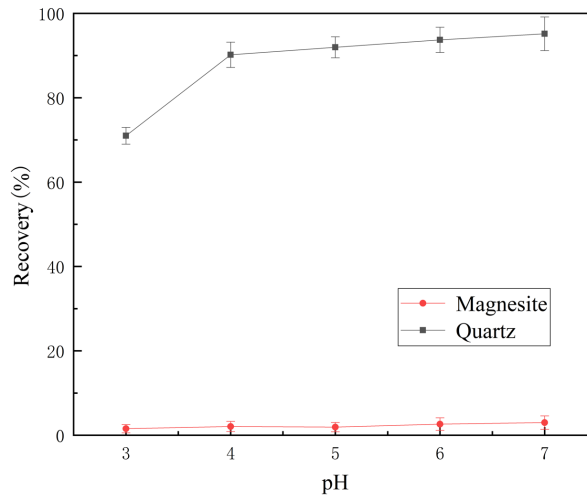


Fig. 3. Effect of pH on the floatability of magnesite and quartz

According to the graph, when pH=3, the difference in recovery rates between quartz and magnesite facilitated by ODD approaches 70%. Within the pH range of 3-7, the recovery of quartz shows a slight increasing trend as pH increases. At pH=7, the recovery of quartz reaches 95.16%. The recovery of quartz increases with rising pH and peaks at pH=7, where the recovery of quartz is 95.16% and that of magnesite is 2.99%. The difference in recovery between the two minerals is the largest at pH=7. Considering all factors, pH=7 was selected as the optimal choice.

##### 3.1.2. Effect of ODD dosage on the floatability of single minerals

The effect of ODD dosage on the floatability of magnesite and quartz was investigated at pH=7, and the experimental results are depicted in Fig. 4.

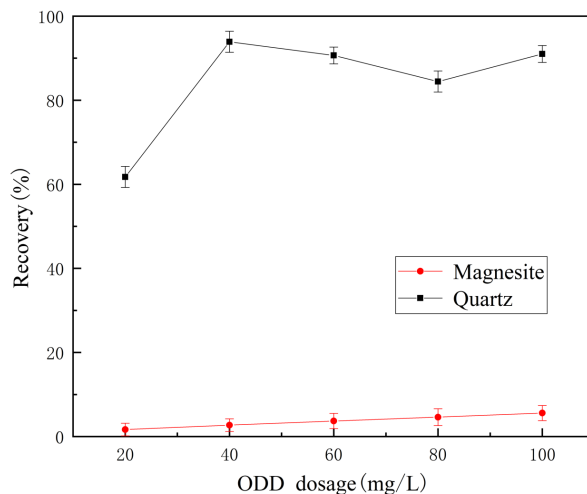


Fig. 4. Effect of ODD dosage on the floatability of magnesite and quartz

As shown in Fig. 4, with an ODD dosage of 20 mg/L, the recovery of quartz is 61.75%, while that of magnesite is only 1.67%. When the ODD concentration increases to 40 mg/L, the recovery of quartz reaches 93.90%. With further increases in reagent dosage, the recovery of quartz shows a slightly decreasing trend. At an ODD dosage of 100 mg/L, the recovery of magnesite is only 5.59%, consistently remaining below 10%. Comparing the recovery of the two minerals at different ODD dosages, it can be seen that at an ODD dosage of 40 mg/L and a pulp pH of 7.0, there is a significant difference in the flotation recovery of quartz (93.90%) and magnesite (2.73%).

### 3.1.3. Influence of flotation machine speed on the floatability of single minerals

The ODD dosage was fixed at 40 mg/L and pH=7, and the effect of flotation machine speed on the floatability of magnesite and quartz was investigated. The results are depicted in Fig. 5.

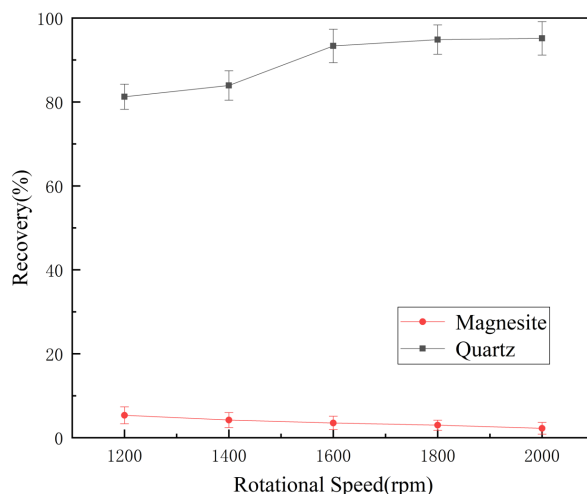


Fig. 5. The effect of flotation machine speed on the floatability of magnesite and quartz

From Fig. 5, it can be seen that the recovery of quartz increases with the flotation machine's rotating speed. At a speed of 1200 rpm, the recovery of quartz reaches 81.23%, while that of magnesite is 5.35%. When the speed reaches 2000 rpm, the recovery of quartz reaches 95.16%, and that of magnesite is 2.99%. At this speed, the difference in the recovery of the two minerals is the largest. Based on the above experimental results, it can be concluded that the optimal rotating speed for flotation experiments is 2000 rpm.

### 3.2. Artificial mixed ore flotation separation experiment

Artificial mixed-ore flotation separation tests were carried out in accordance with the protocol depicted in Table 2 in order to improve the flotation separation efficiency of magnesite and quartz under various flotation conditions. 2.0 g of ore samples were used in each artificial mixed ore flotation test, with the mixed ore ratio adjusted at magnesite: quartz = 1:1, 7:3, and 9:1.

Table 2. Results of Artificial Mixed Ore Experiments

| Mixed Ore Proportion  | Product     | Yield/% | Grade/% |                  | Recovery/wt. % |        |
|-----------------------|-------------|---------|---------|------------------|----------------|--------|
|                       |             |         | MgO     | SiO <sub>2</sub> | Magnesite      | quartz |
| Magnesite: Quartz=1:1 | Concentrate | 38.14   | 35.31   | 6.18             | 73.44          | 4.83   |
|                       | Raw Ore     | 100.00  | 23.81   | 50.00            | 100.00         | 100.00 |
| Magnesite: Quartz=7:3 | Concentrate | 66.40   | 45.04   | 1.24             | 93.68          | 2.74   |
|                       | Raw Ore     | 100.00  | 33.33   | 30.00            | 100.00         | 100.00 |
| Magnesite: Quartz=9:1 | Concentrate | 86.19   | 45.82   | 0.72             | 95.32          | 6.22   |
|                       | Raw Ore     | 100.00  | 42.85   | 10.00            | 100.00         | 100.00 |

From the table 2, it can be seen that, except for the artificially mixed ore with a ratio of 1:1, where the concentrate had an MgO grade of 35.31% and a SiO<sub>2</sub> grade of 6.18%, the artificially mixed ores with ratios of 7:3 and 9:1 had higher MgO grades. In these ratios, the MgO grade of the concentrate remained above 45%, and the SiO<sub>2</sub> grade was below 1.3%. This indicates that ODD can effectively separate quartz and magnesite during the flotation process.

### 3.3. Mechanism analysis

#### 3.3.1. Zeta potential analysis

The influence of flotation agent on mineral surface often alters their properties, subsequently impacting the flotation behavior of mineral particles (Zhang et al., 2023; Song et al., 2023). Zeta potential detection and analysis are widely used in the field of mineral flotation separation to elucidate the nature of interactions between mineral surfaces and flotation chemicals (Han et al., 2021; Zgheib et al., 2024). The results of zeta potential analysis for magnesite and quartz before and after interaction with flotation chemicals are depicted in Fig. 6.

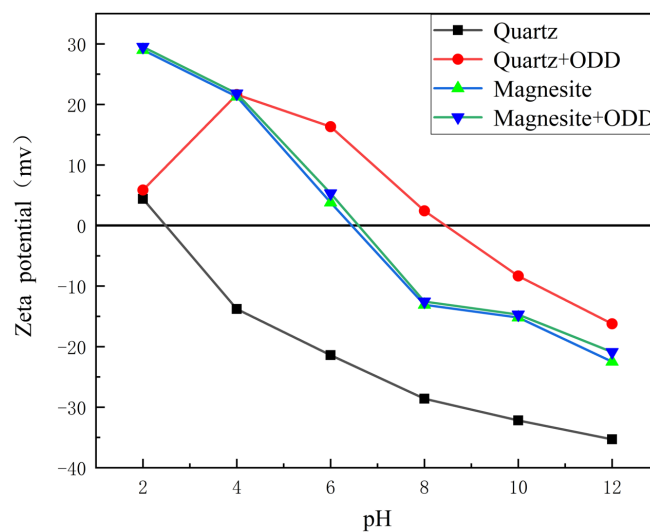


Fig. 6. Relationship curve of mineral surface zeta potential with slurry pH before and after ODD

Zeta potential measurements indicate that as the pH increases, the zeta potential of the mineral surface gradually decreases. At pH 7.00, the zeta potential of quartz (-21.4 mV) is more negative than that of magnesite (3.8 mV). Due to electrostatic attraction, at pH 7.00, positively charged ODD is more easily adsorbed onto negatively charged quartz than onto positively charged magnesite, resulting in a significant change in the zeta potential of quartz from -21.4 mV to 16.33 mV. After the addition of the reagent, the surface potential of magnesite changes only slightly, indicating that most of the ODD is adsorbed onto the surface of quartz, causing a significant change in its potential, while only a small portion of ODD was adsorbed onto magnesite. Overall, hydrogen bonding and electrostatic attraction interactions facilitate strong adsorption of ODD onto quartz, making the effective collection of ODD on quartz possible.

#### 3.3.2. Contact angle measurement

The hydrophilicity of a mineral surface can be visually indicated by the contact angle, and the size of the angle significantly impacts the mineral's floatability (Bai et al., 2020; Ling et al., 2024). A higher contact angle indicates that the mineral surface is more hydrophobic. Therefore, we can determine the floatability properties of quartz and magnesite by comparing the changes in contact angle before and after the minerals interact with chemicals.

Prior to the addition of the ODD agent, the initial contact angles of quartz and magnesite measured 12.61° and 13.92°, respectively, as shown in Fig. 7 (a) and (c), indicating strong hydrophilicity. The addition of ODD significantly altered the surface properties of quartz, increasing the contact angle from

12.61° to 45.89°, indicating strong hydrophobicity. In contrast, the contact angle of magnesite increased only slightly from 13.92° to 14.01°, with almost no change in hydrophobicity. This indicates that while the addition of ODD increased the surface hydrophobicity of both magnesite and quartz, the enhancement was much greater for quartz. This suggests that ODD exhibits a high degree of selectivity in altering the surface hydrophobicity of magnesite and quartz.

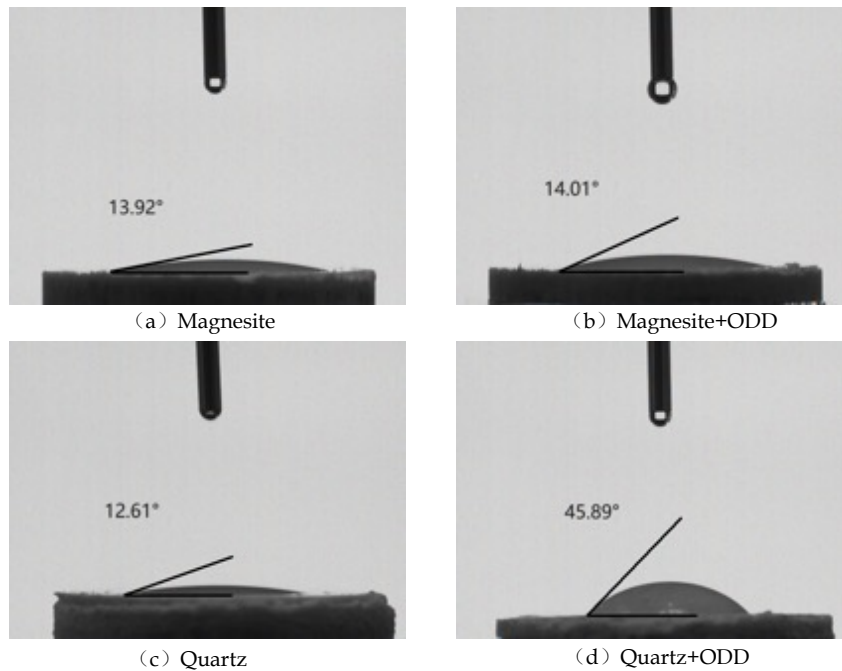


Fig. 7. Contact angle characteristics of magnesite and quartz before and after the action of flotation chemicals

### 3.3.3. X-ray photoelectron spectroscopy

The elemental composition, chemical state, molecular structure, and surface content of a material can all be determined using X-ray photoelectron spectroscopy (XPS), a crucial surface analysis method (Qin et al., 2021; Zhang et al., 2024). XPS detection and analysis were carried out to gain insights into the mechanism underlying the activity of the collector ODD on quartz and magnesite. Previous studies have indicated that ODD primarily adsorbs onto the surfaces of quartz and magnesite through hydrogen bonding and electrostatic interactions. Therefore, to study the adsorption strength of ODD collectors on the surface of minerals, the XPS spectra of the anionic element O on the surfaces of magnesite and quartz were analyzed.

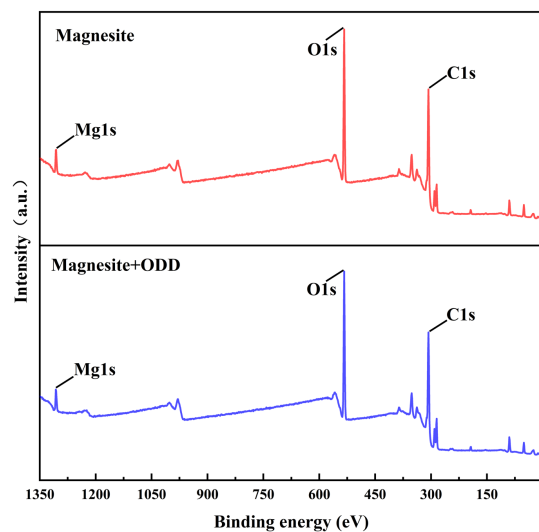


Fig. 8. Mineral X-ray photoelectron full spectrum of quartz before and after ODD action

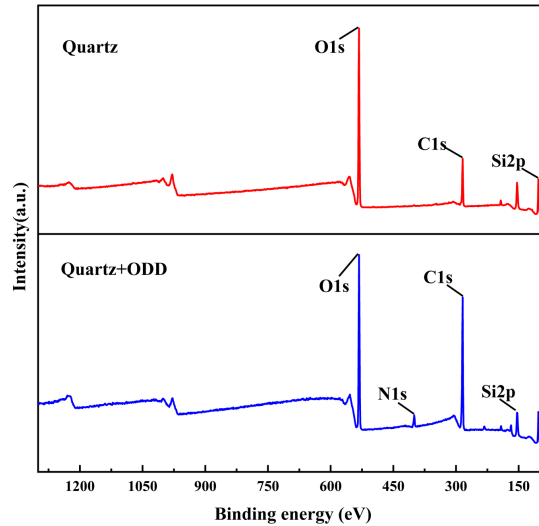


Fig. 9. Mineral X-ray photoelectron spectra of magnesite before and after ODD action

Previous studies have shown that the Mg1s peak of magnesite typically appears around 1304 eV, attributed to Mg<sup>2+</sup> ions on the surface of magnesite (Gong et al., 2024; Sun et al., 2024), while the Si2p peak of quartz was typically observed at approximately 103 eV (Wang et al., 2024; Liu et al., 2023). The N1s peak was also expressed at around 401 eV and can be attributed to the ODD molecule, as ODD is the sole nitrogen source.

With a 40 mg/L ODD dosage and a slurry pH of 7.0, XPS analysis was conducted on the mineral surfaces to investigate the adsorption mechanism of ODD on minerals. Fig. 10 and 11 depict the results, including the complete X-ray photoelectron spectra and their corresponding surface O elemental energy spectra.

Table 3. Analysis of relative content of mineral surface elements

| Mineral       | Elemental content% (Binding Energy/ Ev) |                   |                   |                   |                  |
|---------------|---|-------------------|-------------------|-------------------|------------------|
|               | Mg1s                                    | Si2p              | C1s               | O1s               | N1s              |
| Magnesite     | 5.26<br>(1305.85)                       | 0                 | 39.36<br>(284.80) | 55.38<br>(531.84) | 0                |
| Magnesite+ODD | 4.79<br>(1305.87)                       | 0                 | 40.93<br>(284.80) | 54.13(531.68)     | 0                |
| Quartz        | 0                                       | 22.31<br>(103.06) | 29.80<br>(284.80) | 47.89<br>(532.96) | 0                |
| Quartz+ODD    | 0                                       | 13.46<br>(103.10) | 54.27<br>(284.80) | 29.75<br>(533.43) | 2.51<br>(400.27) |

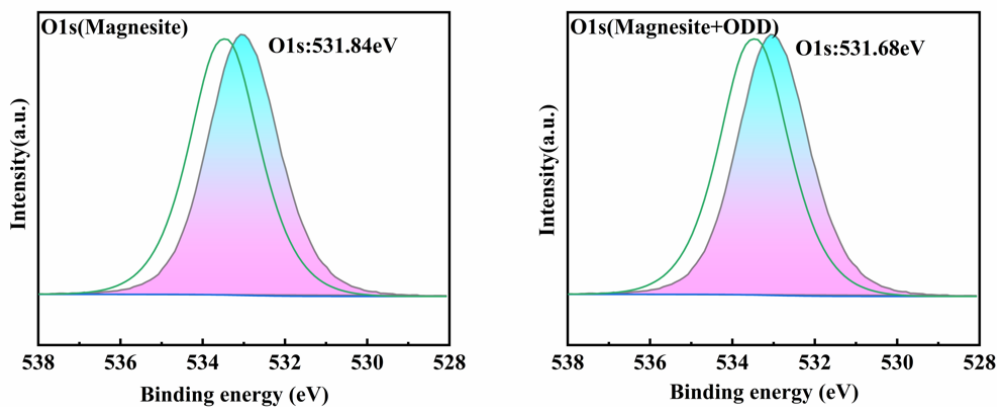


Fig. 10 O binding energy on magnesite before (a) and after (b) ODD treatment



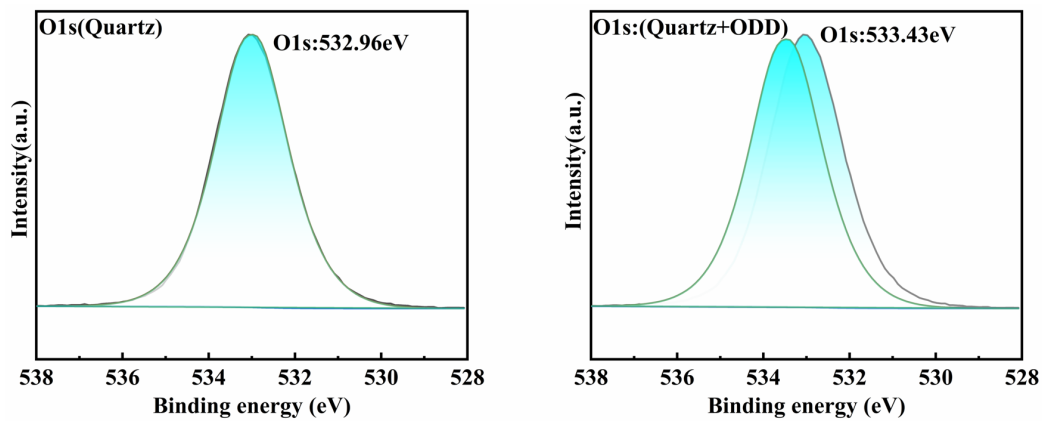


Fig. 11 O binding energy on quartz before (a) and after (b) ODD treatment

The X-ray photoelectron spectra of the untreated magnesite single mineral, as shown in Fig. 10(a), and the ODD-treated magnesite single mineral, as shown in Fig. 10(b), exhibit similarity, the absence of a distinct peak at the binding energy of 402 eV suggests that ODD did adsorb onto the surface of magnesite. On the contrary, table 3 shows that a clear N1s peak(2.51eV) was observed at a binding energy of 400.27 eV on the quartz surface treated with ODD, indicating that ODD was adsorbed there. Upon examining Figs. 10 and 11 it is evident that following ODD treatment, the binding energies of the O1s peaks corresponding to the O elements of quartz and magnesite were 533.43 eV and 531.68 eV, respectively. The binding energies of the O1s peaks for the quartz and magnesite surfaces exhibited slight shifts, by 0.47 eV and 0.16 eV, respectively, compared to the absence of agents. Additionally, the shift in the binding energy of the O1s peak on quartz surfaces (0.47eV) is about 2.93 times that of magnesite (0.16 eV). This indicates that the action of ODD on the surface of quartz is much stronger than on magnesite, resulting in the selective adsorption of ODD collectors on the surface of quartz rather than magnesite.

### 3.4. Modeling the separation of magnesite and quartz

Magnesite belongs to carbonate minerals. During the stirring of the aqueous solution, lattice anions and cations ( $Mg^{2+}$  or  $CO_3^{2-}$ ) continuously dissolve from the surface, undergoing hydrolysis and protonation reactions in the aqueous solution to form  $MgOH^+$  and  $HCO_3^-$  in the solution phase. Meanwhile, the mineral surface can react with adsorbed water molecules, generating surface functional groups (or sites), such as  $-MgOH^+$  and  $-HCO_3^-$ . The generation of mineral surface electrical properties is directly related to the uneven content of magnesite and quartz (Zeng et al., 2024; Tian et al., 2024). Under different conditions, the functional groups formed on the surface of carbonate minerals exhibit differential adsorption of anions and cations in solution. This disparity eventually leads to variances in the kinetic characteristics of the mineral surface.

At a slurry pH of 7.0, the cationic quaternary ammonium salt group of the heptadecylamine ethyl imidazoline molecule facilitates preferential adsorption of ODD onto the surface of the quartz. Quartz's surface is more negatively charged, making it a better candidate for ODD adsorption. The XPS test results reveal a significant change in the binding energy of the O site on the surface of the quartz, indicating the formation of  $N \cdots H-O-Si$  hydrogen bonding between O sites on the quartz surface and ODD molecules. This bonding enhances the stability of the ODD molecule attached to quartz. Furthermore, ODD, as a quaternary ammonium salt, possesses a chemical structure comprising a hydrophobic group (heptadecenyl) and a hydrophilic group (imidazoline quaternary ammonium salt group). During adsorption, the hydrophilic group interacts with the quartz surface, rendering it hydrophobic due to exposure of the hydrophobic group. Consequently, quartz becomes more likely to adhere to bubble surfaces during flotation, in contrast, when magnesite adsorbs ODD, its surface still exhibits high hydrophilicity, causing it to sink, thereby achieving flotation separation between magnesite and quartz. Under the action of the ODD collector, the separation model of magnesite and quartz is shown in Fig. 12.

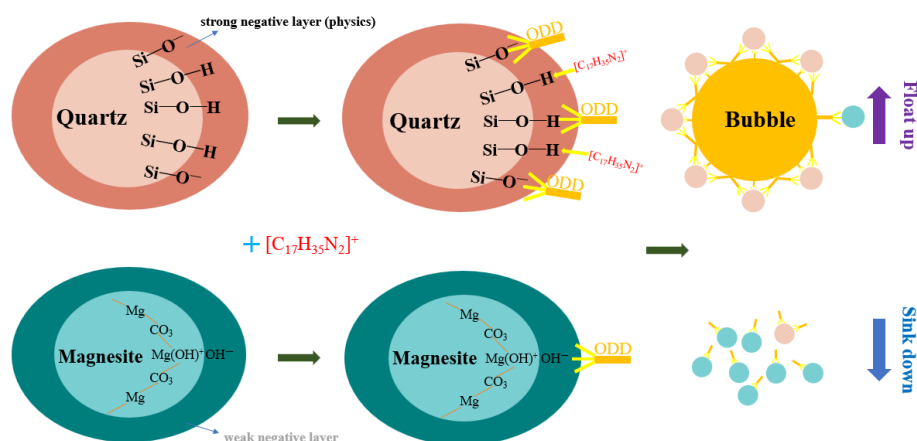


Fig. 12 Schematic representation for adsorption models of ODD on magnesite and quartz surfaces

#### 4. Conclusions

1. The results of the single-mineral flotation test revealed a significant difference in flotation behavior between magnesite and quartz when the ODD dosage was 40 mg/L and the pH was maintained at 7.0. Subsequently, flotation separation tests were conducted using different ratios of artificially mixed minerals under these conditions. It was observed that ODD facilitated a more effective flotation separation of magnesite and quartz.
2. The results of the zeta potential analysis revealed that the addition of the ODD collector shifted the zero electric point of both magnesite and quartz surfaces positively. Specifically, the potential value of quartz at pH 4.0 increased from -13.75 mV to 21.644 mV, indicating the superior selectivity of ODD.
3. The results of the contact angle measurements indicated a significant increase in the hydrophobicity of the quartz surface after ODD treatment, whereas the surface properties of magnesite remained relatively unchanged. When combined with the results of the XPS analysis, it becomes evident that ODD is more readily adsorbed onto the O site of the quartz surface, with weaker interaction observed at the Mg site. Consequently, ODD exhibits selective adsorption on the surface of quartz.

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