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# Effect of ultrasonic pretreatment on flotation purification of quartz

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**Abstract:** Quartz sand purity dictates its applications, with current research focusing on flotation purification. To investigate the effects of ultrasonic pretreatment on quartz flotation, an RKIII single-tank flotation machine was employed at a neutral pH of 6.8, and the impacts of varying ultrasonic powers (120-300 W) and different treatment durations (0-25 min) were discussed. Additionally, ultrasonic pretreatments were carried out in acidic and alkaline environments simulated by 1%-5% solutions of hydrochloric acid and sodium hydroxide, respectively. Through the analysis of impurity content in quartz sand, it was found that under natural pH conditions and a power range of 120-300 W, the optimal purification effect was achieved by adding 100 g of quartz sand to 1200 $cm^3$ of deionized water and subjecting it to ultrasonic treatment for 10-15 min. As the ultrasonic power increased, the purification effect was enhanced. The results showed that the removal of $Fe_2O_3$, $TiO_2$, and $Al_2O_3$ was increased by 10.4%, 3.3%, and 1.2%, respectively, compared with that of the conventional flotation after ultrasonic pretreatment for 15 min with ultrasound power 240 W in a neutral environment. In the optimal 5% HCl solution, the removal rate of $Fe_2O_3$ was 11.2% and 21.6% higher than that of the control group and the untreated group, respectively. The removal rate of $TiO_2$ was 4.6% and 7.9% higher, respectively. The removal rate of $Fe_2O_3$ increased by 23.2% and that of TiO2 increased by 9.1% with 240 W ultrasonic treatment in 4% NaOH solution.

***Keywords:*** ultrasonic pretreatment, quartz, purification, flotation

## 1. Introduction

With the continuous advancement of technology, there is a growing demand for high-purity quartz sand within high-tech industries. This sand, primarily composed of silicon dioxide, undergoes a rigorous purification process using top-quality quartz ore (Zhang et al., 2023). High-purity quartz sand boasts exceptional attributes such as superior heat resistance, high transparency, thermal shock resistance, low thermal expansion coefficient, robust anti-crystallization properties, high insulation, corrosion resistance, and distinctive optical qualities (Vatalis et al., 2014). As a result of these characteristics, it finds widespread application in premium sectors like optoelectronics, optical fiber communication, semiconductor integrated circuits, and aerospace, playing a pivotal role in the development of the countries (Zhong et al., 2022). In recent years, China has increasingly emphasized the significance of clean energy with the introduction of the "carbon peak and carbon neutrality" concept, elevating the importance of clean energy nationwide. Photovoltaic power generation, being a critical clean energy source, has gained significant traction. However, existing technologies' limited effectiveness in purifying ore restricts the production of high-quality photovoltaic quartz sand. Consequently, how to effectively purify quartz has become the research focus of the current and future (Pan et al., 2022a).

Flotation is one of the important methods for the separation of granular minerals, to enhance mineral flotation, methods like strengthening through external factors such as magnetic fields (Özbayoğlu et al., 2009; Sun et al., 2020; Xu et al., 2022), electric fields (Azmi et al., 2020; Deng et al., 2021; Hakizimana et al., 2017), and acoustic fields (Mao et al., 2019; Ng et al., 2020, 2021) are explored. Among these, ultrasonic strengthening technology stands out due to its rapid development. The main effects of ultrasonic treatment on a flotation system are characterized by cavitation and are accompanied by a local increase in pressure and temperature. As solid-liquid interactions are weaker than liquid cohesion forces, solid-liquid interfaces are more amenable to the formation of cavitation. The unsettled conditions

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caused at a solid-liquid interface might modify the surface properties of minerals, leading to changes in the adsorption of reagents at mineral surfaces, hence their flotation responses (Gungoren et al., 2017; Ozkan, 2012). Therefore, in recent years, the utilization of ultrasound for enhancing mineral flotation has gradually increased (Kang and Li, 2020). According to the literature, ultrasonic treatment is employed at various stages in flotation processes, including during reagent conditioning, the flotation process itself, both during conditioning and flotation or as pretreatment before flotation (Farmer et al., 2000; Gungoren et al., 2019; Mao et al., 2020; Peng et al., 2018). Pandey et al. (2010) reported that impurities such as aluminum oxide, silicon dioxide, and phosphorus in iron ore can be more effectively removed after ultrasonic treatment. Videla et al. (2016) studied the use of ultrasonic technology to enhance the recovery rate of copper flotation. Their research suggests that introducing ultrasonic technology during pulp conditioning and flotation processes can effectively improve flotation efficiency, resulting in a 3.5% increase in copper recovery. The cavitation effect of ultrasound can efficiently cleanse particle surfaces and reduce the slime coatings on tailings particles, thereby facilitating the action of reagents. Ozkan et al. (2017) investigated the effects of synchronous ultrasonic technology on the flotation of hard coal slime with a $d_{80}$ particle size of minus 0.2 mm. Their study demonstrates that ultrasonic treatment can cleanse mineral surfaces, thereby enhancing the adsorption environment of reagents on coal particle surfaces and reducing the optimal flotation reagent (Ekofol-440) dosage from 120-125 g/Mg to 95-100 g/Mg. Within the optimal reagent dosage, over 90% combustible recovery and 70% separation efficiency values were achieved during this study.

Although significant results have been achieved in ultrasound-enhanced flotation of different types of coal slurry (Ambedkar et al., 2011), magnesite slimes (Ozkan, 2002), sulphide ores (Aldrich and Feng, 1999; Cilek and Ozgen, 2009b) and other minerals (Fang et al., 2020; Kursun, 2014), ultrasound-enhanced flotation of low-grade quartz has been less studied, especially in the ultrasound pre-treatment of quartz in acidic solution and alkaline solution has been rarely involved in the study. In this sense, this study proposes the use of ultrasonic technology as pretreatment to improve quartz flotation, and quartz flotation experiments with different ultrasonic power, pretreatment time, and treatment environments were carried out to investigate the effect of ultrasonic pretreatment on quartz flotation.

## 2. Materials and methods

### 2.1. Materials

The quartz sand used in the experiments was sourced from quartz sandstone originating from Hanzhong, Shaanxi Province. The obtained quartzite samples were first crushed using a jaw crusher (MT-10Y/S/MT), ground with a planetary ball mill (YXQM-1L), and then sieved through 70- and 140-mesh screens to select particles ranging from 109 to 212 μm. The results of X-ray diffraction (XRD, Nishi-Smart Lab 9KW, Japan) spectral analysis of the quartz sand are shown in Fig. 1, and the results of X-ray fluorescence (XRF, Thermo Fly -ARL PERFORM, USA) chemical analysis are shown in Table 1, which indicate that the silica content in quartz sand is 99.42%, and the main impurity elements are Al, Ti, and Fe. After preliminary experiments (Bai et al., 2023), the flotation process employs soluble starch as the depressant for quartz, dodecylamine (DDA), sodium oleate (NaOL), and sodium dodecyl sulfonate (SDS) as collectors for feldspar, and pine oil as the frother. Deionised water is used throughout the flotation process.

### 2.2. Analysis and characterization

The elemental content in the samples was measured using an Agilent 725 ES inductively coupled plasma optical emission spectrometer. FEI NOVA NANO450 SEM was used to observe the morphological changes on the surface of quartz sand particles before and after sonication. The changes of functional groups on the surface of quartz before and after ultrasonication were analysed by using Germany-broke-tensor II FTIR. A certain amount of quartz sand before and after ultrasonication pretreatment was weighed, washed with deionised water, dried, and then ground with an onyx mortar and sent to the laboratory for testing. PHI 5000 VersaProbe 4 XPS was used to qualitatively and quantitatively analyse the elements on the surface of the quartz sand before and after ultrasonic pretreatment and to determine the effect of ultrasonic pretreatment on the impurity content of the quartz sand surface.

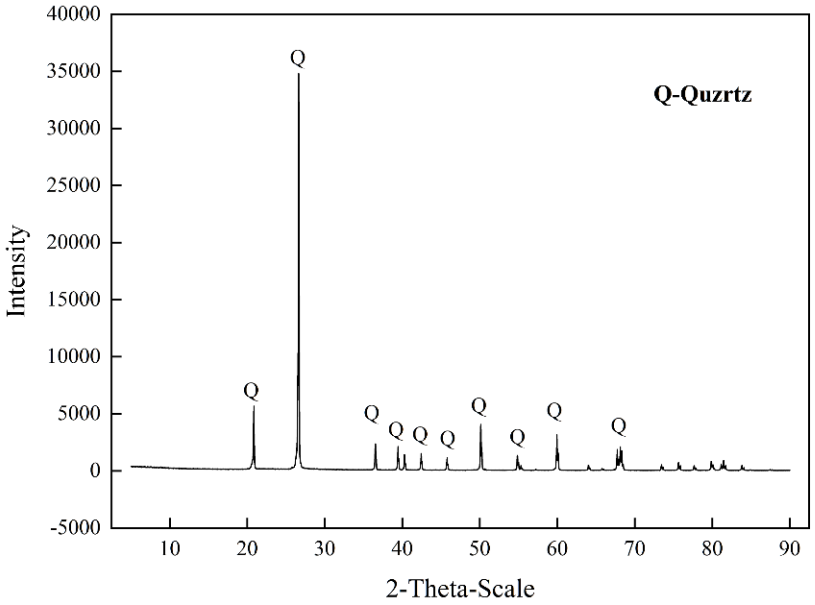


Fig. 1. XRD analysis result of the quartz sample

Table 1. Chemical components of the quartz sample

| Components | $SiO_2$ | $Al_2O_3$ | CoO | $TiO_2$ | $Na_2O$ | $Fe_2O_3$ | $K_2O$ | CaO | MgO | ZnO |
|---|---|---|---|---|---|---|---|---|---|---|
| Content (%) | 99.42 | 0.2267 | 0.0318 | 0.0334 | 0.0256 | 0.0215 | 0.0132 | 0.0077 | 0.0045 | 0.0026 |

### 2.3. Flotation procedure

#### 2.3.1. Conventional flotation

The flotation experiment utilized a single-tank flotation machine of RK Ⅲ type, with a spindle speed of 2300 r/min. The flotation temperature is set at 25°C, and reverse flotation is used, as shown in Fig. 2(a). In the flotation experiment, 100 g of quartz sand was mixed with 1200 $cm^3$ of deionised water and put into the flotation tank, $H_2SO_4$ and NaOH were used to adjust the pH value, 250 g/Mg of starch was added, 400 g/Mg of NaOL was added after stirring for 5 min, 250 g/Mg of SDS was added after stirring for 5 min, the slurry was adjusted for 5 min, and 500 g/Mg of DDA was added, the slurry was adjusted for 5 min, and then 10 $cm^3$ of pine oil was added, and the flotation was started. After flotation, the quartz sand was washed with anhydrous ethanol and deionised water and dried in an oven at 70℃. The froth and the sink products were collected separately after drying for analysis of the quartz yield the impurity removal (Jiang et al., 2020). The quartz yield was calculated using Eq. (1):

$$Quartz\ yeld(\%) = \frac{m_1}{m_1+m_2} \times 100\% \quad (1)$$

where $m_1$ is mass of the concentrate, g; $m_2$ is mass of the tailings, g.

The method for calculating the removal rate of impurity elements is shown in Eq. (2) (Mowla et al., 2008).

$$Impurity\ removal\ rate(\%) = \frac{C_{origonal} - C_{concentrate}}{C_{origonal}} \times 100\% \quad (2)$$

where $C_{original}$ represents the content of a specific impurity in the raw quartz ore, ppm; $C_{concentrate}$ represents the content of the same impurity in the flotation concentrate, ppm.

#### 2.3.2. Neutral environment ultrasonic pretreatment flotation

With the exception of the blank control group experiments, all experiments were preceded by ultrasonic pretreatment. Ultrasonic pretreatment was performed using an ultrasonic cleaning machine (CR-040S) with a capacity of 10 $dm^3$. The maximum ultrasonic power was 360 W, and the temperature could be adjusted from room temperature to 80°C. 100 g of quartz sand was mixed with 1200 mL of deionised water and placed into the flotation tank. The tank was then subjected to ultrasonic treatment at four

power levels: 120 W, 180 W, 240 W, and 300 W. Each power level underwent treatments for 5 min, 10 min, 15 min, 20 min, and 25 min. After ultrasonication in a neutral environment, the quartz sand is filtered out and added to 1200 $cm^3$ of clean deionised water in an ultrasonic cleaning machine to adjust the slurry. The steps for adding reagents and the conditioning time were the same as those described in the conventional flotation above. The pre-treatment temperature for ultrasound is set at 50°C. Following ultrasonic pretreatment, the flotation tanks were mounted on the flotation machine for the subsequent flotation experiments. The ultrasonic pretreatment flotation process is illustrated in Fig. 2(b).

### 2.3.3. Acid or alkaline ultrasonic pretreatment flotation

Ultrasonic pretreatment experiments are conducted in both alkaline and acidic solutions, with the pretreatment temperature set at 50°C. The ultrasonic power is maintained at 240 W, and the duration is 15 min. For the alkaline solution pretreatments, sodium hydroxide solutions of 1%, 2%, 3%, 4%, and 5% were prepared, resulting in respective solution pH values of 12.41, 12.73, 12.90, 13.00, and 13.13. In the acidic solutions, HCl solutions of 1%, 2%, 3%, 4%, and 5% were prepared using deionized water, with corresponding pH values of 1.58, 1.28, 1.10, 0.94, and 0.82. Subsequently, 100 g of quartz sand was weighed and added to 1200 $cm^3$ of each NaOH and HCl solution for ultrasonic treatment. Subsequently, the pulp was adjusted in the ultrasonic cleaning machine, with the amounts and adjustment times of depressants and collectors added being consistent with the reagents used in the conventional flotation process (2.3.1). Flotation could commence only after the completion of pulp conditioning.

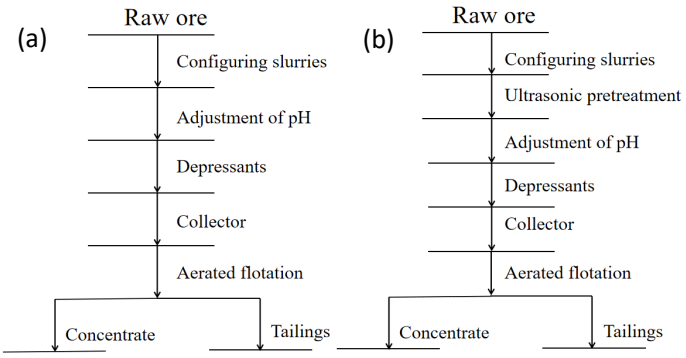


Fig. 2. Flowsheet of the flotation (a) Conventional Flotation (b) Ultrasonic Pretreatment Flotation

## 3. Results and discussion

### 3.1. Effect of ultrasonic pretreatment on flotation in neutral environment (pH=6.8)

From Fig. 3, it is evident that ultrasonic pretreatment has no significant impact on the recovery rate of quartz concentrate, with the recovery rate stabilizing above 80%. The optimal removal efficiency for $Fe_2O_3$ and $TiO_2$ occurs with a 10-min ultrasound pretreatment at 120 W, resulting in an increase of 4% and 4.8% in the removal rates of $Fe_2O_3$ and $TiO_2$, respectively. However, with a further extension of the pretreatment time, the removal efficiencies of $Fe_2O_3$ and $TiO_2$ begin to decline. Concerning $Al_2O_3$ removal, after a 5-min ultrasonic pretreatment, the removal rate drops from 75.4% to 66.6%. Increasing the pretreatment time to 10 min leads to a recovery in $Al_2O_3$ removal, although it still falls short of the blank control group level. Beyond 15 min, the removal efficiency of $Al_2O_3$ decreases again. In terms of overall impurity removal, ultrasonic pretreatment at 120 W does not significantly enhance the flotation efficiency, possibly due to the low power not adequately cleaning impurities attached to the surface of quartz sand. The removal effects of the three main elements show only minor fluctuations, indicating that low-power ultrasonic treatment has a limited impact on quartz sand flotation.

Fig. 4 demonstrates that ultrasonic pretreatment at 180 W has a negligible impact on the recovery rate of quartz sand concentrate, with no significant influence observed across different pretreatment durations, maintaining above 80%. Regarding $TiO_2$ removal efficiency, there is a slight decrease after 5 min, dropping from 71.4% to 69.1%. It rises to 73.8% at 10 min (2.4% higher than the blank control group) but declines again after 15 min. For $Fe_2O_3$ removal, there is minimal fluctuation at 5 min, a rebound to

47.3% at 10 min, followed by relative stability around 45%. Unfortunately, the removal efficiency for $Al_2O_3$ is suboptimal, with a slight decrease to 69.6% at 5 min, reaching 70.3% at 10 min, and maintaining around 60% for the remaining durations, all inferior to the blank control group.

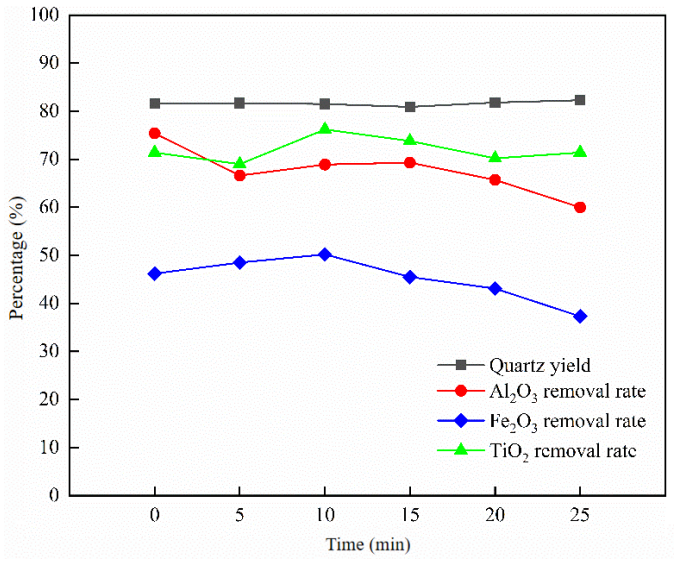


Fig. 3. The effect of 120 W ultrasonic pretreatment on the flotation of quartz and the removal rate of $Al_2O_3$, $Fe_2O_3$, and $TiO_2$ at the natural pH of 6.8

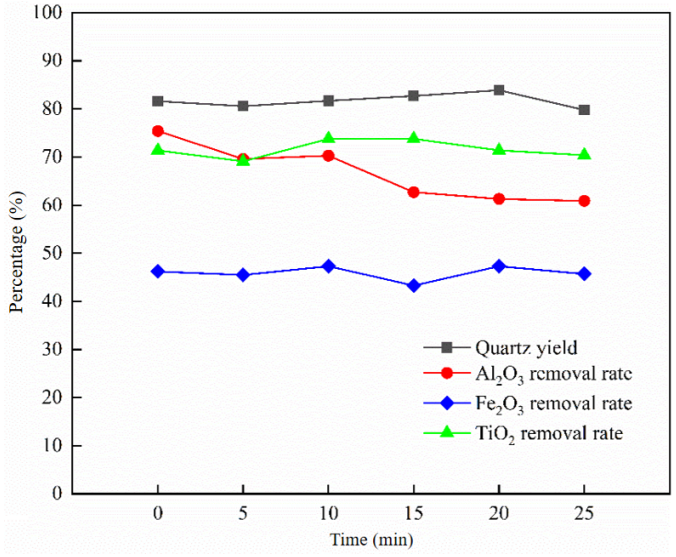


Fig. 4. The effect of 180 W ultrasonic pretreatment on the flotation of quartz and the removal rate of $Al_2O_3$, $Fe_2O_3$, and $TiO_2$ at the natural pH of 6.8

Fig. 5 reveals that, at a power of 240 W, ultrasonic pretreatment has minimal impact on the recovery rate of quartz sand concentrate, with different pretreatment durations showing no significant influence and maintaining a level above 80%. Regarding the removal efficiency of $Fe_2O_3$, there is a slight decrease at 5 min of pretreatment (from 46.2% to 40%), followed by a significant increase, reaching 52.7% (a 6.5% improvement compared to the blank control group). At a 15-min pretreatment, the removal rate of $Fe_2O_3$ stabilizes at 56.6% (a 10.4% increase compared to the control group). $TiO_2$ shows a steady increase within the first 15 min, reaching 74.7%. The removal efficiency of $Al_2O_3$ experiences a minor decline at 5 and 10 min of pretreatment but significantly rebounds to 76.6% at 15 min. In summary, at 240 W power, a 15-min ultrasonic pretreatment demonstrates the most significant impurity removal effect on quartz sand. This could be because high-power ultrasonication enhances the exposure of fine impurity minerals from the pores to the solution, facilitating subsequent flotation removal (Altun et al., 2009).

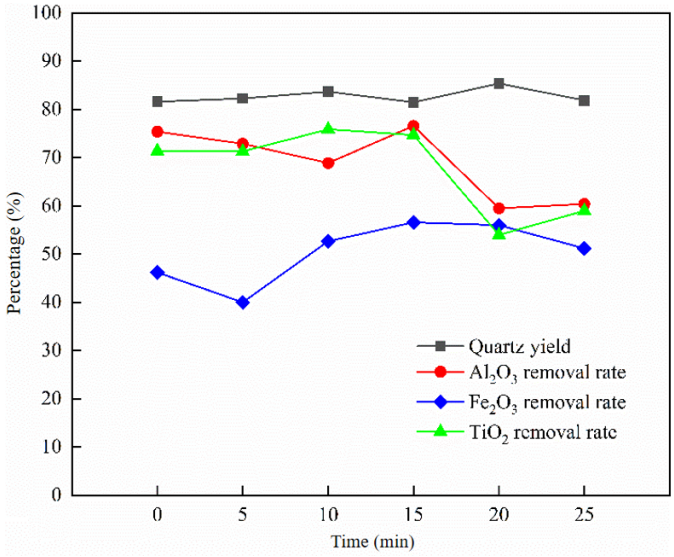


Fig. 5. The effect of 240 W ultrasonic pretreatment on the flotation of quartz and the removal rate of $Al_2O_3$, $Fe_2O_3$, and $TiO_2$ at the natural pH of 6.8

As shown in Fig. 6, at 300 W power, ultrasonic pretreatment introduces slight fluctuations in the recovery rate of quartz sand concentrate. During the 5-min pretreatment, there is a slight improvement in the recovery rate, followed by a minor decline with prolonged pretreatment, yet maintaining an overall stability of around 80%. Regarding the removal efficiency of $Fe_2O_3$, the 300 W ultrasonic pretreatment proves to be effective. At the 5-min pretreatment, the removal rate of $Fe_2O_3$ increased by 14.7% compared to the blank control group. With an increase in pretreatment time, there is a slight improvement, reaching 64.2% at 15 min (an 18% increase compared to the control group). However, with further increases in pretreatment time, the removal rate of $Fe_2O_3$ experiences a minor decline but remains consistently at a relatively high level. The removal effect on $TiO_2$ is minimally affected by the 300 W ultrasonic pretreatment, except for a slight decrease at 10 min, maintaining a level above 70% for other pretreatment durations, similar to the blank control group. Conversely, the removal effect on $Al_2O_3$ shows a negative impact, with a noticeable decrease as the pretreatment time increases. At 5 min, the removal rate of $Al_2O_3$ drops from 75.4% to 69.6%, reaching 58.4% at 15 min. In conclusion, at 300W power, ultrasonic pretreatment effectively removes $Fe_2O_3$, has minimal impact on $TiO_2$, but adversely affects the removal efficiency of $Al_2O_3$.

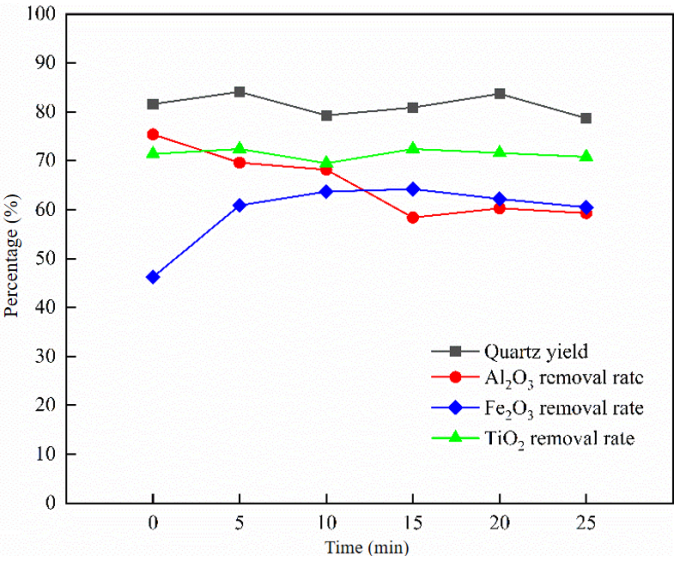


Fig. 6. The effect of 300 W ultrasonic pretreatment on the flotation of quartz and the removal rate of $Al_2O_3$, $Fe_2O_3$, and $TiO_2$ at the natural pH of 6.8

### 3.2. Effect of ultrasonic pre-treatment on flotation effect under acid/alkaline environment

As shown in Fig. 7, when NaOH concentration is 3% or below, ultrasonic pretreatment has a minor impact on the recovery rate of quartz sand concentrate, maintaining overall stability. However, with an increase in the NaOH concentration, the recovery rate begins to decline. Regarding the removal efficiency of $Fe_2O_3$, as the NaOH concentration increases, the removal rate of Fe gradually rises, reaching its maximum at 4% NaOH concentration, with the $Fe_2O_3$ removal rate peaking at 69.4%. Compared to the blank control group with 240 W power ultrasonic pretreatment for 15 min in a neutral environment, there is a 12.8% improvement; relative to flotation results without ultrasonic pretreatment, there is a 23.2% improvement. Ultrasonic pretreatment in a higher-concentration alkaline environment significantly enhances the removal of $Fe_2O_3$. For $TiO_2$ removal, minimal variation is observed in low NaOH solution concentrations, but at 4% NaOH concentration, there is a 5.8% improvement compared to the blank control group and a 9.1% improvement compared to flotation without ultrasonic pretreatment. However, ultrasonic pretreatment in an alkaline environment shows poor effectiveness for $Al_2O_3$ removal. Summarizing the results of ultrasonic pretreatment with different NaOH concentrations indicates that ultrasonic pretreatment in an alkaline environment is effective for removing Fe and Ti elements from quartz sand, especially in cases with high Fe and Ti contents.

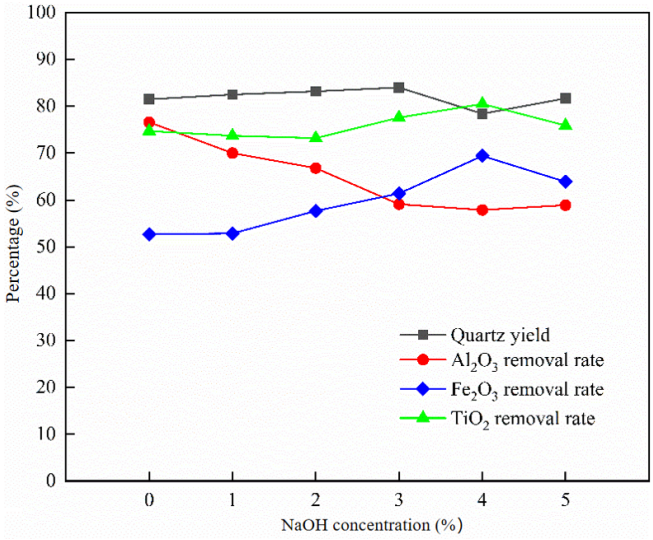


Fig. 7. The effect of 240 W ultrasonic pretreatment on the flotation of quartz and the removal rate of $Al_2O_3$, $Fe_2O_3$, and $TiO_2$ at alkaline conditions at the pH range of 12.41-13.13

In Fig. 8, the impact of different concentrations of HCl solution on the recovery rate of quartz sand concentrate is minimal, demonstrating overall stability. Considering the previously discussed influence of ultrasonic pretreatment on the recovery rate of quartz sand concentrate, it is evident that ultrasonic pretreatment does not affect the overall recovery rate of quartz sand but results in varying degrees of improvement or decline in impurity removal efficiency. At low concentrations, there is a slight fluctuation in the removal efficiency of $TiO_2$, decreasing from 74.7% to 72.4%. As the concentration of HCl solution increases, the removal rate of $TiO_2$ rises, reaching 78.2% at 3% HCl concentration. Compared to the blank control group, there is a 3.5% improvement; relative to flotation without ultrasonic pretreatment, there is a 6.8% improvement. With an increase in HCl concentration to 5%, the removal rate of $TiO_2$ reaches 79.3%. For $Al_2O_3$, low concentrations of HCl solution are ineffective, but when the HCl concentration increases to 5%, the removal rate of $Al_2O_3$ restores to 76.6%, remaining comparable to the blank control group. Regarding $Fe_2O_3$, as the HCl concentration increases, the removal rate rises, reaching 67.8% at 5% HCl concentration. Compared to the blank control group, there is an 11.2% improvement; relative to flotation without ultrasonic pretreatment, there is a 21.6% improvement. In summary, the overall removal efficiency for impurities is optimal when the HCl concentration is 5%.

To more intuitively understand the content of impurity elements in quartz sand after ultrasonic pretreatment and flotation, we measured the impurity elements in samples treated with 240 W

ultrasound for 15 min in a neutral environment (S1), 4% NaOH solution (S2), and 5% hydrochloric acid solution (S3), followed by flotation, using ICP.

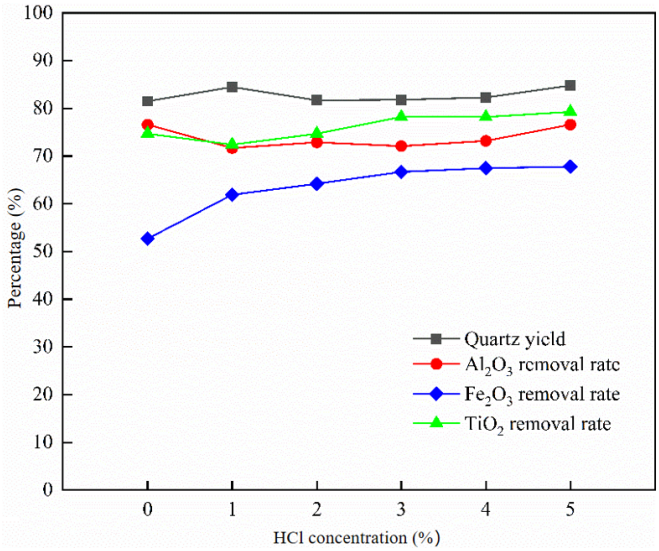


Fig. 8. The effect of 240 W ultrasonic pretreatment on the flotation of quartz and the removal rate of $Al_2O_3$, $Fe_2O_3$, and $TiO_2$ at acidic conditions at the pH range of 0.82-1.52

Table 2. ICP measurement results of quartz sand under different conditions

| sample | impurity element content (ppm) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Al | Co | Na | K | Fe | Ti | Ca | Mg |
| S1 | 287.59 | 192.54 | 121.63 | 96.33 | 66.20 | 54.83 | 50.07 | 25.18 |
| S2 | 484.13 | 206.79 | 112.54 | 87.11 | 47.18 | 39.33 | 48.16 | 24.37 |
| S3 | 278.91 | 188.87 | 116.94 | 89.58 | 49.25 | 40.37 | 51.83 | 25.52 |

### 3.3. SEM analysis

The changes that occur on the surface of quartz sand minerals after ultrasonic pretreatment cannot be directly distinguished with the naked eye and need to be analysed with the help of SEM.

Fig. 9a shows that there are small pieces of bright components in the quartz sand surface, which are associated with minerals and slime attached to the quartz surface. As known from the literature, ultrasonic pre-treatment has a strong cleaning effect on the clay attachment on the mineral surface (Chen and Peng, 2018; Ghadyani et al., 2018). After ultrasonic pre-treatment of quartz, it can be seen that the surface flatness of quartz has been improved to some extent, and the particles attached to the surface have been significantly reduced. Through SEM observation, we can see that ultrasound has a strong cleaning effect on the surface of quartz particles. This also enables the agent to attach more effectively to the quartz surface (Yang et al., 2019).

Fig. 10 shows scanning electron microscope images of quartz treated with different ultrasonic powers in a neutral environment. Cracks on the quartz surface can be observed in Figs. 10f and 10h. This is due to the facilitation of transient cavitation by low-frequency (20-50 kHz) ultrasonic waves. The rupture of transient cavitation bubbles produces high-impact forces that result in effects such as surface cleaning, sonochemical effects, and fragmentation (Chen et al., 2020). The surface cleaning and fragmentation effects generated by cavitation play a positive role in the flotation purification of quartz in this experiment. The surface cleaning effect of ultrasonic cavitation can be well observed in Figs. 9b-d and 10a-h. The different forms of impurities in quartz significantly influence the quality of the quartz and the requirements of subsequent purification processes. Impurities are categorized into three types based on the difficulty of purification: independent minerals, inclusions, and lattice impurities. When elements like Fe, Al, and Ti exist as inclusions, they can be ground to a certain size, or exposed by calcining with water and quenching, and then removed through processes such as flotation and magnetic separation (Pan et al., 2022b). Therefore, ultrasonic pre-treatment of quartz, and the fragmen-

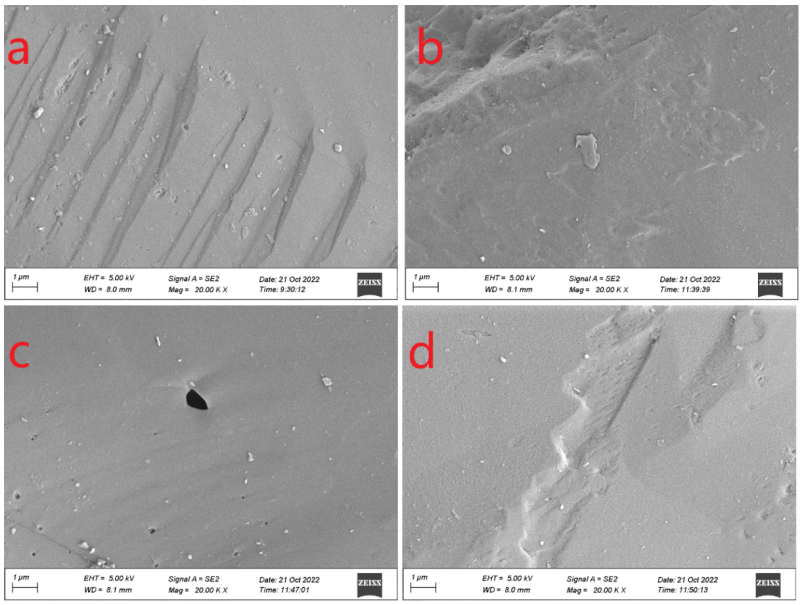


Fig. 9. SEM images of quartz sand (a: Not pretreated by ultrasound, b: 240 W ultrasonic power pretreatment, c: Ultrasonic pretreatment in NaOH solution at 4% concentration, d: Ultrasonic pretreatment in 5% HCl solution).

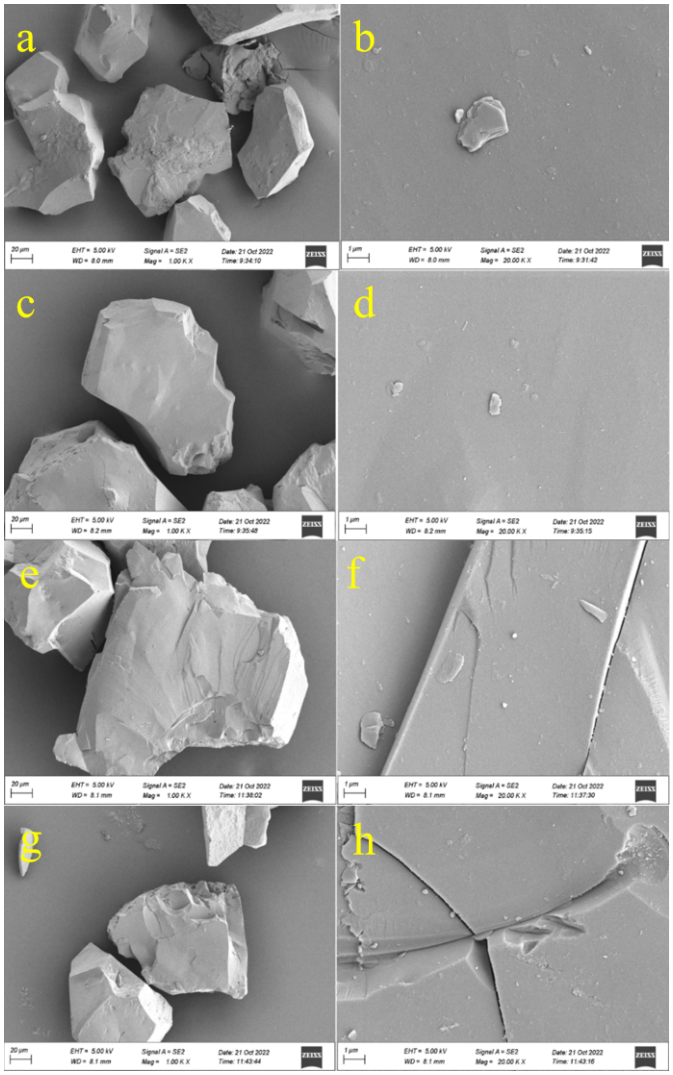


Fig. 10. SEM images of quartz after 15 min of ultrasonic pretreatment under natural pH (a, b: 120 W; c, d: 180 W; e, f: 240 W; g, h: 300 W)

tation effects produced by cavitation, can also expose some fine impurity minerals encapsulated within quartz to the solution, facilitating their removal in subsequent flotation and acid leaching experiments.

### 3.4. FTIR analysis

FTIR spectra of quartz sand before and after ultrasonic pretreatment with different powers and environments are shown in Fig. 11. The overall trend of the infrared spectra of quartz sand treated with different power ultrasound is consistent, and the overall structure of the macromolecules has not changed significantly, only the vibration strength of certain functional groups has changed. Except for the ultrasonic pretreatment in NaOH solution, the vibration intensity of functional groups under the rest of the ultrasonic conditions is larger than that of quartz sand without ultrasonic pretreatment. In comparison, it was found that the anti-symmetric stretching vibration peak of Si-O-Si at 1080.64 $cm^{-1}$, and the peaks at 777.71 $cm^{-1}$, 693.76 $cm^{-1}$, and 456.63 $cm^{-1}$ were the symmetric vibrational stretching peaks of Si-O bonds (Amin et al., 2016; Liu et al., 2011). After ultrasonic pretreatment, these peaks have a certain degree of improvement compared with the quartz sand without ultrasonic pretreatment, which may be due to the ultrasonic pretreatment for the mineral surface to play a certain degree of cleaning effect, reducing the impact of the impurities attached to the surface of the mineral flotation. The surface of the impurity minerals contained in the quartz sand was also effectively cleaned, so that the flotation chemicals could be better adsorbed on the surface of the impurity minerals, reducing the impact of surface impurities on the flotation of impurity minerals. The peak intensity of the quartz sand pretreated by ultrasonic treatment in NaOH solution was not significantly improved, which may be due to a certain degree of reaction between the quartz surface and NaOH.

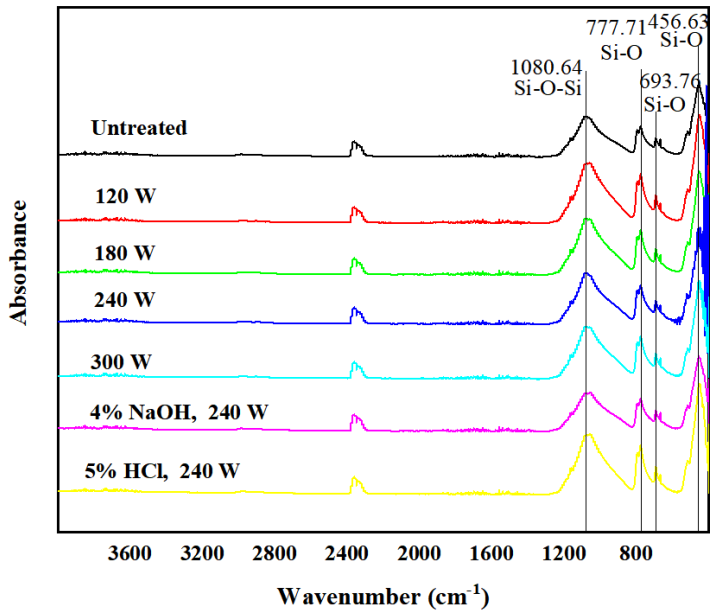


Fig. 11. Infrared spectra after 15 min of ultrasonic treatment at different power levels

### 3.5. XPS analysis

In order to further investigate the effect of ultrasound on quartz, XPS analysis was conducted on quartz samples treated under different conditions.

From Fig. 12, we can see that the signal intensity of O 1s and Si 2p peaks is very strong, which is in line with the molecular structure of quartz, and the spectra of quartz without ultrasonic pretreatment, we can see that the peaks of the three impurity elements of Fe, Al, and Ti, which are discussed in this study, did not appear, which suggests that these three impurity elements are not mainly endowed on the surface of the quartz sand (Hu et al., 2024). Combined with the previous SEM observations, the cleaning effect of ultrasonic pretreatment on the quartz surface is only for the silt on the surface of the quartz, while the main impurity elements are not removed in the ultrasonic pretreatment stage, but are effectively separated in the subsequent flotation step. The intensity of the Si 2p peak under 240 W power

ultrasonic pretreatment can be seen to be larger than that of the quartz sand without ultrasonic pretreatment and under low power ultrasonic pretreatment, which may be due to the more obvious effect of high-power ultrasound on the surface of the quartz sand. The signal intensity of the Si 2p peak of the quartz sand pretreated with ultrasound in 4% NaOH solution is weakened, which may be due to the reaction between the quartz sand and NaOH, and there is a Na 1s peak on the spectrum, which may be due to the residue of the agent caused by the quartz sand not being washed clean.

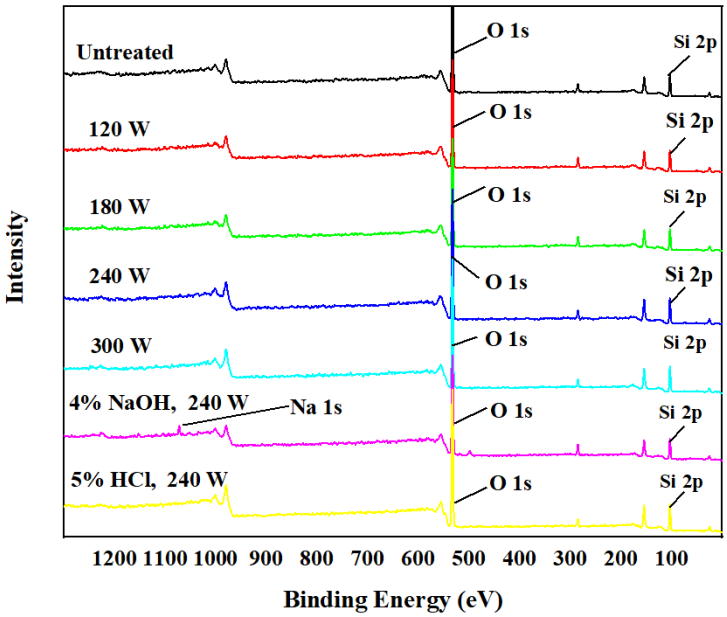


Fig. 12. XPS spectra of quartz sand treated with ultrasound at different power levels for 15 min

## 4. Conclusions

(1) In flotation experiments, ultrasonic treatment has little impact on the quartz recovery rate, and lower-power ultrasound shows limited improvement in quartz flotation. When the ultrasonic pretreatment power increases to 240 W, there is a significant enhancement in quartz flotation, with noticeable removal effects on $Fe_2O_3$, $TiO_2$, and $Al_2O_3$. However, at 300 W power, there is no further improvement in quartz flotation, making 240W ultrasonic pretreatment the most suitable choice.

(2) In an alkaline environment, ultrasonic pretreatment significantly enhances the removal effects of $Fe_2O_3$ and $TiO_2$. At a NaOH solution concentration of 4%, the $Fe_2O_3$ removal rate significantly increases to 69.4%, a 12.8% improvement compared to the blank control group and a 23.2% improvement compared to flotation without ultrasonic pretreatment. For $TiO_2$, the removal effect is 5.8% higher compared to the blank control group and 9.1% higher compared to flotation without ultrasonic pretreatment. In an acidic environment, the optimal HCl solution concentration is 5%, resulting in a removal rate of 76.6% for $Al_2O_3$, which is comparable to the blank control group. For $Fe_2O_3$, at a 5% acid concentration, the removal rate reaches 67.8%, an 11.2% improvement compared to the blank control group and a 21.6% improvement compared to flotation without ultrasonic pretreatment. The removal rate for $TiO_2$ also reaches 79.3%, a 4.6% improvement compared to the blank control group and a 7.9% improvement compared to flotation without ultrasonic pretreatment.

(3) Observations of the quartz sand surface morphology before and after ultrasonic pretreatment reveal a significant cleaning effect, facilitating better action of flotation reagents on the quartz sand surface. Elemental analysis of the quartz sand indicates that ultrasonic pretreatment does not directly remove the main impurities; Instead, the subsequent flotation effect is enhanced by optimizing the surface of the quartz sand.

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