



# Effects of alkyl ether amine and calcium ions on fine quartz flotation and its guidance for upgrading vanadium from stone coal

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## ABSTRACT

Great progress has been made in reverse flotation of fine quartz, especially many new reagents were proved their excellent effects. However, the influences of complex flotation system on fine quartz flotation still need to be scrutinized. In this study, alkyl ether amine (Flotigam EDA-C from Clariant) was used as a collector for the flotation of quartz. It shows better collectability for the flotation of quartz at about pH 10.0 ( $\pm 0.2$ ). The adsorption mechanism of EDA-C on quartz and the influence of calcium ions on the flotation of quartz were investigated by adsorption tests, zeta-potential measurements, FTIR spectra measurements, X-ray photoelectron spectroscopic and atomic force microscopy imaging. Results show that EDA-C adsorbs on the quartz surface in physical adsorption with no new products, changing its zeta potentials and increasing its hydrophobicity. Calcium ion concentration of 20 mg/L, 40 mg/L, 60 mg/L and 1 g/L can inhibit the quartz surface strongly and decrease its recovery sharply. Inhibition action of 2 g/L  $\text{Ca}^{2+}$  decreased, even disappeared when the collector concentration increased to 90 mg/L. Calcium ions changed the chemical environment of Si atoms and O1s on the quartz surface. After positively charged  $\text{CaOH}^+$  adsorbed on quartz surface, the amount of EDA-C adsorbed on quartz surface and the recovery decreased. It was greatly inspired to explore quartz separation application from these results.

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## 1. Introduction

Quartz is one of the most common impurities associated with the natural occurring ores, such as feldspar, mica, talc, pyrite, hematite, smithsonite, and apatite [1]. It needs to be removed in order to achieve a suitable concentrate for the economic extraction of the metal values. The separation of quartz from other minerals has attracted increasing interest in recent years, especially for the separation of feldspar and quartz due to their similar tectosilicate structure [2,3]. To obtain high purity quartz, Froth flotation is a generally used process by which the hydrophobic particles of a finely divided ore are separated from the hydrophilic particles. The addition of selective reagents induces hydrophobicity in the desired mineral particles, which get attached to air bubbles and report in the froth phase [4–6].

Quartz can be satisfactorily floated by anionic collectors, such as sodium oleate [7], sodium alkyl sulfonate [2,8], sodium alkyl sulfate [9], with metal ions as activators, and various amines which play a key role in the reverse flotation of iron ores. Primary monoamines and their corresponding acetate or chloride derivatives have been used as

the cationic collectors for a long time. These years, design and development of cationic collectors with such properties as environment-friendly, high-efficiency, hypothermia resistance, and high water-solubility, have become a hot area [10–13]. And series of novel cationic surfactants have been successfully synthesized and utilized as collector in separation of quartz from iron ores, such as Flotigam SA-B, Flotigam T2A-B, Collector 075/94, HOE F2835–B, Flotigam EDA-B, Flotigam EDA-3B, MG-98-A3, ECNA 04D, Nb 104, Nb112, Colmin C12, etc. [14]. Mixed anionic/cationic collectors were also frequently used in silicon minerals flotation [15–17]. However, there is always a need for developing highly selective collectors to reduce the overall cost of the process with an enhanced quartz recovery.

Metal ion activation has been a classic pretreatment method for quartz flotation [2]. In the practical mineral separations, unavoidable metal ions such as Fe (III), Mg (II), and Ca (II) can profoundly affect the flotation of minerals [18,19]. These ions can activate the gangue minerals, thereby increasing their flotation by promoting collector adsorption. Ejtemaiea et al. and Wang et al. [20,21] found that Fe (III) and Ca (II) ions improve the flotation of spodumene and beryl mineral sand. However, it largely depends on the type of collector used (i.e. anionic, cationic, or amphoteric) [13]. In most cases, metal ions are highly detrimental to the process during the flotation of the oxide minerals. The activation of the silicates by metallic cations impairs the

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selectivity of the separation of the oxides and silicates such as beryllium minerals from quartz [20]. All these metal ions, Fe(III), Mg(II), Ca(II), may have potential as activators in the flotation of albite, spodumene, and quartz minerals.

The objective of the present investigation is to understand the surface behavior of quartz, solution chemistry and effect of Ca(II) ions on quartz flotation and the underlying adsorption mechanism of Fm EDA-C in the quartz flotation. Flotation performance of single quartz using Fm EDA-C as a collector was assessed by flotation tests. Their adsorption on minerals was analyzed by zeta-potential measurement, Fourier transform infrared (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS), and atomic force microscopy (AFM). Effect of Ca (II) ion in flotation system on the quartz floatability was studied by different flotation environment.

## 2. Experimental

### 2.1. Materials

Quartz was obtained from mineral processing laboratory of the Wuhan University of Technology. The chemical analysis showed that it contained more than 99% SiO<sub>2</sub>, which was deemed sufficiently pure for research purposes. Fm EDA-C, an Alkyl Ether Amine (C<sub>10</sub>) provided by Clariant (China), was used as a collector for quartz. It is a new effective cationic collector used for quartz flotation. It has the foaming property after improvement. Other chemicals employed in the test are of A.R. grade. Single mineral experiments were carried out with distilled water only.

The quartz samples used for AFM imaging experiments were purchased from Yunnan Bao Yun Jewelry Company, China. The sample is finely polished, further cleaned by rinsing thoroughly with ethanol and water and a 1 cm × 1 cm sample is used for the AFM experiments.

The vanadium-bearing stone coal was collected from Teng-da Mining and Metallurgy Co. Ltd., Hubei, PR China. Around 200 kg of representative ore samples was crushed to below 2 mm size with two-stage jaw crusher and one-stage roll crusher. The crushed materials were firstly decarbonized in a SXZ-10-B muffle furnace at 700 °C for 90 min, the loss on ignition is 12–13%. Then the decarburization sample was mixed and divided into 200 g samples for flotation.

### 2.2. Methods

#### 2.2.1. Flotation

Single quartz flotation tests were conducted in a single flotation cell of 40 mL volume. In each test, the mineral sample of 2 g was put into flotation cell to disperse for 3 min, adjusted pH value for 3 min, and mixed with the collector Fm EDA-C for 5 min, floated for 5 min. Then the product was filtered, dried, weighed, and analyzed. The decarburization sample of stone coal was well mixed and divided into 200 g samples for quartz flotation, while NaOH and H<sub>2</sub>SO<sub>4</sub> were the modifiers, sodium silicate was the inhibitor, Fm EDA-C was the collector, and neutral oil (2# oil) was the foaming agent. The decarburization sample flotation was conducted in a single flotation cell of XFDII0.5 at a pulp density of 50%.

#### 2.2.2. Chemical analysis

The determination of vanadium, silicon and calcium grade was measured in accordance with Test Methods of Vanadium in Coal Standard (GB/T19226-2003) and potassium dichromate volumetric method. Others were performed with the Xios advanced X-ray fluorescence (XRF) analyzer.

#### 2.2.3. Adsorption tests

Hydroxamic acid concentration was determined using a Shimadzu 2600 UV-vis spectrophotometer at 507 nm based on the ferric hydroxamate method. Adsorption tests were conducted in a 100 mL

glass beaker. Conditioning procedures were similar to that used for micro-flotation tests. After that, 10 mL of the suspension was centrifuged at 9000 rpm for 10 min, and the separated liquid was collected to quantitatively analyze the octanohydroxamic acid concentration.

#### 2.2.4. Infrared spectroscopy

In order to characterize the nature of the interactions between the collector and quartz particles, the infrared spectra of collector as well as the samples with or without collectors pretreated were measured by the KBr technique. FTIR spectra of the samples were recorded with Nicolet Model Nexus 670 (USA) instrument using KBr pellet, at a resolution of 4 cm<sup>-1</sup>.

#### 2.2.5. Zeta potential measurement

The zeta potential of quartz was determined on Coulter Delsa440sx Instrument (Backman Coulter, USA). The measurements were conducted by taking 0.5 g of mineral in 100 mL of surfactant solution. NaOH (1%, mass fraction) solution and H<sub>2</sub>SO<sub>4</sub> (1%, mass fraction) solution were used for adjusting pulp pH value. An average of six measurements was taken for each mobility point.

#### 2.2.6. XPS analysis

XPS analysis was conducted using an ESCALAB250Xi (Thermo Scientific, UK) with Al K $\alpha$  X-ray source. The mineral samples were taken from flotation experiments then washed with distilled water to a similar pH to remove any suspended colloidal particles, finally they were introduced immediately into the fore vacuum of the spectrometers as a slurry.

#### 2.2.7. AFM imaging

Multimode 8 AFM (Bruker, USA) was used in the contact mode for imaging of the quartz surface. Triangular cantilevers with nominal spring constant of 0.12–0.58 N/m were used for surface image measurements. The AFM measurement was commenced after the quartz plate contacts the chemical's solution for a specific time. The images of the quartz surface were obtained at a scan rate of 1 Hz and a scan area of 5  $\mu$ m × 5  $\mu$ m. The images were processed offline using Nanoscope Version 5.31R1 software. Flattening and low pass filtering were applied to remove noise.

## 3. Results and discussion

### 3.1. Single mineral quartz floatability

To investigate the floatability of single mineral quartz, the flotation tests of quartz were carried out using Fm EDA-C as the collector in this part. The results are summarized in Fig. 1. Fig. 1a shows that when the pulp density is 10%, the recovery is highest. The recovery of quartz increases with the concentration of Fm EDA-C. When the concentration is more than 30 mg/L, the recovery reaches a plateau as shown in Fig. 1b. The effect of the pH on the flotation of quartz in the presence and absence of Fm EDA-C is shown in Fig. 1c. The results indicate that the recovery can be improved greatly in alkaline condition, especially at about pH 10.0 ( $\pm$ 0.2) in the presence of Fm EDA-C. The recovery decreases sharply to 77.1% at about pH 7.0. The highest recovery at pH 7.1 is below 30% in the absence of collector. This result indicates that quartz has no natural floatability. It also corresponds to the fact of other research results. The flotation results with the addition of Fm EDA-C or 2# oil in Fig. 1c also indicate that Fm EDA-C not only has collecting property but also has the foaming property. However, the 2# oil does not have the collecting property.

### 3.2. Adsorption of Fm EDA-C on quartz

The adsorption of Fm EDA-C on quartz is also studied at different pH values and surfactant concentrations, the results of which are shown in Figs. 2, 3. Adsorption amount of Fm EDA-C at

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