



Enhancing the purity of magnesite ore powder using an ethanolamine-based collector: Insights from experiment and theory

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ABSTRACT

Magnesite ore powder is the primary raw material used to prepare refractory magnesia. With the depletion of high-grade magnesite, methods to reduce the impurities and improve the quality of magnesite ore powder are urgently required. In this study, an ethanolamine-based surfactant, bis (2-hydroxyethyl) dodecylamine (BHDA), was first introduced into the flotation of magnesite ore as a novel collector, and the possibility of improving the purity of the ore was investigated by experiment and theory. Flotation tests, X-ray photoelectron spectroscopy, Fourier-transform-IR spectroscopy, and zeta potential measurements were carried out to study the behaviors and mechanisms by which the collector enhances the purity of magnesite ore powder. The flotation results revealed the noticeable difference in the floatability of quartz, magnesite, and dolomite when combined with the collector over a wide range of pulp pH values (4.18–9.17). The analytical results obtained from FTIR spectra, XPS, and zeta potential measurements indicate that BHDA was readily adsorbed on the surface of quartz, mainly by electrostatic and hydrogen bonding interactions, whereas it was adsorbed on the surfaces of magnesite and dolomite by weak electrostatic interactions. By introducing a hydroxyethyl group to the tertiary amine collector, more hydrogen bonds can form with the surface of quartz, strengthening the adsorption stability. Thus, compared to the traditional collector, dodecylamine, BHDA showed better desilication and purification performance for magnesite ore powder. Meanwhile, our results show that the simpler reagent system and process flow makes BHDA promising for the industrial purification of magnesite ore powder.

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1. Introduction

Magnesia (MgO) is one of the most important raw materials in the refractory industry. Magnesia refractory is usually used as the main material of lining materials for industrial kilns in high-temperature manufacturing industries, such as iron and steel making, nonferrous metals treatment, and the preparation of building materials and glass, as well as the petrochemical industries [1–3]. China possesses the most abundant reserves of magnesite, which is the largest producer and exporter of magnesia refractories in the world [4, 5]. Magnesite is the primary Mg-rich carbonate minerals, as well as the main source of magnesia. In recent years, with the depletion of high-grade magnesite, the extraction and use of low-grade magnesite ores have increased. However, approximately 500,000 tons of waste magnesite powder which during magnesite processing every year could be utilized [6]. Thus, the reduction in the impurity contents (for example, SiO₂ and Ca) to improve the quality of magnesite ore powder has become crucial.

Pure ore is required to meet the demands of the high-temperature manufacturing industry and also promote the environmentally friendly and sustainable development of the magnesium salt manufacturing industry in China [7, 8].

Froth flotation is the most widely utilized technique for the separation and enrichment of valuable minerals. As early as the 1930s, flotation had been used for the separation of silicate minerals from magnesite ore [8]. Recently, flotation is gradually accepted as the primary method of desilication and quality improvement of low-grade magnesite. The separation and enrichment are based primarily on the physicochemical differences between the surfaces of the valuable and gangue minerals [9, 10]. However, in addition to the natural characteristics of minerals, the surface properties are also affected by the surfactants. In particular, collectors are selectively adsorbed on the surface of minerals, enhancing the surface hydrophobicity. The hydrophobic mineral particles attach more air bubbles and float [11]. Hence, studies on the utilization of collectors for the efficient separation of magnesite ore have been carried out around the world. Due to the cationic collectors generally express the ability of high-efficiency separation, a series of novel cationic surfactants have been synthesized and proven to have outstanding flotation performance for minerals such as quartz.

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These surfactants include tertiary amines, amides, ester amines, polyamines, quaternary ammonium salts, and organosilicones [12–15]. Recently, the use of mixed collectors (amines/nonionic or cationic) has also been investigated [16–18]. However, studies concerning the flotation of magnesite have focused on the traditional cationic collector (dodecylamine, (DDA)), and most studies concerning the separation of fine particles have focused on the application of modifying agents such as depressants (inorganic salts or organic acids and salts) [19]. However, as early as 2001, a commercial mono ether amine as a collector was introduced in the flotation of magnesite, but few studies have been conducted and reported about the application of the novel collectors for the flotation of magnesite ore. Brezání showed that a gemini surfactant (benzyl dodecyl dimethyl ammonium bromide, BDDAB) could be efficiently utilized in the reverse flotation of magnesite ore [8, 20]. However, the mechanism of the selective adsorption of cationic collectors has not been verified. Most investigations into the adsorption mechanisms of cationic collectors only relate to the surface of quartz, which limits the application and development of novel collectors for the flotation of magnesite ore.

In this study, an ethanolamine-based surfactant, bis(2-hydroxyethyl)dodecylamine (BHDA), was used as a novel collector for the reverse flotation of magnesite ore powder. To investigate the mechanism of the purity improvement of the ore, experimental and theoretical investigations were carried out. BHDA, which has two hydrophilic groups, is an amino alcohol. Compared with dodecyl dimethyl amine, the addition of an alcoholic hydroxyl group to the molecule enhances its solubility and alters the flotation performance [21]. This paper aims to investigate the flotation behaviors and adsorption mechanisms of BHDA as a collector for quartz, magnesite, and dolomite. The selective adsorption behaviors and mechanisms on magnesite ore were systematically investigated by Fourier transform (FT) IR, X-ray photoelectron spectroscopy (XPS) analysis, and zeta potential measurements. Flotation experiments were also conducted to evaluate the flotation performance and the potential use of BHDA for the industrial purification of magnesite ore.

2. Materials and methods

2.1. Minerals and reagents

Hand-picked natural specimens of magnesite, dolomite, and quartz were obtained from Haicheng, Liaoning Province. The samples were crushed, ground in a porcelain laboratory ball mill and wet-sieved to obtain the $-74 + 15 \mu\text{m}$ fractions. High purity silica for flotation studies was prepared by leaching with hydrochloric acid. Magnesite and dolomite samples were further purified using a magnetic separator and shaking table. Chemical compositions of magnesite, dolomite, and quartz are shown in Table 1. As shown by the data in Table 1, the magnesite, dolomite, and quartz had high purity 99.07%, 99.32%, and 99.42%, respectively, and meet the requirements for the following studies.

The collector (BHDA, $\text{C}_{12}\text{H}_{25}\text{N}(\text{CH}_2\text{CH}_2\text{OH})_2$) with a purity of 99.5%, was purchased from Aladdin Reagents. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used to adjust the slurry pH. All chemicals used in the experiments were of analytical grade and obtained from Sinopharm. Distilled water was used throughout all the experiments.

Table 1
Chemical compositions of the pure minerals (wt%).

Sample	MgO	SiO ₂	CaO	Fe ₂ O ₃	Al ₂ O ₃	P ₂ O ₅
Magnesite	47.18	0.16	0.40	0.12	0.09	<0.01
Dolomite	21.28	0.39	30.66	0.06	0.07	<0.01
Quartz	0.006	99.42	0.004	<0.01	0.178	<0.01

2.2. Flotation tests

The flotation of single minerals and the separation of artificially mixed samples were carried out in a XFG₁₁₅₋₃₅ flotation machine (Jilin Exploration machinery plant, China) with a 30 mL flotation cell. In each flotation test, 5 g samples were introduced to the cell with 25 mL water and agitated with a mechanical impeller for 3 min at a speed of 1920 rpm. In addition, pH regulators were added to the cell to regulate the pulp pH and maintain the pH value in the process of flotation. Then, the collector was introduced into the pulp. After agitation for 1 min, flotation was performed for a total of 5 min. The concentrates and tailings were separately weighted and assayed after drying. All the presented results are the averages of duplicate flotation experiments (experimental error, $\pm 3\%$). Finally, the recovery was calculated on the basis of the weight and elemental contents of the products.

2.3. XPS measurements

XPS spectra were measured with America Thermo VG ESCALAB250 spectrometer using Al K_{α} X-rays (1486.6 eV) generated from the sputtering source at a power of 150 W (15 kV, 10 mA). The XPS measurements were performed inside the analysis chamber operating in a high vacuum of about 5.0×10^{-7} Pa. A binding energy of 284.8 eV was used as the C (1s) calibration energy. In each test, 1 g mineral samples were added to 50 mL aqueous solution and stirred for 0.5 h. Then, the samples were filtered, washed with distilled water three times, and vacuum dried at about 45 °C. The XPS spectra were immediately recorded to analyze the surface compositions.

2.4. FTIR spectra analyses

FTIR spectra of the samples were recorded by Nicolet 740 FT-IR spectrometer at 25 °C between 400 and 4000 cm^{-1} over KBr disc pellets. Prior to the test, the purified mineral samples were ground to $<15 \mu\text{m}$ in an agate mortar. Then, 1 g of the samples were added to 30 mL aqueous solutions with or without reagents and stirred for 0.5 h. Then, the samples were filtered, washed with distilled water three times, and vacuum-dried at around 45 °C.

2.5. Zeta potential measurements

The zeta potentials of the untreated minerals and those treated with the collector were measured using a Nano-ZS90 zeta potential analyzer (Malvern Instruments Ltd. Britain) at 25 °C. Prior to the test, the purified mineral samples (20 mg, below $-5 \mu\text{m}$) were added to a 50 mL aqueous solution containing 1×10^{-3} mol/L KCl as a supporting electrolyte and conditioned by magnetic stirring for 15 min. Then, the suspension was settled for 10 min, and the supernatant of the dilute fine particle suspension was prepared for zeta potential measurements. A series of pH values were prepared by adding 1% HCl or NaOH solutions to the slurry. At each condition, the zeta potentials of the minerals were used at least five times, and the averages were adopted as the final results. The repeated tests showed a measurement error of less than ± 2 mV.

3. Results and discussion

3.1. Flotation performance

The flotation performances of magnesite, dolomite, and quartz were investigated using BHDA as a collector, and the results are shown in Figs. 1 and 2.

The effects of the collector concentration on the flotation recoveries of magnesite, dolomite, and quartz at the natural pH of the slurries (7.04, 8.16, and 9.17, respectively) are shown in Fig. 1. The results indicate that the flotation recovery of quartz slowly increased with increasing BHDA concentration until maximum of 97.51% at a BHDA

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