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Utilization of novel surfactant N-dodecyl-isopropanolamine as collector for efficient separation of quartz from hematite



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ABSTRACT

In this paper, a novel surfactant N-dodecyl-isopropanolamine (NDIA) has been synthesized and utilized as a collector for flotation separation of quartz from hematite. The flotation performances and adsorption mechanisms of NDIA on quartz are investigated by flotation tests, FT-IR spectra studies, zeta potential measurements, and XPS analysis. Flotation results demonstrate that NDIA has excellent floatability and selectivity on quartz at a wide range of pulp pH (about 4.5–8.0). And the artificially mixed minerals of hematite and quartz could be separated effectively. The concentrate with iron recovery of 93.40% and iron grade of 67.16% is obtained at the slurry pH of 6.65 with 33.33 mg/L NDIA and 13.33 mg/L starch. FT-IR spectra studies, zeta potential measurements and XPS analysis reveal that NDIA could absorb on quartz surface in the forms of strong electrostatic and hydrogen bonding interaction.

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1. Introduction

Researches on iron ore flotation began in 1931 and have caused a lot of concerns recently. As such properties of complex intergrowth and fine dispersion of iron ore become serious, flotation, especially reverse flotation, has been considered as one of the most widely utilized technology for iron ore separation [1–3]. Flotation is achieved for physicochemical difference between desired and undesired mineral components, and flotation reagents especially collector play a key role in this process. Hence, studies on use of flotation reagents for mineral separation have been carried out widely around the world. Meanwhile, some researches confirm that reverse cationic flotation of quartz is more sensitive, comparing with reverse anionic flotation [4–7].

Cationic collectors, such as various amines, play a key role in reverse flotation of iron ores. 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. And series of novel cationic surfactants have been successfully synthesized and utilized as collector in separation of quartz from iron ores, such as quaternary ammo nium salts (CS-22, M-302), N-dodecyl-β-amino-propylamide, N-laurel-1,3-diaminopropane, N-(6-(hydroxyamino)-6-oxohexyl) octan-amide, N-dodecylethylenediamine, and N-(2-aminoethyl)-

1-naphthalene acetamide [2,8–12]. However, the existence of such disadvantages as high cost, cohesive bubble and poor selectivity has hindered the utilization of these cationic collectors [13]. Thus, development of new collectors for economic flotation of valuable minerals has been attempted by many investigators.

Recent research indicated that the insertion of polar groups into amine structure could promote its adsorption performance at liquid–gas interface, thereby decreasing the interface surface tension and reducing gas bubble coalescence [14]. Thus, hydroxypropyl group, which could improve water-solubility of surfactant, is introduced to dodecylamine molecule to form new cationic surfactant. And then, the conceived surfactant, N-dodecylisopropanolamine (NDIA), was synthesized and used as collector for separation flotation of quartz from hematite. The floatation performance and selectivity of NDIA on quartz were evaluated by flotation experiments, and the adsorption mechanism of this new collector on mineral surface was also discussed by FT-IR spectra studies, zeta potential measurements and XPS analysis.

2. Materials and methods

2.1. Minerals and reagents

Hand-picked natural specimens of quartz and hematite were obtained from Qidashan Iron Mine of Anshan Steel Company in China. Hematite was crushed, ground and wet-sieved to obtain the $-74 + 15 \,\mu$ m fraction. And then, shaking table was used to

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elevate the purity of hematite for flotation studies. The quartz sample was crushed, ground in a laboratory porcelain ball mill, and wet-sieved to obtain the $-74 + 15 \,\mu$ m fraction. High purity silica was obtained by leaching with dilute hydrochloric acid for flotation studies. Chemical compositions of hematite and quartz were analyzed and shown in Table 1. X-ray diffraction patterns were used to determine the mineral composition of hematite and quartz and the results were shown in Fig. 1. From Table 1 and Fig. 1, it can be concluded that hematite and quartz with high purities of 98.27% and 99.36% could meet the desirable requirement for the following studies. Artificially mixed mineral with Fe content of 41.27% was prepared by mixing hematite and quartz particles at the mass ratio of 3:2.

One novel hydroxypropyl amine surfactant, N-dodecylisopropanolamine (NDIA), was used as collector and synthesized in laboratory, and its structure was confirmed by Infrared spectrometer (Nicolet, FTIR-740, America), ¹H NMR (Bruker AvanceIII400, Switzerland) and MS Spectra (Bruker APEX IV FT-MS, America). HCl and NaOH were used for pH adjustment. All chemicals used in experiments were analytical grade and obtained from Sinopharm. Non-modified corn starch, provided by Qidashan plant, was used as depressant, and the starch solution was freshly prepared to avoid degradation. Distilled water was used during all the experiments.

2.2. Flotation tests

Flotation of single minerals and separation of artificially mixed samples were carried out in a XFG_{II} 5-35 flotation machine with a 30 mL flotation cell. The slurry was agitated with a mechanical impeller at speed of 1920 r/min. 5.0 g of the minerals were placed into the cell with distilled water and stirred for 2 min at room temperature $(25 \pm 2 \,^{\circ}\text{C})$. The pH adjustment and starch were added into the cell continuously with 2 min interval. And then, collector was introduced into the pulp with agitation of 1 min and followed by flotation for 5 min. The froth products and tailings were weighted and assayed separately after drying, and the recovery was calculated based on weight and elemental contents of the products obtained.

2.3. Froth collapse study

The static foam collapse tests were carried out in a 1000 mL measuring cylinder loading the froth products [8]. Froth products were supplied by the flotation of iron ore (the products of high-intensity magnetic separation process, Qidashan Iron Mine's Concentrator Branch) using NDIA (33.33 mg/L) or dodecylamine (DDA, 74.14 mg/L) as collector. The flotation of iron ore (300 g) were carried out in a XFD-0.75 L flotation machine. The height and volume variation of froth with time were recorded.

2.4. FT-IR spectra analysis

Fourier transform infrared (FT-IR) spectra of samples were recorded by Nicolet 740 FT-IR spectrometer at room temperature $(25 \pm 2 \text{ °C})$ in the range from 400 cm⁻¹ to 4000 cm⁻¹ over KBr disk pellets. Prior to test, the pure minerals were ground to less than 15 µm in an agate mortar. Then 1 g of the samples were added into

Table 1				
Chemical com	position of p	oure miner	als (wt.%).	
C 1	F 0	610	D O	6.0

Sample	Fe ₂ O ₃	SiO ₂	P_2O_5	CaO	Al_2O_3	MgO
Hematite	98.27	1.65	0.011	<0.01	0.049	<0.01
Quartz	0.43	99.36	0.005	<0.01	0.178	<0.01



Fig. 1. X-ray diffraction patterns of hematite (a) and quartz (b).

30 mL aqueous solution with or without 33.33 mg/L NDIA at pH 6.65, and conditioned for 0.5 h. After that, the samples were filtered, three times washed with distilled water, and dried in a vacuum oven at 35 °C for 24 h.

2.5. Zeta potential measurement

Zeta potentials of minerals before and after interacting with collector were measured by Nano-ZS90 zeta potential analyzer (Malvern Instruments Ltd. Britain). Finely ground minerals (20 mg) below $-5 \,\mu$ m were added to 50 mL aqueous solution containing 1×10^{-3} mol/L KCl as a supporting electrolyte, and conditioned by magnetic stirring for 15 min so that the suspension was homogenized. A series of pH values were acquired by adding 1% HCl or NaOH solution to slurry. At each condition, the zeta potentials of minerals were measured and an average value of at least ten individually measurements was accredited.

2.6. XPS measurements

XPS spectra were measured with America Thermo VG ESCALAB 250 spectrometer using Al K α X-rays (1486.6 eV) as 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 5.0×10^{-7} Pa. A value of 284.7 eV was adopted as the standard C (1s) to calibrate binding energy [15].

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