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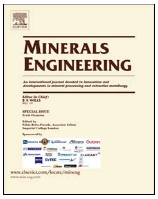

The flotation behavior and adsorption mechanisms of 2-((2-(decyloxy)ethyl) amino)lauric acid on quartz surface

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ABSTRACT

A novel surfactant 2-((2-(decyloxy)ethyl)amino)lauric acid (LDEA) – substituting 3-(decyloxy)propan-1-amine (DPA) on the α-carbon position of lauric acid was firstly synthesized and utilized as a collector for the flotation of quartz mineral at a relatively low temperature. The flotation performances and adsorption mechanisms of LDEA collector on quartz mineral surface were investigated by zeta potential measurements, Fourier transform infrared (FT-IR) spectroscopy, and X-ray photoelectron spectroscopy (XPS), in conjunction with the results of quartz micro-flotation tests. The flotation results showed that LDEA exhibited an excellent collecting performance on quartz mineral at a wide pulp pH range (about 6–11) with a low collector concentration 15.0 mg/L at a relatively low temperature 15 °C, where above 95.0% of the quartz mineral could be floated. Zeta potential measurements, FT-IR spectra, and XPS analyses reveal that the LDEA collector could be adsorbed on the surface of pure quartz in the forms of electrostatic and hydrogen bonding interactions at pulp pH 10.0.

1. Introduction

Flotation is a physicochemical separation technique based on chemical differences in the surface characteristics of the mineral species present in pulp. The process utilizes chemical additives to modify the minerals' surfaces to maximize the selective separation of mineral species (Wills and Napier-Munn, 2006). In the beneficiation of iron oxide resources (hematite and magnetite), anionic reverse flotation has been considered as one of the most widely utilized technologies in China (Luo et al., 2016; Weng et al., 2013). The main technical feature of this process is to add anionic collectors into the pulp of iron ores, in order to float out the main gangue quartz mineral, while the iron oxides are kept depressed with the help of starches (Cao et al., 2013; Lima et al., 2013; Liu et al., 2016).

The most extensively used anionic collectors are long carbon chain length fatty acids and their salts (Quast, 2006). In order to improve the solubility and activity of these type collectors, the flotation pulp should be maintained at a relatively higher pulp temperature (35–45 °C) which leads to a great deal of energy consumption and promotes the researches on design and development of new type collectors in hopes of finding a solution to cut the non-renewable energy consumption, thus minimizing the emission of carbon dioxide (CO_2) and other greenhouse gases for the protection of the global environment. Japanese scientist Ogata et al. (1979) found that the modified saturated fatty acids with chlorine (Cl) atom introduced to the α-carbon position could improve the solubility of these reagents. Also, the enhancement of electronegative of fatty acid by Cl atom resulted in a stronger adsorption among active sites of O atoms of carboxyl group with hydroxyl of quartz. Based on these findings, RA series (RA-315, RA-515, RA-715, and RA-915), LKY, and KS-II collectors were synthesized and utilized as the reverse flotation collectors (Lin et al., 1993; Mei et al., 2009; Zhang and Liu, 2003). Besides, bromine atom also has been introduced into the α-carbon position by solvent-free method (Hell-Volhard-Zelinski reaction), and the novel surfactant α-bromo fatty acids were obtained with the advantages of high yield, easy operation and realization in laboratory at ambient pressure (Luo et al., 2015; Zhu et al., 2015). Compared with α-chloro fatty acid, the Br modification product α-bromo fatty acids also show a good flotation ability for quartz mineral at a comparatively lower temperature (Luo et al., 2015; Zhu et al., 2015). In the meanwhile, the α-bromo fatty acids could also be intermediate products followed by displacement of bromide ion with hydroxyl (−OH) and amino group ($-NH_2$) to enhance the solubility in water solution (Wade, 2010).

The attempt of this paper is to substitute the 3-(decyloxy)propan-1-amine – containing imino group and a hydrophilic oxygen atom onto the α-carbon position of lauric acid that the new novel surfactant 2-((2-

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Table 1
The chemical composition of the single quartz (wt.%).

SiO_2	Al_2O_3	Na_2O	K_2O	Fe_2O_3	CaO	Loss
99.90	0.02	0.02	0.01	0.02	0.01	0.02

(decyloxy)ethyl)amino)lauric acid could be synthesized. Compared with the origin saturated fatty acids, the solubility and dispersity in water, adsorption ability on quartz surface, and foaming ability of froth stable layer may be enhanced for the modification (Huang et al., 2009). The collecting performance and adsorption mechanism of the new collector on quartz mineral surface were also studied by means of zeta-potential, FT-IR, and XPS measurements, in conjunction with the results of quartz mineral micro-flotation tests.

2. Experimental

2.1. Mineral

The pure quartz samples supplied by Anqian iron mine plant (Liaoning Province, PR China), were carefully hand-picked, crushed, ground, and wet-sieved to obtain the −0.10 + 0.038 mm size fraction for micro-flotation tests. Chemical compositions of quartz samples were shown in Table 1. As seen in Table 1, it is obvious to be seen that the quartz consists of 99.90 wt.% SiO_2, which meets the desirable requirement of purity for the experiments.

2.2. Reagents

The novel surfactant 2-((2-(decyloxy)ethyl)amino)lauric acid (LDEA) was firstly synthesized by α-ammonolyzed reaction using 3-(decyloxy)propan-1-amine (DPA) and α-bromolauric acid (α-BLA) in laboratory, the synthetic route of LDEA was given in Scheme 1. DPA was synthesized through decyl alcohol and acrylonitrile, and then were reduced by hydrogen gas under Raney-Ni catalysis (Qin, 2008). α-BLA was synthesized by Hell-Volhard-Zelinski reaction (Carey, 2008).

Decyl alcohol (79.14 g, 0.5 mol) and 3.73 wt.% potassium hydroxide (KOH) 10.0 mL solution (W_{KOH}: $W_{dodecyl\ alcohol} = 0.4\%$) was placed in a 250 mL flask. Under mechanical stirring, raising the temperature to 140.0 °C and kept for 1 h. After the reaction system temperature cooled down to 35.0 °C, the acrylonitrile (29.18 g, 0.55 mol) was added into the system dropwise in order to keep the system temperature at 50.0 ± 2.0 °C, preventing the self-polymerization reaction of acrylonitrile. After the addition, the mixture was kept stirring for 2 h to ensure synthesized reaction to be carried out thoroughly. Then, the mixture and catalyst Raney-Ni were moved into a high-pressure reactor, and the hydrogenation reaction was conducted under the conditions of stirring speed 800 rpm, 2.0 MPa and 130.0 °C. After the reducing reaction, the obtained DPA and former achieved product α-BLA (133.0 g, 0.48 mol) was dripped into a 500 mL three-neck round bottom flask, and then the mixture was agitated for 5 h at a certain temperature of 90 °C. Continually, the product was purified by adjusting the product solution to low pH 4.0 and high pH 12.0 successively to remove the unreacted α-BLA and DPA, respectively. H_2O was then removed from the mixture by atmospheric distillation at 100 °C for 1 h. Finally, the target product α-ammonolyzed fatty acid LDEA was obtained.

Potassium bromide (KBr, Sinopharm) of spectroscopic purity was used to conduct the experiments of infrared spectra. Potassium chloride (KCl, Sinopharm) of analytical grade was used as the background supporting electrolyte in the aqueous solution. Ultra-pure water was used in all tests.

2.3. Micro-flotation tests

Flotation tests were carried out in a 50 mL cell of a XFGII50 laboratory flotation machine. Prepared quartz samples (2 g) were placed in a plastic cell, in which 40 mL ultra-pure water was filled. The pulp was stirred for 2 min at a rotating speed of 1900 rpm before adding the HCl or NaOH solution to adjust the pulp pH to the specified value. The reagents were added into the cell every 2 min continually in the order of pH regulator and collector LDEA, respectively. The suspension was agitated for 3 min after adding all the desired amount of reagents, and the flotation pulp was conducted for 4 min. Finally, the froth products and tailings were weighed respectively after drying, and the recovery was calculated based on the weight of the products obtained.

2.4. Zeta potential measurements

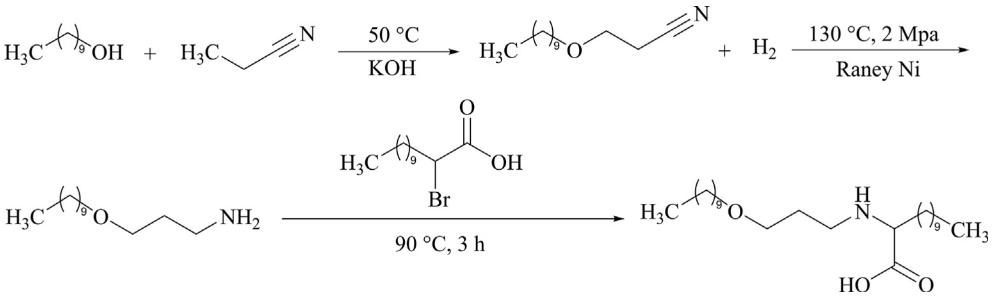
Zeta potentials of quartz mineral before and after treated by the collector LDEA in a 1 mM KCl background electrolyte solution were measured by Malvern Instruments Nano-ZS90 zeta potential analyzer. The suspension containing 0.04 wt.% −0.005 mm quartz solid was agitated for 15 min with a magnetic stirrer at room temperature 25 °C. Pulp pH was regulated with 0.10 M HCl or 0.10 M NaOH. The measurement tolerance was within ± 2 mV after at least 3 measurements.

2.5. FT-IR spectroscopy

Fourier transform infrared (FT-IR) spectra of samples were recorded by Nicolet 380 FT-IR spectrometer in the range of 4000–400 cm^{-1} with 4 cm^{-1} resolution using KBr pellets about 1 mg sample accompanied with 100 mg KBr usually. The mineral samples used for this purpose were ground to −0.002 mm in an agate mortar. Then, 2 g of mineral samples were added to 40 mL aqueous solution in absence and presence of 15.0 mg/L LDEA at pH 10.0, 15 °C. After being stirred for 0.5 h, the mineral samples were filtered, washed with distilled water for three times, and dried in a vacuum oven at 40 °C for 24 h (Huang et al., 2012; Jiang et al., 2011). 256 scans were collected for each specimen.

2.6. X-ray photoelectron spectroscopy

XPS spectra of quartz particles untreated and treated by 15.0 mg/L LDEA at pH 10.0 were collected from a surface of size about 2 mm × 2 mm on an America Thermo VG ESCALAB250 spectrometer using Al K_α X-rays (1486.6 eV) as the sputtering source at a power of 150 W (15 kV × 10 mA). The pressure in the analysis chamber was



Scheme 1. The synthetic route of LDEA.