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The flotation of low-grade manganese ore using a novel linoleate hydroxamic acid



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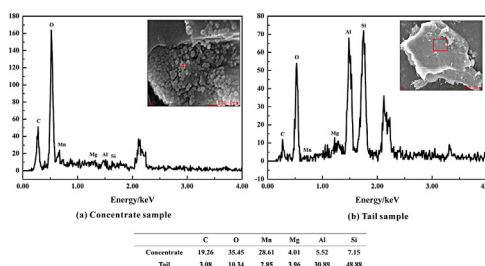
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HIGHLIGHTS

- LHA was investigated as a new collector for rhodochrosite flotation.
- The selectivity of LHA is better than that of the traditional collector oleate acid.
- We investigated key factors in flotation and the optimum values were obtained.
- Chemisorption is the major adsorption mechanism in the flotation.

GRAPHICAL ABSTRACT



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ABSTRACT

We designed and synthesized a novel linoleate hydroxamic acid (LHA) which demonstrated high selectivity and strong collecting capacity for the beneficiation of manganese ore by selective flotation. The performance of this LHA and oleic acid (OA) used for anionic froth flotation was compared, and the critical factors of rhodochrosite flotation were investigated. It demonstrated that the use of sodium carbonate instead of sodium hydroxide as the pH regulator, dosages of depressant and collector, and the addition of synergist are essential to the effective recovery of Mn in the flotation. And the concentrate grade of 18.3% Mn had been produced from a feed mixture with 10.7% Mn, and the Mn recovery could be achieved as highly as 97% when the LHA was used for the flotation separation of rhodochrosite under the optimum operating conditions. It was found that compared to OA, the novel LHA achieved superior results. Moreover, the analyses of Zeta-potential, Fourier Transform Infrared Spectroscopy (FT-IR), and X-ray diffraction (XRD) indicated that chemisorption accounted for the flotation mechanism. Besides, results of the Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) revealed that the concentrate obtained had significantly higher proportion of manganese-bearing grains (e.g., rhodochrosite and pyrolusite) relative to the tail and implied that Mn-LHA compounds or chelates might be produced through the interaction between the rhodochrosite and LHA.

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1. Introduction

Conservative estimates indicate that over 90% of Manganese resources in China are classified as low-grade poor manganese ores characterized by complicated composition and finely disseminated grains, which need to be beneficiated [1]. The conventional techniques used for manganese ore beneficiation include gravity separation, magnetic separation, flotation, pyrometallurgy and

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hydrometallurgy. Among them, froth flotation, regarded as an effective and favorable mineral processing method, shows various advantages over the other techniques in terms of applicability, adoptability, ease of operation, efficiency and economics [2–4]. As a selective processing method, flotation has been used to obtain specific separations from complex ore (e.g., copper-zinc, lead-zinc), and has been expanded to include oxides, oxidized minerals, and non-metallic ore [5–9]. However, very limited information on rhodochrosite flotation is available in the literature.

Flotation reagent plays a critical role upon the separation process of flotation [10]. Traditional collectors including oleic acid, tall oil, oxidized paraffin wax soap and other anionic organic compounds have been used for rhodochrosite flotation but the results from these collectors are far from satisfactory [11]. On the other hand, hydroxamic acid used as the collector has been extensively applied in the flotation of rare earth minerals due to the fact that it has nitrogen and oxygen atoms, which contain lone pair electrons to coordinate with metal atoms [12–14]. Investigations on the interaction of hydroxamic acid with manganese ore, was not systematically investigated, in spite of its use as the flotation collector of manganese ore even at plant level.

In this paper, a novel hydroxamic acid had been synthesized for the flotation separation of rhodochrosite from manganese ore, and the flotation performance by the resultant was studied to compare with that by the oleic acid. As an aliphatic hydroxamic acid, the structure of LHA is characterized by a single double bond being replaced by conjugated double bonds, which is similar to oleic acid. In hydroxamic acid –CONHOH group, both O and N atoms are recommended as coordination atoms in all kinds of complexes, and the characteristic structure determines the strong chelating ability of the hydroxamic acid with metal ions [15]. The objective of this work was to develop a novel collector with better selectivity for the flotation of rhodochrosite against quartz and illite in the manganese ore. Flotation tests were conducted to investigate the flotation performance of LHA. The effects of important factors such as pH regulator type, temperature, dosages of depressant and collector, and the addition of synergist were investigated on the separation of rhodochrosite from manganese ore and the optimum values of these parameters were reported. Besides, zeta-potential, FTIR, XRD, and SEM-EDX were performed to study its mechanisms [16].

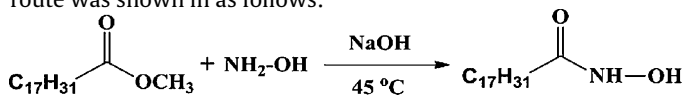
2. Experimental and methods

2.1. Materials and reagents

The manganese ore samples used in the flotation studies originated from Changsha, China. X-ray fluorescence (XRF) chemical analysis of the ore samples indicated that the samples contained 13.8% MnO, 39.07% SiO₂, 6.02% CaO, 7.30% Al₂O₃, 6.62% Fe₂O₃, 2.55% MgO, and 3.30% K₂O. Combining with the information obtained from XRD, possible compositions of the ore samples were rhodochrosite (20–22%), quartz (26–28%), illite (29–31%), albite (12–14%), pyrite (5–7%), gypsum (3–5%), pyrolusite (2–4%). The reagents utilized in this study were listed in Table 1.

2.2. Synthesis of LHA

Linoleate hydroxamic acid was synthesized referring to the synthesizing method of hydroxamic acid [17,18], and its synthetic route was shown in as follows:



All chemicals used for synthesis were analytically pure. Infrared spectrum apparatus (Nicolet6700) was applied to identify the

Table 1
Reagents used in experiments.

Chemical	Purity (%)	Source	Role
Oleic acid	Analytical purity	Made in Tianjin	Collector
Linoleate	>70	Synthesized in lab.	Collector
hydroxamic acid			
sodium dodecyl benzene	Chemically Pure	Made in Shanghai	Synergist
Sulfonate sodium dodecyl sulfate	Chemically Pure	Made in Shanghai	Synergist
Tween-80	Chemically Pure	Made in Shanghai	Synergist
Tritonne X-100	Chemically Pure	Made in Shanghai	Synergist
Sodium hydroxide	Analytical reagent	Made in Shanghai	pH regulator
Sodium silicate	Industrial goods	Made in Hunan	Depressant
Pin oil	Analytical reagent	Made in Shanghai	Frother

structure of synthesized products, and High Performance Liquid Chromatography (Labtech, LC600, China) was used to analyze their purity.

One equivalent of linoleic acid in absolute methanol was mixed with 0.4 equivalent of 98% sulfuric acid, and then the solution was refluxed with stirring for 3 h. The excess methanol was eliminated on a rotary evaporator, and sulfuric acid was neutralized with an aqueous solution of 10% sodium bicarbonate until formation of carbon dioxide ceased. The residue was purified by distillation to afford methyl linoleate (with 80% yield and 95% purity).

One equivalent of hydroxylamine hydrochloride in absolute ethanol was added to ethanol solution containing one equivalent of sodium hydroxide. Sodium chloride precipitated from the solution was removed by suction filtration, and then ethanol solution with one equivalent of potassium hydroxide was added to the filtrate. Subsequently, synthesized methyl linoleate dissolved in ethanol was added to the hydroxylamine solution and refluxed with stirring for 2 h at 45 °C. The precipitate was recrystallized in ethyl acetate and acidified to pH 4.5 by using aqueous solution 5% hydrochloric acid to get the desired product LHA (with 60% yield, and 80% purity).

2.3. Flotation test

Flotation tests of manganese ore were carried out to investigate collecting performance of the novel synthesized LHA compound. The samples were ground to 90% passing 74 μm. The pulp was conditioned at 25 wt.% solids in an XFD-type laboratory flotation cell with a volume of 0.75 L for 3 min and the impeller speed was fixed at 2000 rpm. Analytical grade reagents sodium hydroxide or sodium carbonate solution were used as a pH regulator by adjusting the pulp pH to a desired value and was conditioned for 10 min. Sodium silicate was used as a depressant and conditioned for 5 min, after which the slurry was conditioned with collectors (LHA or OA) for 5 min. Finally, pine oil was used as a frother (120 g/t) with a conditioning time of 2 min. The froth was scraped for 10 min to collect the concentrate. The concentrate (floated fractions) and tailing (unfloated fractions) were dried separately and weighed to calculate the productivity and the recovery (R%). R% is calculated using the following formula

$$R\% = \frac{\text{Concentrate Grade\%} \times \text{Concentrate Weight\%}}{\text{Feed Grade} \times \text{Feed Weight\%}} \times 100 \quad (1)$$

2.4. Zeta potential measurement

Zeta potential was measured with zeta potential analyzer (Malvern, Zetasizer Nano, United Kingdom). The suspension containing 0.01% solids by weight less than 5 μm was dispersed in a beaker for 15 min with the use of 1 × 10⁻² mol/L NaOH and HCl solution as a pH regulator. After measuring the pH, collectors at a

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