



Flotation behavior of four C18 hydroxamic acids as collectors of rhodochrosite



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ABSTRACT

The number of double bonds effects on the flotation of low-grade poor manganese ores using hydroxamic acid was studied. Four types of hydroxamic acids, namely stearic hydroxamic acid (SHA), oleic hydroxamic acid (OHA), linoleate hydroxamic acid (LHA) and α -Linolenic hydroxamic acid (α LHA) were synthesized and tested to float manganese ores containing 10.7% Mn. In all of the collectors screened, the study showed that LHA is a particularly good collector and has an excellent selectivity in rhodochrosite flotation. The surface tension experiment indicates that the values of surface tension cannot always function as a reliable indicator of the effectiveness of flotation collectors. The results of zeta potential and Fourier transform infrared spectroscopy (FTIR) demonstrate that chemisorption accounts for the flotation mechanism. Besides, the formation of five-membered chelate ring of rhodochrosite with LHA was proposed. The scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) data indicate that the flotation separation using LHA is significantly efficient, and the adsorption of LHA onto rhodochrosite leads to the formation of hydrophobic agglomerates of rhodochrosite particles which enhance the floatability of rhodochrosite.

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1. Introduction

China has abundant manganese resources, more than 90% of the resources are classified as low-grade poor manganese ores with complicated composition and finely disseminated grain.

The hydroxamic acid is a kind of organic compounds containing —CONHOH groups which has been extensively applied in the flotation of minerals as collectors. Both O and N atoms in the —CONHOH groups are recommended as coordination atoms in all kinds of complexes and its characteristic structure determines the strong chelating ability of the hydroxamic acid with metal ions. In recent years, the chelating performance of hydroxamic acids with transition metals including Cu, Ni, Zn, Fe, Cr, Mn and Co has been studied through theoretical and experimental methods (Boddien et al., 2011; Buglyó et al., 2007; McSweeney et al., 1997; Zhao et al., 2013; Zhou et al., 2015).

Fuerstenau (1984) and Quast (2000) had made comprehensive reviews on the application of alkyl hydroxamic acid collectors in mineral flotation. These reviews indicated that detailed basic studies on the flotation with alkyl hydroxamic acid reagents were

carried out mainly on minerals containing either transition metal or rare-earth metal ion in their mineral lattice. In a recent study, Jiang et al. (2011) designed compounds containing mono-carboxyl group and double-hydroxycarbonyl groups, which demonstrated better selectivity on the flotation of diasporite against aluminosilicate minerals. The study of chelation between hydroxamate and alkaline-earth metals (Be, Ca) and heavy metal (Pb) has also been reported (Ali and Fridgen, 2011; Bátka and Farkas, 2006). These investigations posited that one metal ion can chelate 2 or 3 hydroxamate molecules to form five member rings complexes according to the valence of metals ions. For example, Cu (II), Ni (II) and Zn (II) can chelate two hydroxamate molecules forming complexes with two five-member rings. Correspondingly, complexes with three five-member rings were formed for Fe (III) and Co (III). However, very limited information on the nature of the hydroxamate–rhodochrosite interaction is available in related literature.

In this study, hydroxamate compounds with different degrees of unsaturation were designed in order to develop collectors with better selectivity on the flotation of rhodochrosite. The difference in their collecting performance was due to the differing polar group, like electronegativity and hydrophobicity. Collectors used here are 18-carbon chain fatty hydroxamic acids including SHA, OHA, LHA, and α LHA. The numbers of double bonds in the chain are zero (SHA), one (OHA), two (LHA) and three (α LHA) respectively. The different degrees of unsaturation was expected to play

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an important role during flotation separation of rhodochrosite from manganese ores. The flotation efficiency of these four collectors would be compared and analyzed in terms of the structure–activity relationship of collectors. In addition, surface tension, zeta potential, infrared spectra and SEM–EDX techniques were used to study the mechanism of rhodochrosite flotation. A proposed mechanism is used to explain the flotation results obtained.

2. Experimental methods

2.1. Synthesis of collectors

Four hydroxamic acids were synthesized following the procedure reported by Jiang et al. (2012) and comprehensively tested as collectors. The synthesis scheme and the structures of the hydroxamic acids used in our study are given in Table 1 along with their abbreviated names. All synthesized hydroxamic acids were white crystalline solids and were recrystallized in ethyl acetate and acidified to pH 4.5 by using aqueous solution 5% hydrochloric acid to give the desired product. The solids had a very long shelf-life and did not decompose. All chemicals used for synthesis were analytically pure. Infrared spectrometer (Nicolet6700) was applied to identify the structure of synthesized products and High Performance Liquid Chromatography (Labtech, LC600, China) was used to analyze their purity.

2.2. Rougher flotation tests on a laboratory scale

Real manganese ores originated from Hunan province in China were crushed to about –3 mm. Based on the X-ray diffraction (XRD) analysis, the composition of the manganese ores were rhodochrosite (20–22%), quartz (26–28%), illite (29–31%), albite (12–14%), pyrite (5–7%), gypsum (3–5%), pyrolusite (2–4%). X-ray fluorescence (XRF) chemical analysis 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.

The ores were ground to 90% passing 74 μm and flotation tests were carried out in flotation machine (RK, XFD-0.5L, China). The pulp was conditioned at 25 wt.% solids and the impeller speed was fixed at 2000 r/pm. Analytical grade reagent sodium carbonate was used as a pH regulator by adjusting the pulp pH to 9 and was conditioned for 5 min. A desirable sodium silicate (1000 g/t), collector (1250 g/t), Tween-80 (400 g/t) and pin oil (120 g/t) were added and agitated in sequence for 5, 5, 3 and 2 min respectively. The floated fraction was then gathered for 5 min. The concentrate (floated fractions) and tailing (unfloated fractions) were dried separately and weighed to calculate the yield and the recovery.

2.3. Surface tension measurement

The surface tension was determined at 20 °C applying the Wilhelmy plate method using a JK99B Digital Tensionmeter with

0.2 wt.% hydroxamic acids. A platinum plate length of $w = 19.9$ mm and $d = 0.2$ mm was used. During measurement, complete wetting of the platinum plate occurs (i.e. the contact angle is 0°) (Schuster et al., 2014). Hydroxamic acids solutions were measured at 20 ± 0.2 °C in triplicate. The average standard deviation between these measurements was 0.08 mN/m. The plate was first cleaned by washing with DI water and ethanol and then flamed to remove remaining ethanol before each measurement.

2.4. Zeta potential measurement

Zeta potential was measured with zeta potential analyzer (Malvern, Zetasizer Nano, United Kingdom). The suspension containing 0.01% Mn ore 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 concentration of 2.0×10^{-4} mol/L were added while stirring on a magnetic stirrer for 3 min. The suspension was immediately transferred to the tested square sample vessel for the measurement of the zeta potential. The experiments were carried out at room temperature (20 °C). Three independent suspensions were prepared in the same condition and measured to obtain the average value of the zeta potential.

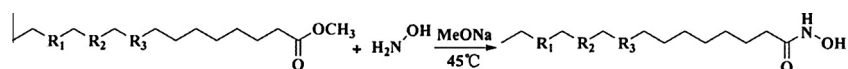
2.5. Infrared spectra

In order to characterize the nature of the interaction between the collector and the Mn ore, the infrared spectra of mineral samples conditioned with or without the collector were obtained by Nicolet6700 infrared spectrometer. Spectra were acquired in the range of 4000–500 cm⁻¹ with 0.5 cm⁻¹ resolution and processed using OMNIC ESP software. To obtain the tested sample, 50 mg of the mineral samples below 5 μm were added to 30 mL aqueous solution with or without 2.0×10^{-4} mol/L collectors at pH 7 and 25 °C, stirred for 30 min, filtrated, and then dried in a vacuum oven at 35 °C for 24 h. A tablet was prepared by mixing 5 mg of the tested sample with about 500 mg KBr which was dried at 70 °C for 3 h. Then pressed into a transparent tablet of 1 cm in diameter, and then the infrared spectra were immediately recorded.

2.6. SEM–EDX

SEM–EDX was used to observe the adsorption of these four different collectors onto the real Mn ore. After adsorption, the ore samples were thoroughly washed with deionized water of the same pH to remove loosely attached composition. Before observation, the floated fractions were dried in an oven at 60 °C and gold coated under vacuum using an ion coater. Images were acquired using a Hitach SU8010 field emission scanning electron microscope.

Table 1
Scheme for synthesis and the structures of hydroxamic acids synthesized.



#	R ₁	R ₂	R ₃	Name	Abbreviation
1	–CH ₂ –CH ₂ –	–CH ₂ –CH ₂ –	–CH ₂ –CH ₂ –	Stearic hydroxamic acid	SHA
2	–CH ₂ –CH ₂ –	–CH ₂ –CH ₂ –	–CH=CH–	Oleic hydroxamic acid	OHA
3	–CH ₂ –CH ₂ –	–CH=CH–	–CH=CH–	Linoleate hydroxamic acid	LHA
4	–CH=CH–	–CH=CH–	–CH=CH–	α-Linolenic hydroxamic acid	αLHA

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