



Behaviour of ilmenite as a gangue mineral in the benzohydroxamic flotation of a complex pyrochlore-bearing ore



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ABSTRACT

One challenge identified in the flotation of niobium bearing minerals with hydroxamates is the recovery of gangue minerals which can result in concentrate grade dilution. Micro-flotation of single and mixed minerals and bench scale flotation tests on a low-grade complex pyrochlore-bearing ore were conducted to assess the flotation behavior of ilmenite as a gangue mineral in the flotation of pyrochlore using benzohydroxamic acid (BHA) as a collector and sodium hexametaphosphate (SHMP) as a dispersant/depressant. In micro-flotation, high dosages of BHA resulted in high pyrochlore recovery and low ilmenite recovery at pH 8. It was observed that the order of addition of SHMP in relation to BHA had a strong effect on pyrochlore recovery, with high recovery maintained only when SHMP was added after BHA. When ilmenite and pyrochlore were floated in the same system, it was found that reagent addition order played an important role in selective flotation of pyrochlore. Flotation of pyrochlore was more selective over ilmenite when SHMP was added before BHA, similar to a cleaner flotation stage in real-ore flotation. Contrary to micro-flotation results titanium recovery was high (more than 60% TiO₂ recovery in most cases) under similar pulp conditions in bench scale flotation, an indication that to some extent titanium minerals were recovered by a mechanism other than genuine flotation. Similarly, in mixed mineral micro-flotation, the recovery of ilmenite increased in the presence of pyrochlore. Two possible scenarios, one physical and the other chemical in nature, have been proposed to explain this observed phenomenon. First, it is possible that moderately floatable titanium mineral particles were recovered in the froth by association with highly floatable pyrochlore, particularly at starvation levels of a dispersant (SHMP) through the hydraulic entrainment of fines. The second possibility is that titanium minerals experienced inadvertent activation in multi-mineral systems, perhaps with metal-hydroxamates as the activating species, forming hydroxamate complexes involving ferric and/or niobium species. This research area is under further investigation.

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1. Introduction

Pyrochlore (Na,Ca)₂Nb₂O₆(OH,F) is the most economically important niobium-bearing mineral and is typically upgraded by flotation using an amine-based collector. The pulp pH is maintained between 5.5 and 7 in the rougher circuit and is gradually dropped to as low as pH 2 in the final cleaner stage (Gibson et al., 2015a; Bulatovic, 2010; Guimares and Weiss, 2003; Filho et al., 2002; Oliveira et al., 2001; Gendron et al., 1981). This bene-

ficiation process is reported to be associated with many challenges, including (Ni, 2013; Gibson et al., 2015d):

1. Complicated flowsheets.
2. Process sensitivity to the presence of slimes.
3. Need for anticorrosive equipment to withstand acidic pulp conditions.
4. Difficulty meeting final concentrate purity specifications.

Globally, there are numerous high grade undeveloped pyrochlore deposits which cannot be effectively upgraded by flotation because ultra fine grinding is required to achieve adequate liberation of highly complexed pyrochlore grains (de Oliveira Cordeiro et al., 2011; Bulatovic, 2010; Aral and Bruckard, 2008). Advancement of these deposits is dependent on the development

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of beneficiation process that can tolerate slimes and selectively recover fine grained pyrochlore from associated gangue minerals.

Flotation using hydroxamic acids (chelating reagents) as collectors has been demonstrated as an effective means to upgrade ultra fine grained material (Pradip and Fuerstenau, 1988). Although hydroxamates have historically not been widely used in industry in western countries due to lack of commercial availability, an alkyl hydroxamic acid product named 'IM-50' is reported to have been in commercial use in Russia (former Soviet Union) since the 1960s and China since the 1970s (Nagaraj, 1987). Hydroxamic acids are now commercially available worldwide (from reagent manufacturers such as Cytec), warranting more testwork on real ore systems.

Hydroxamates can form complexes with nearly all transition metals, although stability is stronger with iron, copper and highly charged cations than with other transition metals (niobium, tantalum, titanium etc.) (Nagaraj, 1987). As a result, selective flotation of transition metals from one another using hydroxamic acids is challenging and tends to vary depending on the type of hydroxamic acid used. Factors affecting collector selectivity include both mineral and chelate solubility; chelate solubility being primarily affected by the stability constant (Nagaraj, 1987).

The application of alkyl hydroxamic acid as a collector in flotation of pyrochlore-bearing ores at the laboratory scale has been well documented (Ni and Liu, 2012a; Gorlovskii et al., 1968). Other hydroxamic acid types such as benzohydroxamic acid have shown to be effective for pyrochlore recovery through single mineral micro-flotation studies by Espinosa Gomez et al. (1987) and on real ore systems by Gibson et al. (2015a). Independent of the type of hydroxamic acid, a major issue identified in the flotation of niobium bearing minerals with hydroxamates is the recovery of other transition metal gangue minerals, such as magnetite, hematite, pyrite, ilmenite and rutile (Ni, 2013) which result in concentrate grade dilution.

The present work aims to investigate the flotation of behavior of ilmenite as a gangue mineral in the flotation of pyrochlore using benzohydroxamic acid as a collector.

2. Materials and methods

2.1. Mineral sample preparation

A high purity sample of pyrochlore flotation concentrate was obtained from laboratory and pilot plant flotation testwork conducted at SGS in Lakefield, Ontario as this mineral was not available at sufficiently high purity from other sources. In order to ensure de-activation/purification of the pyrochlore concentrate, it was leached with hydrochloric acid at a concentration of 20 g/L at 20% solids concentration for 4 h at a temperature of 50 °C. The leach residue was then filtered and washed five times with de-ionized water. The dried leach residue was then heated to 200 °C for 45 min to further ensure destruction of any remaining organics on the mineral surface. The purified mineral sample contained ~86% pyrochlore assaying 42.8% Nb determined through semi-quantitative XRD and X-ray Fluorescence, respectively. The ilmenite sample was from mineral storage at the Mining Department at Queen's University. It indicated 35.6% Ti. Subsequent XRD analysis indicated rutile and pseudorutile as minor impurities. In preparation for testing, the ilmenite was de-slimes after repeated ultrasonic treatment and decantation/washing at each particle size of interest. The XRF results for the pyrochlore concentrate and ilmenite samples are presented in Table 1.

Mineral samples were prepared as two size fractions for microflotation tests. The 'coarse' particle size range was -150 µm/+53 µm, and the 'fine' particle size range was -53 µm/+20 µm.

Table 1
XRF Analysis of pyrochlore and ilmenite used in micro-flotation tests.

XRF results	Assay (%)	
	Pyrochlore	Ilmenite
Nb	42.8	–
Nb ₂ O ₅	61.2	–
SiO ₂	4.80	0.95
Al ₂ O ₃	0.63	1.01
Fe ₂ O ₃	3.11	31.5
MgO	0.07	0.26
CaO	9.95	0.14
Na ₂ O	2.78	0.12
K ₂ O	0.34	0.05
TiO ₂	3.39	61.5
P ₂ O ₅	0.19	0.27
MnO	0.06	1.48
LOI	3.68	2.06

Samples were ground using a porcelain mortar and pestle and required microsieves.

2.2. Micro-flotation

Micro-flotation tests were carried out in a 70 mL modified Halimond tube (Partridge and Smith, 1971). The shaft of the micro-flotation cell was approximately 250 mm long and opened into a rounded concentrate launder. A medium porosity piece of fritted glass was located approximately 10 mm above the gas inlet. A schematic of the micro-flotation cell together with a photo showing the cell in action is illustrated in Fig. 1. A Teflon magnetic stirring bar from Fisher Scientific was used to suspend mineral particles in the cell during testing. For all tests, the magnetic stir bar was rotated at a rate of 1000 rpm.

The image in Fig. 1 (right) shows the separation of pyrochlore (brownish froth) from ilmenite (black particles at the bottom remaining mostly unfloatable). It is important to note the presence of practically barren bubbles in the collection zone separating the concentrate product and tails after about 30 s into flotation (pH: 8.1, BHA: 1.04×10^{-3} M, MIBC: 60 ppm).

For all tests the cell was filled with approximately 40 mL of de-ionized water. The pH in the cell was measured and then adjusted using sodium hydroxide to reach alkaline conditions. The sample was conditioned at pH 8 for two minutes. Benzohydroxamic acid collector with 99% purity from Alfa Aesar (BHA at 2% by wt.) was then added and the sample was conditioned for an additional three minutes. The BHA dosage ranged from 1 kg/tonne (1.04×10^{-4} M) to 20 kg/tonne, based on weight of the mineral added (1 g). The sodium hexametaphosphate (or SHMP (NaPO₃)₆ purchased as 'Calgon'), dosage ranged from 0.5 kg/tonne (1.17×10^{-5} M) to 5 kg/tonne. The conditioning period was exactly the same whether the SHMP was added before or after the collector. MIBC was added as a frother in the last minute of conditioning at a rate of 60 mg/L (provided by a tiny drop of pure MIBC from a syringe). After conditioning, the air was turned on and adjusted to a flowrate of 36 mL/min. Concentrate was collected from the overflow launder for 1 min while water (de-ionized) was added at the top of the cell (~70 mL/min) to facilitate a continuous overflow of the concentrate slurry into the launder. Washing water was added only to the launder and was not used to change the cell level during concentrate collection. In the tests involving sodium hexametaphosphate (SHMP), its addition was made directly into the cell in powder form.

2.3. Bench scale flotation

A Box-Behnken experimental design was conducted on a low-grade, niobium-bearing carbonatite gravity tailings product to

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