



Zeta potential study of pentlandite in the presence of serpentine and dissolved mineral species



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ABSTRACT

Pentlandite and serpentine specimens were obtained from a sample of an ultramafic deposit to explore interactions that might be factors in processing by flotation. Zeta potential was measured under conditions designed to progressively approach the environment under which flotation will be attempted: *viz.* pentlandite and serpentine alone with indifferent background electrolyte; pentlandite conditioned in the presence of serpentine with indifferent background electrolyte; pentlandite conditioned in the presence of serpentine with supernatant derived from an ore suspension as background electrolyte. The “single mineral” experiments showed that hetero-aggregation is expected and was confirmed in the “mixed mineral” experiments. In the presence of supernatant uptake of $Mg(OH)_2$ precipitates was detected and confirmed by tests using solutions doped with Mg ions and by surface analysis using scanning electron microscopy.

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1. Introduction

Since its introduction over a century ago, flotation has become one of the most important techniques for mineral beneficiation. It has expanded into other areas, such as water treatment, deinking of paper and separation of plastics for recycling (Nguyen, 2013). In the mineral processing industry, the process requires size reduction of the ore (commonly to less than 100 μm) to liberate the valuable minerals. The underlying principle is straightforward: suspended particles are introduced to tanks where air is introduced to form bubbles. The surface of a particular mineral (commonly the valuable one) is rendered hydrophobic, thus particles of this mineral will preferentially attach to air bubbles, forming aggregates. These aggregates will rise to the top of the tank, where they are collected as the concentrate.

An important aspect regarding the separation via flotation is related to the electrical state of the surface, responsible of some relevant behavior of the system; surface charge and surface potential may determine particle-chemical interactions (*e.g.* collector adsorption) and particle-particle interactions (*e.g.* hetero-aggregation).

Electrokinetic phenomena (*e.g.* electrophoresis, streaming potential and electro-osmosis) are manifestations of the electrical of particle charge, thus the techniques associated with them are

important. Electrokinetic studies (*viz.* zeta potential) of minerals are frequently used to predict and understand their behavior in aqueous systems.

Examples of such surface chemistry studies are numerous, including: adsorption of flotation reagents (Moudgil et al., 1988; Fuerstenau, 2001; Jordens et al., 2014); oxidation of sulfide minerals (Prestidge and Rowlands, 1997; Fullston et al., 1999; Peng and Zhao, 2011); slimes coating (Hunter and Neville, 1980; Vergouw et al., 1998a,b; Feng et al., 2012); accidental activation of gangue (Fuerstenau and Palmer, 1976; Malysiak et al., 2004; Ejtemaei et al., 2012); and exchange of released ions among sulfide minerals (Zhang et al., 1992; DiFeo et al., 1997; Ikumapayi et al., 2012).

Experiments, however, are often performed under ideal conditions (*e.g.* pure minerals using an indifferent electrolyte as background), which are far from actual flotation conditions. Vergouw et al. (1988a,b) studied the agglomeration of several sulfide minerals through zeta potential analysis of mixed mineral systems. They observed electrokinetic behavior that suggested surface oxidation, which was attributed to galvanic interactions between the minerals.

In addition to the galvanic interactions, effects between minerals and chemical species (such as those present in the process water or product of mineral dissolution) should be considered. Acar and Somasundaran (1992) showed that predictions based on single mineral tests often failed in the mixed mineral case, due to the fact that most minerals release some ion species to solution. The released species can undergo reactions such as hydrolysis,

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complexation, adsorption and precipitation. The nature of the complex equilibria involving such reactions determine the interfacial properties of the particle, and consequently its floatability. It is therefore necessary to mimic the pulp environment as closely as possible.

In this paper, zeta potential measurements were used to study pentlandite, Pn, $((\text{Ni}, \text{Fe})_9\text{S}_8)$ from an ultramafic ore, which is the main nickel phase in this type of ore. The study attempted to approximate to a more realistic system, specifically, the presence of serpentine ($\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$), the main gangue in the ore, and some mineral dissolved species, released from main gangue minerals. Electron microscopy was used to support the electrokinetic measurements. The approach used to study this particular ore may be used to detect problems in the processing of other ores.

2. Experimental

2.1. Minerals

Pentlandite was isolated from a sample of concentrate obtained by processing an ultramafic ore from Thompson (Manitoba, Canada) in the Vale mini-plant (Xu et al., 2003). The concentrate contained mostly pentlandite, pyrrhotite and serpentine. The concentrate was screened and the fine material ($-75 \mu\text{m}$) discarded. Pentlandite content was upgraded by gravity by means of a Mozley table and magnetic concentration using a lab scale magnetic drum separator (Carpco, USA, model WD20). The $-106 + 75 \mu\text{m}$ fraction was cleaned with acetone (Sigma-Aldrich, HPLC grade) to remove flotation reagents and stored in a freezer to limit further surface oxidation. The elemental analysis of the sample by atomic absorption spectroscopy (AAS) (Varian, USA, model AA240FS) is shown in Table 1 along with electron microprobe (JEOL, Japan, model JXA-8900) analysis of 20 pentlandite grains. The slightly elevated iron content of the sample (and low nickel assay) indicates some residual contamination, primarily by pyrrhotite.

Serpentine was isolated from the same ultramafic ore sample by gravity and magnetic separation. Examination by X-ray diffraction (Phillips, the Netherlands, model P1770, with rotating Cu anode set at 40 kV and 20 mA; X'Pert Quantify and X'Pert High Score software were used for data acquisition and phase identification) showed clinochrysotile with minor pyrrhotite. Clinochrysotile is the most common of the three known polytypes of chrysotile, a fibrous polymorph of serpentine, with a 2-layer monoclinic cell (Wicks and Whittaker, 1975).

Both minerals were ground to produce a $-25 \mu\text{m}$ fraction for electrophoretic studies and a $+45/-75 \mu\text{m}$ fraction as the 'coarse' fraction for the mixed mineral tests.

2.2. Zeta potential

2.2.1. Single minerals

An electrophoretic method (Brookhaven Instruments, USA, model ZetaPlus) which uses the Smoluchowski approximation

Table 1

Measured elemental composition of the sample determined by AAS compared to ideal pentlandite composition and composition determined by microprobe analysis on identified pentlandite grains.

| Pentlandite | Fe | Ni | Co | Zn | Cu | S | Insoluble |
|----------------------------------|-------|-------|------|------|------|-------|-----------|
| Ideal composition ^a | 31.26 | 32.85 | – | – | – | 35.89 | – |
| Electron microprobe ^b | 30.70 | 35.00 | 1.20 | – | 0.07 | 33.00 | – |
| Atomic absorption spectroscopy | 40.35 | 30.99 | 0.88 | 0.15 | 0.04 | N.D. | 8.56 |

^a Ideal is based on $((\text{Ni}, \text{Fe})_9\text{S}_8)$.

^b Electron microprobe analysis based on the analysis of 20 pentlandite grains.

was used to measure the zeta potential from the measured electrophoretic mobility. Suspensions were prepared using 0.1 g of $-25 \mu\text{m}$ solid in 500 mL of deionized water (resistivity, $18.3 \text{ M}\Omega/\text{cm}$). The suspensions were conditioned for 24 h before measurement to reach chemical equilibrium based on prior research (Alvarez-Silva et al., 2010). The pH was adjusted using either KOH or HNO_3 over the pH range 3–10. The pH was recorded 15 min after adjustment. The results reported are the average of a minimum of two full repeats with the 95% confidence interval indicated.

For pentlandite, prior to suspension preparation approximately 0.2 g of mineral was ultrasonicated (Crest Ultrasonics, USA, model 275DA) several times to eliminate pentlandite fines; the pentlandite was then placed in a flask with dilute HCl solution (pH ca. 2) and agitated on an orbital shaker model G-33 (New Brunswick Scientific, USA) to dissolve any oxidation products. The sample was finally filtered and rinsed several times with distilled, deoxygenated water and placed in a desiccator. When dry, the pentlandite was ground to $-25 \mu\text{m}$ in an agate mortar and pestle and 0.1 g placed in the test solution (N_2 purged for 20 min).

The supernatant was prepared from the ore (P_{80} ca. $100 \mu\text{m}$) suspension (10% by weight solids) agitated for 1 day. The solution was recovered by filtration (first, filter paper grade 1 and, then, a nylon syringe filter with pore size: $0.45 \mu\text{m}$). The solution was analyzed by atomic absorption spectroscopy (for metal ions), argentometric titration (for chloride ions) and turbidimetry (for sulfate ions).

The tested solutions were: KNO_3 (10^{-2} mol/L and $5 \times 10^{-3} \text{ mol/L}$); supernatant; and, in the case of pentlandite, magnesium added as $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (Anachemia, analytical grade). In the latter two cases, to adjust the ionic strength 10^{-2} mol/L KNO_3 was added to the solution.

Chemical analysis by atomic absorption spectroscopy (for the metals) and titrimetric analysis (for Cl^- and SO_4^{2-}) determined the composition of the supernatant. Magnesium, calcium and chloride ions were dominant, with concentrations of 88.5, 37.6 and 332.5 mg/L , respectively. Analysis was conducted for metal ions, including Cu, Fe, Ni, Zn, Si and Al, which were not detected.

2.2.2. Mixed minerals

The technique was based on work by Zhang et al. (1997). Different size fractions of pentlandite and serpentine ($-25 \mu\text{m}$ and $+45-75 \mu\text{m}$) were mixed to facilitate subsequent separation prior to zeta potential measurements. The coarse fraction (either pentlandite or serpentine) was allowed to settle and a sample of the still-suspended fine particles was taken for zeta potential measurements. The series of background test solutions for the mixed mineral case were as described above.

2.3. Scanning electron microscopy

For scanning electron microscopy, pentlandite ($-106 + 75 \mu\text{m}$) was conditioned in a similar manner as in the zeta potential experiments. Samples were dried in a desiccator and placed in holders before being gold coated. When the pentlandite was pre-conditioned with serpentine, the mineral particles were rinsed with the solution in which they were suspended (but free of solids). In this way, the slimes that were not attached to the surfaces were removed. The Philips XL30 field emission gun system scanning electron microscope (FEM-SEM) was used for morphological characterization (secondary electrons) and elemental analysis (energy dispersive spectroscopy, EDAX system).

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