



Surface properties of fractured and polished pyrite in relation to flotation



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ABSTRACT

This research was designed to answer the question: can conventionally polished mineral surfaces be used in electrochemical studies in relation to flotation where minerals are fractured? Pyrite surfaces were prepared by fracturing and polishing, separately, and then subjected to electrochemical studies including open circuit potential (OCP), cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) measurements as well as X-ray photoelectron spectroscopy (XPS) analyses. Differences on polished and fractured pyrite surfaces were observed in the initial stage of electrochemical measurements, but became minor after conditioning in solution for a short time as the surfaces tended to reach an equilibrium quickly. In the presence of xanthate, dixanthogen formed on both the fractured and polished pyrite surfaces through xanthate oxidation. Immediately after fracturing, pyrite surfaces displayed the stoichiometry nearly in accordance with FeS₂, while polished pyrite surfaces showed an iron loss and the formation of polysulfide. However, the surface composition of the fractured pyrite and polished pyrite became similar after conditioning in buffer solution. It was also similar to that on samples directly taken from flotation. It is concluded that polished pyrite surfaces can be used in electrochemical studies representing fractured pyrite surfaces in relation to flotation.

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1. Introduction

Sulfide minerals, such as chalcopyrite (CuFeS₂), chalcocite (Cu₂S), covellite (CuS) and galena (PbS), are a class of minerals containing sulfide (S²⁻) as the major anion. They are economically important as the source of base metals. Flotation which exploits the difference in surface wettability has been widely used to recover sulfide minerals from their ores for over a century. Xanthate (ROCSS⁻) is the most extensively used collector in the flotation of sulfide minerals to render the value mineral surface hydrophobic. The adsorption of xanthate on sulfide mineral surfaces is an electrochemical process involving the oxidation of xanthate coupled with oxygen reduction (Haung and Miller, 1978; Leppinen et al., 1989; Miller et al., 2002). During flotation, sulfide minerals can also be oxidized to form hydrophobic sulfur-rich species or hydrophilic sulfoxo species on the surfaces and therefore display different flotation behaviors. Due to the electrochemical reactions taking place in the flotation of sulfide minerals, electrochemical measurements on sulfide mineral surfaces are the key to understand the underpinning mechanisms responsible for the recovery or rejection of sulfide minerals in flotation.

Commonly used electrochemical techniques include cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), open circuit potential (OCP), polarization curves, potentiometry, coulometry, etc. For example, Tolley et al. (1996) and Giannetti et al. (2001) used CV to study the oxidation and reduction of sulfide minerals, Hicyilmaz et al. (2004) used CV to study copper activation on pyrite surfaces, and a number of other researchers used CV to study the interaction of collectors and depressants with sulfide minerals (Janetski et al., 1977; Huerta-Cerdán et al., 2003; Guo et al., 2015; Mu et al., 2016a,b). EIS has been used to study the physical and chemical processes occurring at mineral/electrolyte interfaces. De Wet et al. (1997) and Mu et al. (2016b) investigated the surface modification of pyrite by cyanide and lignosulfonate biopolymers using EIS to explore their depression effects in flotation. Liu et al. (2011) used EIS to evaluate the oxidative dissolution of pyrite. OCP measurements have been used to characterize the state of mineral surface oxidation under open circuit conditions. Mielczarski and Mielczarski (2005) examined the potential variation of sulfide minerals alone and in galvanic contact by open circuit potential. Huang and Grano (2005) and Qin et al. (2015) used polarization curves to quantify the galvanic interaction such as the galvanic current between grinding media and sulfide minerals. In all these studies, mineral electrodes were prepared and their surfaces were refreshed by wet polishing before any electrochemical

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measurement. This is a standard procedure to conduct the electrochemical studies on sulfide minerals.

However, surface polishing is different from mineral grinding which prepares the surface for flotation by fracturing. Velásquez et al. (2002) studied the properties of polished and fractured enargite surfaces by XPS analyses. They found that the fractured surface revealed the stoichiometry in accordance with its chemical formula (Cu_3AsS_4), while the polished surface shows a loss of As and Cu leading to the formation of polysulfide at the sample surface. In another study, the fractured and polished pyrite electrodes showed different impedance due to the different surface morphologies and chemical states (Mendiratta, 2000). However, fracturing the surface for electrochemical studies is impractical since it is impossible to refresh a fractured surface or prepare a new one with the same physiochemical properties such as surface area and surface topography. It is noted that the studies by Mendiratta (2000) and Velásquez et al. (2002) were not conducted within the context of mineral flotation requiring the conditioning and the presence of oxygen. In fact, it has been widely reported that during wet grinding the loss of metallic ions into solution occurs and polysulfides also form on sulfide minerals (Buckley and Woods, 1987; Ahlberg et al., 1990; Tao et al., 2003; Chandra and Gerson, 2010). Therefore, the conventional surface treatment by polishing for electrochemical studies related to mineral flotation cannot be judged inappropriate based on the studies by Mendiratta (2000) and Velásquez et al. (2002).

In this study, electrochemical studies were conducted on both polished and fractured pyrite electrodes in parallel. Unlike the studies by Mendiratta (2000) and Velásquez et al. (2002), this study considered the flotation condition, i.e., the presence of oxygen and xanthate, and pulp conditioning. The surface properties of polished and fractured pyrite electrodes were also compared with the surface properties of pyrite particles directly taken from a flotation cell. The objective of the research was to answer the question: can conventionally polished mineral surfaces be used in electrochemical studies in relation to flotation where minerals are fractured?

2. Materials and methods

2.1. Materials and reagents

Pyrite was obtained from GEO discoveries, Australia. The chemical composition of the pyrite sample analyzed by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) is shown in Table 1. The elemental content of iron and sulfur is 45.4 wt.% and 50.5 wt.%, respectively. The analysis of the sample reveals the composition of 96 wt.% pyrite with minor quartz, galena and copper minerals.

The collector, potassium amyl xanthate (PAX), was provided by Orica Australia Pty Ltd. It has been widely used in pyrite flotation. 0.1 M potassium dihydrogen orthophosphate (KH_2PO_4) buffer solution was prepared with deionized water to maintain pH at 5.0 at which pyrite displays good floatability. All solutions used in the experiment were prepared with concentrated analytical grade reagents, diluted with deionized water and prepared just prior to the experiment.

2.2. Methods

2.2.1. Electrochemical measurements

In this work, open circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) measurements were conducted. The experiment was performed with CHI 920D Scanning Electrochemical Microscope (CH Instruments, US) with a conventional three-electrode electrochemical cell. A double layer wall glass reactor was used as the electrochemical cell with an effective volume of 200 mL. A Ag/AgCl electrode with 3 M KCl as electrolyte, a platinum plate electrode with a surface area of 1 cm^2 and the fractured/polished pyrite electrode were used as the reference, counter and working electrode, respectively. Potentials were measured against the Ag/AgCl reference electrode which has a potential of +220 mV against a standard hydrogen electrode (SHE) (Bates and Macaskill, 1978).

Pyrite samples were cut into thin rods of approximately $3 \times 3 \times 10 \text{ mm}$ using a low speed diamond saw. A copper wire was attached to one of the $3 \times 3 \text{ mm}$ faces by silver conductive epoxy (MG chemicals). The electrical contact was covered with an electrochemically inert epoxy resin (MG chemicals). To make the pyrite electrode to be polished, the sample was mounted in a glass tube using the same non-conductive epoxy resin leaving one face of the pyrite exposed. The surface was regenerated by wet polishing using 1200 grit silicon carbide paper and rinsed with deionized water for several times (Mu et al., 2016b). To make the fractured pyrite electrode, the sample was mounted in a glass tube using the non-conductive epoxy to cover approximately half of the electrode with the other half protruding out. A fresh surface was created when the electrode was fractured by blowing a sharp tap on the protruding part through a cell port (Mendiratta, 2000). The polished pyrite electrode and fractured pyrite electrode were referred as to PPE and FPE, respectively. Studies conducted by Mendiratta (2000) and Velásquez et al. (2002) have shown that different electrode preparation procedures lead to differences in surface morphologies and chemical state and further affect their electrochemical responses.

The experiments were conducted in open air like flotation. The electrodes were inserted into solution immediately after the generation of new surfaces. OCP sweep was conducted before each type of electrochemical measurements. The impedance spectra were obtained at the OCP by applying a sinusoidal excitation signal of 10 mV in the frequency range from 10 mHz to 10 kHz. The EIS diagram was displayed as Bode and Nyquist plots and analyzed using the ZView software. In CV studies, cycles were performed from the OCP to 0.6 V on an anodic scan, then to -0.6 V on a cathodic scan and then back to the OCP at a scan velocity of 20 mV/s. Five cycles were performed for each test and the second cycle was adopted because the shape and the peak intensities representing oxidized or reduced species became stable and similar since the second scan. Experiments were performed in the absence and presence of PAX. When PAX was added, the pyrite electrode was conditioned for 15 min in electrolyte solution before the electrochemical measurement.

2.2.2. X-ray photoelectron spectroscopy (XPS)

XPS measurements were performed with a KRATOS Axis Ultra (Kratos Analytical, Manchester, UK) with monochromatic Al $K\alpha$

Table 1
Chemical compositions of the pyrite sample.

Mineral	Species present (wt.%)									
	Fe	S	Cu	Bi	Pb	Al_2O_3	SiO_2	Ti	Zn	
Pyrite	45.40	50.50	0.10	0.02	0.13	0.03	0.35	0.04	0.05	

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