



Pyrite oxidation inhibition by organosilane coatings for acid mine drainage control



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ABSTRACT

By using electrochemical techniques and conventional chemical leaching approach, the potential of organosilane coatings to suppress the pyrite oxidation under acidic conditions was investigated, including γ -mercaptopropyltrimethoxysilane (PropS-SH), γ -aminopropyltrimethoxysilane (APS) and vinyltrimethoxysilane (VTMS). The coated pyrite samples were analyzed by Fourier transforms infrared reflection (FT-IR) spectroscopy and X-ray photoelectron spectrometer (XPS). The results from electrochemical tests have indicated that all the studied coatings could suppress the oxidation of pyrite and the inhibition efficiency should follow the sequence of PropS-SH > VTMS > APS. The results of chemical leaching experiments have showed that pyrite oxidation was decreased by 49.4%, 71.4% and 89.2% (based on Fe release) respectively by using APS, VTMS and PropS-SH coatings. The mechanism of inhibition action could possibly be the formation of a cross-linking network of Si–O–Si and Si–O–Fe bonds on the pyrite surface which could restrain the permeation of aggressive species.

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1. Introduction

Pyrite (FeS_2) is a common metal sulfide mineral in tailings and valuable mineral raw materials (Peppas et al., 2000). Pyrite is unstable in oxidizing environments. When exposed to liquid water and oxygen, pyrite can undergo rapid weathering (Joeckel et al., 2005). Weathering of pyrite results in the production of sulfuric acid and dissolved ferric ions, which in turn increase the dissolution of pyrite and accelerate the release of toxic metals from coexisting minerals (Jiang et al., 2000). The toxic heavy metals of pyrite leachate could enrich in organisms, on the other hand, it could enter into the human body through food chain and do harm to the health of people (Sánchez-Chardi et al., 2009). These processes lead to the formation of acid mine drainage (AMD) (Akcil and Koldas, 2006; Sheoran and Sheoran, 2006). In addition, fewer studies (Sracek et al., 2004; Strömberg and Banwart, 1999) have documented the higher pyrite content in waste rocks, the faster release of heavy metals on account of acid production. These are the reasons why many researchers have selected pyrite as research subject to study to AMD formation. AMD is often characterized by low pH water with elevated concentrations of iron, sulfates and heavy metals. As a result, AMD is a serious and persistent problem in watersheds with active or abandoned mines.

In the past decades, several post-treatments have been proposed to control AMD (Coulton et al., 2003; Kalin and Caetano Chaves, 2003). The most commonly used treatment method is chemical neutralization using a suitable alkali such as lime, soda ash, caustic soda and magnesium oxide (Caraballo et al., 2009; Mackie and Walsh, 2012). This method works well for the remediation of AMD, however, it has main drawbacks of high cost for large doses of alkaline materials and producing bulky iron-rich sludge as secondary pollutant (Johnson and Hallberg, 2005). These drawbacks limit the use of chemical neutralization on a large scale.

Recently, methods for the source control of AMD by suppressing the oxidation of sulfide minerals have been developed. One such treatment involves the passivation of pyrite, in which a thin organic or inorganic protective coating is formed on the surface of pyrite to prevent its contact with the air, water and other oxidants (Lu et al., 2001). To date, numerous passivation agents have been reported, including silicate (Kargbo and Chatterjee, 2005), phosphate (Kargbo et al., 2004; Zhang et al., 2003), 8-hydroxyquinoline (Lan et al., 2002), triethylenetetramine (TETA) (Chen et al., 2006; Liu et al., 2013) or sodium triethylenetetramine-bis(dithiocarbamate) (DTC-TETA) (Shu et al., 2013) that create ferric silicate, ferric phosphate or organic iron complexes on the surface of pyrite and subsequently reduce the rate of pyrite oxidation. Unfortunately, each of these passivation agents has their own disadvantages. For example, the formation of phosphate, silicate and 8-hydroxyquinoline coatings require initial surface oxidation using

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hydrogen peroxide (H_2O_2), which is difficult to handle in real application. Other passivation agents, such as triethylenetetramine and sodium triethylenetetramine-bisdithiocarbamate (DTC-TETA), are toxic to the environment. Thus, it is necessary to seek more environmentally friendly alternatives. Organosilanes, a kind of harmless and environmentally friendly materials, have been widely applied in anticorrosion field of metals and their alloys, which are identified as excellent coupling agents owing to generating Me–O–Si bond on the metal substrate and simultaneously creating a crosslinked Si–O–Si network (Zhou et al., 2013; Zhu and van Ooij, 2003) to protect metals efficiently against corrosion. However, knowledge on the application of organosilanes for suppression of pyrite oxidation is scarce. Additionally, most of the protection effects of coatings on pyrite have been evaluated by chemical methods. Pyrite dissolution is usually interpreted as electrochemical behavior (Ogunsola and Osseo-Asare, 1987), but only a limited number of investigations on pyrite passivation by electrochemical methods have been reported.

In the present work, three kinds of organic silanes with different functional groups, including γ -mercaptopropyltrimethoxysilane (PropS-SH), γ -aminopropyltrimethoxysilane (APS) and vinyltrimethoxysilane (VTMS), were selected as coating agents to suppress oxidation of pyrite for remediation of AMD. A preliminary assessment of inhibitory efficiencies for pyrite coatings was provided by using electrochemical tests, such as cyclic voltammetry (CV), Tafel polarization and electrochemical impedance spectroscopy (EIS). Traditional chemical leaching tests were also used to further verify the effects of organic silanes on the rate of pyrite oxidation. Furthermore, the chemical characteristics of these organic silane films were analyzed with X-ray photoelectron spectrometer (XPS) and Fourier transform infrared reflection (FT-IR) spectroscopy. The aim of this study is focusing on seeking an effective passivant to suppress oxidation of pyrite. There is a need for more systematic studies on the application of selected passivant to sulfide mineral wastes and the simplification of coating procedure in the future research.

2. Experimental

2.1. Materials preparation

The pyrite sample used in the study was purchased from ChangSha Mineral market. The chemical composition analyzed by X-ray fluorescence (Table 1) shows that the purity of FeS_2 is 99.56% and the main impurity is Al_2O_3 . Pyrite samples with fine crystallization were cut into cubes with side length of 1 cm and polished sequentially into mirror-like surface with waterproof abrasive papers (grit size 600–2000). After polishing, the pyrite cubes were rinsed with ultrapure water and acetone to remove any species that may have remained on the electrode surface. The remaining samples were ground with an agate mortar and then sieved to isolate particles with diameter less than 75 μm . All samples prepared were stored in a vacuum desiccator at room temperature. All reagents used in the experiment were of analytical grade and ultrapure water was used to prepare solutions.

Table 1
Chemical composition of the studied pyrite sample.

Compound	Mass (%)
FeS_2	99.56
Al_2O_3	0.35
NiO	0.08
P_2O_5	0.01

2.2. Coating processing

The silanol solutions were prepared by adding organosilane compound (PropS-SH, APS or VTMS) to a mixture of ultrapure water and ethanol. The volume ratio of organosilane/ultrapure water/ethanol was 1:1:8. Hydrochloric acid was used to adjust the pH to 4. The silanol solutions were stirred for 1 h at 40 °C to make it workable. Then pyrite cubes or pyrite powders prepared previously were dissolved in the silanol solutions and then the solutions were kept stirring for 2 h at 50 °C after adjusting the pH to 9 with 0.5 M ammonium hydroxide. The coated samples were separated from solutions and dried at 100 °C for 12 h.

2.3. Anticorrosive effect experiments

2.3.1. Electrochemical test

A conventional three-electrode system was adopted in the electrochemical experiments with pyrite electrode as working electrode, Pt foil (15 mm \times 15 mm) as counter electrode and saturated calomel electrode (SCE) with a Luggin's capillary as reference electrode. Pyrite electrode was prepared as follows: each coated or uncoated pyrite cube was electrically connected to a copper wire with conductive silver adhesive and mounted in epoxy resin with one side exposed.

The electrochemical measurements were performed at CHI 660 Electrochemical Workstation at room temperature and all potentials quoted in this paper were referenced to the SCE. The electrolyte was 0.2 M sodium sulfate solution with pH of 2, which was adjusted by sulfuric acid. Cyclic voltammetry tests were conducted at a sweep rate of 100 mV/s and the scan ranged from -0.7 V/SCE to $+0.7$ V/SCE. Tafel polarization curves were recorded over the range of OCP \pm 0.2 V/SCE at a scan rate of 1 mV/s. The EIS tests were measured at open circuit potential over the frequency range of 10⁵ Hz–0.1 Hz with the AC signal amplitude of 10 mV. The EIS data were analyzed by using ZSimpWin 3.00 software.

2.3.2. Chemical leaching test

Chemical leaching test was operated by adding 1.0 g sample of coated and uncoated pyrite into 120 ml 3 wt% hydrogen peroxide solution with the natural pH of 3.1. The mixture was placed in vapor-bathing constant temperature vibrator with rotating speed of 150 rpm and the constant temperature is 25 °C. A 5-ml sample of leaching solution was collected at the following time: 0.5, 1, 2, 4, 6, 8, 10, 12, 24, 36, 48 and 60 h. Prior to analysis, the collected leaching solution was filtered through a 0.22 μm pore size filter to get rid of impurities. The pH value, total Fe and SO_4^{2-} concentration of the solution were measured. The pH value of the leaching solution was determined by a waterproof hand-held pH/mV meter (Eutech instruments pH 310, USA). Total Fe of the leaching solution was analyzed by o-phenanthroline spectrophotography (UV-752N, Shanghai Analytical Instrument Co. Ltd., China) and the absorbance reading was taken at 510 nm. SO_4^{2-} concentration of the leachate was determined by the baryta yellow spectrophotometric method. All chemical leaching tests were repeated 3 times to ensure repeatability and accuracy of the measurements.

2.4. Analytical methods

FT-IR Spectrometer (Nicolet 380, USA) was applied to characterize the chemical structure of the coated pyrite samples. A measured amount of organosilane coatings or pyrite samples were mixed with KBr powder and pressed into pellets at the pressure of 8 MPa. The FT-IR spectra were collected in a transmittance mode from 4000 to 400 cm^{-1} .

The surface compositions of coated and uncoated pyrite were analyzed by X-ray photoelectron spectrometer (XPS). The XPS

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