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Pyrite oxidation inhibition by hydrophobic films for acid mine drainage control at the source

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Abstract: Acid mine drainage (AMD), which is also known as acid rock drainage (ARD), can cause serious environmental pollution, especially for surrounding aquatic and terrestrial ecosystems due to its low pH, high metal and sulfate concentration. Acid mine drainage is an urgent environmental problem for the worldwide ore mining industry. In this paper, we demonstrated that hydrophobic films can inhibit the oxidation of pyrite-bearing tailings to achieve the control of at-source AMD. The results of chemical leaching testing showed that the hydrophobic films formed by linoleic acid can suppress the oxidation of pyrite-bearing tailings and reduce the AMD production. In addition, the presence of hydrophobic films of linoleic acid on the surface of pyrite-bearing tailings was confirmed by the results from Fourier transform infrared (FTIR) analyses and scanning electron microscopy with energy dispersive spectrometry (SEM/EDS).

Keywords: hydrophobic film, pyrite, suppression, linoleic acid, acid mine drainage

1. Introduction

A large amount of tailings and waste rocks containing metal sulfides can be produced from the mining and mineral processing of sulphides, precious metals, uranium, or coal ores, especially pyrite and pyrrhotite. The annual emissions of global mine tailings have exceeded 10 billion tons (Adiansyah et al., 2015). At present, China has more than 8,000 state-owned mines, 110,000 township enterprises (Pan et al., 2014), more than 25 billion tons of tailings, and about 12,000 tailings reservoirs (Yin et al., 2011). When these metal sulphide minerals (such as pyrite or pyrrhotite) in tailings are exposed to oxygen, water, or microorganisms, acid mine drainage (AMD) is produced by the oxidation via inorganic and biologically mediated pathways (Singer and Stumm, 1970). Acid mine drainage (AMD), which is also known as acid rock drainage (ARD), has low pH and high metal sulfate concentration (Parbhakar-Fox and Lottermoser, 2015). Acid mine drainage can cause serious environmental pollution, especially for soil, water resources, and aquatic communities (Galhardi and Bonotto, 2016). It is considered as an urgent environmental problem for the worldwide ore mining industry, especially in the process of mining, processing or disposal of sulphide-containing ores (Singer and Stumm, 1970; Sarmiento et al., 2011). In order to control AMD, numerous end-of-pipe treatment and at-source control techniques have been studied in the past (Johnson and Hallberg, 2005). The aim of the end-of-pipe treatment techniques is to control the AMD after it is produced (Luptakova et al., 2010). However, once AMD is generated, the treatment is difficult and expensive (Aguiar et al., 2016). The at-source control techniques are aimed to control the formation of AMD at source (Luptakova et al., 2010) based on the removal of oxygen, and/or bacteria from the system (Johnson and Hallberg, 2005). This can be done by several engineering techniques for treating the mine solid wastes, such as drying (Villain et al., 2013; Lu et al., 2014; Smart et al., 2010), water amendment (Demers et al., 2017; Kefeni et al., 2017; Nicholson et al., 1989), organic

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amendment, or oxygen consuming covers (Lu et al., 2014; Smart et al., 2010; Demers al., 2015; Peppas et al., 2000). Recently, a promising alternative technique, namely passivation, has been developed to control the formation of AMD at-source by changing the surface properties of pyrite and other sulfide minerals and inhibiting the atmospheric and aqueous oxidation of pyrite and other sulfide minerals. In passivation, chemical reagents are added to form a thin densely packed hydrophobic chemically protective coating (microencapsulation) on the surface of pyrite or other sulfide minerals to minimize the contact between the sulfide minerals and the air or other oxidants (Kang et al., 2016; Evangelou, 2001). So far, a large number of passivators have been reported, including inorganic passivators (e.g. silicates (Evangelou, 2001; Kargbo and Chatterjee, 2005), phosphate (Georgopoulou et al., 1996; Elsetinow et al., 2001), FeOOH (Huminicki and Rimstidt, 2009), and organic passivators (e.g. 8-hydroxyquinonline (Lan et al., 2002), triethylenetetramine (Chen et al., 2006; Liu et al., 2013). However, all of these passivators have their own disadvantages. For example, the formation of coatings from silicates, phosphates, phospholipids, and 8-hydroxyquinolines requires the initial oxidation of pyrite with hydrogen peroxide, which severely limits the practical application of these passivators. Triethylenetetramine may not be suitable for field applications due to its toxicity to the environment. In order to address the above issues of the passivators, it is necessary to develop environment-friendly and easily treated passivators. Based on the principle of flotation, a novel approach to inhibit the oxidation of pyrite was proposed. In the proposed approach, the pyrite was immersed into a surfactant solution with hydrophilic and hydrophobic groups to form a film on the surface of the pyrite, which can change the surface properties of pyrite from hydrophilicity to hydrophobicity. Due to the strong chemical interaction between the head (hydrophilic group) of the surfactant and the iron atoms, the tail (hydrophobic group) of the surfactant faces outward, which causes a uniform and compact hydrophobic film to be formed on the surface of pyrite. Due to the non-conductivity of the hydrocarbon chains, electrons or holes cannot react with pyrite through the insulating layer. The surface hydrophobic film is impermeable to oxygen, thereby retards the driving force of pyrite oxidation and inhibits the oxidation of pyrite. In this study, the effectiveness of passivation was evaluated by coating the surfaces of pyrite-bearing tailings with a densely packed hydrophobic film using linoleic acid. The coated hydrophobic film can prevent air from attacking pyrite. Although air is a natural oxidant, the oxidization of pyrite by air is very slow (especially in the oxidization without microbial intervention). Thus, hydrogen peroxide was used to simulate the long-term oxidation of pyrite. In order to achieve the ultimate goal of reducing the oxidation pyrite and controlling the production of AMD, a series of experimental data were reported to demonstrate the feasibility of using linoleic acid as the passivators in real situations.

2. Materials and methods

2.1. Samples and materials

The pyrite-bearing tailings samples used in this study were obtained from the Nanjing lead-zinc flotation plant in the northeast of Nanjing, Jiangsu Province, China. The main chemical composition of the samples was analyzed by X-ray fluorescence as listed in Table 1. The distribution of the particle size of the pyrite-bearing tailings is shown in Fig. 1. The average particle size was measured by sieving. These samples were produced by drying the tailings slurry at 80 °C and stored in plastic bags at room temperature before use. They were dark brown and fine-grained solids. The following chemicals purchased from Chemical Reagent Co. Ltd. (Nanjing, China) were used in this study: linoleic acid (60%-74%), sodium hydroxide (analytical reagent), barium chloride (analytical reagent), potassium chromate (analytical reagent), hydrogen peroxide (30%), hydrochloric acid (37%), and nitric acid (65%-68%).

Table 1. Chemical composition of the pyrite-bearing tailings

Chemical composition	SiO_2	CaO	SO_3	MnO	Fe_2O_3	Al_2O_3	MgO
Content (wt%)	27.94	20.58	10.56	7.66	7.28	3.88	1.84
Chemical composition	BaO	K_2O	ZnO	TiO_2	PbO	Loss of ignition	
Content (wt%)	0.68	0.50	0.18	0.13	0.11	18.39	

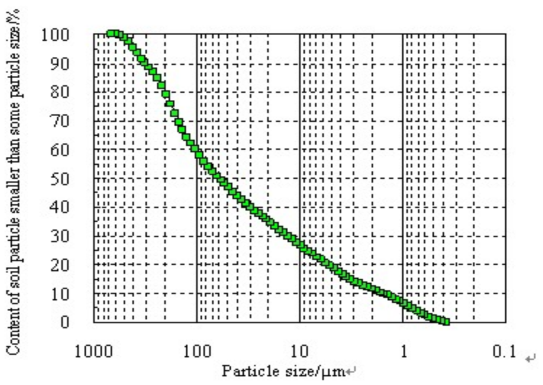

Fig. 1. Particle size distribution curve of the pyrite-bearing tailings

2.2. Surface coating on the pyrite-bearing tailings samples

The linoleic acid solutions with different concentrations were prepared by dissolving linoleic acid into different volumes of anhydrous alcohol. The pyrite-bearing tailings samples (5 g) were placed in 150 ml conical bottle and the conical bottle was shaken to make the pyrite-bearing tailings samples evenly spread on the bottom. Then 3 mL linoleic acid solution with specific concentration was added into the conical bottle and mixed for a specified time to achieve the coating treatment. All samples were allowed to dry in a fume hood overnight.

2.3. Leaching/oxidation experiments

The experimental procedure included the preparation of suspensions containing 5 g pyrite-bearing tailings that had been coated and 30 ml hydrogen peroxide with the concentration of 3 wt%. The suspensions were placed in a vibrator with rotating speed of 10 rpm and the constant temperature of 25 ℃ through water bath. After a specific time, the suspensions were taken out for analyses. Each suspension was filtered through a filter with the pore size of 0.45 μm and the filtrate was analyzed for Fe^{2+} and SO_4^{2-}. The concentration of Fe^{2+} of the leachate was analyzed by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). The concentration of SO_4^{2-} of the leachate was determined by Water quality-determination of sulfate-barium chromate spectrophotometry (HJ/T342-2007). Each experiment was repeated 3 times to ensure repeatability and accuracy of the measurements.

3. Results and discussion

3.1. Effect of linoleic acid dosage on passivation

Fig. 2 shows the oxidation of pyrite-bearing tailings as a function of linoleic acid dosage. In the experiment, both pyrite-bearing tailing samples, one uncoated and the other one coated by 3 mL linoleic acid solution for 8h, were leached by 30 ml hydrogen peroxide with the concentration of 3 wt% under the condition of 24-hour oxidization. From Fig. 2, based on the comparison of sulfate and ferrous ion release between coated and uncoated pyrite-bearing tailings, the linoleic acid coating was able to reduce the oxidation of the pyrite-bearing tailings. The concentration curves of both sulfate and ferrous ion have the same trend with the change of linoleic acid dosage. Before the linoleic acid dosage reached 0.6 g per kg of tailings, which was calculated by the following equation, $(3\ \text{ml} \times 10^{-3} \times 1000\ \text{mg/L})/5\ \text{g} = 0.6\ \text{g/kg}$, the concentrations of sulfate and ferrous ion gradually decreased with the increase of linoleic acid dosage. When the linoleic acid dosage was less than 0.6g/kg tailings, as the linoleic acid dosage increased, the hydrophilic groups that interact with iron on the surface of pyrite increased, the hydrophobic groups on the surface of pyrite-bearing tailings increased, the passivation film on the surface of pyrite-bearing tailings was thickened, and the opportunity for pyrite to contact with oxygen and water was decreased. As a result, the oxidative capacity of pyrite was weakened and the production of AMD was reduced. On the other hand, when linoleic acid dosage was above 0.6 g/kg tailings, the concentrations of sulfate and ferrous ion gradually increased with the increase of linoleic acid dosage. When the linoleic acid dosage was higher than 0.6 g/kg tailings, the amount of linoleic acid was sufficient to completely coat the surface of pyrite-bearing tailings, and the excessive linoleic acid was agglomerated (Nishiike et al., 1997) and adhered to the surface of the passivation film, which caused the film on the surface of pyrite-bearing tailings to fall off. As a result, the pyrite was in contact with oxygen and water in the surrounding environment, the oxidation reaction occurred, and the AMD was produced.

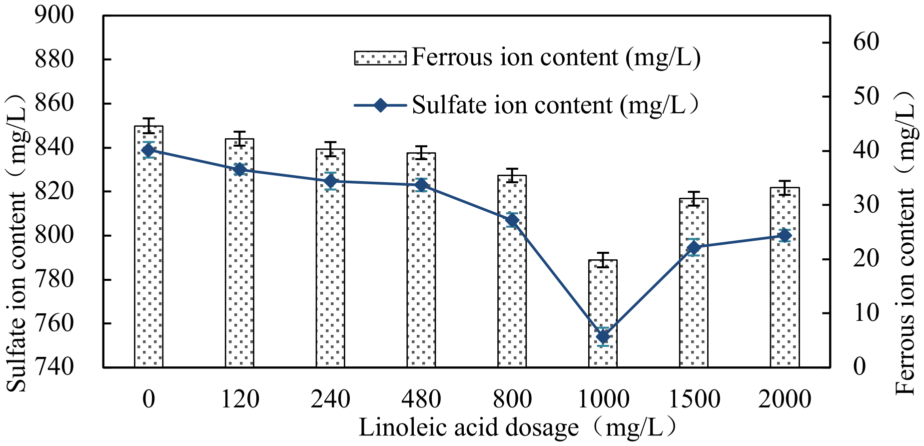

Fig. 2. Release of sulfate and ferrous ion as a function of linoleic acid dosage for the pyrite-bearing tailings oxidation in the presence of 30ml 3 wt% hydrogen peroxide

3.2. Effect of film formation time on passivation

Fig. 3 shows the trend of pyrite-bearing tailings oxidation as a function of the formation time of the film. In the experiment, the pyrite-bearing tailings was coated by 3 mL linoleic acid solution (1000 mg/L) and then leached with 30 ml hydrogen peroxide (3 wt%) under the condition of 24 hour oxidization. From Fig. 3, the formation time of the film has a significant influence on the passivation. The concentrations of both sulfate and ferrous ion have the same trend with the change of the formation time of the film. Before the film formation time reached 4 hours, the concentration of sulfate and ferrous ion decreased sharply with the extension of the formation time. The results showed that the reaction between linoleic acid and the surface material of tailings occurred immediately upon their contact and lasted for a period of time. In a certain period of time, as the passivation time was longer, the contact time between linoleic acid and tailings was longer, more chelates were formed by the reaction between the hydrophilic groups in linoleic acid and the metals on the surface of pyrite-bearing tailings, and more films were coated on the surface of pyrite-bearing tailings. However, after the formation time of the film reached 4 hours, the concentration of sulfate and ferrous ion increased slowly with the prolongation of the formation time. Linoleic acid can remain stable in the air for 48 h; however, after 96 h, the linoleic acid is easily oxidized (Shen, 2010), which causes the passivation film to fall off.

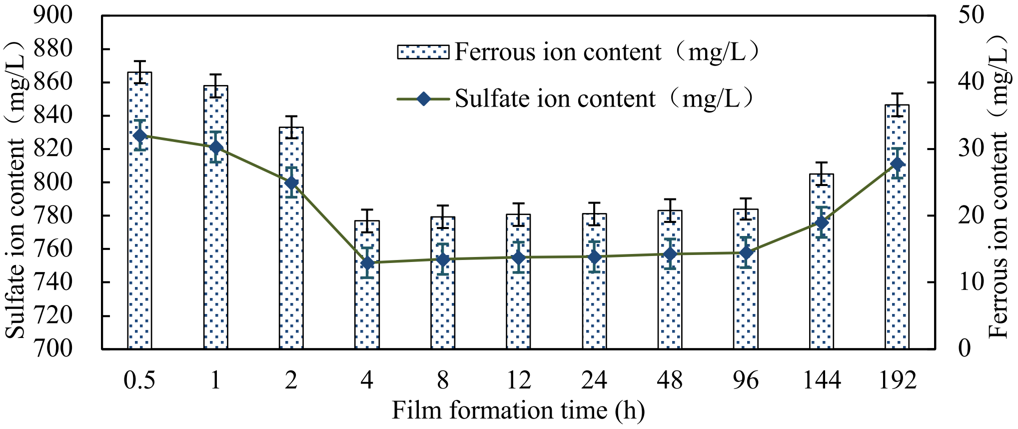

Fig. 3. Release of sulfate and ferrous ion as a function of film formation time for the pyrite-bearing tailings oxidation in the presence of 30ml 3 wt% hydrogen peroxide under 24 h oxidizing condition

3.3. Effect of storage time on passivation

Fig. 4 shows the oxidation of pyrite-bearing tailings as a function of storage time. In the experiment, pyrite-bearing tailings were coated by 3 mL linoleic acid solution (1000 mg/L) with 4-hour film formation and then leached with 30 ml hydrogen peroxide (3 wt%) under the condition of 24-hour oxidization. From Fig. 4, the storage time has a great influence on the passivation. The concentrations of both sulfate and ferrous ion have the same trend with the change of the storage time. The concentrations of sulfate and ferrous ion had no significant change with the prolongation of storage time when the indoor storage was shorter than 4 days. After 4 days of indoor storage, the concentration of sulfate and ferrous ion increased sharply with the prolongation of storage time. The results presented in this study showed that the linoleic acid can form a densely packed hydrophobic film on the surface of pyrite-bearing tailings, weaken the oxidative capacity of pyrite, and reduce the production of AMD. However, linoleic acid can be biodegraded, which causes the surface to be available for oxidation again. How to solve the biodegradability issue of linoleic acid? According to the results on the effect of the formation time of film in this study, linoleic acid should be added before the pyrite-bearing tailings pulp is concentrated, which can ensure sufficient formation time of film. Then the concentrated pyrite-bearing tailings pulp is fully extruded into a dry sheet-like tailing cake, in which the water content is reduced to only about 20% by dewatering equipment. Subsequently, the dry sheet-like tailing cake is transported to a tailings dry yard by belt conveyor or truck to form layered and compacted stacking. According to the results of film storage time in this study, after dehydration, the pyrite-bearing tailings cannot be oxidized before being compacted. After the dehydrated pyrite-bearing tailings are compacted and stacked, the permeability coefficient of the stack body decreases, which can effectively reduce the contact with oxygen and suppress the production of AMD.

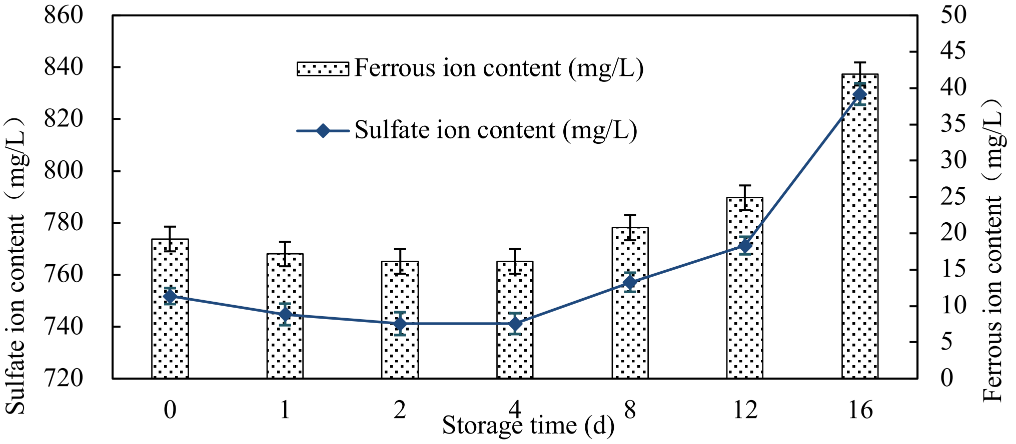

Fig. 4. Release of sulfate and ferrous ion as a function of storage time for the pyrite-bearing tailings oxidation in the presence of 30ml 3 wt% hydrogen peroxide under 24 h oxidizing condition

3.4. Mechanism of pyrite-bearing tailings oxidation suppression by linoleic acid coatings

To better understand the mechanism of the linoleic acid coatings on pyrite-bearing tailings, FTIR measurements were used to investigate the oxidation suppression of the pyrite-bearing tailings coated by linoleic acid. For comparison, the corresponding measurements were also performed on the samples of uncoated pyrite-bearing tailings. From Fig. 5, the infrared spectrum of the pyrite-bearing tailings is relatively flat and does not have any characteristic absorption peaks, which indicates that pyrite-bearing tailings do not have some functional groups. There are five distinct characteristic absorption peaks on the infrared spectrum of linoleic acid, which are located at 2918 cm-1, 2856 cm-1, 1659 cm-1, 1549 cm-1, and 1393 cm-1, which represent the characteristic bands of CH2, CH3, C=C, C=O, and C-O in linoleic acid, respectively. The five characteristic bands also appear at 2918 cm-1, 2856 cm-1, 1663 cm-1, 1543 cm-1 and 1415 cm-1 on the infrared spectrum line of the pyrite-bearing tailings coated by linoleic acid.

The same characteristic absorption peaks at 2918 cm-1 and 2856 cm-1 in the infrared spectra of both linoleic acid and the coated pyrite-bearing tailings indicated that linoleic acid was adsorbed on the surface of pyrite-bearing tailings, and CH2 and CH3 in linoleic acid did not react with pyrite-bearing tailings. The characteristic absorption peaks of C=C, C=O and C-O appeared at different locations in the infrared spectra of linoleic acid and coated pyrite-bearing tailings (located at 1659 cm-1, 1549 cm-1 and 1393 cm-1 in the spectrum of linoleic acid, and located at 1663 cm-1, 1543 cm-1 and 1415 cm-1 in the infrared spectrum of coated pyrite-bearing tailings). The different locations indicated that these characteristic functional bonds were also adsorbed on the surface of pyrite-bearing tailings and reacted with pyrite-bearing tailings, resulting in the shifts of the absorption peaks of these characteristic function bonds.

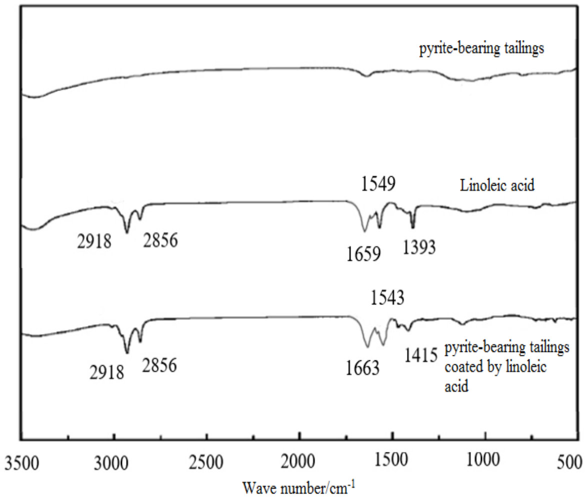

Fig. 5. FTIR spectra of pyrite-bearing tailings; linoleic acid; pyrite-bearing tailings coated by linoleic acid

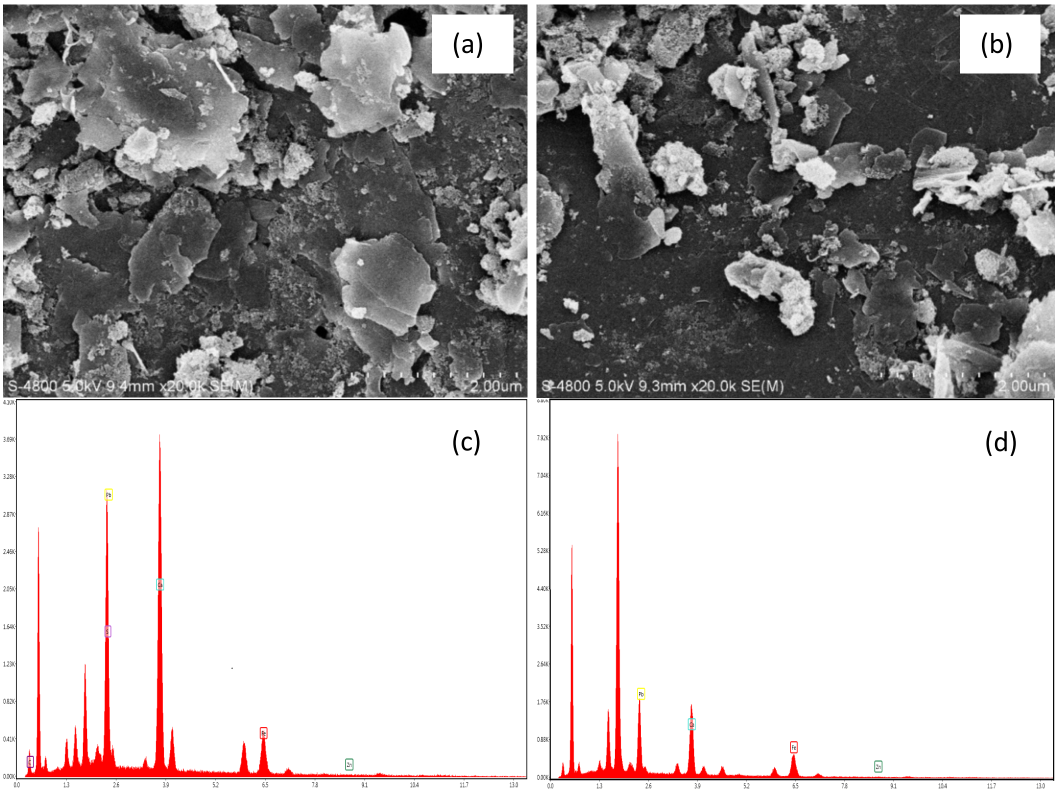

Fig. 6. SEM images of (a) pyrite-bearing tailings, (b) pyrite-bearing tailings coated by linoleic acid and EDS results of (c) pyrite-bearing tailings, (d) pyrite-bearing tailings coated by linoleic acid

The pyrite-bearing tailings samples coated by linoleic acid were collected and dried for further analysis. The SEM-EDS was used to determine the densely packed hydrophobic films formed on the surface of the pyrite-bearing tailings. The results were compared before and after the treatments by linoleic acid. The surface morphology images were obtained from both the uncoated and the linoleic-acid-coated pyrite-bearing tailings using SEM and EDS spectrum, as shown in Fig. 6. Compared with the uncoated pyrite-bearing, significant morphological changes of the coated pyrite-bearing tailings were observed due to the formation of the surface coating by linoleic acid (Fig. 6). The surface of uncoated pyrite-bearing tailings was flaky and layered, while that of pyrite-bearing tailings coated by linoleic acid was smooth and clean, with less flaky or layered appearance. Based on EDS analysis data, the contents of Pb, Ca, and Fe in the uncoated pyrite-bearing tailings were 22%, 30% and 9%, respectively (all values in the unit of wt.%). However, the contents of Pb, Ca, and Fe in the coated pyrite-bearing tailings were decreased to 18%, 16% and 7%, respectively. These results suggested a densely packed hydrophobic film was indeed formed.

From Fig. 2 to Fig. 4, the change of ferrous ions is almost consistent with that of sulfate ion. Are the changes of both ferrous ions and sulfate ions in the filtrate mainly caused by the oxidation of pyrite? The oxidation of pyrite can be described with the following equation: $2FeS_2(s) + 7O_2+2H_2O \rightarrow 2FeSO_4(aq) + 2H_2SO_4(aq)$. In theory, the molar number of sulfate ion should be twice as large as that of ferrous ion. According to the changes of ferrous ion in Fig. 2, the corresponding theoretical changes of sulfate ion can be inferred. The experimental and theoretical changes of sulfate ion are shown in Fig. 7.

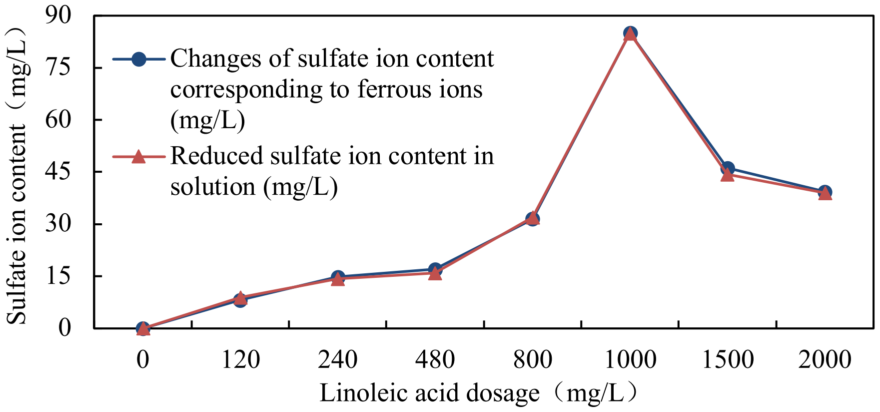

Fig. 7. Comparison of the actual and theoretical changes of sulfate ion in the experiment based on the influence of linoleic acid dosage on the oxidation of pyrite-bearing tailings

From Fig. 7, the theoretical change of the sulfate ion with the linoleic acid dosage is consistent with that from the test. These results indicated that the changes of ferrous ions and sulfate ions in the filtrate are mainly caused by the oxidation of pyrite. The results proved that the linoleic acid can form a densely packed hydrophobic film on the surface of pyrite-bearing tailings, weaken the oxidative capacity of pyrite, and reduce the AMD production.

4. Conclusions

In this study, the feasibility of using environment-friendly densely packed hydrophobic films to suppress the oxidation of pyrite-bearing tailings and control and acid mine drainage at the source has been demonstrated. Experimental results have verified that the linoleic acid can form a densely packed hydrophobic film on the surface of pyrite-bearing tailings. Due to the hydrophobic layer on surface of pyrite-bearing tailings, the oxidation of pyrite-bearing tailings can be effectively suppressed and the production of AMD can be reduced.

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