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Transactions of Nonferrous Metals Society of China

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Trans. Nonferrous Met. Soc. China 28(2018) 1241-1247

Flotation and electrochemical behaviors of chalcopyrite and pyrite in the presence of *N*-propyl-*N*'-ethoxycarbonyl thiourea

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Received 4 May 2017; accepted 30 January 2018

Abstract: The characteristics of *N*-propyl-*N*'-ethoxycarbonyl thiourea (PECTU) were studied in the flotation experiments of chalcopyrite and pyrite compared with butyl xanthate (BX). The interaction mechanism between mineral and PECTU was investigated according to zeta potential and electrochemistry measurements in the presence of PECTU. The results proved that PECTU performed a stronger ability to capture chalcopyrite and a better selectivity against pyrite. The zeta potential of chalcopyrite was positively shifted after interacting with PECTU, which indicated that the collector PECTU was obviously adsorbed on chalcopyrite surface. The cyclic voltammetry and Tafel curves results indicated that the oxidation and corrosion rates of chalcopyrite surface were limited in the presence of PECTU, while the effect of PECTU on pyrite in weak alkaline solution can be neglected basically according to the results of zeta potential and electrochemical tests.

Key words: chalcopyrite; pyrite; flotation; N-propyl-N'-ethoxycarbonyl thiourea; electrochemical behavior

1 Introduction

Chalcopyrite is the main copper-bearing ore and accounts for approximately 70% of the Earth's copper resources [1,2]. Although extensive efforts have been made to extract copper from low-grade copper sulfide ore by leaching and bioleaching, there are lots of difficulties in practical application [3–5]. Pyrometallurgy is still the main method to extract copper from sulfide mineral. And flotation is a requisite procedure before pyrometallurgy to concentrate low grade mineral and separate chalcopyrite from other valuable minerals.

In most porphyritic copper mines, chalcopyrite and pyrite are the main sulfide minerals. Usually, pyrite is associated with chalcopyrite intimately and may host gold [6,7]. Pyrite must be separated from chalcopyrite by flotation in order to obtain high purity copper concentrate. However, it is difficult considering the accidental activation of pyrite by dissolved Cu^{2+} and galvanic interactions between minerals [8,9]. In a long time, as powerful and low cost collectors, xanthates are the most widely used thiol collectors in the flotation of sulfide minerals. However, they suffer from practical

disadvantages due to the instability and lack of selectivity [10]. In modern industrial practice, it is obliged to carry out in high alkaline pulp by adding a large amount of lime in order to depress the pyrite, which leads to the loss of precious metals such as Au and Ag and high lime consumption [11–13]. Meanwhile, the pyrite depressed with excess lime is difficult to float.

Researchers have made efforts to separate chalcopyrite and pyrite in weak alkaline solution. An effective way is to utilize selective collectors to achieve pyrite inhibition. It has been demonstrated that thiourea derivatives are efficient collectors for selective capture of chalcopyrite. NAGARAJ and BRINEN [14] revealed that the adsorption of modified thionocarbamate and thiourea on the surface of pyrite was negligible. LIU et al [15] discussed the adsorption of N-isopropoxypropyl-N'-ethoxycarbonyl thiourea (iPOPECTU) on chalcopyrite surfaces and stated that the iPOPECTU bonded with copper ions on the surface of chalcopyrite, instead of Fe ions and Cu-iPOPECTU complexes formed on chalcopyrite surface. XIAO et al [16] indicated N-butoxypropyl-N'-ethoxycarbonylthat thiourea (BOPECTU) owned stronger collecting ability to chalcopyrite than to sphalerite or pyrite.

Foundation item: Project (51374249) supported by the National Natural Science Foundation of China

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LIU et al [17] confirmed that N, N'-diisopropoxypropyl-N'', N'''-oxydiethylenedicarbonyl bis (thiourea)(DiPOPOCTU) exhibited superior hydrometallurgical performance for flotation recovery of chalcopyrite. FAIRTHORNE et al [12,18] provided evidence that there was no reaction between butyl ethoxycarbonyl thiourea (BECTU) and ferrous, ferric and lead ions; however, BECTU reacted readily with cupric to form an insoluble Cu-BECTU complex. In the flotation of a refractory copper porphyry ore, LIU et al [13] increased the grades and recoveries of Cu, Au and Mo in the copper concentrates with mixed collectors of N-propyl-N'-ethoxycarbonyl thiourea (PECTU) and sodium butyl xanthate (SBX), compared with single SBX.

It has been realized that there are electrochemical reactions between sulfide minerals and collectors, and electrochemical measurements have been powerful instrument to discuss the interaction between sulfide minerals and collectors [19–23]. Although the interaction of chalcopyrite and thiourea collectors has been researched by various methods, few papers focused on the electrochemical interaction between chalcopyrite and thiourea collectors. In this paper, the floatation behaviors of chalcopyrite and pyrite were compared in the presence of PECTU or BX, and the interaction between minerals and PECTU was investigated using electrochemical measurements and zeta potential tests, which could give a more comprehensive understanding of action mechanism of thiourea collectors.

2 Experimental

2.1 Materials

Chalcopyrite and pyrite were obtained from Daye, Hubei Province (China) and Yunfu, Guangdong Province (China), respectively. The mineral samples were crushed by geological hammer and dry ground in a porcelain ball milling. The fraction with size of 38-74 µm obtained by screening was used for flotation. Chemical analysis showed that chalcopyrite contained 32.35% Cu, 30.04% Fe and 33.62% S, and pyrite contained 43.75% Fe and 51.92% S. XRD analysis (Fig. 1) confirmed that the main mineral phases are chalcopyrite and pyrite, respectively. The mineral electrode used in the electrochemistry study was cut from highly mineralized rock specimens and was made into $d12 \text{ mm} \times 5 \text{ mm}$ cylinders with a grinding wheel. The mineral electrode was mounted in epoxy resin with an exposed area of 1 cm². Butyl xanthate of an industrial grade was obtained from Zhuzhou Flotation Reagents Limited Company, China. PECTU was supplied by the Zhonghong Chemicals Limited Company, China.



Fig. 1 XRD patterns of chalcopyrite (a) and pyrite (b)

2.2 Micro-flotation tests

The flotation tests were carried out using an XFG flotation machine with a flotation cell of 40 mL. The rotational speed of impeller was 1890 r/min. A sample of about 2.0 g with 38-74 µm size fractions was weighed on an analytical balance and was mixed with 50 mL distilled water in an 100 mL beaker. The mineral suspension was placed in an ultrasonic bath for exactly 5 min to remove the surface oxides and then quickly removed from the bath and settled for 2 min. The upper liquid layer was decanted and the mineral was moved into the flotation cell with 40 mL distilled water. The mineral pulp was conditioned for 1 min and was adjusted to a desired pH value with HCl or NaOH stock solutions. Collector and frother were sequentially put into the flotation cell with the conditioning time of 3 min and 1 min, separately. The flotation time was 3 min and the flotation recovery was calculated according to the mass ratios of floated and un-floated fractions.

2.3 Zeta potential measurement

The zeta potential measurement was carried out using a Malvern Zetasizer Nano-ZS90. Before each test, 0.05 mg mineral which was ground to $-5 \mu m$ in advance was weighed, and mixed with 50 mL solution of

1 mmol/L KCl in a 100 mL beaker. The mineral suspension was cleaned for 5 min in an ultrasonic bath to remove the oxidized film. Then, the mineral pulp was conditioned for 5 min using a magnetic stirrer and was adjusted to a desired pH value with HCl or NaOH stock solutions. PECTU was added into the pulp during the conditioned period in the test with the collector. Part of upper solution was injected into rectangular electrophoresis cell to test.

2.4 Electrochemical measurement

Electrochemical measurement was performed using a Princeton Galvanostat/Potentiostat 283. A conventional three-electrode system was used which consisted of a reference electrode (Ag/AgCl), a counter electrode (graphite rod) and a working electrode (mineral electrode). The pH 9.2 tetraborate (0.05 mol/L $Na_2B_4O_7 \cdot H_2O$) buffer solution was applied with 0.1 mol/L potassium nitrate (KNO₃) as the supporting electrolyte. Before each test, the mineral electrode was polished by 600, 800 and 1200 grid silicon carbide paper successively to regenerate a fresh surface, and then rinsed with distilled water and quickly transferred to the cell. The cyclic voltammetry measurement was performed with a scan rate of 20 mV/s and the Tafel curves were tested from -250 to 250 mV (vs open circuit potential, OCP) with a scan rate of 1 mV/s. All the potentials reported in this paper were converted to the standard hydrogen electrode (SHE).

3 Results and discussion

3.1 Flotation

Figure 2 shows the flotation behaviors of chalcopyrite and pyrite in the presence of BX or PECTU. Chalcopyrite exhibited a good flotability whether in the presence of BX or PECTU even though the recovery had a slightly drop at pH 12. The recovery of chalcopyrite was above 90% throughout pH values using BX as the collector, and with PECTU, the recovery was higher than 92%. The floatability of pyrite was not as good as that of chalcopyrite and the recovery of pyrite was more sensitive to pH. When BX was used as the collector, the recovery of pyrite was around 80% in acid solution environments, and dropped sharply when pH value was above 8. However, the recovery was still around 30% when pH value was 10.5. This is the reason why the separation of chalcopyrite and pyrite is operated in a high alkaline environment. With PECTU as the collector, the recovery of pyrite decreased from about 70% at pH 4 to 22.78% at pH 9.0 and was less than 10% at pH 10.5. The results proved that the PECTU presented a better selectivity than BX against pyrite.



Fig. 2 Effect of pH value on flotation recoveries of chalcopyrite and pyrite with 1×10^{-4} mol/L PECTU or BX

The effect of initial concentration of collectors on the recoveries of chalcopyrite and pyrite at pH 9.0 is given in Fig. 3. The flotation recoveries of chalcopyrite and pyrite increased with increasing BX or PECTU concentration. The recovery of chalcopyrite increased sharply before the initial concentration reached 0.5×10^{-4} mol/L and then remained stable. With BX as the collector, the recovery of pyrite also ascended quickly and maintained at about 70% until the concentration of BX up to 2×10^{-4} mol/L. However, the recovery of pyrite increased slowly and still lower than 40% in the presence of PECTU at the dosage of 3×10^{-4} mol/L.



Fig. 3 Flotation recoveries of chalcopyrite and pyrite as function of collector concentration at pH 9.0

3.2 Zeta potential measurement

It is proved that the zeta potential test plays an important role in investigating the action mechanism between the minerals and reagents as well as the adsorption behavior of reagent on the mineral surface [24,25]. The zeta potentials of chalcopyrite and pyrite at different pH values with or without PECTU are presented in Figs. 4 and 5, respectively. As shown in

Fig. 4, the zeta potentials of chalcopyrite showed a positive shift after interaction with PECTU, especially at low pH values, which demonstrated that the PECTU could be adsorbed on the chalcopyrite surface adequately. FAIRTHORNE et al [18] obtained the similar results and according to their conclusion about the direction and the magnitude changes of the zeta potential, the chalcopyrite surface was positively charged after PECTU adsorption and had a higher adsorption efficiency at low pH values.



Fig. 4 Zeta potentials of chalcopyrite at different pH values with and without PECTU



Fig. 5 Zeta potentials of pyrite at different pH values with and without PECTU

However, it was noted that the PECTU had almost no effect on the zeta potential values of pyrite (Fig. 5). The zeta potential values of pyrite increased slightly in the acid solution and almost had no change in the neutral and alkaline solution environments. The changes of zeta potentials of chalcopyrite and pyrite with/without PECTU confirm that the PECTU shows a good selective adsorption on the surface of chalcopyrite.

3.3 Cyclic voltammogram

The influence of PECTU on the voltammogram of chalcopyrite in the pH 9.2 buffer solution is presented in Fig. 6. The voltammogram of chalcopyrite obtained in

the absence of collector showed an anodic peak at about 250 mV in the positive-going direction. The anodic peak was attributed to the oxidation of chalcopyrite according to Reaction (1), as proposed by GARDNER and WOODS [26]. The cathodic peak of the negative-going sweep was referred to the reduction of the oxidative products. It can be seen that no new peak appeared on the voltammogram of chalcopyrite in the presence of PECTU. On the contrary, current density of the existing peaks obtained without PECTU decreased due to the presence of PECTU, as found by GÜLER [27] in the presence of dithiophosphate. It was implied that the adsorption of PECTU resulted in the formation of a passive layer on the electrode surface and the oxidation of chalcopyrite was limited. The in situ scanning electrochemical microscopy images obtained by LIU et al [15] also indicated that the preferential adsorption *N*-isopropoxypropyl-*N*'-ethoxycarbonyl of thiourea (iPOPECTU) on chalcopyrite surface resulted in a decrease in the probing electrochemical current response. According to the previous research [15,18], the Cu atoms from the chacopyrite may bond to the N and S atoms from PECTU and form Cu-PECTU complex, which coated on the chalcopyrite surface.

$$CuFeS_2 + 3H_2O \longrightarrow CuS + Fe(OH)_3 + S^0 + 3H^+ + 3e$$
(1)

The voltammograms of pyrite (Fig. 7) also show an anodic peak in the positive potential region and a cathodic peak in the reverse direction, respectively. The anodic peak was attributed to the oxidation according to Reaction (2) [28]. And the cathodic peak presented the reduction of the oxidative products. However, what was different from chalcopyrite was that the addition of PECTU did not affect the voltammogram, which indicated that there was no interaction between the pyrite and PECTU in the weak alkaline solution environment. It was consistent with the flotation results.

$$FeS_2+3H_2O \longrightarrow Fe(OH)_3+2S^0+3H^++3e$$

(2)



Fig. 6 Cyclic voltammograms of chalcopyrite in the absence and presence of PECTU in pH 9.2 buffer solution



Fig. 7 Cyclic voltammograms of pyrite in the absence and presence of PECTU in pH 9.2 buffer solution

3.4 Tafel curves

Tafel curves of chalcopyrite in pH 9.2 buffer solution are illustrated in Fig. 8 and the Tafel parameters are given in Table 1. After the addition of PECTU, the corrosion potential (φ_{corr}) declined from 103.8 to 68.33 mV and the corrosion current density (J_{corr}) dropped from 1.496 to 0.794 μ A/cm². The reduction of the φ_{corr} and J_{corr} demonstrated that the adsorption of PECTU on the surface of chalcopyrite led to the formation of a passivation coating on the surface of electrode, which inhibited the corrosion of chalcopyrite electrode.



Fig. 8 Tafel curves of chalcopyrite in the absence and presence of PECTU in pH 9.2 buffer solution

 Table 1 Tafel parameters of chalcopyrite in the absence and presence of PECTU in pH 9.2 buffer solution

$c(\text{PECTU})/(\text{mol} \cdot \text{L}^{-1})$	$\varphi_{\rm corr}$ (vs SHE)/mV	$J_{\rm corr}/(\mu {\rm A}\cdot {\rm cm}^{-2})$
0	103.8	1.496
5×10 ⁻⁴	68.33	0.794

Figure 9 and Table 2 present Tafel curves and parameters of pyrite with and without PECTU, respectively. It can be seen that the presence of PECTU almost did not affect the Tafel curve of pyrite. Both the corrosion potential and corrosion current were basically unchanged before and after adding PECTU, which also indicated that the pyrite could not react with PECTU.



Fig. 9 Tafel curves of pyrite in the absence and presence of PECTU in pH 9.2 buffer solution

Table 2 Tafel parameters of pyrite in the absence and presence of PECTU in pH 9.2 buffer solution

$c(\text{PECTU})/(\text{mol}\cdot\text{L}^{-1})$	$\varphi_{\rm corr}({\rm vs~SHE})/{\rm mV}$	$J_{\rm corr}/(\mu {\rm A}\cdot {\rm cm}^{-2})$
0	267.6	4.237
5×10^{-4}	263.1	4.199

4 Conclusions

1) PECTU performed a stronger ability to capture chalcopyrite and selective inhibition capacity to pyrite. Chalcopyrite exhibited a better floatability in the presence of PECTU than butyl xanthate. Pyrite was floated easily in the acid and neutral solutions while the recovery decreased dramatically at pH beyond 8 in the presence of xanthate. Especially, the floatability of pyrite becomes poor in the neutral solution with PECTU as a collector.

2) The zeta potential tests illustrated that the PECTU was adsorbed on chalcopyrite surface throughout pH values and the potential of chalcopyrite shifted in the positive direction. The results of electrochemical measurements indicated that the oxidation and corrosion of chalcopyrite were inhibited due to the adsorption of PECTU on chalcopyrite electrode surface.

3) The zeta potential and the electrochemical tests confirmed that the pyrite could not react with PECTU in weak alkaline solution.

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N-丙基-*N'*-乙氧羰基硫脲对 黄铜矿和黄铁矿的浮选及其电化学行为

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摘 要:通过浮选试验,比较 N-丙基-N-乙氧羰基硫脲(PECTU)和丁基黄药(BX)对黄铜矿和黄铁矿浮选行为的影响,并利用动电位测试和电化学方法研究乙氧羰基硫脲与黄铜矿和黄铁矿的作用机理。浮选试验结果表明,相对于丁基黄药,乙氧羰基硫脲表现出对黄铜矿更强的捕收能力以及更好的选择性。与 PECTU 作用后,黄铜矿表面 zeta 电位向正方向移动,证明 PECTU 在黄铜矿表面发生了吸附。循环伏安曲线和塔菲尔测试结果表明,PECTU 与黄铜矿表面相互作用后,吸附在黄铜矿电极表面的 PECTU 阻碍了黄铜矿自身的氧化和腐蚀。动电位和电化学 测试结果表明,在弱碱性条件下,PECTU 与黄铁矿几乎无相互作用。

关键词:黄铜矿;黄铁矿;浮选; N-丙基-N'-乙氧羰基硫脲; 电化学行为

(Edited by Wei-ping CHEN)