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Short Communication

Flotation of fine pyrite by using N-dodecyl mercaptan as collector in natural pH pulp

Kaile Zhao^{a,b}, Guohua Gu^b, Wu Yan^{a,*}, Xiaohui Wang^{a,*}, Chongqing Wang^c, Longhua Xu^b

^a Institute of Multipurpose Utilization of Mineral Resources, Chinese Academy of Geological Sciences, Chengdu 610041, Sichuan, China

^b School of Minerals Processing and Bio-Engineering, Central South University, Changsha 410083, Hunan, China

^c School of Chemical Engineering and Energy, Zhengzhou University, Zhengzhou 450001, Henan, China

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ABSTRACT

Laboratory and pilot-scale flotation tests were conducted to investigate the effect of N-dodecyl mercaptan (NDM) on the flotation of fine pyrite. At bench scale, NDM resulted in higher recovery, and the mixed collector of NDM and potassium isoamyl xanthate (PIAX) (mass ratio of 2:1) caused the highest recovery of 96.5%. High grade and recovery of pyrite was obtained without adding any activator (CuSO₄) and modifier (H₂SO₄) in a natural pH of the pulp. Pilot-scale tests indicated that a concentrate with the grade of 50.05% S, 42.23% Fe and the recovery of 98.51% S, 96.69% Fe was achieved by using the mixed collector of NDM + PIAX at pH 6.5.

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1. Introduction

Dongbafeng has about 250 million tones of pyrite reserves with S grades as low as 15.04% in Guangxi province, China, and the pyrite mainly coexists with pyrophyllite, kaolinite and carbonates [1]. In the practice of pyrite flotation, the general method is to add modifier H₂SO₄/H₂C₂O₄ and activator CuSO₄ in association with collector xanthates [2–5]. Due to the fine dissemination of pyrite [6,7] (–37 μm 56.8%, –10 μm 20.3%) and the high clay contents in this low-grade sulphide ore [8], H₂SO₄ and CuSO₄ are used as modifier and activator respectively,

and potassium isoamyl xanthate (PIAX) is used as collector [9] in the pyrite flotation. Zhao et al. reported that the recovery of pyrite might decrease by 10–20% in absence of H₂SO₄ [8]. However, using large quantity of H₂SO₄ (8 kg/t) may contaminate the environment, accelerate equipment corrosion and increase production costs.

Dodecyl mercaptan includes N-dodecyl mercaptan (NDM, C₁₂H₂₆S) and tert dodecyl mercaptan (TDM, C₁₂H₂₅SH). TDM has been used as the collector for sulphide ores (sphalerite, arsenopyrite, molybdenite) flotation [10,11], but its application is limited due to high price, complex procedure and bad odor.

* Corresponding authors.

E-mails: yan507@163.com (W. Yan), luomodeyu@163.com (X. Wang).

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TDM is the isomer of NDM, and therefore they have the same molecular formula and functional group [12,13]. NDM is odorless and low price in China and can be used in sulphide ore flotation.

In this work, the introduction of N-dodecyl mercaptan (NDM) into the fine pyrite (clay-type, <37 μm) flotation industry without adding any modifier (H_2SO_4) and activator (CuSO_4) has been successful. The objective of this work is to float fine pyrite in a natural pH pulp at bench scale and pilot scale.

2. Experimental

The raw ore used in this study was obtained from Dongbafeng Sulphur Mine of Guangxi Province, China. The sample was crushed, ground and screened into -3 mm . It contains 15.04% S, 12.95% Fe, 31.29% Al_2O_3 , 24.06% SiO_2 , 3.26% TiO_2 , 1.84% C and 1.12% CaO. The sample is composed mainly of pyrite, pyrophyllite, bauxite, kaolinite, anatase, graphite, chlorite and montmorillonite. Pyrite is the only mineral of economic value in the ore. The grain size of pyrite particles is between 1 and 1000 μm . The degree of liberation of pyrite is below 74 μm . In addition, the $-37\text{ }\mu\text{m}$ and $-10\text{ }\mu\text{m}$ size fractions account for 56.8% and 20.3% of the entire pyrite particles, respectively.

In laboratory and pilot-scale experiments, N-dodecyl mercaptan (NDM, $\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2\text{SH}$, MW 202) and Potassium isoamyl xanthate (PIAX) were used as collectors, and sodium hexametaphosphate (SHMP) was used as the clay dispersant. Due to good natural floatability of pyrophyllite and other clay minerals [6,14], guar gum was used as the clay depressant. Sulphuric acid (H_2SO_4) and copper sulphate (CuSO_4) were used as modifier and activator respectively, and methyl isobutyl carbinol (MIBC) was used as frother. The reagents used in this study were of technical purity. Pulp pH was recorded by the PHS-3 meter. Tap water was used in both grinding and flotation experiments.

In laboratory experiments, the 300 g ore was ground to $-74\text{ }\mu\text{m}$ (97 wt%) in a XMB-70 rod mill at a pulp density of 50 wt%. Flotation tests were performed in a XFD-63 flotation cell (self-aeration), 1.5 dm^3 for rougher-scavenger flotation and 1.0 dm^3 for cleaner flotation, respectively. The impeller speed was 1590 rpm. The froth product was collected with an automatic froth scraper device. In the rougher flotation stage, the desired amount of reagents (SHMP 750 g/t, guar gum 300 g/t, NDM 100 g/t, PIAX 50 g/t, and MIBC 50 g/t) were added in the sequence to the rougher flotation cell with 3 min interval, and flotation time was 6 min. In pilot-scale trials, the processed capacity of the pilot-scale plant was 1.25 t/d (52 kg/h) feed. The pyrite flotation was carried out in a FX2-39 flotation cell (self-aeration) at a feed particle size of $-74\text{ }\mu\text{m}$ (97 wt%, using a XMBL-420 \times 600 mm rod mill). The volume of flotation cells for rougher-scavenger and cleaner flotation were 39 dm^3 and 24 dm^3 , respectively. The products were collected, dried, and weighed. Determination of S was conducted by infrared carbon and sulphur analyzer (Multi EA 2000, Analytikjena Germany), and Fe content was analyzed by volumetric method ($\text{K}_2\text{Cr}_2\text{O}_7$). To assure the accuracy of laboratory experiments, the calculated S grade of feed should be in the range of $15.04 \pm 0.04\%$, otherwise the experiments were repeated. The flotation flowsheet and corresponding reagent

concentrations of closed flotation circuit and pilot-scale tests are given in Fig. 1.

3. Results

Fig. 2a shows the S recovery as a function of PIAX, NDM, or NDM+PIAX dosage in the rougher operation. The recovery of pyrite increased markedly with the increasing of collector dosage. Compared to PIAX, NDM resulted in higher recovery with the same collector addition. Moreover, the mixed collector greatly improved the pyrite recovery, and the highest recovery was 96.5% at 150 g/t NDM + PIAX (preferred mass ratio of 2:1). The effect of PIAX and NDM addition in the rougher operation is shown in Fig. 2b and c, respectively. Compared with PIAX, NDM showed larger effect on the recovery of pyrite. The optimum dosage was 100 g/t NDM and 50 g/t PIAX, achieving high grade pyrite with grade of 41.2% S and recovery of 96.5% S.

Based on the optimum conditions of open circuit tests, locked cycle tests were carried out at batch scale using NDM + PIAX as a mixed collector in natural pH pulp. The comparative tests (S_2) were conducted using H_2SO_4 and CuSO_4 without NDM. As shown in Fig. 1, the closed flotation circuit includes one roughing, three scavenging, and four cleaning steps. Flotation results of the average values are displayed in Table 1 (S_1 , S_2). The dispersive values of the 4th, 5th and 6th cycles and their standard deviations are shown in Table 2 (S_1 , S_2).

In the comparative system S_2 , the reagents used in the flotation were H_2SO_4 of 8 kg/t (pH 4), CuSO_4 of 850 g/t, guar gum of 700 g/t, PIAX of 450 g/t and MIBC of 80 g/t. Under these conditions, a concentrate grade of 45.53% S at a recovery of 87.95% was achieved. To reduce reagent costs and improve the pyrite recovery, a mixed collector of NDM + PIAX (mass ratio of 2:1, the rougher concentration 150 g/t, the scavenger concentration 75 g/t, the cleaner concentration 30 g/t) was adopted in the flotation circuit. A concentrate grade of 49.23% S at a recovery of 98.33% was achieved on a feed of 15.04% S using NDM+PIAX as a mixed collector at pH 6.5. Compared with S_2 , the S_1 procedure increased the grade and recovery of S by 3.70% and 10.38%, respectively. Meanwhile the tailing grade decreased from 2.55% S to 0.36% S, implying that NDM is a powerful and selective collector for fine pyrite particles.

Because of the good collection performance for fine pyrite, collector complexes of NDM + PIAX were selected in the pilot-scale tests. The pilot-scale trials were performed at the Ermei pilot-scale plant located in Sichuan, China. The pilot-scale continuous running tests were carried out more than 120 h (August, 2016). The flotation flowsheet is given in Fig. 1. The average values of flotation results are shown in Table 1 (S_3). The dispersive values of 10 groups of pyrite concentrates (every 12 h) and their standard deviations are shown in Table 2 (S_3).

In S_3 procedure, a concentrate grade of 50.05% S, 42.23% Fe at a recovery of 98.51% S, 96.69% Fe was achieved in the neutral circuit using NDM + PIAX as a mixed collector. Meanwhile, a tailing with 0.35% of S and 0.61% of Fe was produced. For this low-grade sulphide ore, NDM + PIAX showed an excellent collection for fine pyrite ($-37\text{ }\mu\text{m}$ 56.8%), confirmed by the high grade and recovery of pyrite achieved using NDM + PIAX

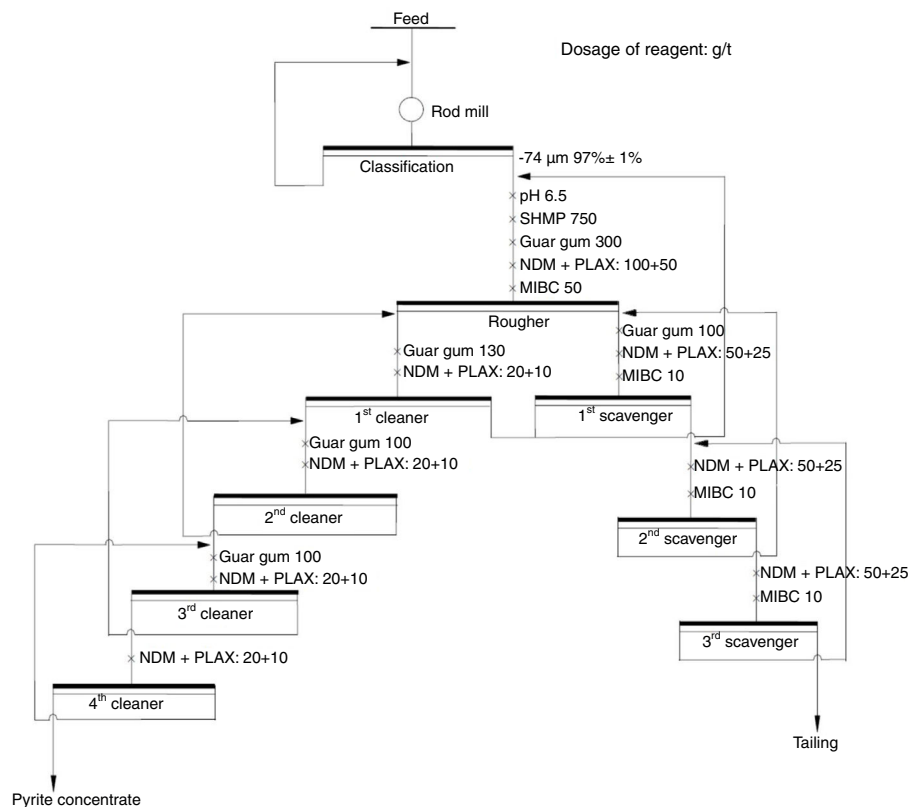


Fig. 1 – The flotation flowsheet and corresponding reagent concentrations of closed flotation circuit and pilot-scale tests.

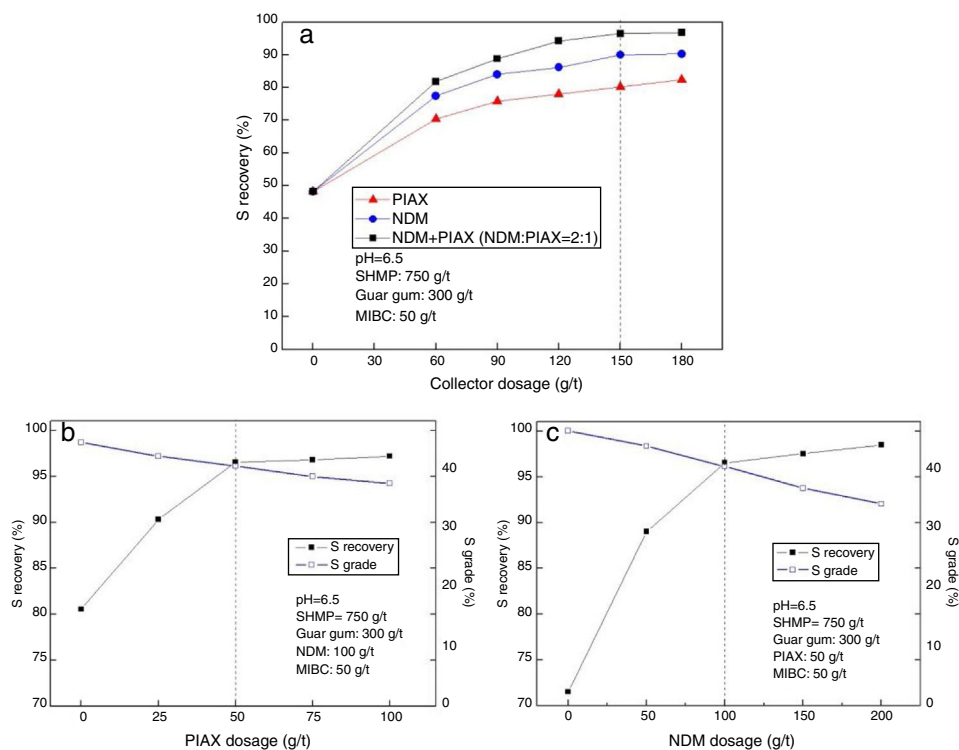


Fig. 2 – The effects of collector (PIAX, NDM, or NDM + PIAX as collector) dosage on the S recovery (a) and grade (b, c) in the rougher concentrates at pH 6.5 without H_2SO_4 and CuSO_4 addition.

Table 1 – The average values of closed flotation circuit (S₁, S₂) and pilot-scale trials (S₃).

System	Products	Ratio (w/%)	Grade (%)		Recovery (%)	
			S	Fe	S	Fe
Locked cycle flotation tests (S ₁)	Pyrite concentrate	30.06	49.23	41.71	98.33	96.81
	Tailing	69.94	0.36	0.59	1.67	3.19
	NDM + PIAX (pH 6.5)	100.00	15.05	12.95	100.00	100.00
Locked cycle flotation tests (S ₂) ^a	Pyrite concentrate	29.02	45.53	38.23	87.95	85.64
	Tailing	70.98	2.55	2.62	12.05	14.36
	PIAX (pH 4)	100.00	15.03	12.96	100.00	100.00
Pilot-scale tests (S ₃)	Pyrite concentrate	29.65	50.05	42.23	98.51	96.69
	Tailing	70.35	0.35	0.61	1.49	3.31
	NDM + PIAX (pH 6.5)	100.00	15.06	12.95	100.00	100.00

^a The comparative system S₂, acidic without NDM (pH 4).

Table 2 – The dispersive values of closed flotation circuit (S₁, S₂) and pilot-scale trials (S₃).

System	Products	Ratio (w/%)	Grade (%)		Recovery (%)	
			S	Fe	S	Fe
S ₁	4th pyrite concentrate	30.11	49.18	41.42	98.42	96.30
	5th pyrite concentrate	29.84	49.47	42.39	98.11	97.52
	6th pyrite concentrate	30.23	49.04	41.31	98.45	96.61
	Average value	30.06	49.23	41.71	98.33	96.81
	Standard deviation	0.199	0.219	0.594	0.188	0.634
S ₂	4th pyrite concentrate	29.24	45.05	37.73	87.74	85.13
	5th pyrite concentrate	29.12	45.23	37.92	87.62	85.35
	6th pyrite concentrate	28.71	46.32	39.03	88.48	86.43
	Average value	29.02	45.53	38.23	87.95	85.64
	Standard deviation	0.278	0.687	0.702	0.466	0.696
S ₃	1–12 h pyrite concentrate	29.74	49.87	41.69	98.48	95.87
	13–24 h pyrite concentrate	29.72	49.75	41.73	98.31	96.09
	25–36 h pyrite concentrate	29.62	50.02	42.26	98.45	96.73
	37–48 h pyrite concentrate	29.54	50.21	43.13	98.56	97.71
	49–60 h pyrite concentrate	29.76	49.69	41.53	98.29	95.74
	51–72 h pyrite concentrate	29.82	49.61	41.36	98.36	95.32
	73–84 h pyrite concentrate	29.56	50.35	42.75	98.78	97.64
	85–96 h pyrite concentrate	29.61	50.11	42.33	98.59	96.86
	97–108 h pyrite concentrate	29.45	50.83	43.37	98.53	98.01
	109–120 h pyrite concentrate	29.69	50.01	42.13	98.72	96.89
	Average value	29.65	50.05	42.23	98.48	95.87
Standard deviation	0.115	0.361	0.682	0.164	0.917	

as a mixed collector. The 2:1 collector mixture NDM + PIAX showed great potential for industrial application. This study solved the problem of flotation separation of fine pyrite and clay minerals, providing technical support for flotation recovery of clay-type pyrite flotation.

42.23% Fe and the recovery of 98.51% S, 96.69% Fe was achieved by using the mixed collector of NDM + PIAX at pH 6.5. The 2:1 collector mixture NDM + PIAX showed great potential for industrial application. The NDM + PIAX flotation procedure will be adopted at the Dongbafeng sulphur mine.

4. Conclusions

In this study, a mixed collector of N-dodecyl mercaptan (NDM) and potassium isoamyl xanthate (PIAX) was developed to float fine pyrite from clay-type sulphide ore. The laboratory experiments indicated that NDM resulted in higher recovery, and the mixed collector of NDM + PIAX (mass ratio of 2:1) caused the highest recovery of 96.5% S. High grade and recovery of pyrite was obtained without adding any activator (CuSO₄) and modifier (H₂SO₄) in a natural pH of the pulp. 120-h pilot-scale tests showed that a concentrate with the grade of 50.05% S,

Conflicts of interest

The authors declare no conflicts of interest.

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