



Diethylenetriamine depression of Cu-activated pyrite hydrophobised by xanthate



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ABSTRACT

In copper sulphide flotation, copper adsorbs on pyrite through superficial oxidation of the copper minerals (e.g. chalcopyrite) which promotes pyrite flotation in the presence of xanthate. This “inadvertent” activation of pyrite by copper ions is undesirable in copper sulphide flotation. In order to minimise this effect, depressants are used to suppress the effect of the activating ions. The effect of diethylenetriamine (DETA) in different combinations (under aerated and non-aerated conditions) on Cu-activated pyrite hydrophobised by xanthate was examined using flotation, spectroscopic and solution analyses, at pH 10. The results showed that DETA affects the flotation behaviour and surface chemistry of pyrite. However, high dosages are required. The depression action of DETA on Cu-activated pyrite was attributed to both the removal of surface copper to form soluble Cu–DETA complex in solution and competition for Cu sites on the activated pyrite surface. The significant depression of pyrite in the presence of DETA under the aerated condition was due to increased amount of iron oxy–hydroxides (Fe–O/OH), copper oxides and Cu(I)–DETA hydrophilic species.

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1. Introduction

Chalcopyrite and gold are the main valuable mineral phases in most polymetallic sulphide ores, with pyrite being the main sulphide gangue mineral. Chalcopyrite and gold are mostly associated with pyrite in these ores; therefore, their economical extraction requires a selective depression of pyrite.

The major problem associated with chalcopyrite and pyrite selectivity is due to accidental activation of pyrite by dissolved Cu^{2+} or Pb^{2+} ions from complex sulphide minerals through oxidation/dissolution reactions, which may be enhanced by galvanic interactions. This enhances pyrite interaction with thiol collectors and hence promotes its floatability. In most processing plants, sodium cyanide is employed to depress pyrite (by removing Cu^{2+} ions from its surfaces) under alkaline conditions. The environmental and health hazards associated with cyanide usage make it an unsafe approach to depress sulphides in selective flotation. As an alternative to cyanide, sulphur–oxygen depressants, in the form of sulphite (SO_3^{2-}), bisulphite (HSO_3^-), metabisulphite ($\text{S}_2\text{O}_5^{2-}$) or sulphur dioxide (SO_2) have been used in depressing pyrite, sphalerite and galena (Grano et al., 1997a, 1997b; Khmeleva et al., 2002, 2003; Shen et al., 2001; Chander and Khan, 2000; Bulut et al., 2011). The general mechanism of sulphide mineral depression using sulphite ions involves the formation of metal sulphite hydrophilic

species, decomposition of xanthate and consumption of oxygen (reduction in Eh) (Misra et al., 1985; Yamamoto, 1980; Miller, 1970). Despite numerous studies on the mechanism of sulphite ions in pyrite depression, little effort has been made to understand the effect of diethylenetriamine (DETA) in selective depression of pyrite in sulphide minerals.

DETA is a polyamine and soluble in water and alcohol (Weast, 1985). Generally, polyamines are excellent complexing or chelating agents. The application of DETA in flotation is to depress sulphide minerals and has been used extensively in pyrrhotite rejection in Ni–Cu ore flotation. It has been observed by Falconbridge Ltd., that DETA was only effective in rejecting pyrrhotite in the presence of SO_2 (Kelebek et al., 1995). The mechanism of DETA in the rejection of pyrrhotite was linked to the removal of activating ions (e.g. Cu^{2+} and Ni^{2+}). The removal of activating ions from pyrrhotite surfaces in the presence of DETA resulted in a decreased xanthate adsorption and hence reduced its floatability (Yoon et al., 1995). In another related operation (Inco operations), it was observed that DETA was only effective when pyrrhotite was extensively oxidised. This is because the activation product on the pyrrhotite surface (CuS species) is insoluble in the reducing condition which is not soluble in DETA (Yoon et al., 1995). However, when pyrrhotite is oxidised, the activation product is converted to oxide/hydroxides of copper and nickel, which are more soluble in DETA. The effective role of DETA in depressing pyrrhotite has led to an investigation into its applicability in depressing other sulphide minerals like pyrite. Sui et al. (1998) have shown that DETA

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can be a good depressant for pyrite. The depression mechanism of DETA on pyrite involves the removal of activating ions (in this case, Pb^{2+}) and the formation of a hydrophilic Pb–DETA complex which adsorbs back onto the pyrite surface to compete with xanthate for adsorption sites. Although the use of DETA for depressing pyrite is as good as sulphur–oxy depressants, limited work has been conducted on this depressant (Sui et al., 1998; Zanin and Farrokhpay, 2011).

In this study, the effect of DETA in combination with aeration of Cu-activated pyrite in flotation was investigated. Surface (X-ray spectroscopic, XPS and EDTA extraction technique) and solution (Ultraviolet visible spectroscopic, UV–visible and inductively coupled mass spectroscopy, ICP–MS) analytical techniques were used to understand the mechanisms of DETA on Cu-activated pyrite depression.

2. Materials and methods

2.1. Chemical composition

A high grade pyrite sample was obtained from a copper mine in Peru. The chemical/elemental compositions of the pyrite sample analysed by inductively coupled plasma mass spectroscopy (ICP–MS) is shown in Table 1.

2.2. Reagents

All the chemical reagents were of analytical grade. Potassium amyl xanthate ($CH_3CH_2OCS_2K$, abbreviated PAX) and methyl isobutyl carbinol (MIBC) were used as collector and frother, respectively. PAX and MIBC were supplied by Orica Mining and Cytec Chemicals, respectively. Copper sulphate ($CuSO_4$) was used as an activator for pyrite during the conditioning time. Diethylenetriamine ($NH_2-CH_2-CH_2-NH-CH_2-CH_2-NH_2$, abbreviated DETA, 99% pure) supplied by Sigma–Aldrich, and air were used as depressants. The pulp pH was kept constant at 10 by means of lime and hydrochloric acid additions. Ethylene diaminetetraacetic acid di-sodium salt (abbreviated EDTA, 99% pure), supplied by AJAX CHEMICALS. The reagents concentrations used were; $CuSO_4$ (200 g/t), PAX (100 g/t), MIBC (40 g/t), DETA (200 and 500 g/t) and air (2 dm³/min for 5 min). Each reagent was prepared as 1% solution strength before used in the flotation process.

2.3. Sample preparation and flotation

The experiment was carried out in a 0.5 dm³ Gliwice mechanically agitated flotation cell. Ceramic agate mortar and pestle were used to grind the pyrite sample to produce particles of $d_{80} = 38 \mu m$. The particle size distribution of the feed pyrite was determined by Malvern MasterSizer (Malvern Instrument Ltd., UK). The ground products were divided into 50 g each, kept under a desiccator to remove all moisture content before storing in a freezer below $-4 \text{ }^\circ C$ to minimise surface oxidation. After grinding, 50 g of the sample was pulped in the flotation cell and agitated for 5 min before reagent conditioning. In these tests, the effect of depressant, DETA (at different dosages) under aerated and non-aerated conditions

was examined, at pH 10. The experimental procedure is shown schematically in Fig. 1.

2.4. Rest potential measurement

The rest potential for the pyrite in the pulp during conditioning and flotation was measured using a TPS 90-FLMV pH/Eh/DO combined meter. The pH and Eh probes were calibrated using pH buffer solutions of 7 and 10 and Light's solution (Light, 1972).

2.5. X-ray photoelectron spectroscopy (XPS)

The X-ray spectroscopy (XPS) measurements were performed using a Kratos Axis-Ultra X-ray photoelectron spectrometer equipped with a delay-line detector. A monochromatised Al K α X-ray source was used, operating at $\sim 300 \text{ W}$ and the spectrometer analysis area was $\sim 0.3 \times 0.7 \text{ mm}$ dimensions. The broad scan survey and high-resolution spectral data were processed using CasaXPS version 2.3.5. The Cu-activated pyrite samples were taken from the flotation experiments (Fig. 1) and the following sample streams (Py + Cu + X, Py + Cu + X + 500 g/t DETA, Py + Cu + X + Air and Py + Cu + X + Air + 500 g/t DETA) were analysed. For the XPS analysis, each sample was analysed for Cu, Fe, S, O, C and N in terms of their surface atomic concentration (%). High resolution scans were also collected of the C 1s, O 1s, Fe 2p, Cu 2p and S 2p photoemission lines.

2.6. EDTA extraction technique

Ethylene diaminetetraacetic acid (EDTA) was used to extract metal oxidation products (e.g. oxide/hydroxide, sulphate, carbonate, etc.) from the pyrite surface (Kant et al., 1994; Rumball and Richmond, 1996; He, 2003), in the following: Py + Cu + X, Py + Cu + X + 500 g/t DETA, Py + Cu + X + Air and Py + Cu + X + Air + 500 g/t DETA sample streams. A pulp volume of 0.1 dm³ was mixed with a 3% AR grade EDTA solution and conditioned for 5 min while purging with nitrogen to prevent further oxidation of the pyrite surface. The EDTA solution was purged with nitrogen for 10 min before extraction to remove residual oxygen. The amount of surface oxidation products extracted by EDTA was measured in solution by inductively coupled plasma mass spectroscopy (ICP–MS).

2.7. UV–visible spectroscopy

An Evolution 201 UV–visible spectrophotometer was used to determine the concentration of xanthate remaining in solution before and after treatment with DETA. Identifying the peaks (for xanthate and DETA) in the process water was difficult due to the presence of other mobile ions, hence demineralised water at neutral pH was used for all the experiments. A 0.1 dm³ beaker containing demineralised water was maintained at neutral pH 7 prior to potassium amyl xanthate addition. All spectra were recorded over the range 200–400 nm. In addition to xanthate in solution, adsorption spectrum was recorded for Cu-activated pyrite induced by xanthate in the presence of DETA.

3. Results

3.1. Flotation

3.1.1. Effect of DETA and aeration

The effect of DETA on pyrite rejection was studied under two different test conditions; (1) non-oxidising (or non-aerated) and (2) oxidising, (or aerated) condition. The experimental results

Table 1
Chemical composition of the pyrite sample.

	Elements (wt.%)									
	Fe	S	Cu	Ca	Si	Pb	Mg	Zn	Mn	Py
Pyrite	44.9	53.5	0.13	0.44	0.69	0.02	0.08	0.18	0.02	>98

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