



## Agitation and column leaching studies of oxidised copper-cobalt ores under reducing conditions



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### ABSTRACT

Sulphuric acid agitation and column leaching tests of an oxidised copper-cobalt-bearing ore (1.44% Cu and 1.04% Co) were performed to investigate its amenability to copper and cobalt metals extraction under reducing conditions. Oxidised copper-cobalt-bearing ores from the Congolese Copperbelt contain a large proportion of cobalt in trivalent form, which is readily soluble in solutions of sulphuric acid, provided a reducing agent is also present. Sodium metabisulphite (Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>) is one of the most commonly recommended reducing agents, but its addition during the leach phase could cause significant environmental problems and could affect copper recovery if present in large amounts. During leaching, sodium metabisulphite reacts with sulphuric acid and dissociates to form SO<sub>2</sub> which in fact reduces Co(III). However, SO<sub>2</sub> is only partially utilised, and much of it is entrained unreacted in the leach liquor. A large accumulation of SO<sub>2</sub> can become uncontrollable and therefore escape to the environment if not consumed by the ore. The paper presents and discusses the effect of recirculation of the leach liquor after pH readjustment to 1.5 on the minimisation of unreacted SO<sub>2</sub> entrained in the leach liquor, sulphuric acid consumption; and the overall improvement of copper and cobalt extraction yields. The most important information found is that recirculation of the leach liquor proved to be very useful in reducing the amount unreacted SO<sub>2</sub> and significantly increasing recovered metals yields with little acid consumption. Moreover, the injection of Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution at 75% of the column bed-height considerably reduced the potential risks of SO<sub>2</sub> emanation.

### 1. Introduction

The treatment of copper-cobalt-bearing ores by hydrometallurgical process requires a preliminary leaching step. However, the majority of copper- and cobalt-bearing ores mined in the Democratic Republic of Congo, and particularly in the Katanga province, contain cobalt as a heterogenite with a fairly large proportion as trivalent cobalt (Kime, 2017). This form of cobalt is insoluble during leaching and requires the presence of a reducing agent for its solubilisation in a sulphuric medium (Mwema et al., 2002; Barriga et al., 2004; Kime and Makgoale, 2016). Common reducing agents for dissolving the trivalent cobalt include ferrous ions and sulphur dioxide. Unlike ferrous ions, a number of environmental and operational issues have been raised concerning the use of SO<sub>2</sub> as a reducing agent for cobalt (Ferron, 2008; Ferron and Henry, 2008). These include environmental and safety risks associated with gaseous SO<sub>2</sub> emissions, even when using SO<sub>2</sub> as sulphurous acid (Ferron, 2008; Ferron and Henry, 2008; Apua and Mulaba-Bafubiandi,

2011). These risks are minimised to some extent by using SO<sub>2</sub> derivatives such as sulphite (Na<sub>2</sub>SO<sub>3</sub>) or metabisulphite (Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>) (Ferron, 2008). During reductive leaching with SO<sub>2</sub> or its derivatives, a decrease in copper extraction is also observed. This is mainly due to the precipitation of “Le Chevreult’s salt” (CuSO<sub>3</sub>·Cu<sub>2</sub>SO<sub>3</sub>·2H<sub>2</sub>O) which occurs when leaching is carried out at pHs greater than 2 (Ferron, 2008). The chemical equation of the formation of “Le Chevreult’s salt” is given by:

$$3\text{CuSO}_4 + \text{SO}_2 + 6\text{H}_2\text{O} \rightarrow \text{CuSO}_3 \cdot \text{Cu}_2\text{SO}_3 \cdot 2\text{H}_2\text{O} + 4\text{H}_2\text{SO}_4 \quad (1)$$

The techniques used to dissolve the minerals are mainly percolation and agitation leaching. Agitation leaching generally involves dispersing the finely ground ore in water to obtain a pulp containing 40–70% by weight of the solids and subsequently adding the acid or base solution. Agitation makes it possible to avoid the sedimentation of the particles, but also promotes leaching kinetics, since the reactions are heterogeneous (Chong et al., 2013a, 2013b). Agitation leaching always leads to better solubilisation efficiencies for metals, but requires a large

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investment in reactor maintenance and energy for the implementation of agitation compared to percolation leaching (Ghorbani et al., 2016; Oxley et al., 2016; Staden et al., 2017). As far as percolation leaching is concerned, it has the advantages of having a low investment cost required for the implementation of the leaching operations and a relatively low operating cost. In addition, percolation leaching is typically conducted at a coarser size fraction than agitated tank leaching, thus reducing the power requirement and reagent consumption. These economic benefits make the use of heap leaching technique beneficial for the extraction of precious metals, copper, nickel and cobalt from low-grade minerals and mining waste (Quaicoo et al., 2014; Ghorbani et al., 2016; Panda et al., 2015). In percolation leaching, the lixiviant percolates by gravity through the metalliferous mass which remains static. In general, the ore is arranged in heaps, trays or columns.

As has been shown, SO<sub>2</sub> emissions and related environmental problems may even be exacerbated in reductive percolation leaching by using sodium metabisulfite if a controlled mechanism is not implemented. The efficiency of reductive leaching of oxidised copper-cobalt ore with sulphuric acid is highly dependent on pH (≈ 1.5). The pH can be maintained by the addition of make-up acid. Moreover, the amount of reducing agent used to reduce Co(III) is only partially utilised (Miller, 2008), a good part of it is therefore found unreacted in the leach liquor in forms of SO<sub>2</sub> or sulphite species. Although much research work on the dissolution of cobalt (III) has been reported, most of it was done to demonstrate that cobalt (III) could be readily dissolved under reducing conditions (Mwema et al., 2002; Apua and Mulaba-Bafubandi, 2011; Ndalamo et al., 2011; Chong et al., 2013a, 2013b; Kime et al., 2016). However, how to lower the high consumption of the sulphuric acid and the amount of reducing agents that entail high operating costs and associated environmental issues has been left unexplored. It is with this in mind that the present work was initiated in an attempt to seek ways of improving cobalt extraction during the H<sub>2</sub>SO<sub>4</sub> column leaching of copper-cobalt-bearing ores using sodium metabisulphite as the reducing agent while minimising the amount of unreacted SO<sub>2</sub> and the amount of sulphuric acid used.

## 2. Material and methods

### 2.1. Materials

An oxidised copper-cobalt-bearing ore was used in this study. 1200 kg of the ore sample were sourced from Kwatebala mine (operated by Tenke Fungurume mine). This mine is located 177 kilometres north-west of the city of Lubumbashi in Katanga province, in a 1600 square kilometre concession. The particle size distribution of the as-received sample is given in Table 1.

10 kg, carefully quarried out of the ore sample was ground down in a closed circuit with a 75 µm limiting screen using a ball mill for the agitation leaching tests. Another 1 kg of the ore sample was ground down using a series of roller mills and then pulverised to dimensions of less than 10 µm for chemical and mineralogical analyses.

### 2.2. Characterization

Chemical analysis was carried out by using inductively coupled

**Table 1**  
Particle size distribution of the as-received ore sample.

Sample size fraction (mm)	(wt.%)
+ 25	13.88
– 25 + 12	12.08
– 12 + 3	25.36
– 3 + 1	14.91
– 1	33.77

plasma-atomic emission spectroscopy (ICP-AES) for analysing major elements and compounds. The ore sample was dissolved in aqua regia (HCl with HNO<sub>3</sub> (3:1)) for the determination of the total copper and total cobalt, and was dissolved in sulphuric acid for the determination of copper and cobalt in oxide form. Table 2 presents the chemical assays of the ore sample.

XRD analyses were carried out on Rigaku Ultima IV diffractometer. The diffractometer functions in optimal conditions analysis with 40 kV of tension and 30 mA of intensity. The different mineral phases were identified using software “PDXL” which searches and matches diffraction patterns to reference patterns. Table 3 presents the mineralogical phases of the ore sample.

### 2.3. Agitation leaching

Batch agitation leaching tests were performed using a 1 L polypropylene beaker provided with a pH-temperature electrode to determine the maximum solubilisation yield of cobalt and copper and the proportion of trivalent cobalt in the ore. A total of 200 mL of 75 g/L H<sub>2</sub>SO<sub>4</sub> aqueous solution was placed in the beaker and a weighted ore sample (15% (w/v)) was added. The overall solution was placed on a hot plate and agitated using a magnetic stirrer at room temperature and run for 2 h. Tests were run with and without the addition of a reducing agent. The potential was maintained at ≈ 340–350 mV by the addition of sodium metabisulphite. The pH was kept constant (≈ 1.5) by adding H<sub>2</sub>SO<sub>4</sub> solution at 500 g/L. The leachate and residue were analysed for their Cu, Co, and Fe contents. It was also possible to obtain an estimate of the amount of Co<sup>3+</sup> in the sample by re-leaching the residue obtained from the non-reducing test in a reducing medium. It was also important to look at the total acid consumption (TAC) as it can affect radically operating costs and recoveries. TAC can be expressed as follows:

$$\text{TAC} = \left[ \frac{\text{kg H}_2\text{SO}_4}{\text{t ore sample}} \right] = \frac{m_{AL} - m_{AF}}{m_{TA}} \times 1000 \quad (2)$$

where  $m_{AL}$  is the mass of acid in the lixiviant (in g),  $m_{AF}$  is the mass of acid in the filtrate, and  $m_{TA}$  is the total acid (in g). The free acid in the filtrate was determined by volumetric titration by using a standard 1 M NaOH solution.

### 2.4. Percolation column leaching

Percolation column leaching tests were conducted in two types of columns: small laboratory column and large semi-pilot column. The small column was made of polyvinyl chloride material with a height of 1.2 m and an internal diameter of 9.5 cm. Its loading capacity was 5 kg of ore sample. The large column was made of Plexiglas material, 2 m in height and 0.46 m in internal diameter. This type of column was able to process a mass of 200 kg. Small column leaching tests, with and without the addition of a reducing agent were executed to determine the best values of flowrates and of the durations of the leaching cycles. Large column leaching tests were executed to determine the solubilisation yields to be obtained from pilot scale trials. The schematic diagram of the percolation columns is shown in Fig. 1. Top of the column was kept open for loading the sample as well as supplying the H<sub>2</sub>SO<sub>4</sub> aqueous solution. When the reducing agent was used, it was injected at 75% of the column bed-height. The H<sub>2</sub>SO<sub>4</sub> aqueous solution and sodium metabisulphite solution were fed through spiral shaped perforated hoses using metering pump schemes from two respective storage tanks, 200 L and 50 L, respectively. Before the ore sample was loaded, a screen was placed at the bottom of the columns to allow only the percolate to pass through. A hose restrictor with an opening of 1 cm of diameter was used as the outlet. The leach liquor was made to pass through the ore sample continuously by gravity and cyclically re-circulation, after readjusting the pH to the setpoint of 1.5, until a sufficient concentration of copper

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