



# Production of copper cathode from oxidized copper ores by acidic leaching and two-step precipitation followed by electrowinning

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## ABSTRACT

A step by step hydrometallurgical process for the production of copper cathode was developed after a two-step precipitation from leaching solution of copper oxidized ore, followed by copper concentrate leaching and electrowinning. The copper oxidized ore was primarily comminuted to a size below 100  $\mu$ , followed by acidic leaching at 25 °C for 40 min in H<sub>2</sub>SO<sub>4</sub> solution, in which recovery of copper and iron were 95.95% and 12.63%, respectively. To remove iron impurity, at the first step of precipitation, NaOH was added to increase pH from about 1.5 to the optimum pH of 3.8 at 60 °C for 60 min; thus iron precipitation with recovery of over 80% was achieved. Copper precipitate as concentrate was obtained in the same method from iron-removed solution. The optimum condition of copper precipitation was found to be pH of 5.5, 25 °C and 45 min with 98.69% recovery. One of the advantages of this process was production of Na<sub>2</sub>SO<sub>4</sub> with 99.1% purity after vaporization of the remaining solution from two-step precipitation. The obtained copper concentrate was leached at approximately the same condition of the first leaching step, and then the provided pregnant solution proceeded to an electrowinning cell with lead alloy anode contained antimony and steel sheet cathode under the following condition: temperature of 50 °C, reaction voltage of 2 V and current density of 300 Am<sup>-2</sup>. Finally, a scale-up experiment was carried out and the copper cathode with 99.99% purity produced.

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## 1. Introduction

The copper commonly produced from sulfide and oxide copper ores is extracted through pyrometallurgical and hydrometallurgical processes, respectively. In processing of oxide ores, the first step is to leach ore by using a leaching agent commonly sulfuric acid. Several studies have investigated the leaching of copper oxide ores with different types of acids (Habbache et al., 2009; Moradkhani et al., 2011). In such processes, the major problem is the presence of iron in ore, which comes to leach solution associated with copper ions. In order to remove iron from leach solutions, several methods were applied (Gudeczauskas and Natalie, 1985; Ismael and Carvalho, 2003; Nurmi et al., 2010; Principe and Demopoulos, 2004; Wang et al., 2011). With respect to the type of acid used in the leaching step, the pregnant solution proceeds to one of the following processes such as cementation, ion exchange-electrowinning and or solvent extraction-electrowinning circuits (Bartos, 2002; Harris et al., 2007; Stefanowicz et al., 1997).

In this study, we developed a new systematic hydrometallurgical process for the production of copper cathode from leaching solution

of copper concentrate obtained after leaching of copper oxidized ore, two-step precipitation and copper concentrate leaching, followed by electrowinning step. The remarkable feature of this process is the production of some co-products such as Na<sub>2</sub>SO<sub>4</sub>. In this research, the optimum condition of iron and copper precipitation from the leach solution and electrodeposition of copper from the copper concentrate leach solution were investigated. Finally, scale-up of the process was conducted to reach the developed flowsheet.

## 2. Experimental methods

### 2.1. Materials and reagents

The investigation was carried out on a lean oxidized copper sample from Chodarchai Mine in Zanzan, Iran. The feed of leaching obtained from mine ores was crushed and ground to a particle size below 100 $\mu$ . The feed was not exposed to any process. The composition of the oxide ore is presented in Table 1. The results of X-ray diffraction (XRD) analysis revealed that quartz is major phase and malachite is minor phase. The results also indicated calcite, muscovite and illite are present as trace phases. H<sub>2</sub>SO<sub>4</sub> and NaOH were provided by Merck Company.

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**Table 1**  
Chemical composition of copper oxide ore in the experiments.

Element	Cu	Fe	Zn	Pb	Ni	Cd	Mn	Co	As	Mg	Ca	S	Al
wt (%)	3.01	2.73	0.21	0.20	0.01	0.00	0.08	0.03	0.01	0.13	0.58	0.31	3.5

## 2.2. Leaching experiments

In this research the copper oxide and copper concentrate obtained from copper precipitation step were leached. The leaching experiments were carried out in a flask with mechanical stirrer under the following condition: H<sub>2</sub>SO<sub>4</sub>, 2 molar; leaching time, 40 min; at the room temperature. The L:S ratio (v/w) for the leaching of copper oxide and copper concentrate was conducted at 3.8:1 and 5:1, respectively. The concentration of copper was calculated with respect to correction of volume (Choo et al., 2006).

## 2.3. Iron and copper precipitation experiments

A series of experiments designed by Response Surface Methodology (RSM) was carried out to find the optimized time, pH and temperature for the iron precipitation. Initially, 150 mL of the leach solution was mixed and heated at different temperatures for various times and simultaneously adjusted to a certain pH value using NaOH. At the end of the experiments, the pulp was filtered, and the filtrates and the precipitates were separately analyzed for iron and copper.

After finding the optimized condition for the iron precipitation, a series of experiments was run to find the effect of time, pH and temperature on the copper precipitation with NaOH. For this purpose, after precipitation of iron at the optimized condition, 150 mL of the iron-removed solution was used to investigate the effect of the aforementioned parameters.

## 2.4. Electrowinning experiment

The copper solution achieved from the leaching of copper concentrate was used in electrowinning experiments. The leach solution with respect to the presence of returned spent in scale-up system was diluted. Therefore, we employed an electrolyte containing Cu 54.13 g/L, Fe 1.1 g/L without other elements. A lead alloy anode containing antimony and one stainless steel cathode in a reactor containing 500 mL of the copper solution created the electrowinning system. The areas of cathodes and anodes were 70 cm<sup>2</sup>.

The electrolyte was slightly agitated with a magnetic stirrer (heater stirrer model Yellow line, MST basic C). To supply current, a digital laboratory DC power supply (MEGATEK, model MP-3005D) was used. During electrolysis, the potential was adjusted by varying the current manually. Finally, solid depositions on the cathodes were

**Table 2**  
Concentration of elements in the leaching solution and the leaching rates.

Components	Concentration (mg/L)	Leaching (%)
Cu	7.600 (g/L)	95.947
Fe	1.000 (g/L)	12.625
Pb	10.150	0.128
Zn	166.910	2.107
Cd	0.960	0.012
Ni	1.410	0.018
Mn	0.090	0.001
As	3.000	0.038
Ba	0.300	0.004
Ca	0.490	0.006
Mg	0.000	0.000
Al	9.160	0.116
Co	0.500	0.006
Ce	0.290	0.004
Cl	1.740	0.022

stripped, washed with demineralized H<sub>2</sub>O, dried in the air, weighted and analyzed. All electrowinning experiments were conducted at the temperature of 40–60 °C, reaction voltage of 2 V and current density of 200 Am<sup>-2</sup>.

## 2.5. Chemical analysis

The concentrations of iron and copper in the solutions were analyzed by Perkin-Elmer AA300 model atomic absorption spectrophotometer. The cathode, precipitates and chemical composition of the copper oxide ore were analyzed by XRF. Leaching toxicity extraction test of the leaching residue was performed in accordance with Solid Waste Extraction Procedure for Leaching Toxicity – Horizontal Vibration Method (China GB5086.2-1997 and US EPA Method 1311 TCLP test).

## 3. Results and Discussion

### 3.1. Leaching

The primary leaching test results are shown in Table 2. The leaching rate of copper and iron are 95.95% and 12.63%, respectively. At this condition, copper and iron concentrations are 7.6 g/L and 1 g/L, respectively. The extractions of the other elements Pb, Cd, Ni, Mn, As, Ba, Ca, Mg, Al, Co, Ce and Cl are all less than 15 mg/L and for Zn is 166.91 mg/L. The effect of operational parameters on the leaching has been investigated by Moradkhani et al. (2011) and the results were published elsewhere.

The further leaching was carried out on the copper concentrate obtained from copper precipitation step. At this stage, the leaching rate of copper was 99%. The concentrations of copper and iron in the leaching solution were 97.45 g/L and 1.98 g/L, respectively. This solution is ready to be sent to the electrowinning cell. The impurities of the electrolyte entered to the electrowinning cell are illustrated in Table 3. As seen in this table, the impurities of the electrolyte are lower compared to the copper electrowinning limitations.

### 3.2. Iron Precipitations

The effect of pH, temperature and reaction time on the iron precipitation recovery (RFe) was studied. The optimum condition of parameters and their interactions were investigated by RSM. The effect of pH and temperature on iron precipitation recovery can be seen in Fig. 1a. As seen in the figure, the recovery of iron precipitation increases with increasing pH and no considerable change is observed by raising the temperature. Moreover, Fig. 1b shows that the effect of pH on RFe is

**Table 3**  
Composition of the electrolyte entered to EW cell.

Ref.	Electrowinning limitations (mg/L)	Leaching test (mg/L)	Elements
Habashi (1997)	–	96180	Cu
	2000	1940	Fe
	430	92	Zn
	16000	2.10	Ni
	340	0.02	Sb
	3580	1.20	As
Davenport et al. (2000)	1	0.20	Sn
	100	0.86	Bi
	20–50	1.12	Cl

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