



Leaching of Trace Amounts of Metals from Flotation Tailings in Cupric Chloride Solutions

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Abstract

With decreasing ore grades, the tailings of mining operations are becoming of increasing interest as metal-containing secondary raw materials. The objective of the current work was to investigate chloride leaching of gold, copper, cobalt, nickel, and zinc present in the flotation tailings. In the current study, the effect of cupric ion as an oxidant (0–50 g/L) and NaCl (150–250 g/L) on metals extraction was investigated. The other parameters, such as pH (1.8), temperature (95 °C), solid/liquid ratio (25%), oxygen feed rate (1200 mL/min), leaching time (24 h), and stirring rate (950 rpm), were kept constant. Gold dissolution rate was shown to increase with increase in cupric ion concentrations up to 50 g/L. Also, increase in NaCl concentration up to 250 g/L increased gold extraction. Majority of the copper present in the flotation tailings could be dissolved with all the all solutions investigated. The other base metals, cobalt, nickel, and zinc extractions were also shown to increase with increase in the cupric ion and sodium chloride concentration. However, even in the absence of cupric ion addition ($t = 72$ h, 250 g/L NaCl), the final extraction of Cu, Ni, Zn, Co, and Fe increased up to 98, 93, 83, 76, and 80%, respectively. This shows the power of inherently originating oxidants present in the tailings. Furthermore, the solid analysis of the leach residue indicated that leaching the flotation tailings in pure NaCl may result in partial gold dissolution. The results demonstrate that significant amount of metals present in the tailings could be extracted even with only NaCl as added chemical in the presence of oxygen feed, oxidizing agents originating directly from the raw material. This can provide an advantageous cyanide-free method for extraction of metals from very low-grade tailings with low chemical consumption.

Keywords Sustainable leaching · Waste as raw material · Cyanide-free · Gold · Circular economy

1 Introduction

Extraction of metals from tailings and low-grade ores is of increasing interest due to the continuously increasing need of metals, decreasing amount of valuable metal resources, depletion of mines, and annual production of millions of tons of residues. Mining wastes can have significant amounts of base and precious metals, such as cobalt, nickel, copper, zinc, gold, and silver [10, 24], but also toxic elements like arsenic, lead, and cadmium [1]. Nickel and cobalt are very important critical metals in battery industry and their expected growing

demand is Co 20%, Ni 40% by 2025 [19]. Furthermore, sulfidic flotation tailings can be environmentally hazardous as they oxidize when exposed to water and oxygen. This reaction produces acidic wastewater and sulfates, which are environmentally hazardous [6]. Thus, various hydro- and pyrometallurgical methods and mineral processing technologies have been explored for reprocessing the tailings [4, 5].

Cyanide is the most commonly applied leaching reagent in gold extraction from ores or secondary materials. Nevertheless, there are many disadvantages with the use of cyanide, such as high toxicity and low effectivity for carbonaceous ores. For these reasons, alternative cyanide-free reagents have been investigated both in laboratory scale and in pilot or demonstration scales over the last decades [9, 12, 32]. Alternative reagents for metal leaching include halogens (chloride, bromide, and iodide), thiourea, thiosulfate, thiocyanate, ammonia and glycine, among others [3, 7, 18, 25, 31]. One of the most promising alternative process is chloride leaching as it is less toxic, enables fast dissolution kinetics, poses less problems with gold

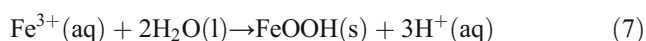
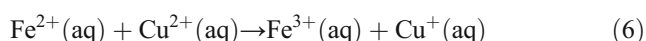
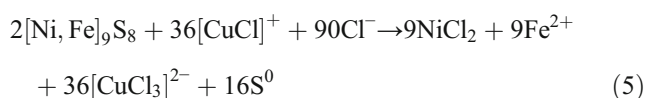
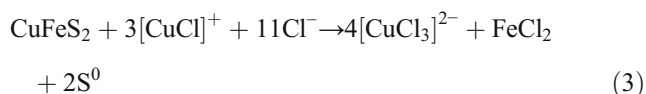
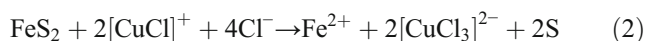
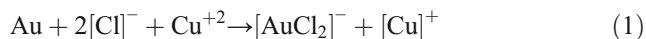
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passivation, and is effective at leaching refractory ores [33]. Chloride leaching is typically performed at ambient pressure, at elevated temperatures, such as 65–95 °C, and with pH kept at a value lower than 3 to keep copper and/or iron soluble. Previous research has utilized chloride as an alternative gold leaching agent for direct gold leaching [3, 18, 25, 31], as a leaching media for gold-copper containing refractory copper concentrates [22, 23], and as leaching media for other base metals [37]. Hypochlorous acid (HClO)/hypochlorite ions (OCl⁻), cupric ions (Cu²⁺), and ferric ions (Fe³⁺) have been used as oxidizing agents whereas chloride acts as the complexants, forming a complex with the dissolved complexing agent for gold. Also, oxygen is used as indirect oxidant to oxidize cuprous ions into divalent stage [2, 11, 14, 33].

Cupric ions are a promising oxidant for chloride leaching, due to their strong oxidizing property and natural and easy diffusion in leach solution [18]. Lundström et al. [22, 23] demonstrated that in concentrated chloride solution with cupric as an oxidant, free-milling gold is dissolved according to Eq. (1). According to Eqs. (2) and (3), pyrite and chalcopyrite also can be dissolved [20]. Other metals (Zn, Ni, Co.), which are in sulfide minerals can be oxidized following Eqs. (4) and (5), and iron can be precipitated as a FeOOH at suitable pH (Eqs. (6) and (7)) [15, 21, 27].



In the typical cupric chloride leaching system for gold, cupric ion concentration is between 1 and 40 g/L, redox potential for the couple [CuCl]⁺/[CuCl₃]²⁻ is between 550 and

700 mV vs. Ag/AgCl and pH, typically kept at lower than 3. Gold dissolution rate increases at elevated temperature (65–95 °C) and with increasing cupric chloride concentration [35]. When the raw material is pure gold, concentration of NaCl has been reported to have a minor effect on the gold dissolution rate, although it affects anodic reactions and complex formation [26, 36].

Currently, Outotec Gold Chloride [27], HydroCopper® ([8].) Intec [29], Dundee [17], and Nippon N-Cho/NIKKO [13] processes have been applied in cupric chloride leaching in an industrial demonstration scale. Table 1 summarizes the concentrations of chloride and cupric ions in the chloride leaching processes operated. In addition to the given values, the processes use bromide to increase the gold complex stability and/or dissolution rate.

The aim of this work was to extract gold present in flotation tailings, but also other valuable metals from flotation tailings by cupric chloride leaching. Experiments were conducted to determine the effect of cupric and chloride ion concentrations on the dissolution of gold, copper, nickel, cobalt, zinc, and iron.

2 Materials and Methods

In this study, the raw material investigated was flotation tailings. The sample was homogenized and divided to the sub-samples and chemically analyzed for elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (Thermo Scientific iCAP 6000) after total dissolution (TOT). Iron, nickel, arsenic, and cobalt contents were analyzed also after bromine methanol dissolution (BM), which enables the quantification of the pyrite and pyrrhotite contents [16, 30]. Gold was analyzed from duplicate samples first by total leaching followed by inductively coupled plasma mass spectrometry (ICP-MS) (Thermo Scientific iCAP Q) and by fire assay method. Main gangue minerals were identified by X-ray diffraction (XRD) and polished sections were prepared for mineralogical study. Gold grains were identified using both manual and automated rare particle search with field emission scanning electron microscope. The mineral composition of the studied flotation tailings was calculated using HSC Chemistry® software.

Table 1 Chloride and cupric ions concentration in industrial development stage processes for gold chloride leaching (modified from [21])

Process	Raw material	[Cl] (g/L)	[Cu ²⁺] (g/L)	[Br] (g/L)
Nichromet/Dundee	Oxidized ore	150	75	*
Nippon N-chlo/NIKKO	Cu sulfide ore with Au	120–200	40–100	*
Intec process	Cu sulfide ore with Au	120–180	~25	*
HydroCopper®	Cu sulfide ore with Au	140–180	From raw material	*
Outotec gold chloride process	All non-pregrobbing feeds	125–225	> 10	*

*Bromide addition used 1–20 g/L

Table 2 NaCl and cupric ion concentrations of experiments (g/L)

Experiment no.	[NaCl] (g/L)	[Cu ²⁺] (g/L)
1	250	10
2	250	30
3	250	50
4	250	0
5	200	30
6	150	30

The particle size distribution of the sample was measured with laser diffraction method by Malvern Mastersizer 3000 analyzer. The samples were ground for 30, 60, or 120 min grinding time in laboratory ball mill at slurry density 65 wt% and 600 rpm rotation speed.

2.1 Leaching Experiments

The cupric chloride leaching tests were performed at Outotec Research Center, Pori, Finland. The tests were run for 24 h in a 3-L titanium reactor, with 950 rpm stirring rate which corresponds to the circumferential speed of 2.68 m/s. The effect of cupric ion (copper (II) chloride dihydrate, CuCl₂·2H₂O, VMR chemicals, technical grade) was investigated with concentrations of 0, 10, 30, and 50 g/L and sodium chloride (NaCl) (MERCK, analytical grade) with concentrations of 150, 200, and 250 g/L. Conditions investigated in batch leaching experiments are presented in Table 2. During the leaching tests, temperature (95 °C), oxygen purging (1200 L_N/min), solid–liquid ratio (1:3), and pH (1.8) were kept constant and adjusted with HCl (37%) and NaOH (20 g/L). Test 4 was performed in different conditions: pH was kept at 1 and the leaching time was prolonged to 72 h. During and after the leaching, samples were taken at 0, 1, 2, 4, 8, 12, 16, and 24 h from the solution and solid–liquid separation was performed by filtration. Metal contents in the solid residues were determined by total leaching and ICP-OES. Gold concentration in the final solution samples was analyzed by ICP-MS and in the residues by fire assay method.

3 Results and Discussion

3.1 Characterization of the Material

Initial material represents the tailings of flotation circuit. The studied sample contains several metals of interest: 3.56% iron, 0.04% cobalt, 0.04% zinc, 0.03% nickel, and 0.09% copper, the main elements being SiO₂ (54.4%), MgO (9.47%), and Al₂O₃ (2.87%). The average gold content of the tailings based on average of two duplicate fire assay measurement is

Table 3 The content of main elements of interest in Kylylahti flotation tailings

Element	Fe (%)	Co (%)	Ni (%)	Cu (%)	Zn (%)	Au (ppm)
Content	3.56	0.04	0.03	0.09	0.04	0.21

0.21 ppm Au (Table 3). The tailings consist mainly of hornblende-type amphibole, quartz, and dolomite that account for 83% of the minerals. Phlogopite, chlorite, and magnetite occur as accessory gangue minerals. Pyrrhotite and pyrite are the most common ore minerals with accessory chalcocopyrite, pentlandite, and sphalerite (Table 4). Chalcocopyrite was encountered mainly as locked particles with pyrite and pentlandite locked with pyrrhotite or as exsolutions (i.e., flame pentlandite) in it. Gold occurs as native gold with a grain size between 1 and 3 μm mainly at the cleavage surfaces and at the rims of gangue and sulfide particles.

The particle size distribution (d_{80}) of original raw material was 150 μm. After the grinding ($t = 30, 60, 120$ min), particle size (d_{80}) was 75, 40, and 26 μm, respectively. The finest particle size used in the leaching tests was 26 μm (Fig. 1).

3.2 The Effect of Cupric Ion Concentration on Elemental Dissolutions

Effect of cupric ion concentration was investigated in three different leaching experiments (nos. 1, 2, and 3) with varying (10, 30, and 50 g/L) additions of cupric chloride into sodium chloride solution (250 g/L). Figure 2 shows the extraction of gold to product liquid solution (PLS). It can be observed that gold extraction increased up to 58% with the increase of cupric concentration to 50 g/L. Gold concentration was under detection limit when using 10 g/L cupric ion as oxidant. Besides, metal concentrations, pH, and redox potential of the

Table 4 Mineralogical composition of Kylylahti flotation tailing

Mineral	Content (%)
Pyrite	1.83
Pyrrhotite	2.85
Pentlandite	0.08
Chalcocopyrite	0.25
Sphalerite	0.07
Hornblende	22.54
Phlogopite	5.04
Chlorite	6.11
Magnetite	0.90
Quartz	35.02
Dolomite	25.33
Total	100

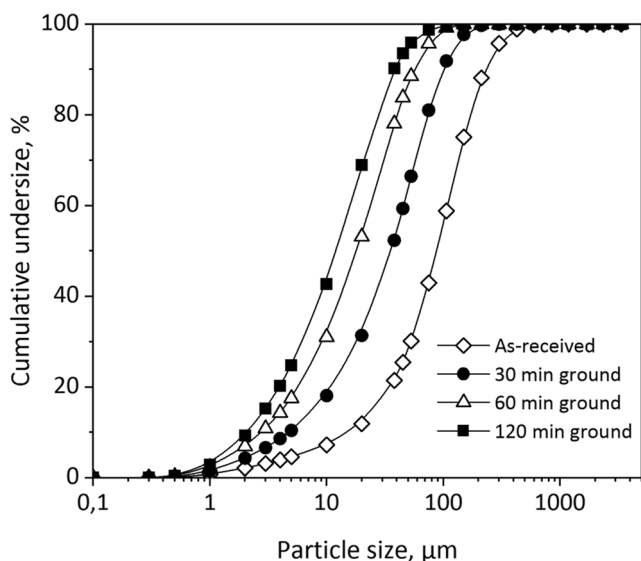


Fig. 1 Particle size distribution of tailings raw material as-received and after grinding by ball mill

final pregnant leach solutions of the experiments are shown in Table 5. These results show that cupric ion concentration has a significant effect on gold extraction. Also, Lampinen et al. [18] have investigated the effect of cupric concentration on gold leaching by cupric chloride solutions, with elemental gold electrode. The gold dissolution rate was increased when cupric concentration increased up to 0.5 M (31.7 g/L Cu^{2+}), and at that concentration the gold dissolution reached its maximum. However, in our work, increase of cupric ion concentrations above 30 g/L was shown to result in further increase in gold extraction into the solution. Also, in the current study, the raw material was gold containing flotation tailings, having

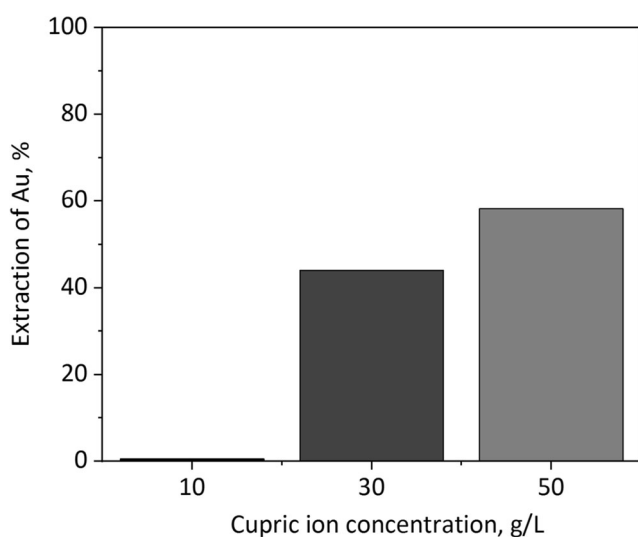


Fig. 2 The effect of cupric ion concentration on gold extraction (%) from the investigated flotation tailings. Solution had 250 g/L NaCl, $T=95\text{ }^{\circ}\text{C}$, $\text{pH}=1.8$, and 24 h

Table 5 Metal contents, pH, and redox potentials of final solution of leaching experiments

Sample name	Au (mg/L)	Co (mg/L),	Ni (mg/L)	Zn (mg/L)	Fe (mg/L)	Final pH	Final redox potential (mV vs. Ag/AgCl)
Exp. 1	<0.01*	50	39	60	337	1.76	580
Exp. 2	0.019	61	45	71	121	1.67	601
Exp. 3	0.03	64	46	73	707	1.60	638
Exp. 4**	<0.01*	71	61	84	7040	1.01	590
Exp. 5	0.012	58	47	71	189	1.8	599
Exp. 6	<0.01*	49	35	56	809	1.78	612

*Below detection limit, **Cu concentration 265 mg/L

different material, particle, and surface characteristics compared to pure gold electrode.

The effect of cupric ion concentration on copper extraction from final residue is shown in Fig. 3. It was observed that increase in cupric ion concentration did not result in a linear response for copper extraction. The highest copper leaching yield (78%) was obtained with the lowest cupric ion concentration (10 g/L) and the lowest (64%) with the medium cupric concentration (30 g/L). However, the copper concentration in the raw material was low (0.03%) compared to the concentration of cupric ion added as oxidant into the solution. It can be concluded that majority of the copper present in the flotation tailings was dissolved at all the conditions ($[\text{Cu}^{2+}] = 10\text{--}50\text{ g/L}$) investigated.

Co, Ni, and Zn extractions as a function of time are shown in Fig. 4a–c, respectively. It can be seen that cobalt dissolution had slower kinetics, maximum extraction being achieved only at ca. 12 h dissolution (Fig. 4a). It can be referred to the slow

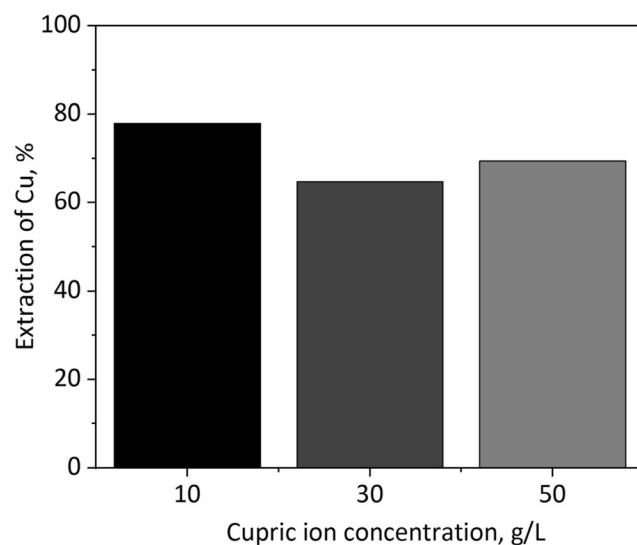
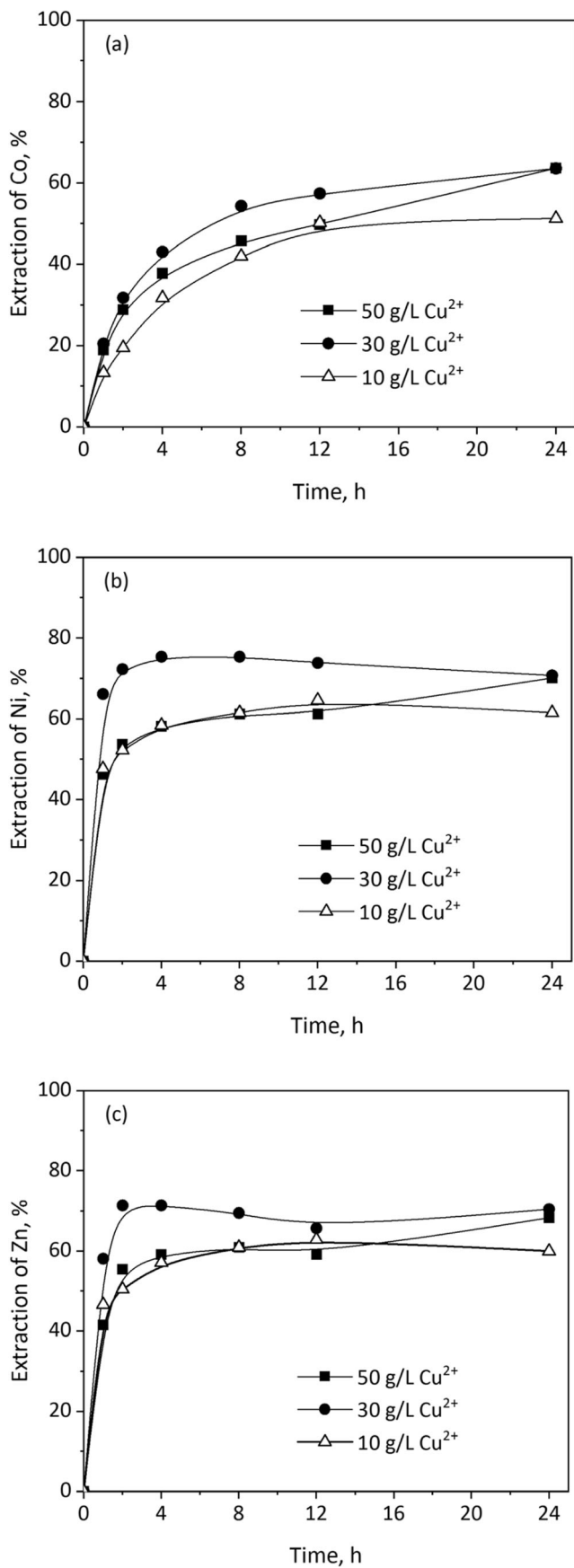


Fig. 3 The effect of cupric ion concentration on copper extraction (%) from the investigated flotation tailings. Solution had NaCl = 250 g/L, $T=95\text{ }^{\circ}\text{C}$, $\text{pH}=1.8$, and 24 h



◀ Fig. 4 The effect of cupric ion concentration on cobalt (a), nickel (b), and zinc (c) extractions (%) from flotation tailings. Solution had 250 g/L NaCl, $T = 95\text{ }^{\circ}\text{C}$, $\text{pH} = 1.8$

leaching kinetic of pyrite mineral matrix, being the main cobalt carrying mineral in the investigated raw material. The dissolution behavior of Zn and Ni was similar, and the maximum extraction into the solution was achieved already during the first 2–4 h (Fig. 4b, c). The main nickel and zinc carrying minerals are pentlandite and sphalerite which have fast leaching kinetics. The maximum extractions for Co, Ni, and Zn (63, 70, and 72%, respectively) were gained at 30 g/L cupric ion and 250 g/L NaCl concentration (Experiment no. 2). For Ni and Zn, this solution showed 10–20% units higher extraction at early hours compared to solutions with $\text{Cu}^{2+} = 10$ and 50 g/L. However, with increase in time the difference decreased.

In order to keep majority of iron at solids, pH was adjusted to value 1.8, iron dissolution into the solution being less than 15%. The only exception was experiment number 4 in which no cupric ions were added but the dissolving iron from tailings was taken as naturally originating oxidizing agent. Thus, pH was kept at 1 to keep the ferric oxidant soluble.

3.3 Elemental Dissolution of Metals in the Absence of Added Cupric Ion

Experiment 4 was performed without oxidant, i.e., cupric ion addition; thus, the oxidizing ions inducing tailings dissolution originated from the raw material itself. Oxygen purging was applied to oxidize and regenerate naturally dissolving metal ions. This experiment conducted for a longer period, 72 h, in

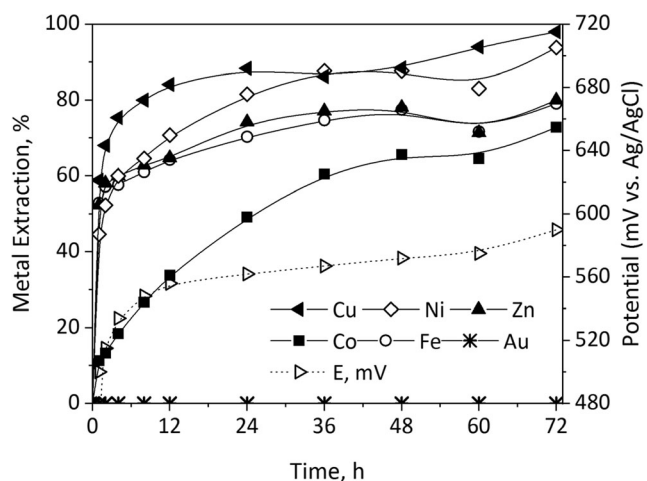


Fig. 5 Extractions of Au, Co, Ni, Fe, Cu, and Zn (%) into the solution without external cupric addition, based on solution analysis. Redox potential (mV vs. Ag/AgCl) presented on the right axis. Solution had 250 g/L NaCl, $T = 95\text{ }^{\circ}\text{C}$, $\text{pH} = 1$, and O_2 feed = 1200 mL/min

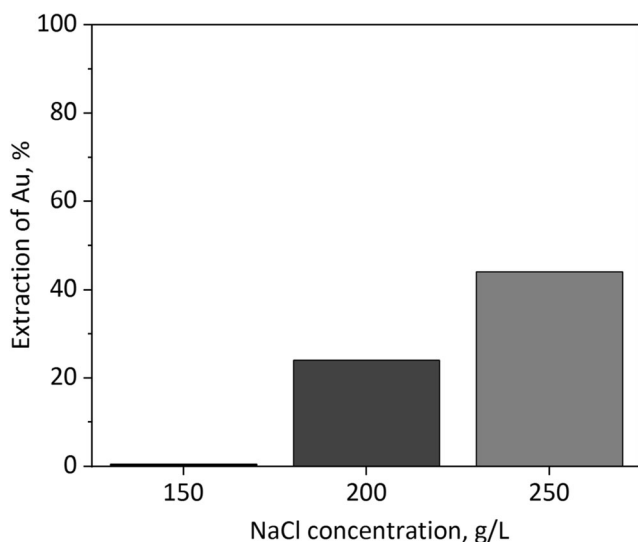


Fig. 6 The effect of NaCl concentration on gold extraction (%). Solution had $[\text{Cu}^{2+}] = 30 \text{ g/L}$, $T = 95 \text{ }^\circ\text{C}$, $\text{pH} = 1.8$, and 24 h

250 g/L NaCl, 1200 mL/min oxygen feed and the pH was kept at 1. It was observed that in the end of the leaching test, the extraction of Cu, Ni, Zn, Co, and Fe into the solution were 98, 93, 83, 76, and 80%, respectively (Fig. 5). It can be observed that while copper, nickel, zinc, and iron had fast dissolution kinetics during the first 12 h, cobalt had lower (30–50% units) extraction and higher redox potential favoring Co-bearing pyrite dissolution. According to solution analysis, gold did not dissolve in this experiment. However, the solid analysis suggested 46% gold extraction into the solution. Even though Velasquez et al. [34] noted that cupric ions are required for chalcopyrite dissolution in chloride media, the results indicate that the dissolution of gold may occur also without cupric addition, having inherently dissolving iron as oxidant, however a longer leaching time (72 h) is required.

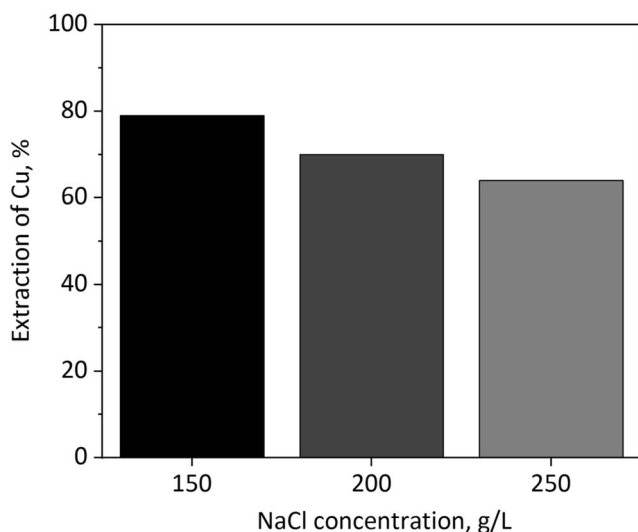


Fig. 7 The effect of NaCl concentration on copper extraction (%). Solution had 30 g/L Cu^{2+} , $T = 95 \text{ }^\circ\text{C}$, $\text{pH} = 1.8$, and 24 h

3.4 The Effect of NaCl Concentration

The effects of sodium chloride concentration (150, 200, and 250 g/L) on the leaching rate of Au, Cu, Co, Ni, and Zn were studied in the at cupric ion concentration of 30 g/L. The effect of NaCl concentration on gold extraction into the pregnant leach solution (PLS) is shown in Fig. 6. The figure shows that no gold could be analyzed at the lowest NaCl concentration (150 g/L); however, gold extraction increased up to 44% by increasing the NaCl concentration up to 250 g/L. Besides, the redox potential of final pregnant leach solution in 250 g/L NaCl concentration was higher (20 mV vs. Ag/AgCl) than 150 g/L NaCl concentration.

Copper extraction was calculated from solid analyses, due to the low amount of Cu in the raw material and high concentrations used as the chemical addition. The effect of NaCl concentrations (150–250 g/L) with 30 g/L cupric $[\text{Cu}^{2+}]$ ion addition for copper dissolution is shown in Fig. 7. It was observed that at all NaCl concentrations, Cu extraction was between 64 and 79% (Experiment nos. 2, 5, and 6). It seems that copper was extracted between 60 and 80% in all experiments, except with longer leaching time ($t = 72 \text{ h}$) in the 250 g/L NaCl solution, the copper extraction into the solution was higher (98%). The original Cu content in the tailings is low (0.09%) and according to results, it seems that already 150 g/L of NaCl addition is enough for dissolving majority of copper into solution. Earlier, Miki and Nicol [28] suggested that the oxidation rate of $[\text{Cu}^+]/[\text{Cu}^{2+}]$ by dissolved oxygen is affected in presence of high chloride concentrations.

The effect of NaCl concentration on Co, Ni, and Zn extractions is shown in Fig. 8. The highest extractions for Co, Ni, and Zn (63, 70, and 72%, respectively) were achieved at the highest NaCl concentration (250 g/L NaCl) whereas with 150 g/L of NaCl, the extractions were 10–15% units lower for all Co, Ni, and Zn. Metal leaching behavior was identical to experiments 1–4, Ni and Zn showing fast dissolution kinetics whereas even at high NaCl concentrations, Co dissolution out of the pyrite matrix was slower.

4 Conclusion

In this work, cupric chloride leaching was applied to dissolve Au, Cu, Co, Ni, and Zn present at low-grade flotation tailings. In order to support the use of this waste fraction as a raw material, grinding pre-treatment process was applied. One hundred twenty minutes of grinding was shown to provide raw material with p_{80} value of 21 μm . The concentrations of Au, Cu, Co, Ni, and Zn elements in raw material were analyzed to be 0.2 ppm, 0.09%, 0.04%, 0.03%, and 0.04%, respectively. The effect of cupric ion (10–50 g/L) and NaCl (150–250 g/L) concentrations were investigated on metal extractions at pH 1.8, redox potential 550–650 mV vs. Ag/AgCl,

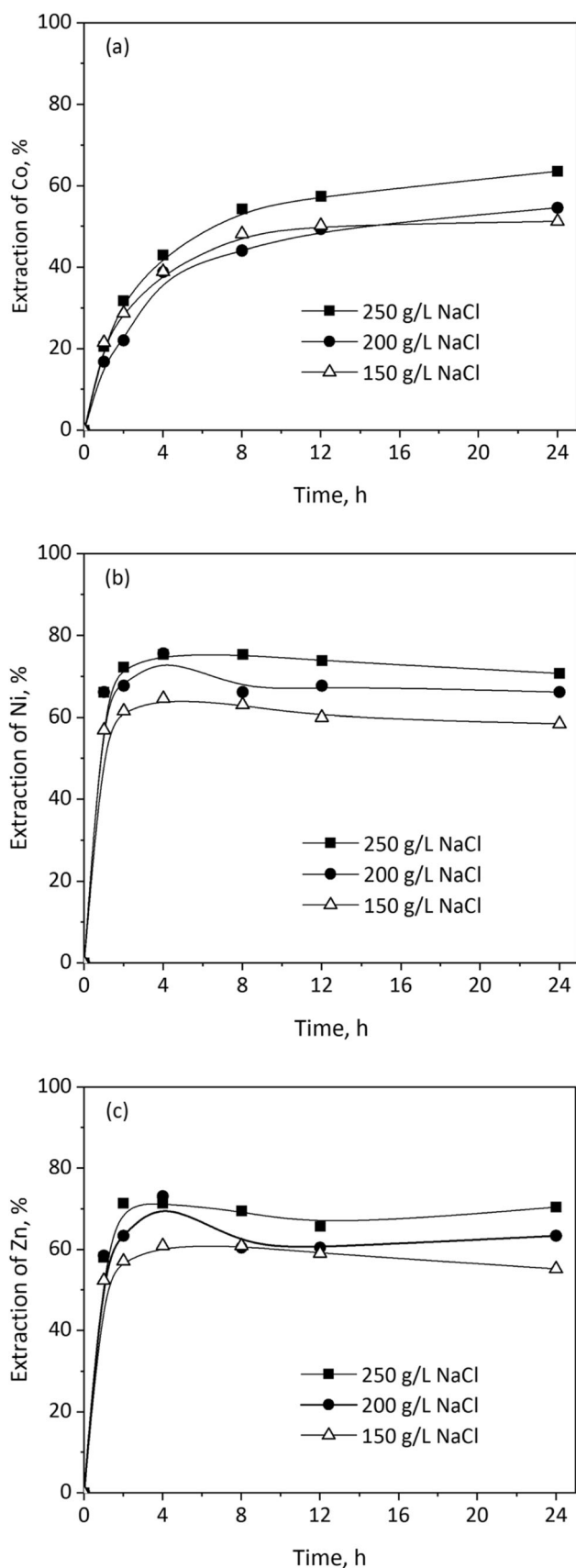


Fig. 8 The effect of NaCl concentration on **a** cobalt, **b** nickel, and **c** zinc extractions

temperature 95 °C, and leaching time of 24 h. The highest gold extraction (58%) was achieved at maximum concentrations of cupric ion (50 g/L) and NaCl (250 g/L) concentration. The copper dissolution into the solution between 60 and 80% was accomplished at all conditions studied. The maximum Co, Ni, and Zn extractions (63, 70, and 72%) were obtained at 30 g/L cupric ion and 250 g/L NaCl concentrations. Cobalt showed slower leaching kinetics, the maximum dissolution being achieved in 12 h whereas the dissolution of Ni and Zn was faster. However, in pure NaCl (250 g/L) solution with leaching time of 72 h in the absence of external oxidant (cupric ion), even higher base metals extraction was evident (Cu 98%, Ni 93%, Co 76%, and Zn 83%). Besides, the solid analysis suggested that 46% Au extraction was achieved in 72 h. The results show that even pure chloride leaching can provide a method for dissolving metals present in trace amounts in the tailings with high simultaneous base metal extraction, the oxidizing ions originating inherently from the tailings raw material. Even though value of copper flotation tailings lies in Au, battery chemical metals such as Co and Ni are of interesting notice for improved metal circular economy and material efficiency.

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Compliance with Ethical Standards

Conflict of Interest The authors declare that there is no conflict of interest.

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