Effects of acid and reductants on iron salt leaching of copper-cobalt oxide ore: A case study from Zebesha Mine, Zambia

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Abstract

Although the conventional acid-reductive leaching method effectively extracts copper (Cu) and cobalt (Co) from Cu–Co oxide ores, efforts to enhance hydrometallurgical performance are ongoing. This study evaluates the influence of sulphuric acid (H_2SO_4) and sodium metabisulphite ($Na_2S_2O_5$) on metal extraction from Zebesha Cu–Co oxide ore using ferrous sulphate heptahydrate ($FeSO_4\cdot 7H_2O$) and ferric sulphate ($Fe_2(SO_4)_3$) as lixiviants. Ore samples (<150 µm) were leached at ambient temperature for two hours, and metal concentrations were determined via X-ray fluorescence analysis. In the $FeSO_4\cdot 7H_2O$ system, Co, Mn, and Ni recoveries increased w=ith $Na_2S_2O_5$ concentration, reaching a maximum of approximately 80% Co at 150 kg/t, while Cu recovery remained below 5%. Conversely, with $Fe_2(SO_4)_3$, Cu recovery peaked at 88% at 50 kg/t $Na_2S_2O_5$, then declined to 79% at 150 kg/t. The addition of H_2SO_4 improved metal recoveries in both systems. Notably, 60 kg/t H_2SO_4 yielded 92% Cu extraction in $Fe_2(SO_4)_3$, while Co, Ni, and Mn remained below 40%. However, in $FeSO_4\cdot 7H_2O$, Co recovery improved from 40% to 84%. These findings suggest $Na_2S_2O_5$ is more effective for cobalt extraction with $FeSO_4\cdot 7H_2O$, whereas H_2SO_4 enhances copper extraction in $Fe_2(SO_4)_3$.

Keywords

copper–cobalt oxide ore, heterogenite, sodium metabisulphite, copper-cobalt extraction, sulphuric acid

Introduction

In recent years, there has been growing interest in the hydrometallurgical extraction of copper-cobalt (Cu-Co) oxide ores from the Central African Copperbelt (CAC) (Crundwell et al., 2011, 2020; Dehaine et al., 2021; Fisher, 2011; Lutandula et al., 2020; Sole et al., 2018; Thabane, 2018; Welham et al., 2015). This surge in attention has been driven by the increasing demand for cobalt and related battery minerals, largely propelled by advancements in electronic technologies, including mobile phones and electric vehicles (Dehaine et al., 2020; Xu et al., 2021).

The primary Cu-Co oxide minerals in the CAC include malachite, the major copper oxide phase, and heterogenite, the dominant cobalt oxide phase, which also contains associated copper, manganese (Mn), nickel (Ni), and iron (Fe) (Crundwell et al., 2011; Sole et al., 2018; Uahengo et al., 2022). Table 1 lists representative Cu–Co oxide minerals from the region. Heterogenite often contains cobalt in both

Table 1

Representative minerals in Cu-Co oxide ore of the Central African Copperbelt

Mineral	Formula
Malachite	$CuCo_3Cu\cdot (OH)_2$
Chrysocolla	$(Cu,Al)_2H_2Si_2O_5(OH)_4\cdot nH_3O$
Heterogenite	$CoOOH$ / $CoO\cdot 2Co_2O_3\cdot 6H_2O$
Siliceous dolomite	$CaMg(CO_3)_2$
Quartz	SiO_2

divalent (Co^{2+}) and trivalent (Co^{3+}) states (Mwema et al., 2002; Stuurman et al., 2014). The average copper content in CAC ores typically ranges between 2%–4% Cu, while cobalt grades range from 1%–3% Co (Annels, Simmonds, 1984; Seo et al., 2013; Slack et al., 2017; Sole, Tinkler, 2016), although local variability exists, for example, the Zebesha Mine in north-western Zambia contains approximately 3.5% Cu and 1.3% Co.

The conventional leaching approach for Cu-Co oxide ores involves the use of sulphuric acid (H_2SO_4) in the presence of a reductant. Trivalent cobalt (Co^{3+}) exhibits low solubility in aqueous media, leading to poor leach efficiency from heterogenite (Crundwell et al., 2020). A suitable reductant is therefore required to reduce Co^{3+} to the more soluble Co^{2+} form. Commonly used reductants include sodium metabisulphite ($Na_2S_2O_5$), sulphur dioxide (SO_2), and ferrous sulphate ($FeSO_4$) (Ferron, Henry, 2008; Kafumbila, 2018; Mwema et al., 2002; Ntakamutshi et al., 2017; Stuurman et al., 2014).

Of these, $Na_2S_2O_5$ is widely favoured in the Copperbelt due to its efficacy in reducing Co^{3+} to Co^{2+}, its environmental acceptability, ease of handling, and logistical suitability for remote sites (Abreu et al., 2012; Bwando, Kanz, 2018; Crundwell et al., 2011; Kafumbila, 2018; Mwema et al., 2002; Stuurman et al., 2014; Sutcliffe et al., 2011; Thabane, 2018; Tshipeng et al., 2017). Its reductive mechanism proceeds through hydrolysis and subsequent SO_2 evolution, as shown in Equations 1 to 4:

$$4CoOOH + Na_2S_2O_5 + 3H_2SO_4 \rightarrow 4CoSO_4 + Na_2SO_4 + 5H_2O \quad [1]$$

$$Na_2S_2O_5 + H_2O \rightarrow 2NaHSO_3 \quad [2]$$

$$2NaHSO_3 + H_2SO_4 \rightarrow Na_2SO_4 + 2SO_2 + 2H_2O \quad [3]$$

$$2CoOOH + H_2SO_4 + SO_2 \rightarrow 2CoSO_4 + 2H_2O \quad [4]$$

Like Co^{3+}, manganese in high oxidation states (e.g., Mn^{4+} in MnO_2) also dissolves under acidic-reductive conditions (Ferron, 2006; Zheng et al., 2023), as shown in Equation 5. Additionally, iron oxides may be reduced to ferrous sulphate under similar conditions (Equation 6):

$$2MnO_2 + Na_2S_2O_5 + H_2SO_4 \rightarrow 2MnSO_4 + Na_2SO_4 + H_2O \quad [5]$$

$$2Fe_2O_3 + Na_2S_2O_5 + 3H_2SO_4 \rightarrow 4FeSO_4 + Na_2SO_4 + 3H_2O \quad [6]$$

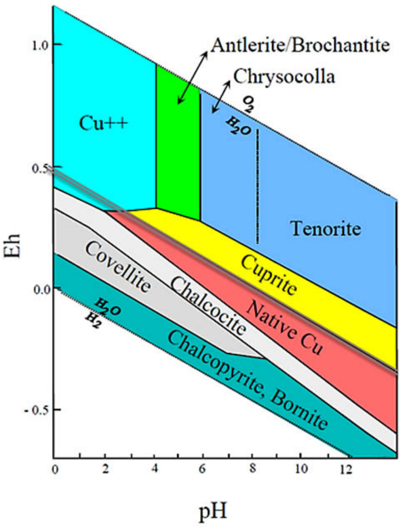

Figure 1—Eh/pH diagrams of Cu leaching at 25°C. Reproduced with permission from Drier (2012)

Copper minerals – including hydroxides, oxides, sulphides, sulphates, carbonates, and silicates – typically dissolve in acidic-oxidative environments, albeit at variable rates depending on mineralogy (Dreier, 2012). Ferric sulphate, especially when combined with H_2SO_4, is often used to facilitate this dissolution.

Despite its effectiveness, $Na_2S_2O_5$ has fallen out of favour due to its high consumption rates, which elevate operating costs (Mwema et al., 2002). Additionally, Na_2SO_4 build-up from $Na_2S_2O_5$ degradation contributes to excess Na^+accumulation in leach circuits. Environmental concerns are also raised by the release of SO_2 gas, especially at higher concentrations (Asadi Dalini et al., 2020; Carvalho et al., 2011). As such, research efforts are increasingly focused on alternative reductants or ways to optimise $Na_2S_2O_5$ usage.

Furthermore, sulphuric acid, though effective, is costly and corrosive. In the Copperbelt, challenges related to sourcing, transporting, and handling H_2SO_4 are exacerbated by inadequate infrastructure, particularly in remote mining areas of Zambia and the Democratic Republic of the Congo (Sole, Tinkler, 2016; Thabane, 2018).

Chemical and mineralogical analysis

The Cu-Co oxide ore sample used throughout the study was sourced from Zebesha Mine, a relatively new copper-cobalt mining operation located in Zambia's North-Western Province. This region forms part of the southern extension of the Central African Copperbelt (CAC), extending from the Democratic Republic of Congo (DRC). To characterise the Zebesha ore, both X-ray fluorescence (XRF) and scanning electron microscopy (SEM) analyses were performed. XRF results revealed relatively high metal content, with average values of 3.69% Cu, 2.28% Co, 0.199% Ni, 0.138% Mn and 3.90% Fe. Figure 2 shows a full elemental distribution thereof.

Mineralogical analysis indicated that copper occurred mainly as malachite, while cobalt was hosted in heterogenite, alongside associated Ni, Mn, and Fe. Quartz, biotite, and talc were identified as the principal gangue minerals, with quartz comprising ~31 wt.%, suggesting a significant silica content (Figure 3). The mineralogy is comparable to other CAC oxide ore deposits (Decrée et al., 2015; Shengo et al., 2016; Stuurman et al., 2014). SEM micrographs in Figure 4 revealed the presence of heterogenite (Het), chrysocolla Fe (Chr Fe), quartz (Qz), biotite (Bi), and chlorite (Chl), among others.

As illustrated in Figure 5, a closer inspection of the mineral phase map reveals that Cu- and Co-bearing phases, such as malachite, heterogenite, and related oxides, are frequently embedded in or rimmed by silicate gangue minerals including quartz, muscovite, biotite, and chlorite. This spatial association suggests partial physical encapsulation of valuable metal phases within the gangue matrix. Such intergrowths may hinder reagent accessibility and contribute to the observed variability in extraction efficiency during leaching. These textural relationships are characteristic of supergene enrichment in deeply weathered oxide terrains typical of the Central African Copperbelt.

Methodology

Materials, reagents and chemicals

The same Cu-Co oxide ore used in the preliminary study was employed in this investigation. All reagents were of analytical grade. Sulphuric acid (98%, Primark Chemicals, South Africa), sodium metabisulphite ($Na_2S_2O_5$), ferrous sulphate heptahydrate

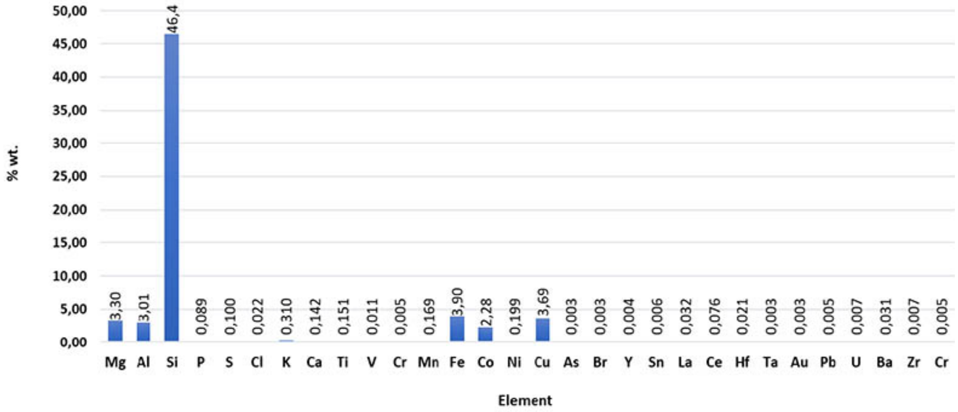

Figure 2—XRF analysis showing elemental composition of Zebesha Cu-Co oxide ore

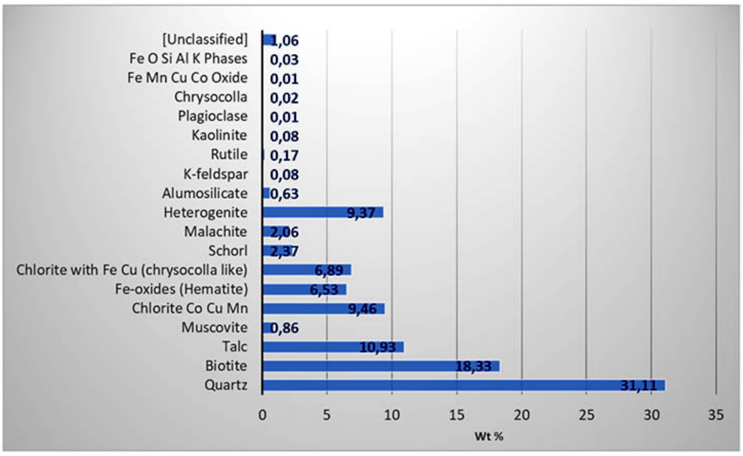

Figure 3—Mineralogical composition of Zebesha Cu-Co oxide ore determined by quantitative X-ray diffraction

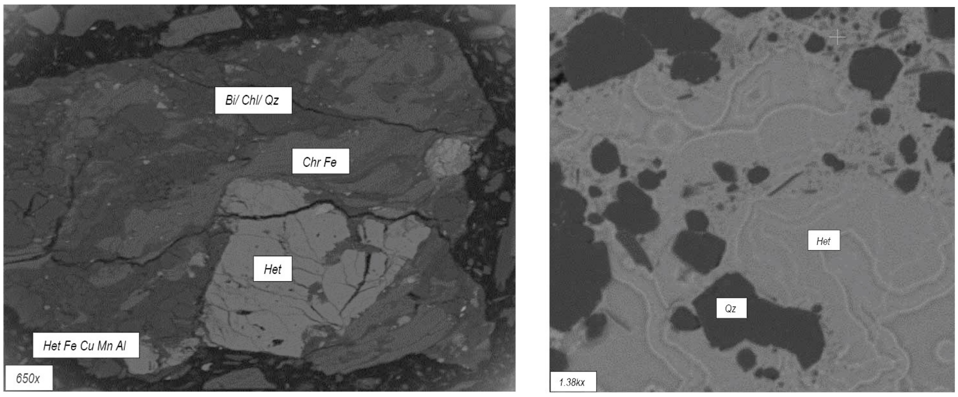

Figure 4—SEM images of Zebesha ore grains at (a) 650× and (b) 1.38× magnification highlighting the distribution of heterogenite (Het), chrysocolla Fe (Chr Fe), quartz (Qz), biotite (Bi), and chlorite (Chl)

($FeSO_4{\cdot}7H_2O$), and ferric sulphate ($Fe_2(SO_4)_3$) were sourced from Associated Chemical Enterprises, South Africa. Iron salt solutions were prepared in distilled water at a concentration of 400kg/t, as determined through preliminary optimisation. Sulphuric acid was added directly in its concentrated form, while $Na_2S_2O_5$ was introduced as a solid.

Experimental design

All experiments were conducted at laboratory scale under ambient conditions (~25°C). The ore was oven-dried, homogenised, and ground to 100% passing 150µm. For feed elemental analysis, a 1kg bulk sample of the Zebesha Cu-Co oxide ore (<1mm) was homogenised and reduced using coning and quartering, followed by rotary splitting to obtain representative subsamples for leaching tests. Initial grade determination was performed by analysing multiple samples using a NEXCG energy dispersive X-Ray fluorescence (EDXRF) instrument to characterise the full feed composition. Subsequently, a handheld X-ray fluorescence (XRF) analyser was used to scan ore samples directly from the sample bag prior to each leaching test.

Preliminary study

Prior to the current investigation, preliminary leaching tests were conducted to establish baseline behaviour and inform reagents

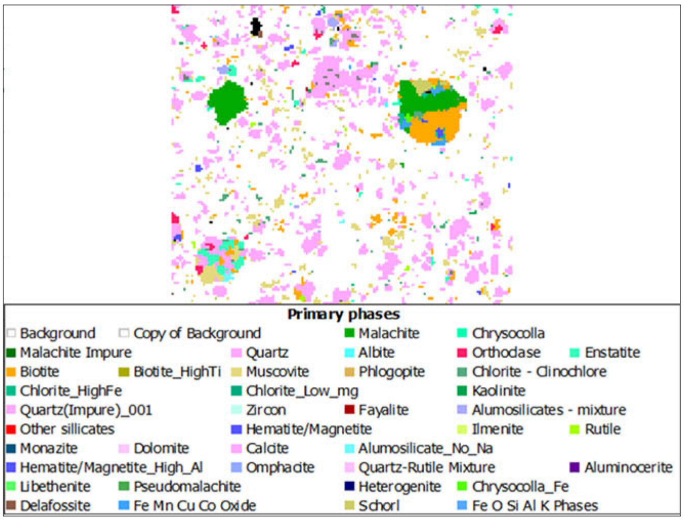

Figure 5—Mineral phase map of Zebesha Cu-Co oxide ore showing Cu- and Co-bearing phases (malachite, heterogenite, Fe-Mn-Cu-Co oxides) spatially associated with gangue minerals including quartz, muscovite, biotite, and chlorite

selection. The objective of the preliminary study was to evaluate the leachability of Zebesha Cu-Co oxide ore using alternative lixiviants to the conventional acid-reductive method. Specifically, iron-based reagents, that is, $FeSO_4{\cdot}7H_2O$ and $Fe_2(SO_4)_3$, were investigated without the addition of sulphuric acid or other reductants/oxidants.

Ferrous and ferric iron salts are known to promote the leaching of certain metals: $FeSO_4$ reduces Co^{3+} to the more soluble Co^{2+}, whereas $Fe_2(SO_4)_3$, acting as an oxidant, facilitates copper solubilisation. For example, CuS and Cu_2S may be leached by $Fe_2(SO_4)_3$ at ambient pressure, as shown in Equations 7 and 8 (Ferron, 2006):

$$CuS + Fe_2(SO_4)_3 \rightarrow CuSO_4 + 2FeSO_4 + S^o \qquad [7]$$

$$Cu_2S + Fe_2(SO_4)_3 \rightarrow 2CuSO_4 + 4FeSO_4 + S^o \qquad [8]$$

Preliminary laboratory leaching tests were conducted using analytical-grade $FeSO_4{\cdot}7H_2O$ and $Fe_2(SO_4)_3$ (Associated Chemical Enterprises, South Africa). These tests were performed at ambient temperature, with variations, initially in preliminary leaching time of 30, 60, 90, and 120 minutes, lixiviant concentrations of 200 kg/t and 400 kg/t, and particle sizes of 80% passing 75 µm, 106 µm, and 150 µm. However, the final preliminary leaching test conditions were fixed at the following parameters:

- 100% passing 150 µm
- 400 kg/t reagent concentration
- 120-minute leaching time
- 33% solids (based on Xu et al., 2023; Zheng et al., 2023).

Leaching tests

Leaching tests were carried out at 33% solids for a fixed duration of 120 minutes under continuous agitation using an overhead stirrer. Each test was conducted in triplicate to ensure reproducibility.

The study aimed to assess the individual effects of $Na_2S_2O_5$ and H_2SO_4 on the leaching performance of $FeSO_4{\cdot}7H_2O$ and $Fe_2(SO_4)_3$. Two separate test series were performed:

- **$Na_2S_2O_5$ series:** Iron salt concentration fixed at 400kg/t; $Na_2S_2O_5$ varied at 0kg/t, 50 kg/t, 100 kg/t, and 150kg/t.
- **H_2SO_4 series:** Iron salt concentration fixed at 400kg/t; H_2SO_4 varied at 0 kg/t, 30 kg/t, 40 kg/t, 50 kg/t, and 60kg/t.

$FeSO_4{\cdot}7H_2O$ and $Fe_2(SO_4)_3$ were applied individually as lixiviants. While leach solutions were not actively pH- or ORP-controlled, measurements of pH, oxidation-reduction potential (ORP), and temperature were recorded at 0, 30, 60, 90, and 120 minutes. Table 2 summarises the leaching conditions for the H_2SO_4-assisted tests. The $Na_2S_2O_5$ series followed a similar matrix. A schematic of the overall procedure is presented in Figure 6.

Analytical procedure and recovery calculations

Following leaching, each pulp was filtered, and the resulting leachates were analysed for Cu, Co, Ni, Mn, and Fe content using X-ray fluorescence (XRF). Each experiment was conducted in triplicate, to confirm the consistency and repeatability of the observed outcomes. The average metal extraction values are reported together with their respective standard errors (SE).

Metal recovery was calculated by mass balance, using Equation 11:

$$\text{Leaching recovery} = \frac{(\%F \ x \ mF) - (\%R \ x \ mR)}{(\%F \ x \ mF)} \ x \ 100 \qquad [9]$$

Where:

%F = % metal content in the feed,
mF = mass of feed (g),
%R = % metal content in residue,
mR = mass of residue (g).

Results and discussion

This study investigated the extraction behaviour of Cu, Co, Ni, and Mn from Zebesha Cu-Co oxide ore using combinations of

Table 2

Leaching conditions for H_2SO_4-assisted tests

Test #	Lixiviant	[H_2SO_4], kg/t
1	$FeSO_4{\cdot}7H_2O$	0
2	$FeSO_4{\cdot}7H_2O$	30
3	$FeSO_4{\cdot}7H_2O$	40
4	$FeSO_4{\cdot}7H_2O$	50
5	$FeSO_4{\cdot}7H_2O$	60
6	$Fe_2(SO_4)_3$	0
7	$Fe_2(SO_4)_3$	30
8	$Fe_2(SO_4)_3$	40
9	$Fe_2(SO_4)_3$	50
10	$Fe_2(SO_4)_3$	60

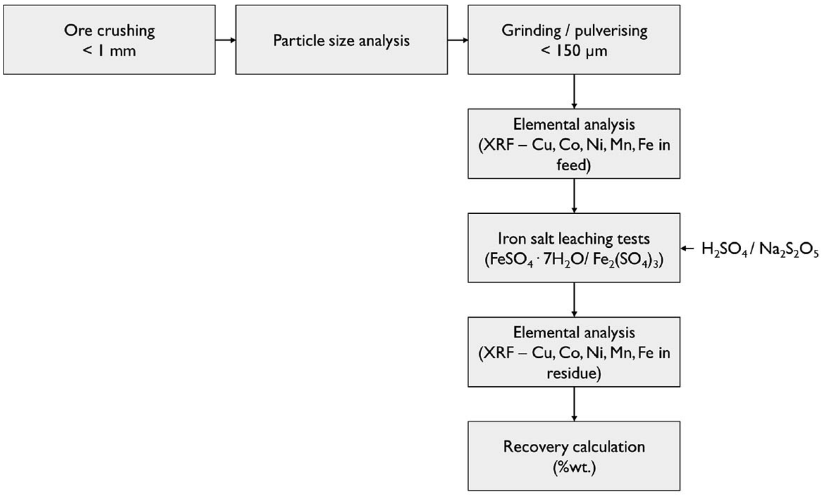

Figure 6—Schematic summary of the experimental procedure

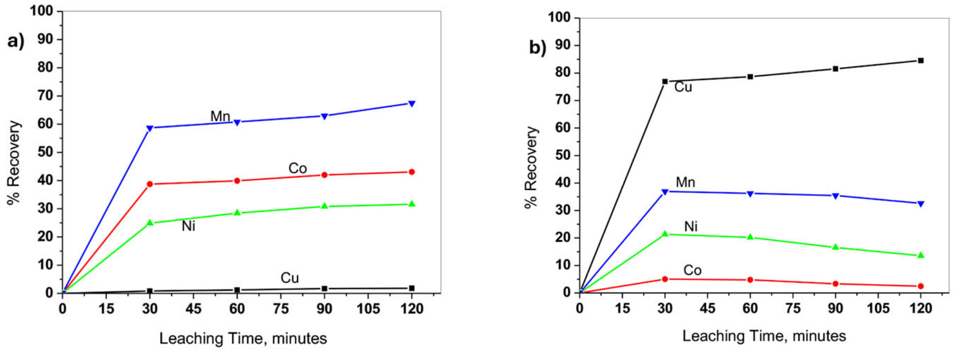

Figure 7—Leaching recoveries of Cu, Co, Ni, and Mn using: (a) $FeSO_4{\cdot}7H_2O$ and (b) $Fe_2(SO_4)_3$ at room temperature, varying leaching times, and 400 kg/t lixiviant concentration

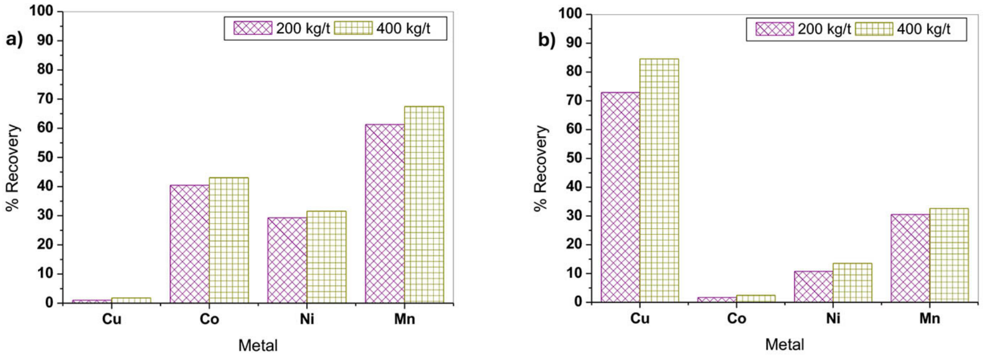

Figure 8—Leaching recoveries of Cu, Co, Ni, and Mn using: (a) $FeSO_4{\cdot}7H_2O$ and (b) $Fe_2(SO_4)_3$ over 120 minutes at 200 kg/t and 400 kg/t lixiviant concentration, respectively

iron-based salts, $FeSO_4{\cdot}7H_2O$ and $Fe_2(SO_4)_3$, with either $Na_2S_2O_5$ or H_2SO_4 at varying reagent concentrations. The data obtained revealed distinct trends in metal extraction efficiency depending on the lixiviant-reagent combination used. Results show that reagent selection critically influences metal selectivity and recovery.

Preliminary test results

The initial preliminary leaching results showed that most metals were leached within 60 minutes, with maximum recoveries observed at 120 minutes using a lixiviant concentration of 400 kg/t, (Figure 7 and Figure 8). Cu and Co extraction showed minimal sensitivity to particle size (Figure 9), and cobalt was hardly leached with $Fe_2(SO_4)_3$.

Final preliminary leaching results shown in Figure 10 demonstrated that $FeSO_4{\cdot}7H_2O$ yielded promising recoveries for Co (43%), Mn (68%), and Ni (35%), but only about 1% for Cu. Conversely, $Fe_2(SO_4)_3$ resulted in high Cu recovery (80%), with substantially lower yields for Co (3%), Ni (13%), and Mn (33%).

Both iron salts hydrolyse to generate H_2SO_4, contributing to their leaching behaviour, as shown in Equations 9 and 10 (Ekmekyapar et al., 2015; Tyrocity, 2012; Welham et al., 2015):

$$FeSO_4 + 6H_2O \rightarrow Fe(H_2O)_4(OH)_2 + H_2SO_4 \qquad [10]$$

$$Fe_2(SO_4)_3 + 6H_2O \rightarrow 2Fe(OH)_3 + 3H_2SO_4 \qquad [11]$$

As $FeSO_4{\cdot}7H_2O$ acts as a reductant and $Fe_2(SO_4)_3$ as an oxidant, their contrasting chemistry yields different extraction profiles. Co^{3+} is reduced to soluble Co^{2+} by $FeSO_4{\cdot}7H_2O$, accounting for its superior cobalt recovery. Conversely, $Fe_2(SO_4)_3$ facilitates copper extraction due to its lower pH (<3.0), whereas $FeSO_4{\cdot}7H_2O$ leaching

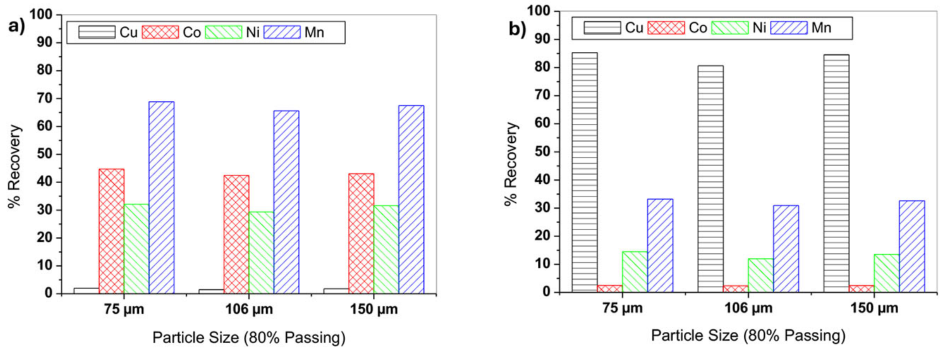

Figure 9—Effect of particle size on metal recovery using: (a) $FeSO_4 \cdot 7H_2O$ and (b) $Fe_2(SO_4)_3$ lixiviants at 400kg/t

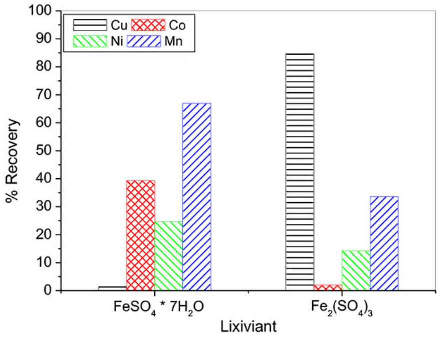

Figure 10—Leaching recoveries for Cu, Co, Ni, and Mn from Zebesha ore using: (a) $FeSO_4 \cdot 7H_2O$ and (b) $Fe_2(SO_4)_3$ at 25 °C, over 120 minutes

pH exceeds 4.5, which is above the optimum range for Cu solubility, which is typically <3.5 (Meshram et al., 2020).

Wang et al. (2021) also demonstrated the effectiveness of $Fe_2(SO_4)_3$ in extracting Cu from malachite, although their material did not include Co, Ni, or Mn.

These preliminary findings highlight the potential of $FeSO_4 \cdot 7H_2O$ for cobalt-selective leaching and $Fe_2(SO_4)_3$ for copper-selective leaching. The study concluded with the recommendation to further investigate ways to enhance overall leaching efficiency. Accordingly, the current study focuses on evaluating the individual effects of H_2SO_4 and $Na_2S_2O_5$ on Cu-Co oxide ore leaching in the presence of iron sulphate salts at ambient conditions.

Effects of sodium metabisulphite addition on metal extraction

Figure 11 illustrates the effect of $Na_2S_2O_5$ concentration on the dissolution of Cu-Co oxide ore using 400 kg/t of either $FeSO_4 \cdot 7H_2O$ or $Fe_2(SO_4)_3$. For both lixiviants, cobalt, nickel, and manganese recoveries increased substantially with rising $Na_2S_2O_5$ concentrations, while copper exhibited little to no improvement. In $Fe_2(SO_4)_3$ systems, copper recovery showed a slight decline with higher $Na_2S_2O_5$ levels, whereas in $FeSO_4 \cdot 7H_2O$, it consistently remained below 5%. The greatest gains in metal recovery from $FeSO_4 \cdot 7H_2O$ leaching were observed for cobalt (~40 %pt.), followed by nickel (~23 %pt.), and manganese (~11 %pt.) over the tested concentration range.

When ferrous sulphate reacts with metal oxides in aqueous solution, it oxidises to ferric sulphate. However, in the presence of $Na_2S_2O_5$, ferric ions are reduced back to ferrous, as illustrated by Equation 12 (Crundwell et al., 2020). This redox cycling effectively increases the concentration of active ferrous ions in solution, enhancing the dissolution of metals that are more readily leached under ferrous-acidic conditions. Additionally, $Na_2S_2O_5$ reduces Co^{3+} and Mn^{4+} to their soluble divalent forms, promoting their extraction in $Fe_2(SO_4)_3$ systems. These redox-mediated transformations account for the observed increase in Co, Ni, and Mn recoveries across both iron-based lixiviants. Thermodynamic support for these reductions is shown in the Gibbs free energy plots in Figure 12.

$$2Fe_2(SO_4)_3 + Na_2S_2O_5 \rightarrow 4FeSO_4 + Na_2SO_4 + 3SO_3 \qquad [12]$$

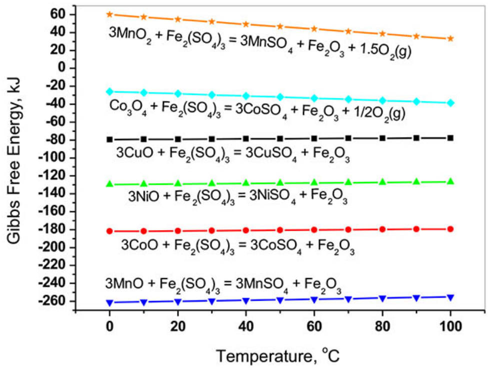

Figure 12—Gibbs free energy change (ΔG) versus temperature for redox reactions of MnO_2, Co_3O_4, CuO, NiO, CoO, and MnO with $Fe_2(SO_4)_3$, generated using HSC Chemistry 6.0 software data

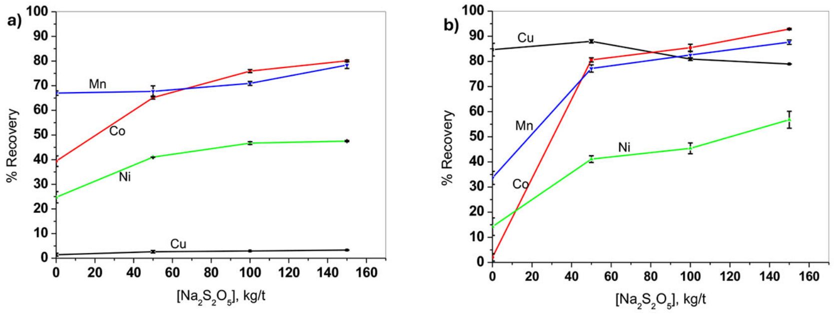

Figure 11—Effects of $Na_2S_2O_5$ concentration on Zebesha Cu-Co oxide ore leaching by using: a) $FeSO_4 \cdot 7H_2O$ and b) $Fe_2(SO_4)_3$

Increasing the $Na_2S_2O_5$ concentration in the $FeSO_4{\cdot}7H_2O$ leaching system resulted in a marked improvement in cobalt recovery, rising from approximately 40% in the absence of $Na_2S_2O_5$ to nearly 80% at 150 kg/t. Concurrently, manganese and nickel recoveries also increased, with Mn leaching improving from ~67% to 78% and Ni from ~25% to 48%, as shown in Figure 11a. The most significant gains were observed between 0 kg/t and 50 kg/t $Na_2S_2O_5$, where cobalt and nickel recoveries rose by 25% and 15%pt., respectively. A further increase of 15%pt. Co and 5%pt. Ni was achieved by increasing $Na_2S_2O_5$ to 150kg/t. In contrast, copper recovery remained consistently low, increasing only slightly from ~1% to below 5% across the tested $Na_2S_2O_5$ range.

Conversely, the addition of 50kg/t $Na_2S_2O_5$ to $Fe_2(SO_4)_3$ leaching led to a modest increase in copper recovery from ~85% to 88%, after which it declined to ~79% at 150kg/t, as shown in Figure 11b. In contrast, $Na_2S_2O_5$ addition substantially improved cobalt, nickel, and manganese recoveries. Cobalt extraction increased markedly from ~2% to ~80% with 50kg/t $Na_2S_2O_5$, reaching a maximum of ~93% at 150 kg/t. Nickel and manganese recoveries followed similar trends, rising from ~14% to 57% Ni and 44% to 88% Mn, respectively, across the tested range. Notably, the greatest improvements in metal dissolution for Co, Ni, and Mn occurred between 0 kg/t and 50kg/t $Na_2S_2O_5$, highlighting the strong initial reactivity of the reductant with Fe^{3+} species and metal oxides in this concentration interval.

As observed in the preliminary iron salt leaching tests, copper exhibited minimal solubility in $FeSO_4{\cdot}7H_2O$ but was readily extracted in $Fe_2(SO_4)_3$ solutions. This discrepancy can be attributed in part to the reductive action of $Na_2S_2O_5$, which converts ferric ions (Fe^{3+}) to ferrous (Fe^{2+}), thereby reducing the availability of ferric sulphate – a key oxidant for copper dissolution. Additionally, effective copper leaching typically occurs under acidic conditions (pH < 2). At higher pH values (e.g., >4), especially in the presence of excess $Na_2S_2O_5$ or SO_2, copper may precipitate as Le Chevreul's salt ($CuSO_3{\cdot}Cu_2SO_3{\cdot}2H_2O$) according to Equation 13 (Crundwell et al., 2020; Ferron, 2006; Ntakamutshi et al., 2017). This precipitation mechanism likely contributed to the low copper recoveries observed in $FeSO_4{\cdot}7H_2O$ systems, even at elevated $Na_2S_2O_5$ concentrations.

$$3CuSO_4 + SO_2 + 6H_2O \rightarrow CuSO_3 \cdot Cu_2SO_3 \cdot 2H_2O + H_2SO_4 \qquad [13]$$

Effects of sulphuric acid addition on metal extraction

Varying concentrations of sulphuric acid were introduced into the leaching system, with both $FeSO_4{\cdot}7H_2O$ and $Fe_2(SO_4)_3$ solutions maintained at 400kg/t to assess the effect of H_2SO_4 on metal extraction efficiencies. The results, presented in Figure 13, show that metal recoveries improved with increasing acid concentration across both systems. However, the enhancement was significantly more pronounced in the $FeSO_4{\cdot}7H_2O$ leaching system, while $Fe_2(SO_4)_3$ exhibited a more subdued response.

The addition of H_2SO_4 to $FeSO_4{\cdot}7H_2O$ leaching significantly improved copper extraction, increasing from ~1% (in the absence of acid) to ~78% at a dosage of 60 kg/t. Concurrently, nickel, manganese, and cobalt recoveries also increased, with Ni rising from ~25% to 56%, Mn from ~67% to 83%, and Co from ~39% to 85%, as shown in Figure 13a. These results indicate that H_2SO_4 addition noticeably enhanced metal solubility in ferrous media, particularly for copper, which otherwise shows limited dissolution in $FeSO_4{\cdot}7H_2O$ alone. This enhancement effectively diminished the initial selectivity gap between Cu and Co under ferrous leaching conditions.

The observed increase in cobalt recovery may also be attributed to acid-driven regeneration of the lixiviant. Under acidic conditions, heterogenite reacts with ferrous sulphate via mechanisms such as shown in Equations 14 and 15:

$$CoOOH + FeSO_4 + H_2SO_4 \rightarrow CoSO_4 + Fe(OH)SO_4 + H_2O \qquad [14]$$

$$2CoOOH + 2FeSO_4 + 3H_2SO_4 \rightarrow 2CoSO_4 + Fe_2(SO_4)_3 + 4H_2O \qquad [15]$$

These reactions suggest that both acid strength and lixiviant regeneration contribute to improved leaching performance. Despite the substantial gains in copper recovery, the highest recoveries were achieved for cobalt (85%) and manganese (83%) under these conditions.

In contrast, the effect of H_2SO_4 addition on $Fe_2(SO_4)_3$ leaching was less pronounced than in the $FeSO_4{\cdot}7H_2O$ system. At 60 kg/t H_2SO_4, copper recovery peaked at ~92%, while cobalt, nickel, and manganese reached ~10%, 18%, and 36%, respectively, as shown in Figure 10b. Manganese recovery increased by only ~3 percentage points across the acid range, while Cu, Co, and Ni each rose by ~8 %pt.

The enhanced copper dissolution can be attributed in part to acid-driven pH reduction, from ~2.7 (without H_2SO_4) to below 2.0 at 60 kg/t, creating more favourable conditions for malachite dissolution. This is reflected in the acid-mediated reaction between malachite and ferric sulphate (Equation 16):

$$2CoOOH + 2FeSO_4 + 3H_2SO_4 \rightarrow 2CoSO_4 + Fe_2(SO_4)_3 + 4H_2O \qquad [16]$$

In contrast, cobalt, nickel, and manganese showed only modest improvements, likely due to their limited presence in readily soluble divalent forms under $Fe_2(SO_4)_3$ leaching conditions. This highlights the relative selectivity of $Fe_2(SO_4)_3$–H_2SO_4 systems for copper over other base metals.

The performance comparison of H_2SO_4 and $Na_2S_2O_5$ reagents addition on leaching Zebesha Cu-Co oxide ore with iron sulphate salt showed that the acid performance was higher in most cases compared to sodium metabisulphite performance. As presented

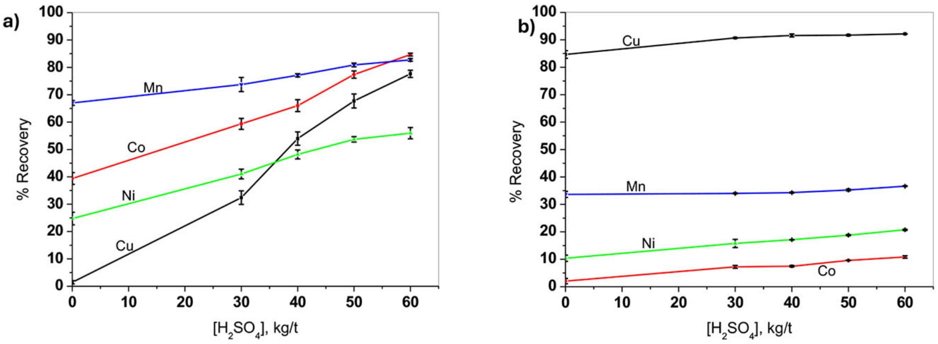

Figure 13—Effect of H_2SO_4 concentration on Zebesha Cu-Co oxide ore leaching using: a) $FeSO_4{\cdot}7H_2O$ and b) $Fe_2(SO_4)_3$ as lixiviants

in Table 3, for instance, at a reagent concentration of 50 kg/t. Ferric-based leaching with H_2SO_4 achieved the highest copper recovery (91.70%), while ferrous-based leaching with $Na_2S_2O_5$ favoured cobalt extraction (65.17%) with minimal copper dissolution (2.64%). H_2SO_4 addition generally improved extraction performance across all metals, with $FeSO_4{\cdot}7H_2O$ + H_2SO_4 yielding high recoveries for both Co and Mn. While the overall objective was to enhance recoveries, particularly for Cu and Co, the ability to selectively leach these elements remains equally important for potential process optimisation.

These findings underscore the synergistic effects of lixiviant-reductant combinations on selective metal extraction. While H_2SO_4 consistently enhanced metal recovery, $Na_2S_2O_5$ exhibited greater selectivity for cobalt relative to copper. Understanding these interactions is essential for optimising leaching conditions to achieve targeted metallurgical outcomes.

The low standard error values observed across most data points, such as 91.70% ± 0.20% Cu using $Fe_2(SO_4)_3$ + H_2SO_4 and 77.37% ± 1.33% Co with $FeSO_4{\cdot}7H_2O$ + H_2SO_4 (Table 3), indicate high precision and reproducibility in the experimental procedures. Slightly higher variability in certain cases such as manganese extraction (67.69% ± 2.25% Mn), likely reflects intrinsic differences in leaching behaviour but remains within acceptable limits for laboratory-scale studies. Overall, the data affirm the reliability and repeatability of the extraction process under the tested conditions.

Conclusions

This study examined the selective leaching of copper and cobalt from Zebesha Cu-Co oxide ore using iron-based salts in combination with sodium metabisulfite ($Na_2S_2O_5$) or sulphuric acid (H_2SO_4). The leaching behaviour differed depending on the oxidation state of the iron salt, that is, ferric iron promoted copper extraction, while ferrous iron favoured cobalt recovery, suggesting that a sequential leaching approach may be effective. The addition of $Na_2S_2O_5$ significantly enhanced cobalt recovery, achieving up to 80%, whereas copper extraction remained limited. Introducing H_2SO_4 markedly improved the dissolution of Cu, Co, Ni, and Mn, with copper and cobalt recoveries reaching 78% and 84%, respectively, at 60 kg/t H_2SO_4. Ferrous sulphate combined with H_2SO_4 outperformed ferric solutions, particularly for cobalt, nickel, and manganese. The findings demonstrate the potential for environmentally and economically sustainable metal recovery through tailored reagent selection and process design. However, additional investigation is required to understand the leaching kinetics and validate the feasibility of sequential processing strategies. These insights offer a foundation for developing more efficient and environmentally responsible extraction techniques for Cu-Co ores.

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Credit author statement

Foibe D.L. Uahengo: Experimental work and original draft (PhD student)
Yotamu R.S. Hara: Reviewing and editing (main supervisor)
Olga Bazhko: Reviewing and editing (co-supervisor).

Nomenclature

A list of abbreviations used in the manuscript, including mineral phases, units, and analytical terms

Abbreviation	Definition
%pt.	Percent point
°C	Degrees Celsius
Bi	Biotite (gangue mineral)
CAC	Central African Copperbelt
Chl	Chlorite (gangue mineral)
Chr Fe	Chrysocolla with iron substitution
DRC	Democratic Republic of the Congo
EDXRF	Energy-dispersive X-ray fluorescence
Het	Heterogenite (primary cobalt mineral)
kg/t	Kilogrammes per tonne
mF / mR	Mass of feed / residue (g)
ORP	Oxidation-reduction potential
pH	Measure of solution acidity
Qz	Quartz (gangue mineral)
%F / %R	Metal content in feed / residue (%)
SE	Standard error
SEM	Scanning electron microscopy
wt.%	Weight percent
X	Reacted fraction of metal (%)
XRF	X-ray fluorescence
ΔG	Gibbs free energy change

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Table 3

Average extraction performance (%) of metals using $Na_2S_2O_5$ and H_2SO_4 at 50 kg/t concentration

Lixiviant	Reagent	%Cu ± SE	%Co ± SE	%Ni ± SE	%Mn ± SE
$FeSO_4{\cdot}7H_2O$	$Na_2S_2O_5$	2.64 ± 0.49	65.17 ± 0.61	41.03 ± 0.01	67.69 ± 2.25
	H_2SO_4	67.73 ± 2.56	77.37 ± 1.33	53.69 ± 0.98	80.69 ± 0.67
$Fe_2(SO_4)_3$	$Na_2S_2O_5$	88.00 ± 0.63	80.60 ± 0.82	41.11 ± 1.34	77.18 ± 1.47
	H_2SO_4	91.70 ± 0,20	9.58 ± 0.04	18.79 ± 0.18	35.25 ± 0.31

Note: Values represent average metal extractions from replicate tests. Standard errors (SE) are shown to reflect variability across replicates.

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Appendix: Data used for the graphs in the article, presented as average

Leaching results for the iron-containing salts leaching of Zebesha Cu-Co oxide ore with and without the addition of $Na_2S_2O_5$

Lixiviant	kg/t $Na_2S_2O_5$	Final T. oC	Final pH	Final ORP, mV	Leaching recoveries %Cu ± SE	%Co ± SE	%Ni ± SE	%Mn ± SE
$FeSO_4 \cdot 7H_2O$	0	28	4,84	162	1,44 ± 0,61	39,39 ± 2,16	24,73 ± 2,29	67,00 ± 0,88
$FeSO_4 \cdot 7H_2O$	50	27	4,66	159	2.64 ± 0.49	65.17 ± 0.61	41.03 ± 0.01	67.69 ± 2.25
$FeSO_4 \cdot 7H_2O$	100	27	4,86	154	2,94 ± 0,04	75,93 ± 0,62	46,71 ± 0,54	70,94 ± 0,81
$FeSO_4 \cdot 7H_2O$	150	26	4,57	154	3,30 ± 0,09	80,10 ± 0,35	47,53 ± 0,25	78,36 ± 1,38
$Fe_2(SO_4)_3$	0	26	2,64	300	84,67 ± 2,53	2,03 ± 1,48	14,23 ± 3,49	33,64 ± 2,60
$Fe_2(SO_4)_3$	50	25	2,60		88.00 ± 0.63	80.60 ± 0.82	41.11 ± 1.34	77.18 ± 1.47
$Fe_2(SO_4)_3$	100	24	3,40		80,93 ± 0,54	85,45 ± 1,40	45,40 ± 2,15	82,57 ± 1,35
$Fe_2(SO_4)_3$	150	25	3,30		78,96 ± 0,02	92,86 ± 0,26	56,77 ± 3,37	87,64 ± 0,87

Leaching results for the iron-containing salts leaching of Zebesha Cu-Co oxide ore with and without the addition of H_2SO_4

Lixiviant	kg/t H_2SO_4	Final T. oC	Final pH	Final ORP, mV	Leaching recoveries %Cu ± SE	%Co ± SE	%Ni ± SE	%Mn ± SE
$FeSO_4 \cdot 7H_2O$	0	25	5,04	162	1,44 ± 0,61	39,39 ± 2,16	24,73 ± 2,29	67,00 ± 0,88
$FeSO_4 \cdot 7H_2O$	30	27	4,69	180	32,40 ± 2,49	59,35 ± 2,01	41,03 ± 1,79	73,73 ± 2,57
$FeSO_4 \cdot 7H_2O$	40	25	3,80	196	53,97 ± 2,43	66,02 ± 2,17	48,18 ± 1,65	77,11 ± 0,59
$FeSO_4 \cdot 7H_2O$	50	23	4,15	210	67.73 ± 2,56	77.37 ± 1,33	53.69 ± 0,98	80.69 ± 0,67
$FeSO_4 \cdot 7H_2O$	60	25	3,72	234	77,63 ±1,33	84,67 ± 0,52	55,97 ± 2,05	82,74 ± 0,49
$Fe_2(SO_4)_3$	0	26	2,64	300	84,67 ± 1,39	2,03 ± 0,97	10,35 ± 1,12	33,64 ± 1,10
$Fe_2(SO_4)_3$	30	26	2,28	324	90,69 ± 0,15	7,23 ± 0,51	15,76 ± 1,50	34,00 ± 0,14
$Fe_2(SO_4)_3$	40	27	2,11	333	91,59 ± 0,54	7,45 ± 0,26	17,12 ± 0,07	34,29 ± 0,16
$Fe_2(SO_4)_3$	50	27	1,92	346	91.70 ± 0,20	9.58 ± 0.04	18.79 ± 0,18	35.25 ± 0,31
$Fe_2(SO_4)_3$	60	26	1,98	345	92,16 ± 0,02	10,84 ± 0,45	20,73 ± 0,22	36,64 ± 0,11