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Leaching of copper slags in sulphuric acid and alkaline glycine media

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Copper slag is industrial waste, having fayalite and magnetite as main phases, copper is present in the form of chalcopyrite and chalcosine. However, the complex structure of the slag makes the dissolution process difficult, which is why methods have been used to recover metals with leaching in sulfuric acid media as a traditional technique. however, the use of new leaching agents has been implemented, for instance, glycine. The operating parameters such as concentration, temperature, particle size are compared in these leaching media, highlighting glycine with high selectivity and efficiency unlike sulfuric acid in alkaline conditions to leach copper. In this study, the efficiency of glycine as a leaching agent for copper recovery will be revised.

KEYWORDS

leaching, acid sulphuric, slag, copper, glycine

1 Introduction

Copper slag is an industrial waste product from the non-ferrous metals group, generated during the production of copper anodes (Nazer et al., 2016). In 2020, China reported an annual production of 20 million tonnes of copper slag (Shi et al., 2020). In recent years, pyrometallurgical methods have been employed to treat copper slag, reducing its viscosity and facilitating slag sedimentation (Wang et al., 2024). These pyrometallurgical processes often involve the addition of fluxes such as calcium oxide and silica, resulting in copper slags with complex compositions copper slag mainly contains 29%–45% iron (Fe), 25%–40% silicon oxide (SiO_2), 5%–11% calcium oxide (CaO), 3%–7% aluminum oxide (Al_2O_3), 1.1% copper (Cu) and other non-ferrous metals (Zhang et al., 2021). The recovery of copper from these slags is challenging due to their intricate structure, which is induced by the interlocking of various minerals (Shi et al., 2020). Additionally, the slag typically contains high levels of SiO2 (10%–71%) and FeO (0.6%–62%), along with elements such as copper, lead, zinc, and nickel (Piatak et al., 2015).

Copper slag is mainly composed of fayalite and magnetite phases, with copper present in both, primarily as chalcopyrite ($CuFeS_2$) and chalcocite (Cu_2S) (Nadirov et al., 2020). However, copper slags can be valorised through the recovery of the metals they contain,

TABLE 1 Comparison of previous studies for the leaching of chalcopyrite to different alkaline glycine media.

Ref.	Glycine Concentration [M]	Time (h)	Oxidising Agent	pH	Particle Size (um)	Cu recovery (%)	Temperature (°C)
Oraby and Eksteen (2014)	0.4	48	Hydrogen peroxide	9.4	150–106	90	Ambient
Khezri et al. (2020)	0.7	25	Dissolved oxygen	10.5	11	91.1	60
Khezri et al. (2021)	0.4	25	Dissolved oxygen	10	<20	95	60
Tanda et al. (2019)	0.5	96	Dissolved oxygen	11.5	<10	72.6	60
O'Connor et al. (2018)	0.3	0.8	Dissolved oxygen	10.5	Not specified	Not specified	25
Shin et al. (2019)	1	96	Hydrogen peroxide	11	40	21	Ambient

offering a potential alternative for metallurgical processes (Nazer et al., 2016). Some studies have proposed methods for the recovery of copper (Nadirov et al., 2019), zinc (Najera Ibarra et al., 2024), and cobalt (Song et al., 2019), often employing hydrometallurgical techniques, which are widely used to extract these metallic values. Other researchers have explored the use of various solvents for metal recovery, including ferric chloride (Anand et al., 1980), hydrochloric acid (Nadirov et al., 2020), hydrogen peroxide (Banza et al., 2002), ammoniacal acid (Nadirov et al., 2019), and sulphuric acid (Aghajani, 2016).

Research has indicated that leaching copper slag with sulfuric acid is the most conventional method for copper extraction (Ahmed et al., 2016). But since copper smelting slag contains a large amount of alkaline gangue, dissolving of the alkaline gangue in the leaching process not only consumes a greater amount of acid but also results in a high content of metal ions in the leaching solution. Previous work (Huang et al., 2023; Zhao et al., 2016) has tested chalcopyrite leaching in sulfuric acid at room temperature, for 30 days, obtaining copper extractions of no more than 22%, a low performance. However, the use of some agents such as pyrite under the same conditions achieves a recovery copper over 80%. In contrast authors (Joe et al., 2009) commented that by increasing the leaching time to 120 days and having optimal control of the concentrations of sulfuric acid 23–25 g/L, copper extractions of over 80% were obtained.

Currently, efforts have been made to explore non-traditional leaching agents and to seek new alternatives for the use of green solvents (Petersen and Dixon, 2006). Among these new solvents, glycine, the simplest amino acid, offers several advantages: it is non-toxic, non-volatile, and recyclable (Ballesteros et al., 2014). Glycine consists of a single carbon molecule and an amino group from the carboxyl group, with the formula NH_2-CH_2-COOH. In aqueous solutions, glycine can exist in three different forms: cationic glycine ion, neutral zwitterion, and anionic glycinate (Barton and Hiskey, 2022). Glycine has been employed in leaching processes for the recovery of various metals from solid materials, including sulphide minerals and electronic waste (Jamett et al., 2022). Table 1 provides information on different studies conducted to achieve copper recovery under similar parameters from chalcopyrite.

The use of sulphuric acid for leaching sulphide minerals poses significant safety concerns due to the corrosive and volatile nature of the reagent, particularly at elevated temperatures (Kumar et al., 2017). In response to these challenges, more environmentally friendly and innovative reagents or solvents have been sought. Tanda et al. (2017) confirm that the use of glycine as a reagent for leaching copper slag from sulphidised copper has become a growing focus of research and innovation in recent years.

This review aims to contribute to the knowledge in the area of leaching of copper slag, as there are currently few studies that revise copper slag leaching technology using sulphuric acid or glycine as a leaching agent, supported by recent scientific publications. The objective of this study is to compare the operational characteristics of sulphuric acid and glycine as lixiviants, analysing the parameters that influence copper extraction in both cases.

2 Fundaments

Copper slags typically consist of two phases phases fayalitic and sulfide, containing a small percentage of the mineral species known as chalcopyrite (Nadirov et al., 2020). However, this presents a challenge for copper extraction, as the refractory nature of chalcopyrite leads to low dissolution rates during leaching (Rawlings et al., 2003),. Additionally, the formation of a passivating layer on the mineral's surface (Klauber, 2008) further hinders leaching. This passivating layer is primarily composed of sulphur species such as polysulfides, elemental sulphur, and jarosite. The following Equation 1 is widely accepted as representing the traditional mechanism without the addition of oxidant for leaching mechanism of chalcopyrite.

$$CuFeS_2 + 4\,H_2SO_4 \rightarrow CuSO_4 + FeSO_4 + 2S + 2SO_2 + 4H_2O \quad (1)$$

Guzmán et al. (2013), with the aim of improving the leaching kinetics of chalcopyrite, studied the effect of mechanical activation on this process. Their results showed a copper recovery of 36% at a temperature of 90°C, indicating that higher temperatures are required for greater recovery. However, the dissolution of chalcopyrite can be accelerated when the redox potential is controlled within an optimal range of 0.36–0.5 V (Petersen and Dixon, 2006; Zhao et al., 2015). Under these conditions, chalcopyrite can be reduced to chalcocite (Cu_2S) the authors note that if direct oxidation of chalcopyrite occurs, the redox potential values will be relatively high (Zhao et al., 2016). However, copper smelting slag contains a significant amount of alkaline gangue. During the leaching process, the dissolution of alkaline gangue not only consumes a large amount of acid but also results in a high concentration of metal ions in the solution. Additionally, its dissolution is slow when sulphuric acid is used as a leaching agent (Mussapyrova et al., 2021).

Similar to sulphuric acid, glycine has been shown to selectively leach copper from slag, making it a promising selective copper leaching agent in recent years. Aksu and Doyle (2001) noted that glycine can exist in aqueous solutions in three different forms (Equations 2–4), which enhance the dissolution of copper. These forms are characterised by their ability to improve the solubility of copper ions in aqueous solutions, forming strong complexes with both copper (II) (Equation 3) and copper (I) (Equation 4). Among these, the cupric copper complex exhibits the greatest stability. The equilibrium constants for reactions 1, 2, and 3 are determined by the following values: 8.6, 15.0, and 10.0, respectively.

$$Cu^{2+} + (NH_2CH_2COO)^- \leftrightarrow Cu(NH_2CH_2COO)^- \quad (2)$$

$$Cu^{2+} + 2(NH_2CH_2COO)^- \leftrightarrow Cu(NH_2CH_2COO)_2 \quad (3)$$

$$Cu^{+} + 2(NH_2CH_2COO)^- \leftrightarrow Cu(NH_2CH_2COO)_2^- \quad (4)$$

In alkaline solution of glycine, the copper and chalcopyrite slag present in the slag is leached according to the following Equations 5, 6 (Huang et al., 2023; Tanda et al., 2019)

$$2Cu^{+} + O_2 + 4NH_2CH_2COO = 2Cu(NH_2CH_2COO)_2 + 2H_2O \quad (5)$$

$$CuFeS_2 + 2Gly + 19OH^- \leftrightarrow Cu(Gly)_2 + Fe + (OH)_3 + 2SO_4^{2-} + 8H_2O \quad (6)$$

The complexation mechanism of copper in glycine-containing solutions involves the formation of copper complexes through the carboxyl groups via an ion exchange process. Additionally, during the formation of the glycine-metal complex, there is an interaction between metal and hydrogen ions as the pH increases. This leads to the displacement of hydrogen protons by copper, resulting in the formation of a stable copper-glycine complex (Aksu and Doyle, 2001; Eksteen et al., 2017). This process is illustrated in Equations 7, 8.

$$(N^{+}H_3CH_2COOH) \leftrightarrow NH_3CH_2COO^- + H^+ \quad (7)$$

$$Cu^{2+} + 2(NH_3CH_2COO^-) \leftrightarrow Cu(NH_2CH_2COO)_2 + 2H^+ \quad (8)$$

Studies conducted by Mokhlis et al. (2021) report that adjusting the pH within the range of 8–10, while considering the equilibrium constants of copper-glycine ion complexes, induces the formation of an alkaline hydrogen solution. In this process, glycine loses a hydrogen ion to form Gly^-, after which a stable complex, $Cu(Gly)_2$, forms with Cu^{2+}, as shown in Equation 9, which enhances copper dissolution. It has also been demonstrated by the studies shown (Table 1) that glycine exhibits excellent selectivity for copper leaching, with copper having a greater coordination capacity with glycine (Mokhlis et al., 2021). This implies that copper will achieve a higher recovery compared to other species such as Fe (Equations 10, 11) or Zn (Equation 12). However, over time, copper glycinates may form, which can complicate copper recovery, highlighting the importance of time control in the process.

$$Cu^{2+} + 2Gly^- = Cu(Gly)_2 \quad (9)$$

$$Fe^{2+} + 2Gly^- = Fe(Gly)_2 \quad (10)$$

$$Fe^{3+} + Gly^- = Fe(Gly)^{2+} \quad (11)$$

$$Zn^{2+} + 2Gly^- = Zn(Gly)_2 \quad (12)$$

Huang et al. (2023) achieved high copper recoveries from slags through leaching with selective complexes using glycine as an agent under alkaline conditions. Gly^- forms a strong complex with the copper ion, resulting in the selective dissolution of copper compared to other metallic elements.

Efforts have been made to understand the selective leaching of copper using glycine and sulphuric acid in copper slag, focusing on variables such as time, particle size, initial glycine concentration, sulphuric acid concentration, liquid-solid ratio, stirring speed, and temperature. However, this study will specifically analyse some of these operational parameters to maximise copper recovery from the slags.

3 Operational parameters

3.1 H_2SO_4 concentration effect

The concentration of sulphuric acid is a decisive factor in the acid leaching process for copper slags. In a study conducted by Gargul et al. (2022), leaching tests were performed on copper slags with sulphuric acid concentrations ranging from 50 to 200 g/L at temperatures of 50°C and 75°C over a 60-min period. The study reported copper recoveries of 65% and 80%, with the highest leaching efficiencies achieved at an acid concentration of 200 g/L. While the increase in copper dissolution was not significant beyond this concentration, the rate of iron leaching increased. This phenomenon is attributed to the higher concentration of sulphuric acid, which enhances the dissolution of the mineral phases in the slag (Shi et al., 2020). However, at high sulphuric acid concentrations, the solubility of hematite in the sulphuric acid solution decreases rapidly, leading to faster iron leaching kinetics (Zhao et al., 2019). In addition, Table 2 shows that the pH of sulfuric acid is 0.7, making this medium acidic, unlike a glycine medium.

3.2 Glycerine concentration effect

The concentration of glycine is a crucial variable, as it significantly enhances both the degree of copper dissolution and the leaching kinetics. In research conducted by Eksteen et al. (2017), chalcopyrite was leached as the slag contains a minimal chalcopyrite phase, resulting in copper extractions of 40.1% at a glycine concentration of 0.4 M after 24 h at 60°C, with the addition of 25 ppm of oxygen. They also tested glycine concentrations of 0.2–0.3 M, achieving copper recoveries of less than 30%. A similar study by Aghajani (2016) evaluated copper extraction as a function of time at a temperature of 60°C with different glycine concentrations. At 0.4 M, they achieved approximate recoveries of 45% within the first 6 h, but as time progressed, a decrease in extraction was observed. However, when the glycine concentration was increased to 2 M, copper recoveries of 70% were achieved after

TABLE 2 Comparison of studies on the dissolution of copper slags using glycine and sulphuric acid media.

Experimental Conditions and Results	Huang et al. (2023)	Gargul et al. (2022)
Temperature (°C)	40	50
Copper Slag Particle Size (μm)	<38	<71
pH	10	0,7
H2SO4 Concentration [g/L]	—	100
Glycine Concentration [g/L]	100	—
Copper Recovery after 1.5 h (%)	66	60
Copper Recovery after 2 h (%)	68	70
Copper Recovery after 4 h (%)	78	70
Copper Recovery after 10.5 h (%)	85,97	—

6 h; yet, over time, copper extraction again declined, falling below 55%. Previous Authors (Aksu and Doyle, 2001; O'Connor et al., 2018) suggest that this decrease in copper recovery is due to the precipitation of copper on the surface of the particles. They also note that during precipitation, copper may precipitate in the form of CuO and Cu_2O. However, Aghajani (2016) proposes that the covellite formed can be quickly leached with glycine, particularly in the presence of oxygen or another oxidising agent.

3.3 Temperature

Temperature is a crucial factor in improving the leaching rate of copper in acidic media, as it accelerates the Brownian movement of fine copper particles induced by elevated temperatures, thereby enhancing the efficiency of diffusion and mass transfer during leaching (Wang et al., 2019). Table 2 highlights a study by Gargul et al. (2022), which shows that at moderate temperatures of 50°C, copper recoveries of 60% were achieved within 2 h. In another study, Shi et al. (2020) conducted leaching tests on slags at elevated temperatures of 185°C under oxygen pressure, achieving copper extractions of 97.2% within 1 h. This indicates that increasing the temperature can significantly improve leaching kinetics in acidic media. However, the effect of temperature on leaching is different when using glycine. The results vary, as shown by Huang et al. (2023), who conducted leaching studies on copper slags. They demonstrated that increasing the leaching temperature to 70°C over 10.5 h resulted in copper recoveries of no more than 50%. Conversely, lowering the temperature to 40°C yielded a copper leaching rate of 85.97%. This is because elevated temperatures cause glycine to decompose through decarboxylation, which in turn reduces the rate of copper leaching (Li et al., 2020).

3.4 Particle size

The particle size in leaching is a critical factor that influences energy consumption because decreasing the particle size requires more work; however, working with smaller sizes and optimises the leaching process in both glycine and acidic media (Dreisinger and Abed, 2002). It is also a key element in the kinetic model of leaching (Li et al., 2014). In a study conducted by Chemuta Tanda (2017), chalcopyrite was leached using glycine with particle sizes ranging from a minimum of 10 μm to a maximum of +75–106 μm. The results showed that smaller particle sizes led to higher copper extractions, achieving up to 90% copper recovery. In contrast, (Sokić et al., 2019), performed similar leaching tests on copper slag using sulphuric acid as a leaching agent at a concentration of 1.5 M. Their particle size analysis involved tests with particles smaller than 37 μm and larger than 75 μm. Under the same conditions of 1.5 M acid concentration, room temperature, and the addition of hydrogen peroxide, copper extractions greater than 80% were achieved with particle sizes of 37 μm. However, when the particle size increased to 75 μm, copper recovery decreased to approximately 40%. These studies suggest that smaller particle sizes increase the surface area and reduce the diffusion barrier, thereby enhancing the contact between the mineral and the leaching agent. This results in a more efficient and rapid reaction between the mineral phases and the leaching agents, whether glycine or sulphuric acid, with copper (Shi et al., 2020).

4 Summary

Various studies on leaching processes in media with glycine and sulfuric acid highlight the advantages of each method for extracting copper from slags, although each presents its own challenges and requires individual evaluation. Sulfuric acid, although the most traditional leaching medium, presents difficulties due to its corrosive nature and the formation of a passivating layer on the slags, which hinders their dissolution. However, positive results have been reported with higher acid concentrations and elevated temperatures, achieving copper extractions greater than 80%. Glycine, on the other hand, can form stable complexes with copper, facilitating the selective leaching of copper slags. Table 2 shows that storing glycine at room temperature or around 40°C achieves copper extractions of 85% at concentrations of 100 g/L, particle sizes of 38 μm, and a pH of 10 (alkaline medium). Therefore, glycine represents a promising alternative for copper dissolution, with potential for further optimization by adjusting parameters such as concentration and particle size.

Author contributions

MM: Visualization, Writing – original draft. IC: Validation, Writing – original draft. PH: Conceptualization, Project administration, Writing – original draft. FG: Conceptualization, Investigation, Writing – review and editing. ES-R: Conceptualization, Investigation, Writing – review and editing. JC: Conceptualization, Project administration, Writing – original draft. ÁS: Conceptualization, Writing – review and editing. EG: Writing – review and editing. NT: Visualization, Writing – original draft.

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