Amino Acids and Galvanic Action to improve Copper recovery during Chalcopyrite Leaching

T Thibile, AF Mulaba-Bafubiandi and E Fosso-Kankeu

***Abstract*—** The aim of this report was to investigate the compare the potential of amino acid and galvanic interaction to reduce the formation of passivation layer and improve the overall copper recovery during chalcopyrite leaching. Sample characterization included X-Ray Fluorescence, X-Ray Diffraction, and Scanning Electron Microscopy. Manganese dioxide was used to form galvanic couple with chalcopyrite in a ratio of 4:1 respectively using sulphuric acid and glycine solutions as leaching medias. Parameters that were investigated during the leaching process were temperature (25 to 55 oC) with an increment of 10 oC, pH ranges of 1.5 – 2.5, with changes of 0.5 and time between 2 and 8 hours, with a 2-hour increment. Pregnant liquors were obtained after every 2 hours, filtered and thereafter analysed using Atomic Adsorption Spectrometry to study kinetics.

During the investigation of temperature, the maximum recovery of copper using glycine and sulphuric was 37% and 42% respectively at the temperature of 55 oC, for 8 hours at the above-mentioned optimum pH. This shows that sulphuric acid remains to be the most effective acid in comparison to glycine during the leaching of chalcopyrite using galvanic action. The use of galvanic action was also effective during chalcopyrite leaching since it increased the recovery of copper from 15% up to 42%, showing that galvanic action plays a role in reducing passivation layer during the leaching process. The rate limiting step of the reaction in the leaching process using the glycine and sulphuric acid, was found to be the diffusion-controlled mechanism. This was supported by the lower activation energies for the glycine and sulphuric acid which were found to be 4 and 16 KJ/mol respectively. The mathematical validation proved that the reactions were under the control of diffusion-controlled mechanism.

***Keywords*—** Chalcopyrite, Glycine, Sulfuric Acid, Manganese Dioxide, Galvanic Reaction, Leaching.

I. INTRODUCTION

Copper is a ductile and malleable metal that is highly valued for its electrical and thermal conductivity, and resistance to corrosion. Copper remains to be one of the most used metals across the globe contributing about 0.006 parts per million (ppm) of the earth's crust [1]. While it can occur naturally in its natural form, most copper is extracted from chalcopyrite being the most abundant copper mineral. Chalcopyrite (CuFeS2) is a widely distributed copper sulphide mineral which plays a crucial role in copper production and contributes almost 70% of the world's copper reserves. This cupric (Cu2+-containing) mineral is concentrated in just a few countries such as Chile, China, Peru, United States, and Zambia, thereby making these countries key players in global copper production and supply. According to [2], the major problem associated with CuFeS2 is its refractory nature, making copper extraction challenging through both pyrometallurgical and hydrometallurgical processes.

The existing body of knowledge with studies by [3-5], highlight that pyrometallurgy effectively extracts copper from refractory minerals using high temperature, however, this process is costly and releases harmful pollutants such as sulphur dioxide (SO2). Hydrometallurgy has been considered lower-temperature and is an eco-friendly process but limited by chalcopyrite's tendency to form a passivation layer, slowing down the dissolution process [6]. Leaching is a type of hydrometallurgical process used to dissolve the metal out of its ore mineral and this process uses organic and inorganic acids that act as a lixiviants to leach the metals. Sulphuric acid (H2SO4) is mostly used as inorganic acid during chalcopyrite leaching because it is strong, cheap, readily available, and is highly effective due to its fast dissolution rate [7-8]. However, it produces toxic by- products such as sulphates (SO42-) which cause acid mine drainage, and corrosion in leaching tanks [9-11]. In this report, H2SO4 was used with galvanic action where the galvanic couple was formed to improve copper recovery and reduce the passivation layer [12].

Amino acids were investigated for chalcopyrite leaching in this report. These acids are known for their ability to form amino acid-copper complexes and prevent the passivation layer during the chalcopyrite leaching. They are eco-friendly and nontoxic as compared to their counterpart inorganic acids. However, amino acids are expensive to produce and leach the metals at lower dissolution rate [13]. The commonly used amino acids are glycine, betaine, and lysine and they differ on their ability to leach the metal.

II. METHODOLOGY

A. Materials and apparatus

For this research, a chalcopyrite (CuFeS2) concentrate sample was used as the major solid component of the leaching process. Manganese dioxide sample (MnO2) was used to create the galvanic couple with chalcopyrite. Sulphuric acid

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was a medium in galvanic action. Glycine is the amino acid that was used as a medium in leaching process. Distilled water was used to form glycine and sulphuric acid solutions in the process. Xray fluorescence (XRF) analytical technique was used for identifying the elements in the sample. Xray Diffraction (XRD) analytical technique was used for identifying the mineral phases in the sample, and Scanning Electron Microscope (SEM) was used for analysing the morphology of the sample. Atomic absorption spectroscopy (AAS) was used to identify the amount of copper in the leachate. Analytical Balance scale was used to weigh the samples. A beaker was used to carry out the leaching process. Magnetic stirrer was used for the agitation process, where the solid materials must be kept in suspension. Filter paper and funnel were used to filter out the leachate from the residue.

B. Experimental procedure

 Preparation and characterization of the chalcopyrite sample

100g of chalcopyrite pulverised concentrate sample was received from supervisor and used in the leaching process. 30g of Chalcopyrite concentrate was analysed using XRF, XRD, and SEM techniques. The mass used for each analytical technique was 10g. The remaining 70g of chalcopyrite concentrate was used for leaching with galvanic process.

 Preparation of H2SO4 solution

0.5M of H2SO4 was mixed with distilled water to form a 100ml H2SO4 solution that was used as an electrolyte. To prepare the galvanic interaction, fine chalcopyrite and manganese dioxide (ratio 1:4) respectively were used to create a galvanic couple. The solid: liquid ratio was 1:10 (10%) and the speed of 300rpm was kept constant throughout the leaching process.

 Preparation of glycine solution

3.10g of glycine was mixed with distilled water to form a 100ml glycine solution that was used as an electrolyte. To prepare the galvanic interaction, fine chalcopyrite and manganese dioxide (ratio 1:4) respectively were used to create a galvanic couple. The solid: liquid ratio was 1:10 (10%) and the speed of 300 revolutions per minute (rpm) was kept constant throughout the leaching process.

C. Parameters investigated in the leaching process using amino acid (glycine)

 The pH

2 grams of chalcopyrite and 8 grams of manganese dioxide was put into a breaker and 100ml glycine solution was used as medium. The pH of 1.5, 2, and 2.5 was used at a constant pH of 55oC to study the effect of Ph. The speed of 300rpm was kept constant throughout the process. The pregnant liquor was obtained after 2, 4, 6, and 8 hours using the conical flask and filter paper to filter the mixture, separating the pregnant liquor from solid residue. The leachate was analysed using (AAS) and carry out kinetic study.

 Temperature

2 grams of chalcopyrite was put into a breaker and 100ml glycine solution was used as medium. The temperature varied from 25,35, 45, and 55oC at a constant pH of 1.5 to study the effect of temperature. The speed of 300rpm was kept constant throughout the process. The pregnant liquor was obtained after 2 hours' interval for 8 hours using the conical flask and filter paper to filter the mixture, separating the pregnant liquor from solid residue. The time varied from 2, 4, 6, and 8 hours to study the effect of temperature. The leachate was analysed using AAS and carry out kinetic study.

D. Parameter investigated for galvanic action with sulphuric acid

 The pH

Two (2) grams of chalcopyrite and 8 grams of manganese dioxide was put into a breaker and 100ml H2SO4 solution was used as medium. The pH of 1.5, 2, and 2.5 was used at a constant pH of 55 oC to study the effect of pH. The speed of 300rpm was kept constant throughout the process. The pregnant liquor was obtained after 2, 4, 6, and 8 hours using the conical flask and filter paper to filter the mixture, separating the pregnant liquor from solid residue. The leachate was analysed using (AAS) and carry out kinetic study.

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The above methodology is depicted in figures 1 and 2 which clearly explain each step undertaken in achieving the current methodology for the leaching process..

III. RESULTS AND DISCUSSION

Copper (II) oxide is the most dominant oxide concentration with a percentage of 32.88% making up the Cu with a percentage of 26.26%. This shows that the sample is a copper bearing mineral. Iron is the second dominant element with a percentage of 19.46% making Fe2O3 to have a percentage of 27.82%, indicating the presence of iron oxides typical of chalcopyrite and associated minerals. Other components include silicon (6.52% as SiO2), calcium (3.57% as CaO), magnesium (2.26% as MgO), and aluminium (1.41% as Al2O3), which likely originate from gangue minerals such as silicates and carbonates. Trace elements like phosphorus, potassium, titanium, manganese, cobalt, nickel, zinc, and strontium are also present in trace amounts, suggesting minor mineral phases or impurities.

 XRD analysis

A powdered chalcopyrite concentrate sample was placed in an X-ray diffractometer. As the X-rays interacted with the crystal lattice, they were diffracted at specific angles. In this case, Figure 3 shows the XRD results for the unroasted chalcopyrite concentrate before the leaching process.

XRD

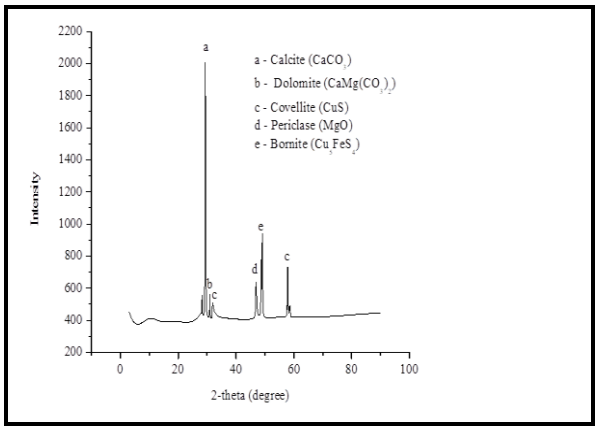


Fig. 1: XRD results of chalcopyrite sample

The dominant peaks correspond to Covellite (CuS) and Bornite ($Cu_5 FeS_4$) confirming this sample as the dominant copper-bearing phase in the sample. However, the additional peaks in figure 3 indicates that the concentrate also contains associated minerals and gangue components. Calcite ($CaCO_3$) has the sharpest peak, suggesting that carbonate minerals are present in significant quantities.

SEM analysis

Figure 4 shows the SEM results of the unroasted chalcopyrite concentrate before the leaching process. Table 2 indicates the elemental compositions at spectrums 1, 2, 3, and 4 shown in EDS results that is obtained from the SEM image.

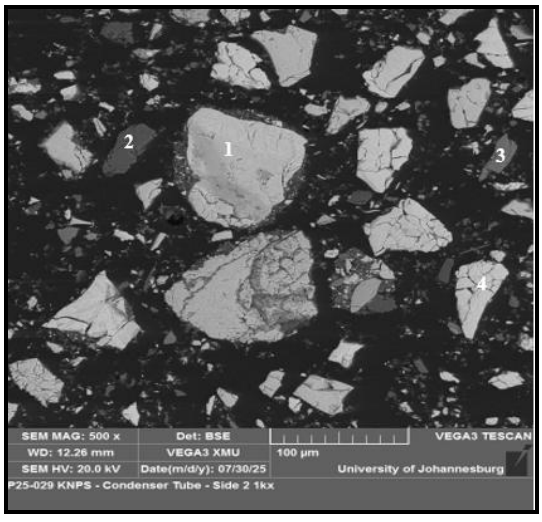
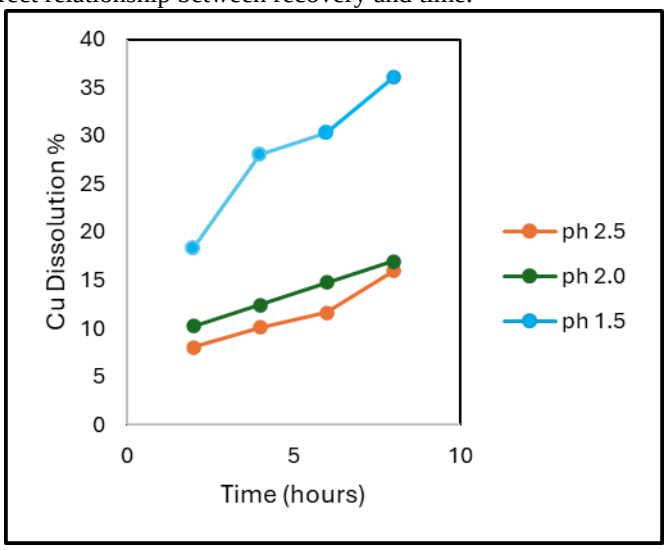


Fig. 1: SEM results of chalcopyrite concentrate

From the EDS results, spectrum 1 region shows high copper percentage (59.7%), sulphur (39.2%), and low iron (1%), indicating a relatively pure and unaltered chalcopyrite phase. Spectrum 2 is dominated by carbon (36%), oxygen (30.3%), magnesium (18.1%), and silicon (9%). This indicates the presence of gangue mineral in the sample. Spectrum 3 has low amounts of copper and contain high levels of carbon (38.6%), fluorine (16%), and oxygen (16%). Spectrum 4 has high amount of copper (41%), sulphur (37%), and iron (21%), consistent with chalcopyrite but with a notably higher iron content.

Baseline effects of pH during chalcopyrite leaching

Figure 3 shows the graph which was conducted to investigate the effect of pH and time on the recovery of copper from chalcopyrite using glycine. The graph shows a direct relationship between recovery and time.



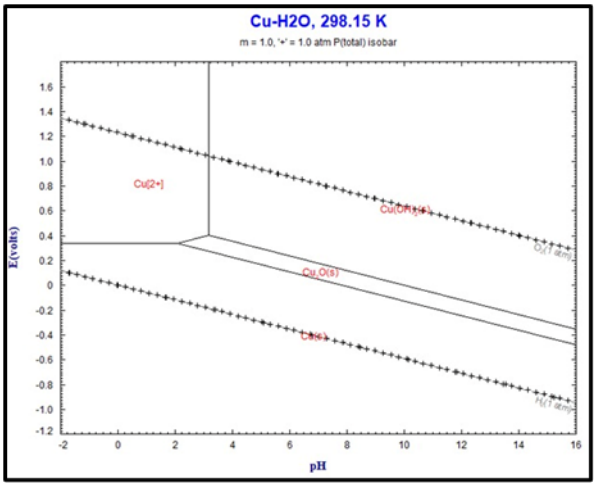


Fig. 3: Recovery of copper using glycine under different pH (A) and Pourbaix diagram of Cu at 25ºC (B)

In Figure 3A, a consistent trend was observed, which was that copper recovery increased with a decreasing pH (2.5, 2.0,

and 1.5) and increasing time from 2 to 8 hours. The graph shows that the pH of 1.5 yielded the most copper recovery at the highest time which was 8 hours. The results correspond to the existing literature which shows that the optimum pH for obtaining maximum chalcopyrite recovery is a pH of 1.5. Also, the Pourbaix diagram of copper- water system in figure 5B supports the actual results that copper dissolves in a pH that is between -2 to 3. The pH of 1.5 was found to be optimum pH for chalcopyrite leaching, therefore it was used for further experiment.

Comparison of two acids based on Cu recoveries using galvanic action

Figure 6 shows the graph which was conducted to investigate the effect of temperature and time on the recovery of copper from chalcopyrite using two different leaching acids which were sulphuric acid and glycine. The graph shows a direct relationship between recovery and time.

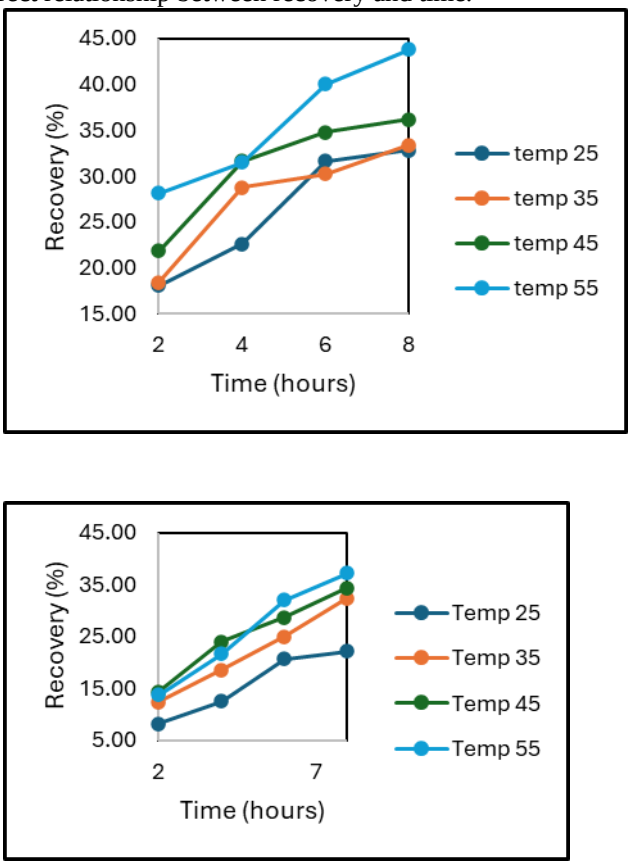


Fig. 4: Recoveries of copper using sulphuric acid (A) and glycine with galvanic action (B)

In both leaching conditions, a consistent trend was observed, which was that copper recovery increased with time from 2 to 8 hours and was highest at elevated temperatures (25, 35, 45, and 55 °C). In the sulphuric acid medium, recoveries from 2, 4, 6, and 8 hours were 30%, 33.44%, 36.19%, and 44.85% respectively, showing that galvanic action becomes more effective over time, likely due to accelerated electrochemical reactions. Literature review supports that optimal recovery occurs between 50 – 55 °C and 7 – 10 hours, with a pH of 1.5 being critical for maximizing copper extraction.

Similarly, in the glycine medium, copper recovery also increased with time and temperature, reaching 22.15%, 32.37%, 34.33%, and 37.24% at 8 hours across the same temperature range. While both acids show improved recovery with time and temperature, sulphuric acid stood out from glycine in terms of copper yield. Therefore, despite its environmental drawbacks mentioned in the literature review, sulphuric acid remains the more effective medium for copper recovery when used in conjunction with galvanic action.

Kinetic analysis

Table 1 presents the calculated correlation coefficients (R^2) corresponding to kinetic models and variables related to sulphuric acid and galvanic action.

TABLE 1:CORRELATION VALUES OF SHRINKING CORE KINETIC MODELS FOR CHALCOPYRITE

Variable	$kt = X$	$kt = 1 - 3(1 - xb)^{\frac{2}{3}} + 2(1 - xb)^3$	$kt = 1 - (1 - xb)^{\frac{1}{3}}$
Temperature, R^2	0,90015	0,92465	0,90538

The regression coefficients obtained by fitting the experimental data to the shrinking core models are too close to deduce the rate controlling mechanism of copper dissolution from CuFeS2 mineral. From the correlation coefficients obtained in table 3, it can be said that the rate limiting step for this reaction is diffusion-controlled mechanism since it has the highest R2 (0.92465). From the literature review, this means that during the leaching process, sulphuric acid solution diffused into the chalcopyrite mineral and dissolved copper into the solution.

Figure 5 shows the Arrhenius plot used to evaluate the temperature dependence of reaction rates. Activation energy (Ea) was obtained from the slop of line which will be discussed for the diffusion-controlled reaction.

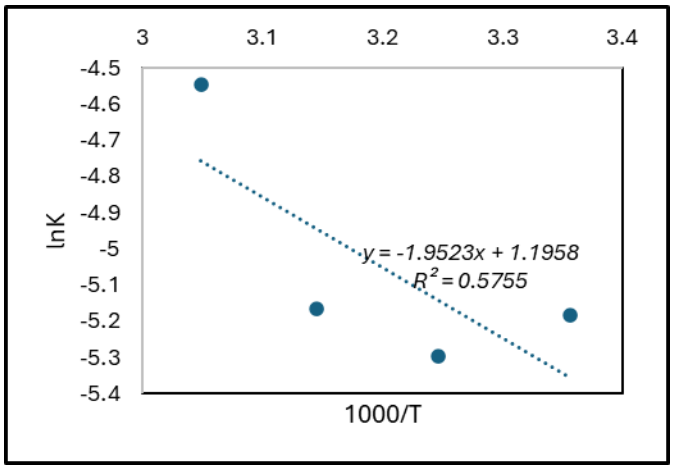


Fig. 5: Arhenius plot graph of the sulphuric acid and galvanic action

Mathematical validation for sulphuric acid

In Figure 7, the Arrhenius plot for sulphuric acid leaching with galvanic action show the temperature dependence of the reaction rate by plotting ln(k) against inverse temperature (1000/T). The linear regression equation derived from the graph is y=-1.9585x-3.015, with an R^2 value of 0.5755, indicating a moderate correlation between temperature and the rate constant. The gradient of the line (1.9523) corresponds to the Ea, which found as 16.234 kJ/mol. The literature review explains that diffusion-controlled mechanism has the activation energy that is smaller than 40KJ/mol which corresponds to the actual results obtained from figure 7. The activation energy also suggest that the reaction is not highly energy-intensive and can proceed efficiently with moderate temperature increases.

Mathematical validation for sulphuric acid

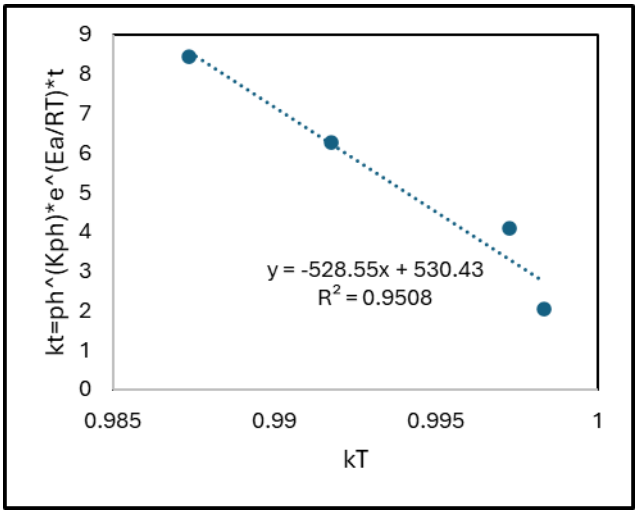


Fig. 6: mathematical validation for sulphuric acid and galvanic action

TABLE II: CORRELATION VALUES OF SHRINKING CORE KINETIC MODELS FOR CHALCOPYRITE

Variables	$kt = X$	$kt = 1-3(1-xb)^{\frac{2}{3}}+2(1-xb)$	$kt = 1-(1-xb)^{\frac{1}{3}}$
Temperature, R^2	0,97688	0,977488	0,96958

From the correlation values obtained in table 4, it can be said that the rate limiting step for the reaction is diffusion-controlled mechanism since it has the highest R^2 (0.977488). This means that during the leaching process, sulphuric acid solution diffused into the chalcopyrite mineral and dissolved copper into the solution [14-19].

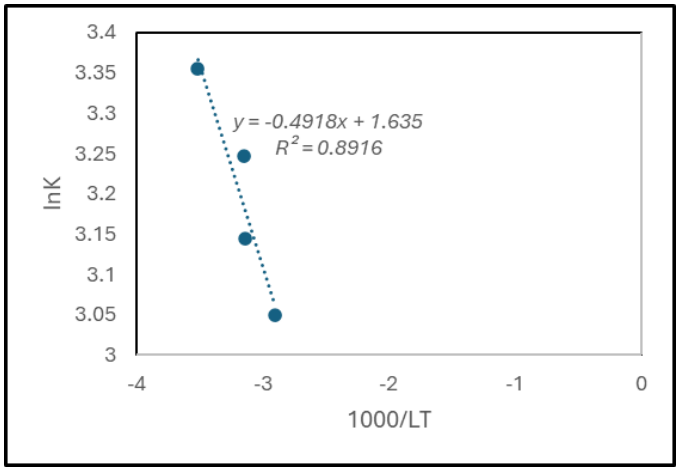


Fig 7: Arrhenius plot graph of glycine

In Figure 7, the graph plots the natural logarithm of the rate constant (lnK) against the inverse of temperature (1000/LT), producing a linear regression with the equation y=-0.4918x+1.635 and an R^2 value of 0.8916. This high R^2 indicates that 89.16% of the variation in the rate constant is explained by changes in temperature, emphasising the Arrhenius model to this system. Activation energy is found to be 4.08 kJ/mol, which was derived from the slope of the line. This supports that the mechanism for the reaction is diffusion-controlled mechanism since from the literature review the activation energy for diffusion-controlled mechanism is < 40KJ/mol. The activation energy obtained suggests that minimum energy is required for the reaction to occur in glycine leaching system than in the sulphuric acid leaching system.

Mathematical validation (glycine)

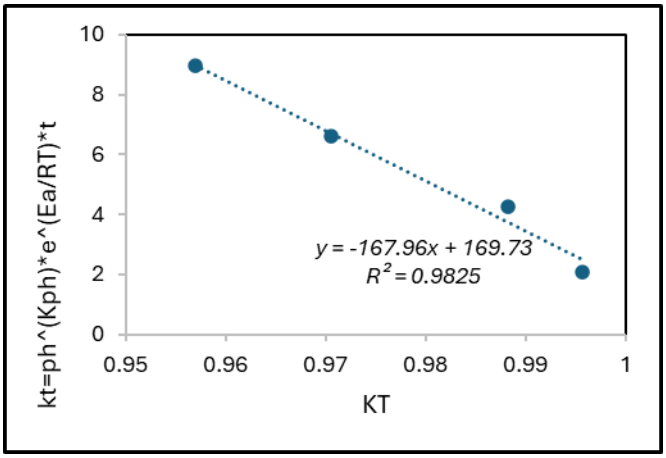


Fig. 8: Mathematical validation for glycine acid

This is the leaching process where glycine was used as a medium, and the mathematical validation reflects a diffusion-controlled mechanism, where the rate-limiting step is controlled by diffusion of glycine solution into the chalcopyrite mineral. According to Figure 4, the leaching process was efficient at a pH of 1.5, with a temperature of 55 °C, at 8 hours where the maximum recovery was 37%.

In Figure 8, the graph plots KT on the x-axis against the rate expression kτ=(kp·h)/(kp·h)·e^(E_a/RT)·t, which simplifies to a temperature and time-dependent kinetic model. The linear regression yields the equation y=-167.96x+169.73 with a high R^2 value of 0.9825, indicating that 98.25% of the variation in the leaching rate is explained by the linear model. This strong correlation confirms that the leaching behaviour is highly predictable and consistent with diffusion-controlled mechanism, where reaction diffusion mechanism dominates and chemical mechanism plays a minimal role.

IV. Conclusion

The aim of this experiment was achieved which was to reduce the passivation layer during the chalcopyrite leaching using galvanic action. Glycine was used as it can form complexes with copper and was compared with sulphuric acid during the leaching process. It was found that the maximum recovery of copper using glycine and sulphuric was 37% and 42% respectively at the temperature of 55 oC, with pH of 1.5, and at 8 hours. This shows that even though sulphuric acid is environmentally unfriendly, it remains to be more effective than glycine during the leaching of chalcopyrite using galvanic action. The use of galvanic action was also effective during chalcopyrite leaching since it increased the recovery of copper from 15% shown in the literature review up to 42% shown by the results obtained. This shows that galvanic action played a role in reducing passivation layer during the leaching process.

The rate limiting step of the reaction in both leaching process using the glycine and sulphuric acid, was found to be the diffusion-controlled mechanism which meant the reagent diffused into chalcopyrite mineral and dissolved copper into the solution. This was supported by the lower activation energies for the glycine and sulphuric acid which were found to be 4 and 16 kJ/mol respectively. The mathematical validation proved that the reactions were under the control of diffusion-controlled mechanism.

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