Comparative Effects of Ferric and Ferrous Sulfate on the Leaching of Chalcopyrite

KB Mphela and E Fosso-Kankeu

***Abstract*—** This study investigates the leaching kinetics of chalcopyrite concentrate using three different lixiviants: sulfuric acid (H_2SO_4), ferrous sulfate ($FeSO_4$), and ferric sulfate ($Fe_2(SO_4)_3$) across a temperature range of 25°C to 65°C. Characterization of the chalcopyrite concentrate via X-ray fluorescence (XRF) revealed copper and iron as the dominant components at 32.88% and 27.82%, respectively, confirming economic viability for copper extraction. X-ray diffraction (XRD) analysis identified a complex mineralogical assemblage including chalcopyrite ($CuFeS_2$), bornite (Cu_5FeS_4), chalcocite (Cu_2S), covellite (CuS), and various gangue minerals. Scanning electron microscopy with energy-dispersive spectroscopy (SEM-EDS) confirmed stoichiometric composition and revealed smooth, angular particle morphology with minimal surface oxidation. Kinetic modeling using the Shrinking Core Model (SCM) demonstrated temperature-dependent mechanistic transitions. For H_2SO_4 leaching, diffusion through the product layer dominated with correlation coefficients (R^2) ranging from 0.90 to 0.99 and activation energies between 51.2 to 79.8 kJ/mol. $FeSO_4$ leaching exhibited chemical reaction control at elevated temperatures (55-65°C) with R^2 values reaching 0.98, while diffusion control prevailed at lower temperatures (25-45°C). $Fe_2(SO_4)_3$ leaching showed the most pronounced mechanistic shift, with diffusion control dominating below 45°C (R^2 up to 0.9959) and chemical reaction control above 55°C (R^2 = 0.9815). Copper recovery increased substantially with temperature across all lixiviants, ranging from 6-10% at 25-45°C to 15-22% at 55-65°C after eight hours. The formation of passivating elemental sulfur layers at lower temperatures and their breakdown at elevated temperatures explain the observed mechanistic transitions. These findings provide critical insights for optimizing industrial hydrometallurgical copper extraction processes from chalcopyrite concentrates.

***Keywords*—** Chalcopyrite, ferric sulfate, ferrous sulfate, sulfuric acid, leaching kinetic.

I. Introduction

Chalcopyrite, the most abundant mineral in copper ore bodies. It's notoriously difficult to process using hydrometallurgical processes due to its refractory nature that's why pyrometallurgical processes are preferred. The declining mineral grades and environmental concerns has encouraged the use of hydrometallurgical processes for the treatment of chalcopyrite This has made hydrometallurgical processes more cost-efficient than pyrometallurgical processes in copper extraction in chalcopyrite [1].

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Leaching is favoured for copper extraction from chalcopyrite in hydrometallurgical processes because it enables efficient, environmentally friendly, and economically viable copper recovery by overcoming the mineral's passivation and refractory nature through controlled chemical and mechanical means [2].

Chalcopyrite leaching is traditionally considered dependent on ferric ions (Fe^{3+}) as the primary oxidant, with ferrous ions (Fe^{2+}) playing a secondary role as a precursor for Fe^{3+} regeneration [3]. The formation of a passivation layer is a significant phenomenon in the leaching of chalcopyrite ($CuFeS_2$), especially when using oxidizing agents. A passivation layer can inhibit further dissolution of the mineral, slowing down the overall leaching process [4].

Studies demonstrate that Fe^{2+} enhances copper extraction more effectively than Fe^{3+} in acidic solutions. Fe^{3+} leaching produces a hydrophobic sulphur-rich layer that passivates the mineral surface, slowing dissolution. Fe^{2+} promotes oxidative dissolution via dissolved oxygen, generating soluble sulphur intermediates and avoiding passivation. This contradiction between theoretical models and experimental outcomes highlights a critical gap in understanding the redox chemistry and surface processes governing chalcopyrite dissolution.

II. METHODOLOGY

A. Equipment

X-Ray Fluorescence (XRF); X-Ray Diffraction (XRD); Atomic Absorption Spectroscopy (AAS)

B. Materials

Unroasted chalcopyrite Concentrate (CuFeS2); Ferric Sulfate (Fe2(SO4)3); Ferrous Sulfate (FeSO4); Sulfuric Acid (H2SO4); Manganese Dioxide (MnO2)

C. Sample Preparation & Characterization

Approximately 500g of chalcopyrite concentrated was collected and sent for pulverization to obtain particle size □75□m. Approximately 50g of sample was extracted from the pulverized sample and sent for sample characterization (XRF, XRD & SEM-EDS).

D. X-Ray Fluorescence (XRF)

10grams of CuFeS2 was mixed 5grams with Sasol wax homogenously. Mixture was placed into aluminium cup and

the sent for pelletisation using a hydraulic press to remove all residual moisture in mixture. Mixture was then sent for XRF analysis to determine the elemental composition of the chalcopyrite sample.

E. X-Ray Diffraction (XRD)

Remaining CuFeS2 was used to fill up a glass container to its maximum. A glass slide was placed on top of the sample to hold down and flatten the surface of the sample. This made it homogenous while being equally spread out all over the glass container. The CuFeS2 was sent for XRD analysis for mineral identification.

F. Scanning Electron Microscopy(SEM-EDS)

10grams of CuFeS2 was then sent for SEM-EDS analysis to determine the imaging and elemental composition of the chalcopyrite sample.

G. Leaching

Ferric Sulfate Solution (0,1M)

Ferric sulphate was dissolved in denoised water to produce 100ml ferrous sulphate solution (0,1M). 10g of manganese dioxide was added to the solution for galvanic coupling. A magnetic stirrer was placed in the ferric sulphate solution before it was placed on hotplate set at 25oC. Once the solution was mixture to homogeneity and heated up, sulfuric acid (18M) was added incrementally to obtain pH 1,5. pH was monitored with a pH meter. CuFeS2 (2,5% w/v) was added to the solution. Watch glass was placed on top of beaker to vapor doesn't escape. Leaching was carried out over 8hrs, leaching solution was extracted every 2hrs. This process was repeated at 35,45,55 & 65oC.

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Control Solution (Sulfuric acid solution only)

10g of manganese dioxide was added to 100ml of deionized water. A magnetic stirrer was placed in the solution before it was placed on hotplate set at 25oC. Once the solution was mixture to homogeneity and heated up, sulfuric acid (18M) was added incrementally to obtain pH 1,5. pH was monitored with a pH meter. CuFeS2 (2,5% w/v) was added to the solution. Watch glass was placed on top of beaker to vapor doesn't escape. Leaching was carried out over 8hours, leaching solution was extracted every 2hours. This process was repeated at 35,45,55 & 65oC. The extracted leaching solution was filtered and placed into a labeled container.

Post-Leaching Analysis

AAS (Atomic Absorption Spectroscopy)

1ml of filtered leach liquor was extracted and placed into a container. 5ml of deionized water was added to the leachate to dilute solution, this process was repeated until the solution was within detection range for copper analysis. This process was repeated for every filtered leach liquor extracted.

III. RESULTS AND DISCUSSION

A. *Sample characterization data.*

XRF

Table 1; XRF results show us that copper is the most abundant component (32.8751%) in the sample. This shows us that the sample is rich in copper (Cu). Iron content is shown to be the second most abundant component (27.8164%) in the sample. This high iron content could slow down the leaching process by encouraging the formation of passivating layers that would surround the chalcopyrite and reduce exposure to the leaching reagents. The high iron and copper contents in the chalcopyrite sample indicate that the chalcopyrite sample is economically suitable for copper extraction. The presence of calcium is considered an impurity (gangue) in the chalcopyrite sample. This could be the results of other gangue minerals as carbonate minerals (calcite or dolomite). Calcium can affect the pH level of the leachate by increasing it. This would cause the leachate to become more basic and reduce leaching rates.

b

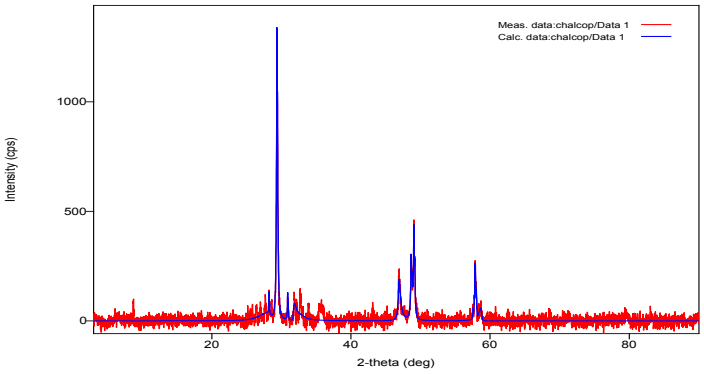

Figure 1:Unroatsed chalcopyrite XRD results

Figure 1, shows the X-ray diffraction (XRD) analysis of the chalcopyrite concentrate show a complex mixture of copper sulfides and gangue minerals. The dominant copper-bearing minerals were chalcopyrite ($CuFeS_2$), bornite (Cu_5 FeS_4), chalcocite (Cu_2 S), and covellite (CuS). Other identified phases included magnetite (Fe_3 O_4), quartz (SiO_2), calcite ($CaCO_3$), dolomite ($CaMg(CO_3)_2$), periclase (MgO), and enstatite ($MgSiO_3$), indicating significant mineral diversity.

The XRD measurements were conducted at 40 kV and 30 mA, across a continuous 2θ range from 3° to 90°.

SEM-EDS

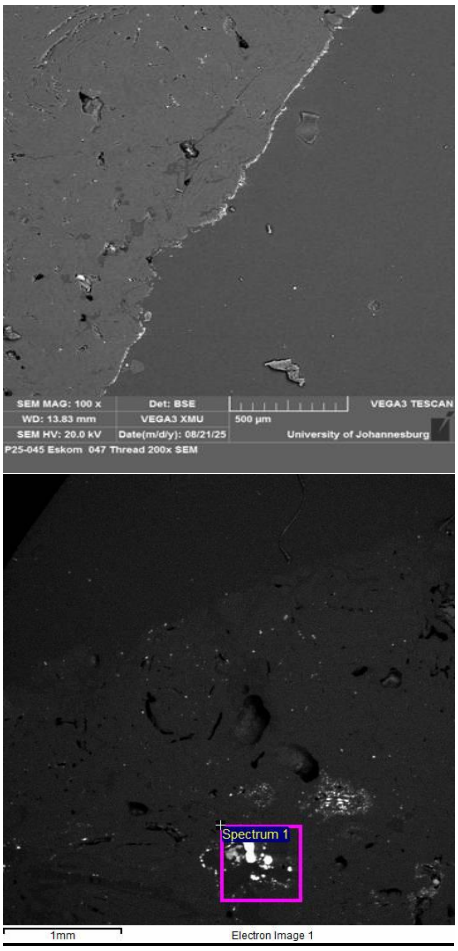

Fig. 2:Unroasted Chalcopyrite SEM-EDS results

Figure 2 shows, the unroasted chalcopyrite concentrate has smooth, well-defined particle surfaces with angular morphology characteristic showing it was mechanically liberated chalcopyrite. Particle size distribution appears heterogeneous with predominantly sub-angular shapes maintaining original crystallographic faces. Surface texture remains uniform without significant roughening or pitting that typically develops during thermal or chemical processing. The preserved crystal edges and faces indicate effective liberation during concentration while maintaining structural integrity.

EDS analysis shown in figure 2, confirms the stoichiometric composition of chalcopyrite as $CuFeS_2$, with copper concentrations ranging from 32.8-35.4 wt% and iron from 29.7-31.8 wt%. The elemental mapping shows uniform distribution across particle surfaces without significant compositional gradients.

Figure 2 shows high-resolution imaging demonstrates excellent preservation of crystallographic features including cleavage planes and surface striations. The crystal lattice remains undamaged by the concentration process, maintaining optimal conditions for chemical reactivity. Minimal porosity or surface defects characterize the unroasted state, contrasting with thermally treated materials that develop surface roughening and increased porosity. This structural integrity affects subsequent leaching kinetics and extraction efficiency.

Copper recoveries

The copper recoveries for each leaching solution all show a distinct recovery patterns, with temperature and time having influence on copper dissolution efficiency. The data clearly shows that time and temperature have a directly proportional relationship to copper dissolution efficiency, with increasing time and temperature copper dissolution efficiency increases.

Ferric sulphate solution (0,1M) obtained the highest overall Cu recovery of 31,81% unlike ferrous sulphate solution (0,1M) which obtained the worst copper recovery of 24,85% at 650C after 8hrs. These findings align with recent investigations, which established that ferric ions play a catalytic role (oxidizing agent) in copper sulphide dissolution by maintaining the desirable redox conditions that prevent occurrence of surface passivation during leaching. The copper recoveries obtained display incremental improvements at every chemical and thermal threshold, underscoring the kinetic favourability of copper dissolution as a function of both oxidant strength and temperature. Córdoba et al. [5], demonstrated that Fe^{3+} ions maintain their oxidizing integrity more effectively at elevated temperatures, which mitigates passivation and increases Cu release. These results further reinforce the Córdoba's findings, validating the observed temperature dependency and vindicating ferric sulphate as a highly effective agent for unroasted chalcopyrite leaching.

Copper recovery for $FeSO_4$ leaching, reveals a significant influence of leaching temperature on copper extraction efficiency over time. At lower temperatures (25°C and 35°C), copper recovery remains relatively minimal throughout the 8hr leaching period, with final recoveries of approximately 10% and 12%, respectively. As temperature increases, notable improvements are observed: recovery at 45°C reaches about 13% after 8 hours, while at 55°C and 65°C, copper recovery rises substantially to approximately 20% and 25%. This trend is consistent with previous studies, which indicate that higher temperatures accelerate leaching kinetics and improve metal recovery, provided that the temperature does not exceed the optimum range where side reactions or excessive reagent consumption may occur.

Ferrous sulphate obtained the lowest overall Cu recovery of 24,85% at 650C after the 8 hrs. This shows us that Ferrous sulphate lacks the necessary oxidizing power to directly attack the CuFe2 lattice unlike ferric sulphate. The principle leaching mechanism relies on ferrous iron first being oxidized to ferric iron, which then reacts with the mineral. In the absence of external oxidants such as dissolved oxygen or additional chemical oxidizers, this process becomes self-limiting and profoundly sluggish. Ferrous sulphate leaching is consistently reported as less effective for copper solubilization from unroasted chalcopyrite. Research done by Sadowski and

Dreisinger [6] highlight that in the absence of active ferric ion regeneration, copper recovery rates in ferrous sulphate solutions stagnate due to the rapid depletion of oxidizing equivalents and the emergence of passivating layers.

Sulfuric acid solution is used as our control reference point to be able to see how does ferric and ferrous sulphate as oxidizing agents affect the chalcopyrite leaching procedure. Our sulfuric acid, where oxidizing agents are absent, functions primarily as a solvent medium rather than an active oxidant for chalcopyrite during leaching. Our control performed moderately, obtaining an overall Cu recovery of 28,70% at 650C after 8hours. The solution was able to obtain better recoveries than ferrous sulphate (24,85% at 650C after 8hrs) but not better than ferric sulphate solution (31,81% at 650C after 8hrs). it showed similar temperature sensitivity to ferric sulphate, where better Cu dissolution efficiency is achieved at elevated temperatures. Olvera et al. [7] demonstrate that acid-alone systems rarely yield recoveries above 20-30%, unless substantial mechanical or thermal pre-treatments or oxidants are included. The current findings, with maximum recoveries from acid leaching below those seen with ferric sulphate, reinforce the established understanding that oxidant addition is essential for efficient chalcopyrite hydrometallurgy.

All solutions show Cu dissolution efficiency is sensitive to temperature, where better Cu dissolution are obtained at elevated temperatures. This points to elevated temperatures lead to improved dissolution kinetics of secondary copper minerals. Muzenda et al. investigated temperature effects during copper leaching and concluded that copper recovery generally increases with temperature, but excessively high temperatures can trigger unfavourable reactions that may reduce recovery in some systems. Similarly, kinetic investigations such as those by Cariaga et al. and recent comprehensive reviews highlight that copper leaching rates are strongly tied to thermally activated dissolution reactions, especially in sulphate systems. Typical laboratory and industrial tests affirm that optimal copper recoveries are achieved at elevated temperatures, aligning with the observation that copper recovery in your data rises markedly at 55°C and 65°C over 8 hours.

Leaching kinetics

The shrinking core model assumes that the chalcopyrite particles maintain their spherical geometry as dissolution proceeds, with the reaction occurring at the interface between the unreacted mineral core and the surrounding solution or product layer. This model has been extensively applied to chalcopyrite leaching systems and has proven particularly suitable for describing the dissolution kinetics under conditions where surface chemical reactions control the overall rate.

Ferrous Sulphate (FeSO4) Solution 1M

Shrinking Core Model – Chemical Reaction Control

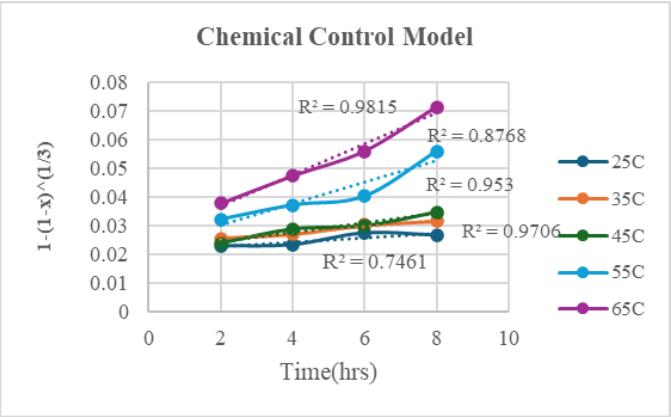

Fig.3: FeSO4 graph

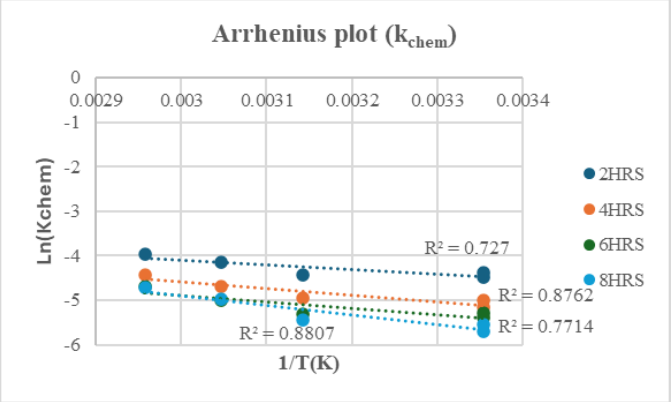

Fig. 4: Arrenhius plot

Figure 3, shows the Shrinking Core Model - chemical reaction control model. The R2 values (coefficient of determination) all exceed 0,90 at 35,45 and 650C. This fit further substantiates that surface chemical reaction is the rate-limiting factor. The chemical reaction control is the dominant kinetic mechanism for chalcopyrite dissolution in ferrous sulphate solution at these temperatures. The R2 value at 250C is 0,727, which indicates that the chemical reaction control isn't the dominant kinetic mechanism in chalcopyrite dissolution. This points to the diffusion layer control being the dominant kinetic mechanism for CuFeS2 dissolution in ferrous sulphate leaching. The R2 value at 550C is 0,8768, this points to a transition from diffusion layer control to chemical reaction control being the more dominant kinetic mechanism. This points more to a mixed system for CuFeS2 dissolution in ferrous sulphate solution. This trend shown in figure3 align with the findings of Dakkoune et al. [1] increasing the temperature and mechanical enhancement both yielded higher reaction rates and more complete copper recovery. It's clear that rate constants increase with both temperature and reaction time. This result implies the system avoids rapid surface passivation and that fresh mineral surface continues to be exposed as the reaction progresses. Such behaviour is similar to what Dakkoune et al [1] described for attrition-enhanced chalcopyrite leaching, where mechanical agitation assists in both particle breakage and removal of

passivating layers, keeping the leaching rate from plateauing prematurely.

Figure 4, shows the Arrhenius plot for ferrous sulphate solution leaching. The plot shows an increase in reaction rate constant with increasing temperature showing the influences temperature has on the reaction rate in CuFeS2 leaching in ferrous sulphate solution. The R2 values (coefficient of determination) all are below 0,90. This linearity shows us that the diffusion control or mixed kinetic processes are the more dominant kinetic mechanisms in the leaching of CuFeS2 in ferrous sulphate solution unlike chemical reaction control kinetic mechanism. The activation energies calculate and shown in table 6 range between 8,6-18kJ/mol. This further proves that the diffusion layer control/ mixed kinetic processes are the dominant kinetic mechanism in the leaching.

Ferric Sulphate (Fe_2 $(SO_4)_3$) Solution 1M

Shrinking Core Model-Chemical Reaction Control

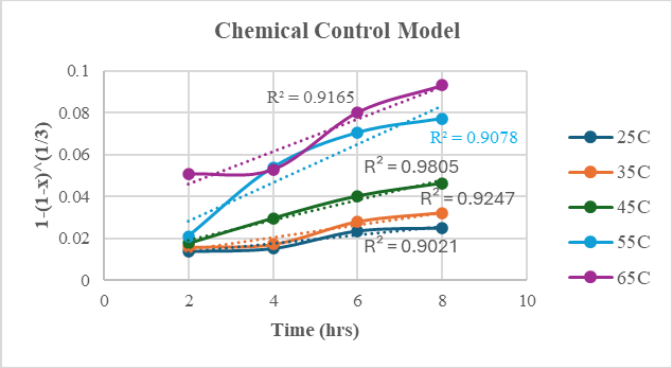

Fig. 3: Ferric sulfate

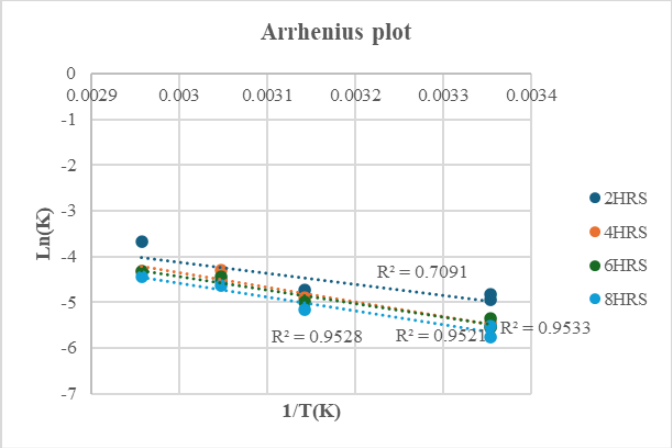

Fig. 4:Ferric sulphate Arrhenius plot

Figure 5, shows the shrinking core model chemical control. Figure 5 shows a temperature dependent kinetic behaviour, with the chemical control model showing excellent correlation with all R2 values (coefficient of determination) all exceed 0,90 with the highest fit being 0,9805 at 450C. This shows that the surface chemical reaction is the rate-limiting factor. The chemical reaction control is the dominant kinetic mechanism for chalcopyrite dissolution in ferric sulphate solution.

Figure 6, shows the Arrhenius plot where great linearity is displayed where R2 exceeds 0,90 at 4-8hrs. The R2 value at 250C is 0,7091, which substantially lower. This could potential lead to the possibility of the diffusion control mechanism potentially being more dominant is the early stages of the leaching process. The activation energies calculated from figure 6 of the ferric sulphate solution range from 19,9-25,3 kJ/mol. Wan and colleagues reported an activation energy of 46 kJ/mol for chalcopyrite dissolution in ferric sulphate media, confirming surface electrochemical reaction control during initial leaching stages. This magnitude substantially exceeds the threshold of 20 kJ/mol characteristic of diffusion-controlled processes, thereby substantiating a mechanism governed by interfacial charge transfer reactions.

Control- Sulphuric acid solution (H2SO4)

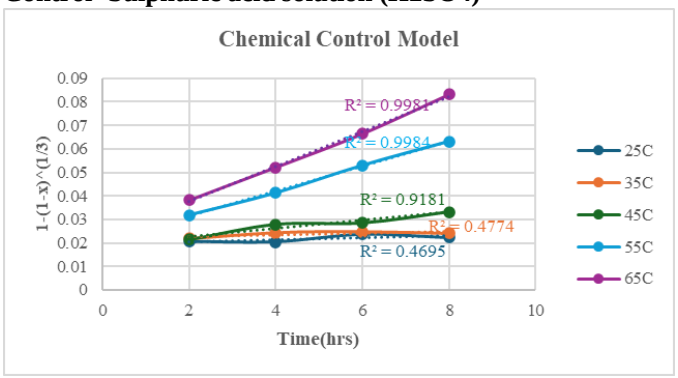

Fig. 5: Shrinking Core Model

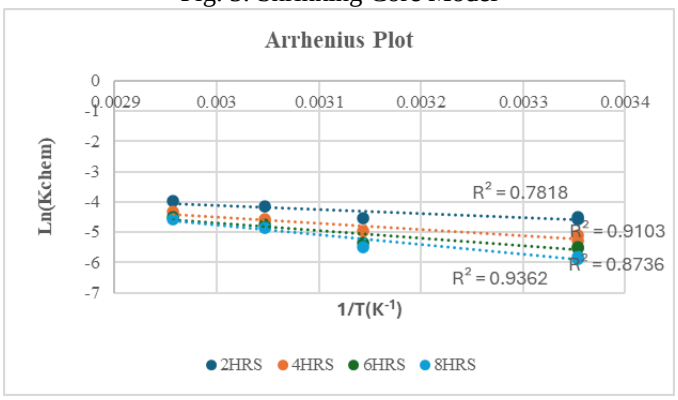

Fig. 6: Arrhenius Plot Chemical Reaction Control for Ferrous Sulphate

All data shown in Figure 7, shows us that reaction rate is influenced by temperature with elevated temperatures obtaining higher reaction rate constants [8-14]. All R2 values exceed 0,90 at 45-650C. This shows that the chemical reaction control is the best fit kinetic mechanism in our control/sulphuric acid solution. The surface chemical reaction is the rate limiting factor.

At lower temperatures of 25-350C, the R2 values are all below 0,5 which shows that the diffusion layer or mixed kinetic process is the more dominant mechanism at these temperatures unlike the chemical control mechanism. This data obtained aligns with Córdoba et al. where he tested all 3

https://doi.org/10.17758/IICBE7.C1125183

Shrinking Core Models (Mixed kinetic processes, chemical and diffusion control mechanisms). He found that the chemical reaction control model provided the best fit at pH 1-2 at moderate temperatures 40-700C, showed R2 values greater than 0,998. This further proves that the chemical reaction control model best describes leaching of CuFeS2 with only sulfuric acid.

Figure 8, shows the Arrhenius plot with good linearity. R2 values from 6-8hrs showing good linearity which further prove that the chemical reaction control mechanism is the dominant kinetic mechanism and surface chemical reaction is the rate limiting factor in CuFeS2 leaching with sulfuric acid only. At 2-4hrs the R2 values are below 0,90 which indicates a diffusion control mechanism or mixed chemical processes is the dominant kinetic mechanism. the 4hr mark having an R2 value of 0,8736 could possible indicate a transition from diffusion layer control or mixed kinetic processes mechanism. the activation energies calculated from figure 8 are show in table 7. The activation energies for the sulphuric acid only leaching of chalcopyrite range between 11,4-26,3kJ/mol.

IV. Conclusion

This investigation of unroasted chalcopyrite leaching kinetics reveals that temperature critically influences both copper extraction efficiency and rate-controlling mechanisms across different lixiviant systems. Characterization confirmed favourable copper content (32.88%) for extraction, though elevated iron levels (27.82%) and calcium-bearing gangue minerals pose challenges through passivation layer formation and pH effects.

Application of the Shrinking Core Model successfully differentiated kinetic behavior among three leaching systems. Sulfuric acid leaching exhibited persistent chemical reaction control across at elevated temperatures (R^2 = 0.90-0.99), with activation energies of 11,4-26,3 kJ/mol indicating mass transfer limitations from elemental sulfur accumulation. Ferrous sulfate demonstrated transitional behavior, shifting from diffusion control at 25-45°C to chemical reaction control at 55-65°C (R^2 = 0.75-0.98). Ferric sulfate showed the most efficient performance with clear mechanistic delineation: diffusion control below 45°C (R^2 0,90) and chemical reaction control above 55°C (R^2 = 0.9815).

Copper recovery increased with temperature across all systems, rising from 6-10% at lower temperatures to 15-31% at 55-65°C after 8 hrs. This temperature dependence shows the importance of thermal activation in overcoming chemical reaction limitations. The formation and properties of sulfur product layers emerged as the critical factor determining rate-limiting mechanisms.

Acknowledgment

The authors acknowledge the financial support from the Faculty of Engineering and the Built Environment at the University of Johannesburg in South Africa.

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