

HYDROMETALLURGICAL EXTRACTION OF ZINC FROM SPHALERITE USING SALTED-OUT ALKALINE (AMMONIA-AMMONIUM SULPHATE): INSIGHTS INTO KINETIC MECHANISMS

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Abstract

This research explores the hydrometallurgical extraction of zinc from sphalerite (ZnS) using a salted-out alkaline leaching system composed of ammonia and ammonium sulphate, with particular focus on the kinetic mechanisms controlling dissolution. Conventional routes for zinc recovery, including pyrometallurgical and acidic hydrometallurgical processes, remain limited by their high energy requirements, elevated operating temperatures, and significant gaseous emissions. In contrast, ammonia–ammonium systems offer an environmentally benign alternative, enabling zinc solubilization through stable ammine complex formation while minimizing the dissolution of impurity metals such as iron and lead. Sphalerite ore sourced from Zurak, Plateau State, Nigeria, was subjected to detailed physicochemical and mineralogical characterization using XRF, XRD, SEM–EDS, and AAS techniques. The sample showed a moderate loss on ignition (4.5%) and a near-neutral pH (7.5), indicating minimal volatile or carbonate content. Elemental composition revealed 5.88 wt% Zn and 16.51 wt% Fe, whereas SEM–EDS micrographs displayed a rough, porous morphology conducive to enhanced leaching. Batch leaching studies were conducted to examine the influence of reagent concentration, temperature (25–80 °C), stirring speed (200–500 rpm), and pulp density (5–20 g) on zinc extraction. Maximum dissolution of approximately 70% was achieved at 3.0 M NH₃–(NH₄)₂SO₄, 80 °C, 400 rpm, and 180 minutes. The dissolution rate increased with temperature and reagent concentration but declined at higher pulp densities. Kinetic interpretation using the shrinking-core model revealed that the reaction was governed by diffusion through a product layer, with an apparent activation energy of 2.445 J mol⁻¹ and an overall reaction order of nearly second order. These results highlight the ammonia–ammonium sulphate medium as an efficient, selective, and environmentally sustainable leaching system for zinc recovery from sphalerite under mild process conditions, offering valuable insight for industrial-scale hydrometallurgical applications.

Keywords: Zinc extraction, Sphalerite, Salted-out alkaline leaching, Ammonia–ammonium sulphate system, Dissolution kinetics, Shrinking-core model

Introduction

Zinc is an essential industrial metal utilized extensively in galvanization, die-casting alloys, battery manufacture, and various chemical applications. The increasing global demand for this metal underscores the need for efficient and environmentally sustainable extraction strategies. Among the primary zinc minerals, sphalerite (ZnS) represents the most significant natural source. However, conventional recovery routes—such as pyrometallurgical smelting and acid-based hydrometallurgical leaching are often constrained by high energy requirements, the release of sulfur dioxide emissions, and the co-dissolution of unwanted impurities including iron and lead (Binnemans *et al.*, 2020; Babu *et al.*, 2002). These drawbacks necessitate the exploration of alternative extraction systems that combine high selectivity with reduced environmental impact.

Alkaline leaching processes, particularly those employing ammonia and ammonium salts, have emerged as promising substitutes for acidic routes. The ammonia–ammonium system is especially advantageous because zinc forms stable ammine complexes that facilitate dissolution while limiting the solubilization of many impurity metals (Babu *et al.*, 2002; Shabalala *et al.*, 2024). Furthermore, the use of ammoniacal media minimizes corrosion problems, allows reagent regeneration, and produces fewer secondary effluents compared to acidic leachants. Despite these benefits, limited studies have systematically explored the kinetic behavior of zinc dissolution from sphalerite under salted-out alkaline environments, such as the ammonia–ammonium sulfate mixture.

This study therefore investigates the leaching kinetics of zinc from sphalerite using an ammonia–ammonium sulfate medium under salted-out alkaline conditions (Ghosh *et al.*, 2002). Previous studies have demonstrated that ammoniacal leaching provides an effective, selective, and environmentally benign route for zinc recovery from sphalerite and related sulfide ores (Ghosh *et al.*, 2002; Babu *et al.*, 2002; Baba *et al.*, 2019). The effects of operational variables including reagent concentration, temperature, agitation rate, pulp density, and leaching duration are quantitatively examined, as these parameters are known to significantly influence zinc dissolution and reaction rates in alkaline and ammoniacal systems (Aydogan *et al.*, 2005; Ghosh *et al.*, 2003; Muktar *et al.*, 2023). The resulting data are interpreted using the shrinking-core model to elucidate the rate-controlling mechanisms and determine the apparent activation energy, consistent with methodologies widely applied in hydrometallurgical kinetic studies (Lin *et al.*, 2023; Baba *et al.*, 2019). Insights gained from this work are expected to enhance understanding of alkaline leaching kinetics and contribute to the development of cleaner, more sustainable hydrometallurgical routes for zinc recovery (Ghosh *et al.*, 2002; Muktar *et al.*, 2023; Lin *et al.*, 2023).

Study Area

The sphalerite ore was sourced from the Zurak mining site in Wase Local Government Area, Plateau State, Nigeria. Wase district (9°5'43"North, 9°57'29"East), Wase Local Government Area of Plateau, Nigeria.

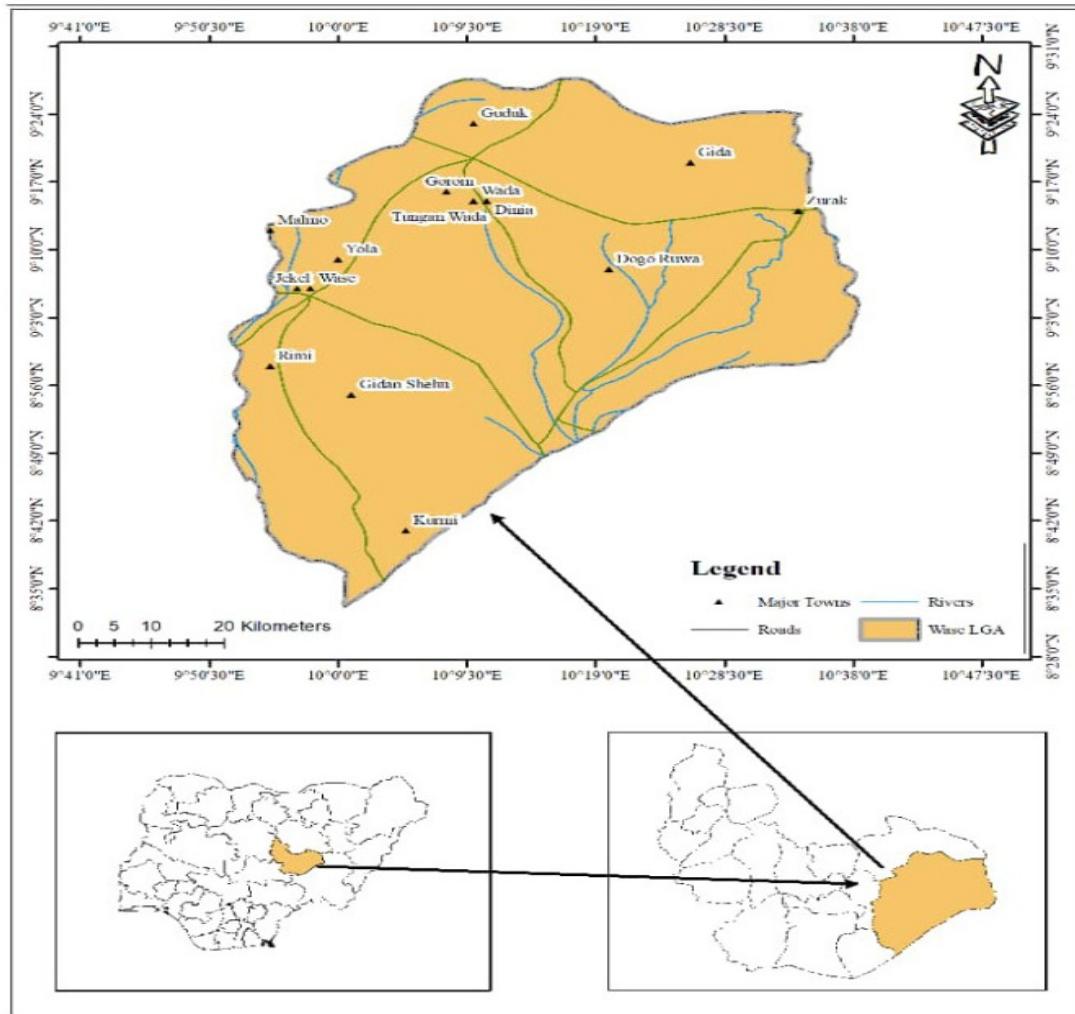


Fig. 1: Map of Wase showing the sampling location

Materials and Methods

Deionized water, $(\text{NH}_4)_2\text{SO}_4$, $\text{NH}_3\text{(aq)}$, Weighing balance, Muffle furnace(VF-14 High Temperature Muffle furnace, China), pH meter(Hannan EIE-108DG12,USA), Hot plate equip with magnetic stirrer, x-ray fluorescence (XRF) machine(X-MET800, England), x-ray diffractometer (XRD)(Powder Ultima Rigaku, Japan), scanning electron microscopy (FEI Nova Nano SEM 230), atomic absorption spectrophotometer (AAS) machine(ice 3000 AA 0213410V Thermoscientific, USA).

Experimental Procedure

Leaching experiments were conducted in 250 mL glass reactors equipped with magnetic stirring and temperature control. The effects of ammonia concentration (2–10 mol/L), ammonium sulphate concentration (0.5–3 mol/L), temperature (25–80°C), and solid-to-liquid ratio (1:10 to 1:50 g/mL) on zinc extraction efficiency were studied. Samples were withdrawn at regular intervals, filtered, and analyzed using atomic absorption spectroscopy (AAS) for zinc content. The experimental setup ensures consistent mixing and temperature control, which are

critical for accurate kinetic studies (Raji *et al.*, 2020; Baba *et al.*, 2019).

Results and Discussion

The essential outcomes of the study are summarized in the Tables and Figures below. The subsequent discussion interprets these results and emphasizes the significant implications derived from the findings.

Table 1: Physicochemical analysis of the ore sample

Parameter	Value
Loss of mass on ignition	4.5±0.005
pH	7.5

Physicochemical Properties

The sphalerite ore analyzed in this study exhibited a loss on ignition (LOI) of $4.5 \pm 0.005\%$ and a slurry pH of 7.5 (Table 1). These parameters provide an initial indication of the ore's volatile gangue content and buffering capacity, the factors that significantly influence both hydrometallurgical and biohydrometallurgical processing behavior.

Loss on Ignition (LOI): Interpretation and Implications

Loss on ignition is widely used as an indicator of volatile, thermally decomposable phases such as organic matter, carbonates, and hydroxyl minerals, which release CO_2 and H_2O upon heating

(Santisteban *et al.*, 2004). The observed LOI value of 4.5% suggests a moderate quantity of these components, implying that the ore is neither highly carbonate-dominated nor completely devoid of volatile matter. Consequently, (1) minimal material and energy losses are expected during thermal pretreatment, and (2) carbonate phases are likely to consume a measurable fraction of the acid during leaching operations. Similar interpretations have been reported in earlier studies (Santisteban *et al.*, 2004; Wang *et al.*, 2011).

pH Characteristics

The slurry pH of 7.5 reflects a near-neutral to slightly alkaline system under ambient conditions. This behavior is consistent with the presence of carbonate minerals (e.g., calcite and dolomite), which buffer the system by neutralizing free hydrogen ions. Practically, such buffering resists rapid acidification, implying a greater acid requirement to attain the acidic environment necessary for effective sulfide dissolution. Environmentally, the near-neutral pH indicates a low short-term potential for acid mine drainage. The pH value recorded in this study is lower than the 5.4 units reported by Baba *et al.* (2011) for Nigerian sphalerite ore, suggesting variations in mineral composition and carbonate content.

Table 2: Elemental Analysis by X-ray Fluorescence (XRF) of the ore sample

Element	Zn	CU	Fe	Al	Pb	Ti	V	Si
Compositions (wt.%)	5.877	0.363	16.510	2.342	0.842	0.037	0.000	3.162

Elemental Composition

The bulk elemental composition (wt.%) of the ore was determined as follows: Zn = 5.877, Cu = 0.363, Fe =

16.510, Al = 2.342, Pb = 0.842, Ti = 0.037, V = 0.000, and Si = 3.162. These values indicate a heterogeneous mixture of sulfide minerals, gangue, and trace

elements, with iron and zinc as the predominant metallic constituents - consistent with sphalerite fractions containing associated iron sulfides or oxides.

The relatively high Fe content (16.510 wt%) compared to Zn suggests that a portion of the iron may occur both as a substituent in the sphalerite lattice (Fe replacing Zn in $Zn_{1-x}Fe_xS$) and as discrete iron-bearing minerals such as pyrite, marcasite, or iron oxides. Iron substitution within sphalerite is well-documented, often reaching several weight percent in Fe-rich varieties (Kelley *et al.*, 2004). The composition observed in this study aligns with the findings of Wei *et al.* (2018), who reported $Zn \approx 5.62$ wt% and $Pb \approx 0.99$ wt% in sphalerite from the Nayongzhi Ore Field.

SEM-EDS Analysis

The scanning electron micrograph of the unprocessed sphalerite ore (Figure 2) reveals a compact, heterogeneous assembly of fine, irregularly shaped particles that are closely aggregated. The particle surfaces exhibit considerable roughness and porosity, indicating minimal weathering and limited crystallographic cleavage features commonly encountered in natural sphalerite prior to any beneficiation (Wang *et al.*, 2021). The abundance of fine grains implies a high specific surface area, which is advantageous in leaching

operations as it enhances contact between the mineral surfaces and leaching reagents (Liu *et al.*, 2020).

Energy dispersive spectroscopy (EDS) analysis linked to the SEM image confirms zinc (Zn) and sulfur (S) as the principal constituents, verifying the primary phase as sphalerite (ZnS). Secondary peaks for iron (Fe) and copper (Cu) suggest partial ionic substitution of Zn by Fe^{2+} and Cu^{2+} within the sphalerite lattice, a phenomenon that is well documented in natural specimens (Adebayo *et al.*, 2022). Minor signals corresponding to aluminum (Al), silicon (Si), and calcium (Ca) point to the presence of amounts of arsenic (As) and chromium (Cr) may stem from accessory sulfide phases (e.g., arsenopyrite, chalcopyrite) or reflect marginal contamination introduced during ore genesis (Chen *et al.*, 2020).

In summary, the SEM-EDS characterization verifies that the ore is predominantly zinc sulfide containing subordinate impurities. This composition supports its prospective suitability for hydrometallurgical or bioleaching strategies targeting zinc recovery. Furthermore, the fine, porous morphology of the particles suggests a potentially high reactivity and favorable dissolution kinetics during leaching - consistent with trends reported in similar studies (Das and Anand, 2020; Bala *et al.*, 2023).

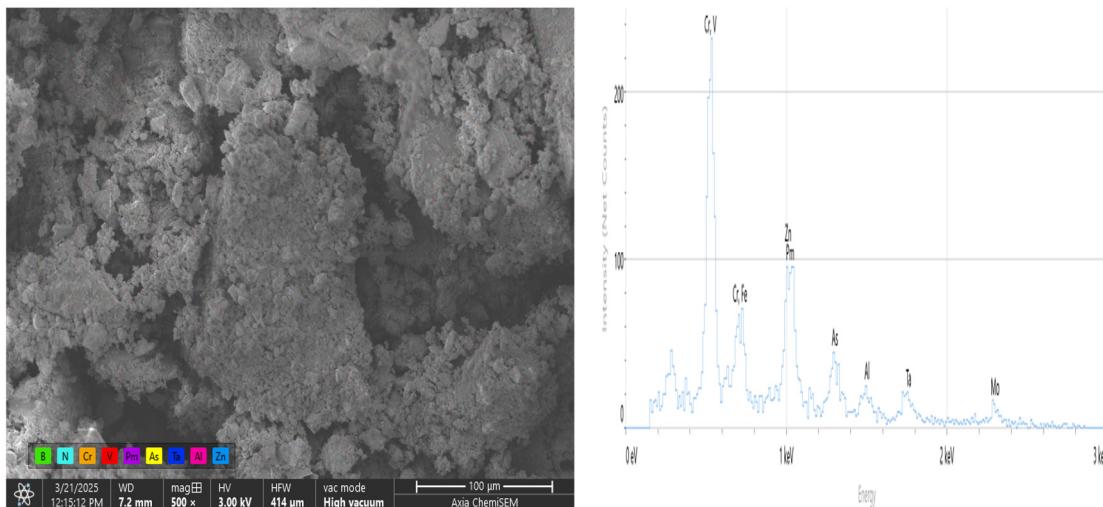


Fig 2: Micrographs of Raw Sphalerite ore

Effect of NH_3 – $(\text{NH}_4)_2\text{SO}_4$ Concentration on Zinc Dissolution

Figure 1 illustrates the influence of reagent concentration (0.5–3.0 M NH_3 – $(\text{NH}_4)_2\text{SO}_4$) on the dissolution of zinc from sphalerite at 50 °C, 400 rpm, and a pulp load of 5 g. The results reveal a progressive enhancement in dissolution rate and extent with increasing reagent concentration. After 180 minutes, the fraction of zinc dissolved rose from approximately 0.30 at 0.5 M to about 0.48 at 3.0 M. The 3.0 M system exhibited the steepest initial slope, achieving ~0.26 dissolution within the first 30 minutes, while the 0.5 M system attained only ~0.13 during the same period.

This behavior suggests that higher ammonia–ammonium sulfate concentrations promote zinc solubilization by enhancing complexation equilibria and increasing the effective activity of $\text{NH}_3/\text{NH}_4^+$ ligands. In comparison, Aydoğan et al. (2005) investigated ammonia leaching of sphalerite concentrates across 1.05–5.20 M NH_3 and observed approximately 45% zinc extraction after 4 h at 3.0 M NH_3 and 90–130 °C. Under the milder conditions of the present study (50 °C, 3 h), a similar or slightly higher extraction (~48%) was achieved, underscoring the efficiency of the NH_3 – $(\text{NH}_4)_2\text{SO}_4$ reagent system at relatively low temperatures.

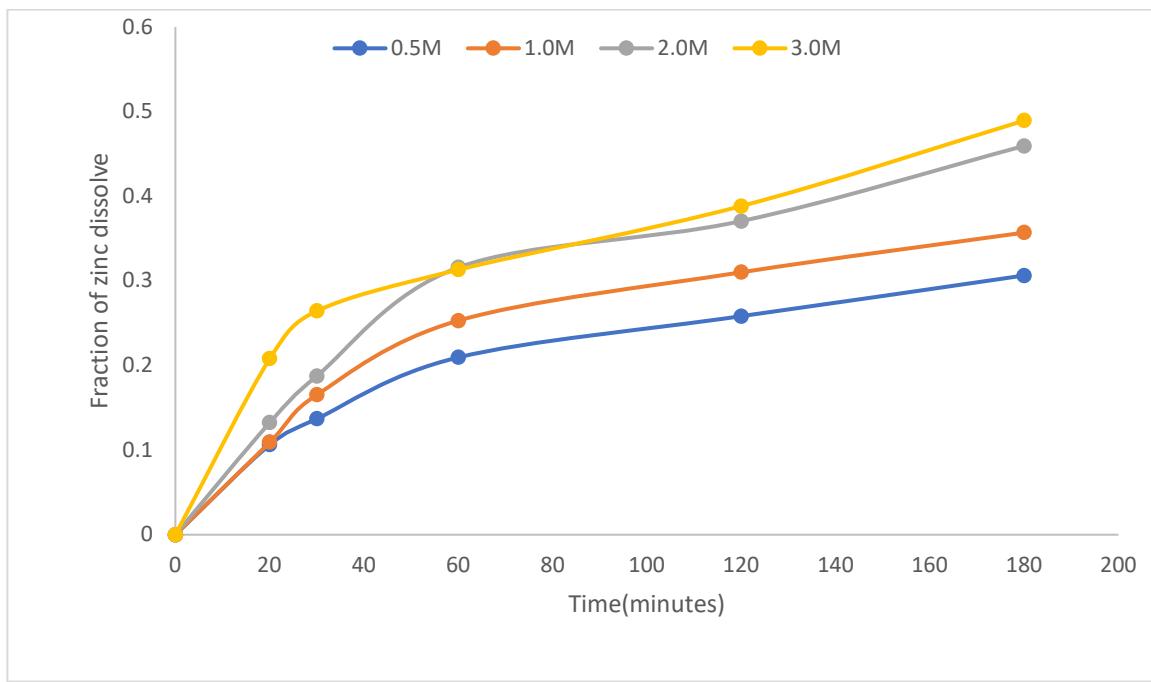


Fig. 3: Effect of Concentration of $\text{NH}_3\text{-}(\text{NH}_4)_2\text{SO}_4$ on the dissolution of sphalerite ore at different leaching time. Conditions: Concentration (0.5-3.0M), pulp density 5g, Temperature 50°C, stirring speed 400rpm, particle size 45 μm .

Effect of Pulp Density on Zinc Dissolution

The influence of pulp density (5–20 g) on zinc dissolution at 3.0 M $\text{NH}_3\text{-}(\text{NH}_4)_2\text{SO}_4$, 50°C, and 400 rpm is presented in Figure 2. An inverse correlation between pulp density and dissolution efficiency was evident. At 5 g, the fraction of zinc dissolved reached ~0.50 after 180 minutes, while at 20 g it decreased to ~0.30. Intermediate densities of 10 g and 15 g yielded extractions of ~0.46 and ~0.36, respectively.

This reduction at higher pulp densities can be attributed to a lower reagent-to-solid ratio, which limits the availability of

complexing species ($\text{NH}_3/\text{NH}_4^+$) and impedes mass transfer across the solid–liquid interface. In contrast, lower pulp densities enhance reagent accessibility and promote more efficient surface reactions. Similar patterns have been reported by Adebayo *et al.* (2020), who observed a decline in zinc recovery from 72.4% to 48.6% as pulp density increased from 2% to 10% (w/v) during bioleaching. Havlík *et al.* (2005) likewise noted a decrease in ammoniacal leaching efficiency from 55% to 34% when pulp density was raised from 5% to 20% at 90 °C.

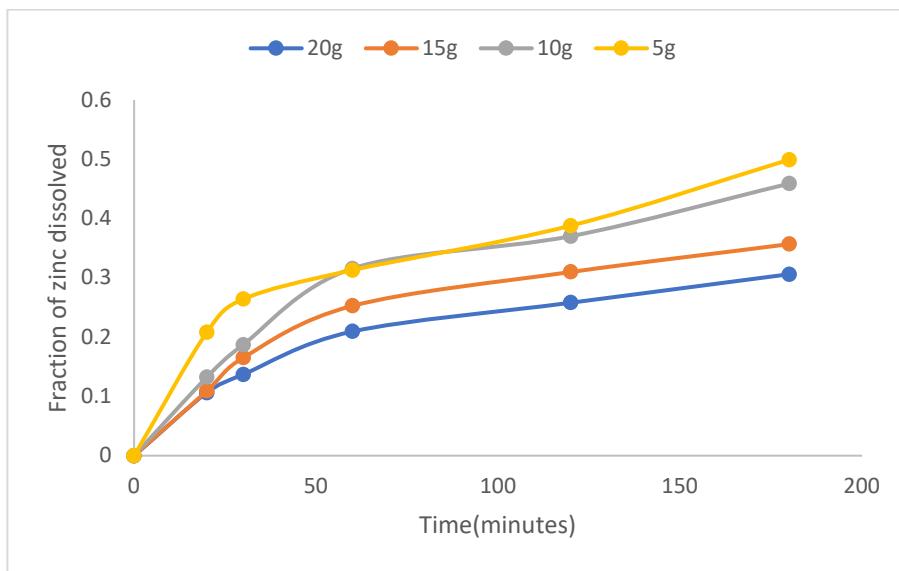


Fig. 4: Effect of pulp density on the dissolution of sphalerite ore at different leaching time. Conditions: pulp density (size 5-20g), Concentration 3.0M, Temperature 50°C

Effect of Stirring Speed on Zinc Dissolution

The impact of agitation speed (200–500 rpm) on sphalerite dissolution in 3.0 M $\text{NH}_3\text{--}(\text{NH}_4)_2\text{SO}_4$ at 50 °C is depicted in Figure 3. At 200 rpm, zinc dissolution was limited (~0.30 after 180 min), while 300 rpm provided moderate improvement (~0.36). The optimum dissolution (~0.60) was achieved at 400 rpm, but a further increase to 500 rpm caused a decline (~0.47).

This indicates that moderate agitation enhances dissolution by improving solid–

liquid contact, minimizing the boundary layer, and facilitating mass transport. Excessive agitation, however, can generate turbulence that promotes fine-particle entrainment or secondary phase re-precipitation, reducing overall recovery. These observations are consistent with Havlík *et al.* (2005), who found optimum leaching at 450 rpm, and with Adebayo and Olanipekun (2019), who reported maximal zinc recovery (65%) during bioleaching at 350 rpm, with higher speeds leading to reduced bacterial activity due to shear stress.

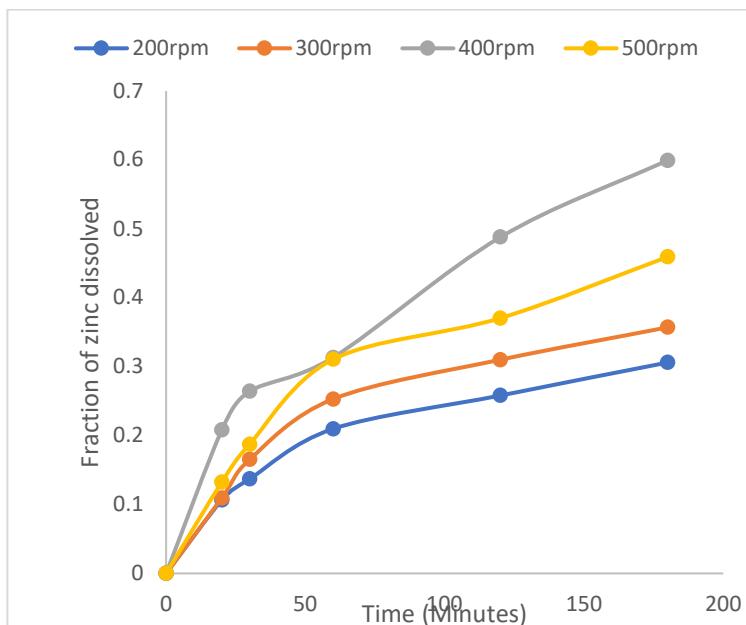


Fig. 5: Effect of stirring speed on the dissolution of sphalerite ore at different leaching time. Conditions: Stirring speed 200 - 500rpm, temperature 50 °C, pulp density 5g, Concentration 3.0M, particle size 45 μm .

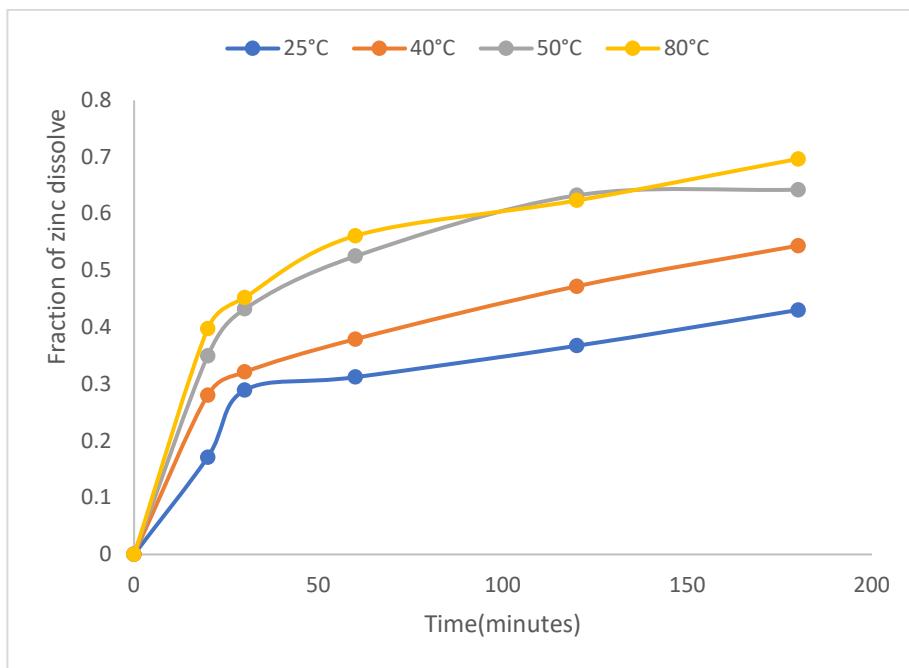


Fig. 6: Effect of temperature on the dissolution of sphalerite ore at different leaching time. Conditions: Temperature 25°C -80 °C, pulp density 5g, Concentration 3.0M , stirring speed 400rpm , particle size 45 μm .

Effect of Temperature on Zinc Dissolution

The influence of temperature on zinc dissolution is presented in Figure 4. The fraction of zinc dissolved increased markedly with temperature and time, approaching steady-state values after approximately 120 minutes. The fractions dissolved were ~0.43 at 25 °C, 0.53 at 40 °C, 0.64 at 50 °C, and 0.70 at 80 °C. This trend conforms to the Arrhenius principle, where elevated temperature enhances reaction kinetics in heterogeneous solid-liquid systems (Aydogan, 2005).

The dissolution process exhibited two distinct stages: an initial rapid phase

within the first 30 minutes, corresponding to surface chemical reactions at active sites, followed by a slower, diffusion-controlled stage likely governed by the formation of a passivating sulfur-rich or iron-hydroxide layer. Such two-stage kinetic behavior is consistent with the findings of Picazo-Rodríguez *et al.* (2020), who observed similar patterns during the direct acid leaching of sphalerite.

Dissolution Kinetics Studies

The dissolution rates of zinc from the zinc-rich fly ash was determined by subjecting the leaching results to three shrinking core model.

$$1 - (1 - \alpha)^{1/3} = Kct \text{ (Chemical reaction controlled)} \quad (1)$$

$$1 - \frac{2}{3}\alpha - (1 - \alpha)^{\frac{2}{3}} = Kpt \text{ (Diffusion controlled process)} \quad (2)$$

$$1 - (1 - \alpha)^{\frac{2}{3}} + \frac{y}{6}[(1 - \alpha)^{1/3} + 1 - 2(1 - \alpha)^{2/3}] = Kmt \text{ (Mixed controlled process)} \quad (3)$$

With diffusion rate constant $K_p = \frac{6MDC_0}{b\rho_{p0}^2}$ and $K_c = \frac{KC_0M}{r_2\rho_b}$, where α is the fraction of the ore that reacted (%), t is the time (minutes) (Baba and Adekola, 2019).

According to rate law expression:

$$\text{Rate} = k \ln[\text{NH}_3\text{-}(\text{NH}_4)_2\text{SO}_4]^n \quad 4$$

Plot of fraction of Zn leached (α) at various concentrations against time has slopes k_1 .

Plot of $\ln k_1$ against $\ln[\text{NH}_3\text{-}(\text{NH}_4)_2\text{SO}_4]$ gives the slope as the order of the reaction.

Plot of fraction of Zn leached (α) at various temperatures against time has slopes k_2 .

Also, the temperature dependence of the specific rate constant can be estimated from the Arrhenius expression:

$$k = Ae^{-Ea/RT} \quad 5$$

Hence, equation 5 can be linearized to obtain equation 6

$$\ln k = \ln A + \left(\frac{-Ea}{R}\right) \frac{1}{T} \quad 6$$

Where k is the rate constant, A is the pre-exponential factor, Ea (kJ/mol) is the activation energy, R is the universal gas constant 8.314×10^{-3} kJ/molK⁻¹ and T is the temperature in Kelvin (Baba *et al.*, 2019).

Plot of rate constant ($\ln k_2$) against the inverse of temperature (T^{-1}) will give the activation energy of the dissolution reaction.

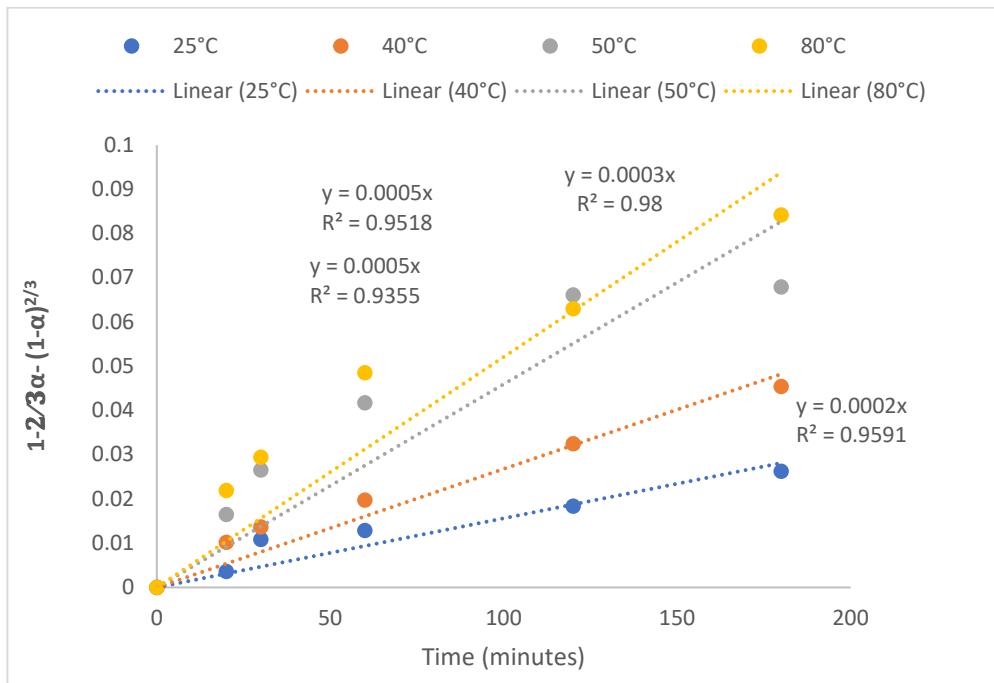


Fig. 7: Plot of $1 - \frac{2}{3}\alpha - (1-\alpha)^{2/3} = k_m t$ for temperature of the salted out alkaline leaching. The experimental rate constant, K was estimated from the slopes of the straight lines of Fig.8, and the results subjected to Arrhenius equations,

$$K = A e^{-Ea/RT} \quad (5)$$

Where A is a constant related to the geometry needed, K is the rate constant, R is the gas constant ($8.314 \text{ Jmol}^{-1}\text{K}^{-1}$), T is temperature in Kelvin (K)

$$\ln K = \frac{-Ea}{R} \left(\frac{1}{T} \right) + \ln A \quad (6)$$

When $\ln K$ is plotted versus the inverse of the temperature ($1/T$) as shown in Figure 8, the value of the slope (m) is equal to $\frac{-Ea}{R}$. Where R is a constant ($8.314 \text{ Jmol}^{-1}\text{K}^{-1}$) (Muktar *et al.*, 2023).

The activation energy (Ea) was estimated to be **2.4455 Jmol⁻¹**

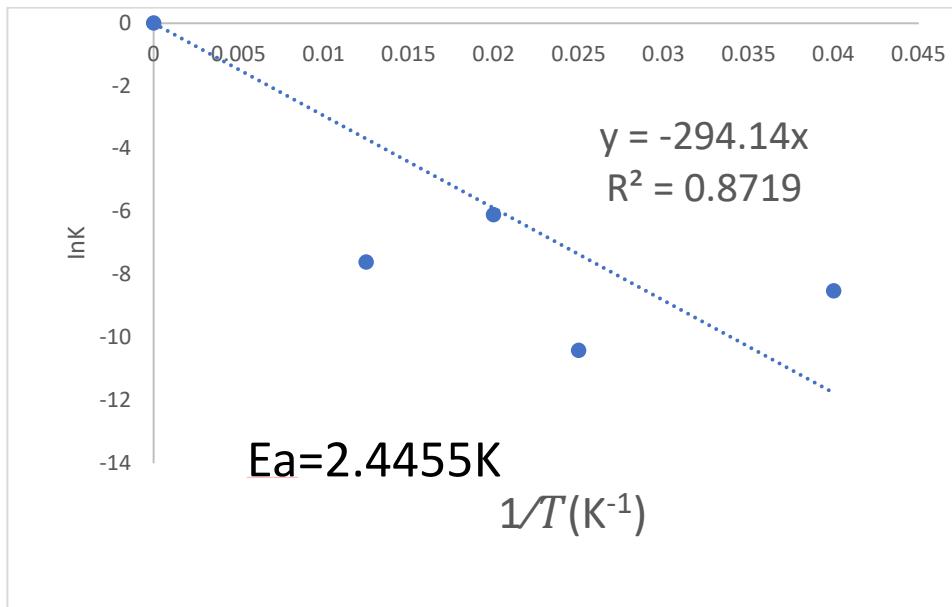


Fig. 8: plot of $\ln K$ against $1/T$

Analysis of the dissolution data reveals that the shrinking core model accurately describes the process, suggesting that the dissolution kinetics are governed by a diffusion control mechanism. Similarly, the result of the fitting test concerning the initial concentration showed that the data fitted perfectly into equation (3), as shown in Fig. 10

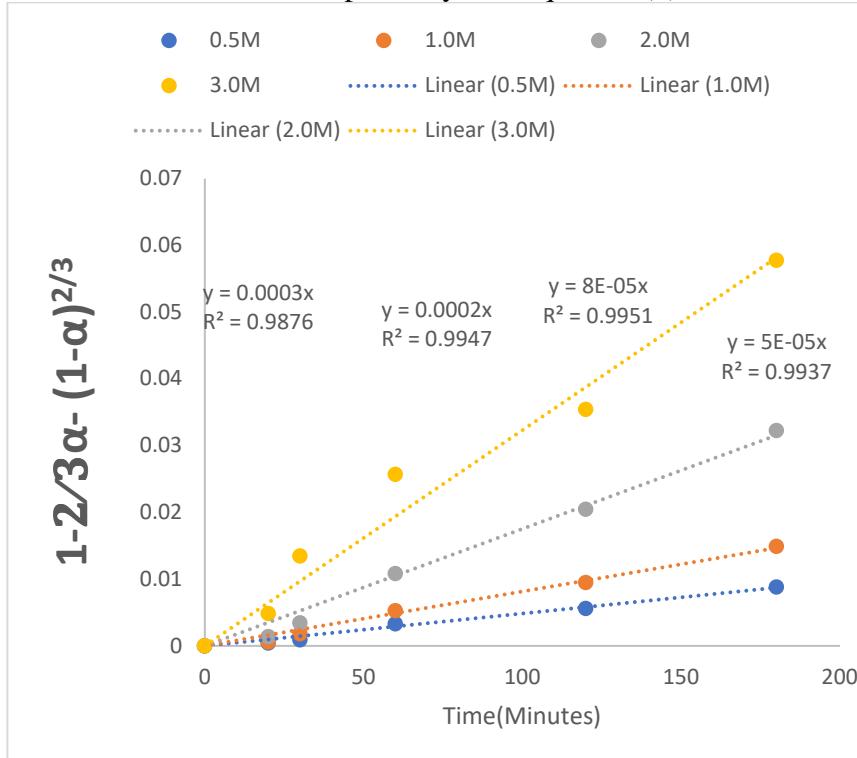


Fig. 9: Plot of $1 - \frac{2}{3}\alpha - (1 - \alpha)^{\frac{2}{3}} = k_m t$ for concentration of the salted out alkaline

The experimental rate constant k_2 for zinc dissolution by was calculated from Figure 9 using the slope of the straight line at different concentration (Chindo *et al.*, 2022).

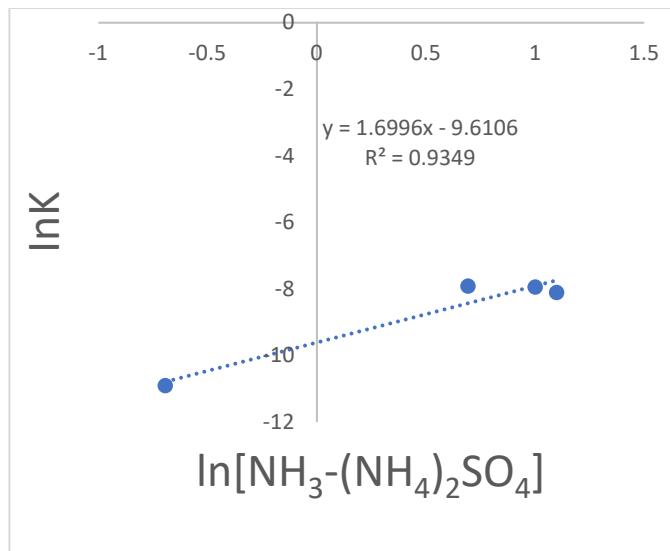


Fig. 10: plot of $\ln k_2$ against $\ln[\text{NH}_3\text{-(NH}_4)_2\text{SO}_4]$

The plot of $\ln k_2$ against $\ln[\text{NH}_3\text{-(NH}_4)_2\text{SO}_4]$ is illustrated in Figure 10, with a slope of 1.6996. This shows that the order of reaction for zinc bioleaching with respect to H^+ ion concentration is 1 with correlation coefficient of 0.9349. This deduction agreed with those reported by Muktar *et al.*, 2023.

Conclusion

Zinc recovery from sphalerite was successfully achieved using a salted-out alkaline leaching system comprising ammonia and ammonium sulphate. The process yielded about 70% zinc extraction under mild conditions (3.0 M reagent, 80 °C, 400 rpm, 180 min). Kinetic analysis based on the shrinking-core model indicated a diffusion-controlled mechanism with an activation energy of 2.445 J mol^{-1} and a reaction order of ~ 2 . The system demonstrated comparable or superior performance to conventional methods while reducing environmental impact. Overall, the ammonia–ammonium

sulphate medium offers a selective, efficient, and sustainable pathway for zinc recovery, with promising potential for industrial scale-up.

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