A baseline study of the ammonia leaching of a sphalerite concentrate

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Finding an economical and ecologically appropriate way to extract Zn from typical industrial zinc sulfide concentrates rich in zinc resources is a significant issue. In this paper, the efficacy of $(NH_4)_2CO_3$, $(NH_4)_2SO_4$, and NH_4Cl as leaching salts in the context of ammonia leaching was investigated, along with the catalytic effects of Cu(II) ions in the Zn extraction process from sphalerite concentrate. Later, studies were carried out to examine the impact of different leaching temperatures, total ammonia concentrations, and pulp density on Zn extraction. According to the research, NH_4Cl was a very effective salt in an oxygen medium. It facilitated Zn extraction effectiveness of up to 91.6% Zn extraction in oxygen within 72 hours. The oxygen medium was more effective than the compressed air medium for zinc extraction. A total ammonium concentration of 6 M, an NH_3/NH_4^+ ratio of 1:1, a pH range of 9-11, a slurry of 2% w/v, a temperature of 238.15 K(55°C), a leaching time of 72 hours, and a stirring speed of 500 rpm were the specific conditions for observing these results. The following parameters were found to be optimal for leaching to extract up to 99.5% Zn: 55°C leaching temperature, 72 hours of leaching, 500 rpm stirring speed, 2% w/v pulp density, 6 M total ammonia concentration (3 M NH_3 and 3 M NH_4Cl), 0.25 g/L Cu(II), pH levels maintained between 9 and 11, and the use of an oxygen-enriched environment. The findings of this study indicated that ammonia leaching facilitated the extraction of Zn from sphalerite concentrate through a surface chemical reaction model and the activation energy needed to dissolve was calculated to be 49.5 kJ/mol.

Keywords: Zinc sulfide, ammonia leaching, zinc extraction, ammonium chloride, Cu(II) ions.

INTRODUCTION

The most abundant source of zinc is sphalerite, which is easily concentrated into a high-grade concentrate by froth flotation. Sphalerite typically contains varying impurities (Fe, Cd, Mn, Cu, Co, Ni, Ge, and In), which replace zinc in the crystal structure (McClung & Viljoen, 2011). They are separated into different concentrations using selective flotation techniques to distinguish between them (Aydogan *et al*., 2005). This concentrate is easily transportable and can be treated in various smelters using the traditional roast-leach-electrowinning procedure (Ghosh *et al*., 2002). This procedure involves roasting the concentrate to produce zinc oxide, leaching the resulting calcine with diluted sulfuric acid, and electrowinning zinc from the purified leachate (Aydogan *et al.*, 2005). Before zinc can be recovered, iron must be precipitated out because iron causes electrochemical short circuits that obstruct zinc's electrolytic deposition (Ismael & Carvalho, 2003). The sphalerite produces SO_2 as a byproduct during the roasting process (Aydogan *et al*., 2005; Ghosh *et al*., 2002). These difficulties have spurred research and development of hydrometallurgical and other alternative metal extraction procedures.

Direct leaching of sphalerite concentrates has drawn a lot of attention recently, even though conventional methods have been used for zinc extraction for a long time (Xu *et al*., 2013).

Since the roasting stage is omitted in the direct leaching process, elemental sulphur rather than sulphur dioxide is produced. This method makes it possible to successfully extract zinc without being impacted by the concentration of iron in the zinc concentrates (de Souza *et al*., 2007). In general, stirred tanks that run at atmospheric pressure or pressure-leaching autoclaves can be used for direct leaching procedures (Buban *et al.,* 2000). The direct dissolution of sphalerite has been the subject of numerous investigations with a variety of reagents, such as ammonia (Ghosh *et al*., 2002), nitric acid (Çopur, 2001), hydrochloric acid (Mizoguchi & Habashi, 1981), acidified solutions loaded with ferric ions (Bobeck & Su, 1985), and aqueous sulfuric acid (Forward & Veltman, 1959). Ammonia has long been known to be a useful solvent in various hydrometallurgical processes and is chosen over other agents due to its unique advantages. Its main advantage is that it may be used to establish a simple leaching environment, which greatly lessens the corrosion problems that are frequently encountered in acidic environments. Through the leaching phase, using an ammonia-based leaching solution also minimises the presence of unwanted elements like iron by transforming them into insoluble oxy/hydroxide forms. As a result, ammonia's reactive nitrogen groups allow precious metals like copper, cobalt, nickel, and zinc to form soluble complexes with it, which frequently leads to increased solubility. This makes the recovery of these metals easier to achieve (Park *et al*., 2007).

Ammonia leaching requires the presence of an oxidising agent for the conversion of sulfide minerals. Various oxidants, such as bromates, chlorates, molecular oxygen, peroxides, and persulfates, have been used in the extraction process of copper sulfides. Among them, molecular oxygen, which may be delivered by compressed air, is the most cost-effective option and has thus been widely used in the solubilisation of base metal sulfides (Bell *et al*., 1995). Sulfide oxidation processes in ammonia solutions usually occur slowly when air or O_2 is present and the temperature is below 323.15 K (50°C) (Ghosh *et al*., 2002). Zinc has been extracted from sphalerite by the ammonia leaching method in several investigations, which have also examined the kinetics of leaching, created kinetic models, and calculated the activation energy (Babu *et al*., 2002; Ghosh *et al*., 2002). Moreover, it has been found that adding ammonium ions can improve the zinc extraction process effectiveness (Ma *et al*., 2018). The investigation by Ghosh *et al.* (2003) showed that the redox pair Cu(II)/Cu(I) was related to the catalytic function of Cu(II). This process involves the oxidation of sphalerite by cupric ammine [$Cu(NH_3)_4^{2+}$], followed by the oxidation of cuprous ammine [$Cu(NH_3)_2^{+}$] by dissolved oxygen. Ghosh *et al.* (1989) reported on the effects of various metal ions and minerals on ammonia leaching of ZnS. They discovered that Cu(I), Cu(II), Ag(I), and Pb(II) all functioned as catalysts, speeding up the process in the following order: Cu(I) ~Cu(II) > Ag(I) >Pb(II). The study also suggested several chemical processes that take place when these metal ions are present and ZnS is exchanged and oxidised (Ghosh *et al*., 1989).

It is possible to explain the leaching process using the shrinking core model. The chemical reaction occurring at the particle's surface or diffusion of an inert particle through its pores can affect the rate at which this reaction develops (Ju *et al*., 2005). The following formula can be used to apply the shrinking core model and characterise the kinetics of the leaching process when the chemical reaction on the surface is the only step that determines the rate (Li *et al*., 2021):

$$[1 - (1 - \alpha)^{1/3}] = k_r t \qquad [1]$$

where α is the fraction of Zn that has been reacted, t is time, and k_r is the reaction rate constant for surface chemical reaction. Similarly, the following expression characterising the kinetics of dissolution is obtained from the shrinking core model if the rate-determining phase is the diffusion of the reagent through a layer of product (Li *et al*., 2021):

$$[1 - 2/3\alpha - (1 - \alpha)^{2/3}] = k_d t \qquad [2]$$

where k_d is the apparent rate constant for diffusion in nonporous particles. Plotting the right-hand sides of Equations 1 and 2 vs. time (t) was expected to produce a linear relationship that would reveal the mechanism at work. The rate constant (k_r or k_d) is represented by the slope of this line. The Arrhenius equation can also be used to find the rate constant's temperature dependency (Ma *et al*., 2018).

$$k = Ae^{-Ea/RT} \qquad [3]$$

This formula has k for the rate constant, A for the frequency factor, E_a for the activation energy of the reaction, R for the universal gas constant, and T for the absolute temperature (Li *et al.*, 2021)

In this study, we looked at employing an ammonia leaching system with several ammonium salts ($(NH_4)_2CO_3$, $(NH_4)_2SO_4$, NH_4Cl) to extract zinc from sphalerite. Using the most effective ammonium salt, we also investigated the effects on zinc extraction efficiency in compressed air and oxygen, varying concentrations of total ammonia, pulp density, leaching temperatures, and Cu(II) ion catalytic effect. The well-known shrinking core model well explains the relationship between the amount of zinc leached and the reaction time. It also makes it possible to calculate the process's apparent activation energy.

Material and methods

Materials

Table I displays the results of measuring the particle size distribution of the concentrate using a Malvern Mastersizer 3000.

Table I. Analysis of the particle size distribution in the concentrate sample

Particle size (µm)	4.11	18.2	58.0
Passing (%)	10	50	90

The UCT Analytical Laboratory conducted inductively coupled plasma-optical emission spectometry (ICP-OES) analyses and digestions, and the UCT Catalysis Research Group carried out X-ray fluorescence and X-ray diffraction (XRD) investigations. Tables II and III present the concentrate's chemical composition and mineralogical composition, respectively.

Table II. Mineral composition of zinc concentrate

Mineral	Sphalerite	Galena	Quartz	Mica	Clinochlore	Plagioclase
Content (wt.%)	81.2	3.2	9.3	2	1.7	2.6

Table III. Chemical composition of sphalerite concentrate

Element	Fe	Mn	Cu	Pb	S	Zn	Al	Si	Mg	Na
Content (wt.%)	8.3	2.6	0.17	2.7	27.8	43.7	0.5	5.8	0.5	0.1

The XRD pattern of the concentrate examined in this work is shown in Figure 1, which indicates that sphalerite, galena, quartz, mica, clinochlore, and plagioclase are the main constituents of the sample.

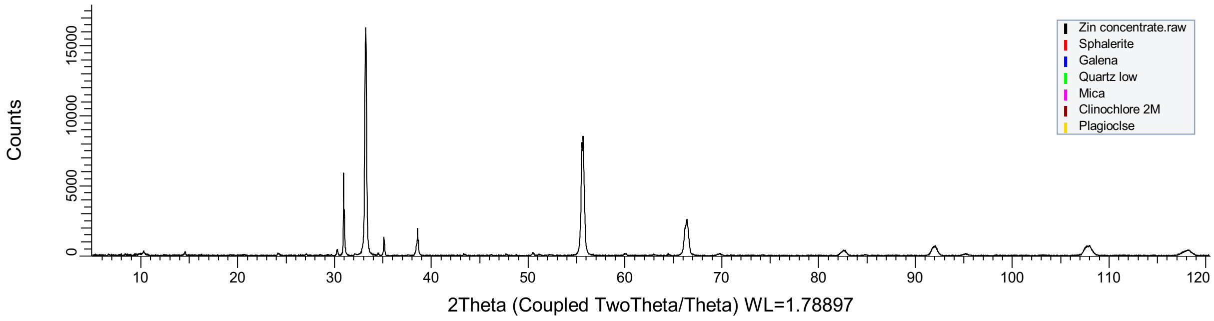

Figure 1. X-ray diffraction analysis of sphalerite concentrate.

Methods

A 2L jacketed reaction vessel from GlassChem, which was fitted with a magnetically connected stirrer for sealed stirring, was used for the leaching tests (see Figure 2). A hot oil circulator with PID control was used to control temperature in the vessel. The first step in every experiment was to fill the reactor with 1000 ml of a particular molarity aqueous ammonia solution and ammonium salt (carbonate, sulphate, or chloride). The mass of sphalerite added was varied based on the required solid/liquid ratio. After sealing, the reactor was gradually stirred while being heated to the desired temperature. The official timing of the experiment began when the temperature reached the set point, at which point oxygen or compressed air was sparged into the solution in the reactor and full agitation was applied. Cu(II) ions catalytic effect, temperature, pulp density, and total ammonia concentration were among the factors that were looked at during the leaching process. After some time, 10 mL of the pregnant leach solution was drawn out, and pH and redox potential were measured and sent to the UCT Analytical Laboratory for ICP-OES measurement of the zinc concentration.

Figure 2. The GlassChem reactor configuration for zinc leaching from sphalerite concentrates using ammonia.

To keep the reactor's volume constant, 10 mL of fresh ammonia-ammonium salt solution was supplied in equal measure. Following that, Equation (4) was used to calculate the zinc recovery rate (Zn%):

$$Zn\% = (C_{zn}V/mw) \times 100\% \qquad [4]$$

$Zn\%$ is the percentage value that indicates the rate of metal ion leaching, C_{Zn} represents the concentration of metal ions in the leaching solution (mg/L), V is the leaching solution volume (L), m is the sample mass (mg), and w is the sample percentage of metal content.

The standard parameters for the experiment were as follows: 500 rpm stirrer speed, 2% slurry density, temperature of 55°C, 6.0 M ammonia concentration, 1:1 NH_3/NH_4^+ ratio, 72 hours of leaching time and oxygenated medium.

RESULTS AND DISCUSSION

Zinc extraction with different ammonium salts

This research investigated the effects of different ammonium salts on the efficiency of zinc extraction under the following conditions: 6 M ammonia total concentration (3 M NH_3 combined with either 3 M NH_4Cl, 1.5 M $(NH_4)_2SO_4$, or 1.5 M $(NH_4)_2CO_3$), 1:1 NH_3/NH_4^+ ratio, temperature of 55°C setting, oxygenated medium, 72-hour reaction time, 500 rpm stirring speed, 2% w/v slurry, pH maintained between 9 and 11, and an oxygen-rich environment.

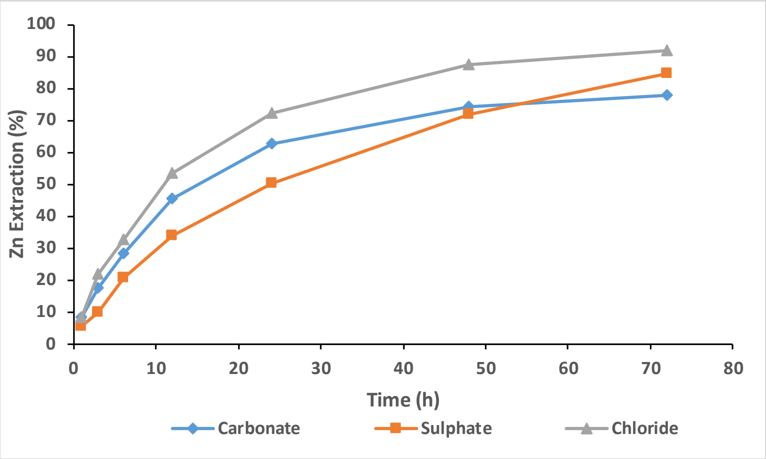

Figure 3. The comparative effectiveness of different ammonium salts in extracting zinc from sphalerite concentrate.

It can be observed from Figure 3 that the extraction of zinc from sphalerite concentrate is impacted by different ammonia-based leaching processes. Zinc extraction was noticeably higher when NH_3 and various ammonium salts were combined. The order of effectiveness was $NH_4Cl > (NH_4)_2CO_3 > (NH_4)_2SO_4$. The extraction rates of the different ammonium salt systems were comparatively similar despite these variations. The NH_3-NH_4Cl system had the greatest Zn extraction after 72 hours at 91.6%. The ability of zinc to form a stable complex with the chloride ion and a coordination complex with NH_3 is principally responsible for its improved performance, as it facilitates the leaching process. Consequently, the NH_3-NH_4Cl system was chosen as the best leaching system for the experiment.

The effect of ammonium salts on the pH

This research also investigated the effects of different ammonium salts on the pH under the following conditions: 6 M ammonia total concentration (3 M NH_3 combined with either 3 M NH_4Cl, 1.5 M $(NH_4)_2SO_4$, or 1.5 M $(NH_4)_2CO_3$), 1:1 NH_3/NH_4^+ ratio, temperature of 55°C setting, oxygenated medium, 72-hour reaction time, 500 rpm stirring speed, 2% w/v slurry, pH maintained between 9 and 11, and an oxygen-rich environment.

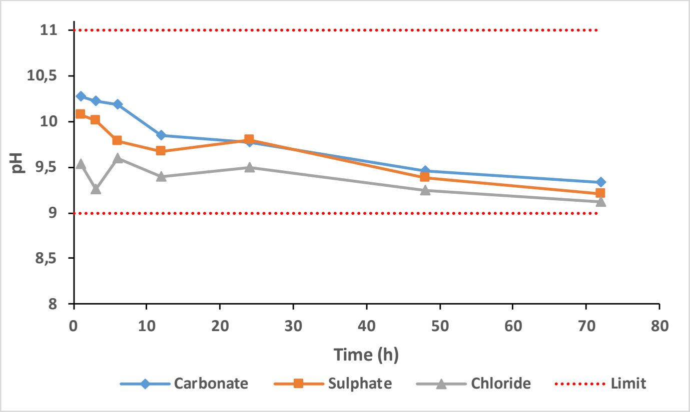

Figure 4. Effect of ammonium salts on pH levels for 72 hours during zinc extraction from sphalerite.

All the ammonia-ammonium salt systems in Figure 4 show a moderate fall in pH, although they all stayed within the pH range of 9–11. Ammonium carbonate is thought to have the strongest buffering ability of all of these. Muzawazi (2013) attributes this to the fact that carbonate (CO_3^{2-}) has a greater pKa value (10.33) than ammonium ions (NH_4^+). When compared to conjugate bases of strong acids like Cl^- (pKa = 2.86) and SO_4^{2-} (pKa = 1.92), which are less effective at buffering, the higher pKa indicates a stronger buffer activity. As a result, the pH of the carbonate system was increased. All the tested ammonium salts, however, were able to maintain a pH between 9 and 11.

The effect of oxidative medium on zinc extraction

The effects of different oxidative mediums on the efficiency of zinc extraction were investigated under the following conditions: 6 M ammonia total concentration (3 M NH_3 and 3 M NH_4Cl), 1:1 NH_3/NH_4^+ ratio, temperature of 55°C setting, 72-hour reaction time, 500 rpm stirring speed, 2% w/v slurry, and pH maintained between 9 and 11.

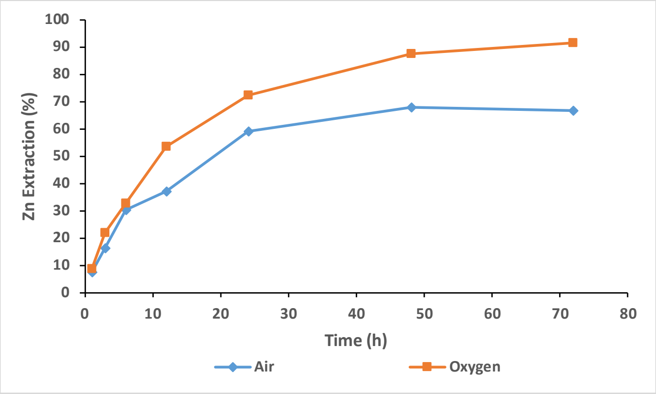

Figure 5. Comparing the effectiveness of oxygen and air in improving zinc recovery rates.

Due to its low cost and lack of corrosive properties, oxygen has been widely used as an oxidising agent to promote sulfide dissolving (Park *et al*., 2007). Since pure oxygen has a larger concentration of oxygen, it promotes zinc sulfide oxidation and facilitates better Zn extraction, while only around 21% of compressed air is oxygen, with the lower partial pressure limiting the gas-liquid mass transfer of oxygen unless operated under pressure leaching conditions. Additionally, the flow-through of nitrogen through the reactor facilitates the evaporation of ammonia from the leach liquor. Consequently, oxygen was chosen as the most effective oxidant for the experiment.

The effect of temperature on zinc extraction

The following leaching parameters were used to evaluate the impact of temperature fluctuations on zinc extraction rates: a total ammonia concentration of 6 M (3 M NH_3 and 3 M NH_4Cl), an NH_3/NH_4^+ ratio of 1:1, a stirring speed of 500 rpm 2% w/v slurry, a 72-hour leaching period, pH maintained between 9 and 11, and an oxygen-rich environment.

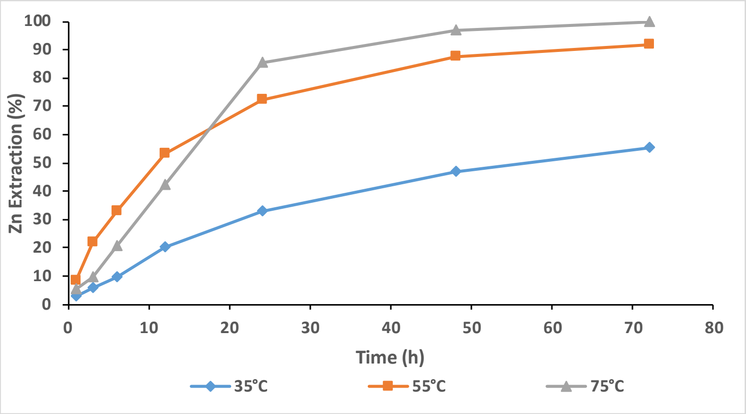

Figure 6. Influence of temperature on ammonia leaching of zinc from sphalerite.

Figure 6 shows how the temperature rose from 35°C to 75°C, gradually increasing the Zn extraction efficiency from 55.2% to 100%. This can be explained by the fact that molecules activate, collide, and diffuse more quickly at higher temperatures, leading to more efficient zinc leaching. But very high temperatures (such as 75°C) cause the ammonia in the NH_3-NH_4Cl leaching system to volatilise, which slows down the rate at which Zn leaching increases by reducing the formation of zinc-ammonia coordination ions. It is observed from the graph that the initial reaction rate at high temperatures (75°C) is slower. It is best to use a lower operating temperature to reduce reagent consumption because the rate of ammonia volatilisation increases with temperature. Consequently, 55°C was chosen as the leaching temperature for the experiment.

Kinetics study for temperature

The main objective of the study of kinetics was the development of a rate equation suitable for use in process modelling and reactor design. Sphalerite leaching was expected to be controlled by the surface reaction or diffusion through the product layer. Nevertheless, attempts to produce linear graphs using the model Equation (2) with the experimental data were unsuccessful.

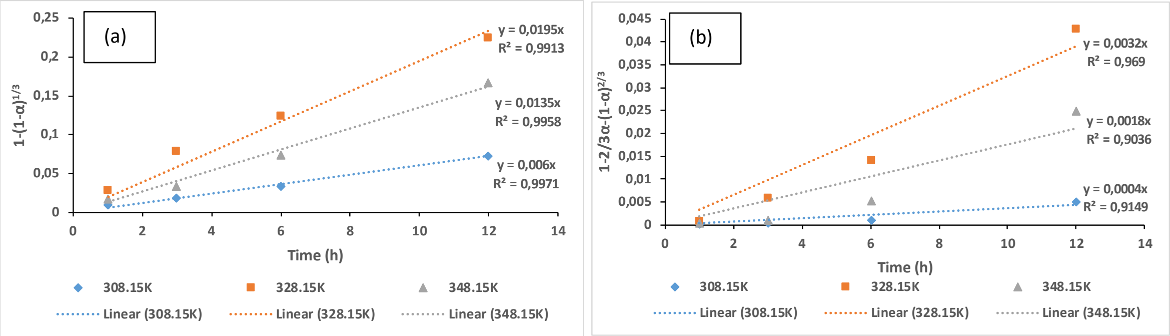

Figure 7. Graph illustrating zinc leaching data based on the shrinking core model, showcasing (a) surface chemical reaction and (b) reagent diffusion through the product layer at different temperatures.

There is a strong linear relationship ($R^2 > 0.99$ for all temperature variations) between $1-(1-\alpha)^{1/3}$ and leaching time, suggesting that the chemical reaction occurring at the particle's surface is the primary driver of the leaching process. Therefore, a chemical reaction model can accurately predict the leaching process kinetics.

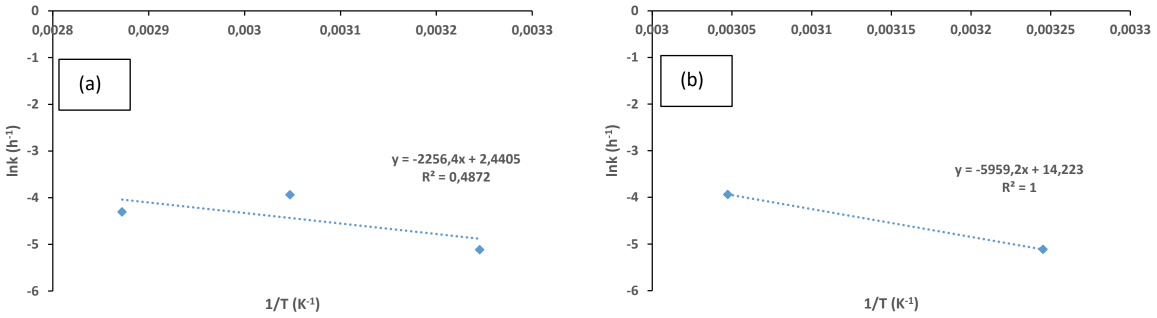

Figure 8. Graphs of Arrhenius for determining activation energy, (a) 75°C temperature included and (b) 75°C temperature excluded.

In Figure 8, the Arrhenius plot for the leaching process is created by plotting ln(k_r) against 1/T, which allows for the activation energy calculation. The initial rate at 75°C is slower due to ammonia evaporation, so this temperature is excluded from the activation energy calculation because the ln(k_r) versus 1/T relationship is not linear when this temperature is included (refer to Figure 8(a)). By using the data from the other two temperatures (35°C and 55°C), an apparent activation energy of approximately 49.5 kJ/mol was determined. The activation energy for a diffusion-controlled model ranges from 4 to 12 kJ/mol. In contrast, for a surface chemical control model, it typically exceeds 40 kJ/mol (Aydogan *et al.*, 2005). Consequently, the calculated activation energy indicates that the leaching process is indeed governed by a surface chemical reaction model in this temperature range.

The effect of total ammonia concentration on zinc extraction

These leaching parameters were used to evaluate the effects of variations in total ammonia concentration on zinc extraction rates: a 1:1 NH_3/NH_4^+ ratio, a temperature of 55°C, a stirring speed of 500 rpm, a slurry concentration of 2% w/v, a leaching duration of 72 hours, pH levels maintained between 9 and 11, and an oxygen-rich environment.

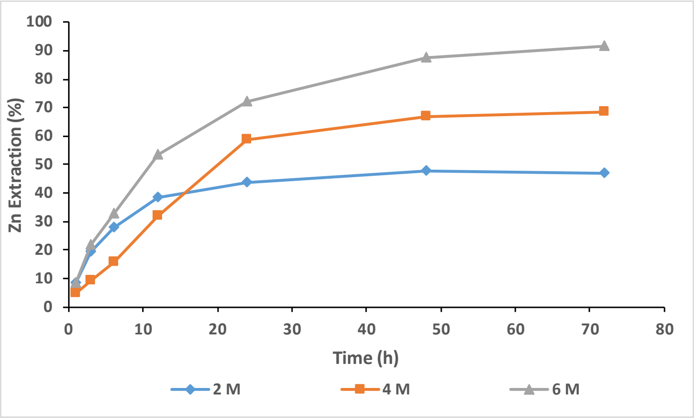

Figure 9. Effects of changing the ammonia concentration total on zinc extraction from sphalerite concentrate.

In the data presented in Figure 9, it was observed that with increasing total ammonia concentrations, the extracted amount of zinc also increased. This trend can be described in qualitative terms: At 2 M, the limited availability of NH_3 prevented effective complexing with Cu and Zn, resulting in zinc extraction of less than 50%. With 4 M, a slightly higher amount of NH_3 was available for complexing with Cu and Zn, leading to a more substantial zinc extraction of over 60% until the NH_3 was depleted. At 6 M, the abundant availability of NH_3 allowed for effective complexing with Cu and Zn, resulting in a high zinc extraction of over 90%.

The effect of pulp density on zinc extraction

The impact of pulp density on zinc extraction rates was evaluated under the following leaching conditions: 6 M ammonia total (3 M NH_3 and 3 M NH_4Cl), 1:1 NH_3/NH_4^+ ratio, temperature of 55°C, 500 rpm stirring, 72-hour leaching period, pH control between 9 and 11, and use of oxygenated medium.

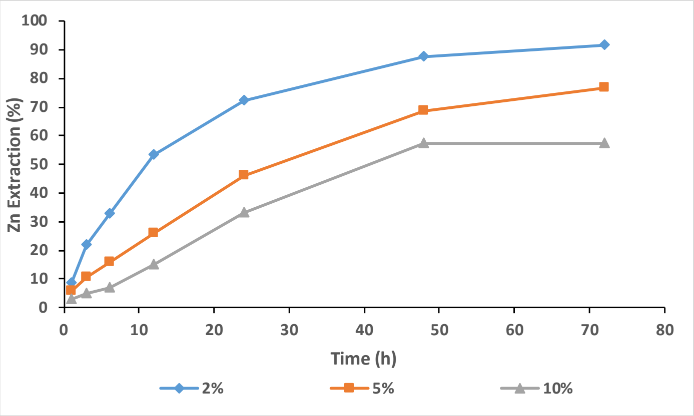

Figure 1. Effects of changing pulp density on zinc extraction from sphalerite concentrate.

The results shown in Figure 10 show that zinc recovery was negatively impacted by an increase in pulp density. Higher pulp densities made the leaching solution more viscous, which limited the amount of leaching agent that may be used when there was a constant leaching dose per unit mass of zinc. As a result of the decreased solvent diffusion rate via the boundary layer, the leaching process was hindered. At high pulp density, the availability of NH_3 for Cu and Zn to complex with was limited, eventually depleting and thus restricting Zn dissolution. At low pulp density, there was sufficient NH_3 available for Cu and Zn to complex with. This explained why Zn extraction was high at low pulp density. Consequently, 2% w/v pulp density was chosen as the most effective pulp density for the experiment.

The catalytic effect of Cu(II) ions on zinc extraction

To evaluate the impact of Cu(II) ions on zinc extraction rates, the subsequent leaching conditions were implemented: A total ammonia concentration of 6 M (3 M NH_3 and 3 M NH_4Cl), 0.25 g/L of Cu(II), a 1:1 NH_3/NH_4^+ ratio, temperature of 55°C, 500 rpm stirring, a 2% w/v slurry concentration, a 72-hour leaching period, pH maintained between 9 and 11, and an oxygen-rich environment were all included.

The function of Cu(II) ions in improving zinc extraction from sphalerite concentrate was investigated in this paper. Results show that adding 0.25 g/L of Cu(II) to the zinc extraction process improved the extraction process from zinc extraction of 91.6% to 99.5%. This implies that Cu(II) ions serve as catalysts throughout the zinc extraction procedure. The redox pair Cu(II)/Cu(I) is the source of the catalytic activity of Cu (II) ions. This involves the oxidation of ZnS by the cupric ammine complex [$Cu(NH_3)_4^{2+}$], which is followed by its reduction to the cuprous ammine complex [$Cu(NH_3)_2^+$]. After that, oxygen (O_2) reoxidises this reduced form (Ghosh *et al.*, 2003; Liu *et al.*, 2012).

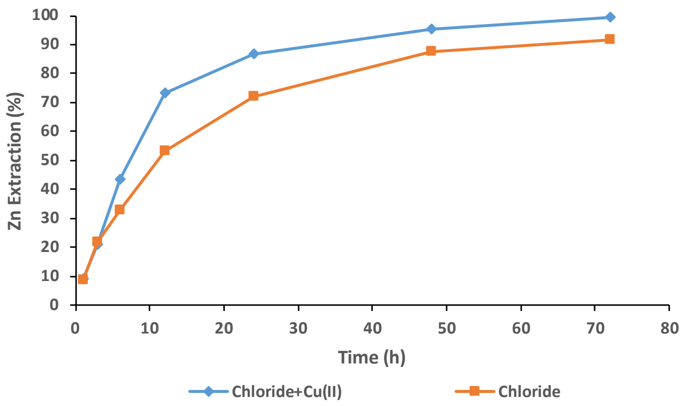

Figure 2. Catalytic effect of Cu(II) on ammonia leaching of zinc from sphalerite concentrate.

CONCLUSIONS

The study on zinc extraction from sphalerite concentrate found that:

- Ammonium chloride was the most effective leaching agent.
- An oxygen medium was more effective than compressed air.
- Zn extractions increased with higher NH_3 concentration and temperature and decreased with higher pulp density; it also improved with the addition of Cu(II) ions.
- The apparent activation energy was determined using the shrinking core model and Arrhenius question to be 49.5 kJ/mol.
- Zn extraction efficiency reached 99.5% under optimal conditions: 6M total ammonia concentration, 0.25 g/L Cu(II), 55°C temperature, 72 hours reaction time, 500 rpm stirring speed, 2% pulp density, 1:1 ammonia/ammonium ratio, and an oxygen-rich environment.

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