



## Kompleks bir Pb-Zn cevherinin sülfürik asit çözeltisinde liçi Leaching of a complex Pb-Zn ore in sulfuric acid solution

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### Abstract

In this study, the leaching behavior of a Gazipaşa-Aydap complex Zn-Pb ore sample in sulfuric acid solutions was investigated. Firstly, the chemical and mineralogical compositions of the ore sample were determined by AAS, ICP-OES, XRF, and XRD analyses. The ore seemed to be very complex, containing major amounts of zinc (16.4%), lead (10.6%), iron (2.6%), and a minor amount of copper (0.2%). The leaching experiments demonstrated that 86.7% Zn, 90% Cu, and 25.2% Fe could be taken into the leach solution under the following conditions: 2 M sulfuric acid concentration, 25 °C leaching temperature, 1/10 solid-to-liquid ratio, and 125 rpm shaking rate. The effects of the leaching temperature on the metal dissolutions were also tested. The experimental results revealed that increasing the leaching temperature had no beneficial effect on zinc dissolution, but increased iron dissolution significantly. Overall, it was determined that zinc could be extracted from the complex ore using a short leaching time (30 min), a low leaching temperature (25 °C), a relatively low sulfuric acid concentration (2 M), and a relatively high solid-to-liquid ratio (2/10).

**Keywords:** Complex Zn-Pb ore, Gazipaşa-Aydap, sulfuric acid, zinc leaching

### 1 Introduction

Zinc holds immense significance as a nonferrous metal extensively utilized in alloy manufacturing and galvanization industries [1]. With a global shift towards reducing sulfur emissions during processing, zinc oxide ores are increasingly favored for zinc extraction, especially amidst sphalerite depletion [2]. Non-sulphide sources might contribute over 10% to global zinc metal production [3], yet untapped reserves remain underutilized due to limited processing methods, notably for low-grade zinc oxide ores with high silicon content [4-5]. In metallurgical sectors, substantial quantities of lead-zinc sulphide ore are floated, resulting in significant non-ferrous and precious metal losses. The rising demand for zinc necessitates thoroughly

### Öz

Bu çalışmada Gazipaşa-Aydap kompleks Zn-Pb cevher numunesinin sülfürik asit çözeltisindeki liç davranışı incelenmiştir. Öncelikle cevher örneğinin kimyasal ve mineralojik bileşimleri AAS, ICP-OES, XRF ve XRD analizleri ile belirlenmiştir. Cevherin yapısının çok karmaşık olduğu ve yüksek miktarda çinko (%16.4), kurşun (%10.6), demir (%2.6) ve az miktarda bakır (%0.2) içerdiği görülmüştür. 2 M sülfürik asit konsantrasyonu, 25 °C liç sıcaklığı, 1/10 katı/sıvı oranı ve 125 rpm çalkalama hızı parametrelerinde gerçekleştirilen liç işlemi ile %86.7 Zn, %90 Cu ve %25.2 Fe'nin liç çözeltisine alınabileceği saptanmıştır. Deneysel sonuçlar, liç sıcaklığının artırılmasının çinko çözünmesi üzerinde yararlı bir etkisinin olmadığını ancak demir çözünmesini önemli ölçüde artırdığını ortaya koymuştur. Genel olarak çinkonun, kısa bir liç süresi (30 dakika), düşük bir liç sıcaklığı (25 °C), nispeten düşük bir sülfürik asit konsantrasyonu (2 M) ve nispeten yüksek bir katı-sıvı oranı (2/10) kullanılarak kompleks cevherden ekstrakte edilebileceği belirlenmiştir.

**Anahtar kelimeler:** Kompleks Zn-Pb cevheri, Gazipaşa-Aydap, sülfürik asit, çinko liçi

exploration of methods to recover zinc from low-grade ores. Projections for 2018 anticipated zinc production and consumption at 13.42 Mt and 13.74 Mt respectively, with a world reserve exceeding 230 Mt [6-7]. Extraction processes like pyrometallurgy and hydrometallurgy offer routes for zinc extraction from oxide ores [8]. While pyrometallurgy suits high-grade ores better, hydrometallurgy emerges as the preferred choice for low-grade ores due to economic, environmental, and technological advantages. Moreover, metals can be directly obtained from pure leaching solutions or recovered from impure ones.

In nature, zinc and lead minerals often coexist within deposits, presenting three primary types of mineralization: sulfide ores, mixed sulfide-oxide ores, and non-sulfide ores, which can undergo weathering in hypogene or supergene

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conditions. Zinc oxide ores, including willemite ( $Zn_2SiO_4$ ), smithsonite ( $ZnCO_3$ ), hemimorphite ( $Zn_4(Si_2O_7)(OH)_2 \cdot H_2O$ ), hydrozincite ( $Zn_5(CO_3)_2(OH)_6$ ), and zincite ( $ZnO$ ), stand out as significant sources of zinc [9]. Treating such intricate and low-grade metal sources, often containing finely dispersed lead and zinc, favors hydrometallurgical processes over pyrometallurgical ones due to their environmental friendliness, lack of harmful emissions or dust, and cost-effectiveness for small-scale operations [10-11]. Common gangue minerals found in zinc oxide ores include hematite ( $Fe_2O_3$ ), goethite ( $FeOOH$ ), dolomite ( $CaMg(CO_3)_2$ ), calcite ( $CaCO_3$ ), and quartz ( $SiO_2$ ), which bring about the significant acid consumption during processing [12]. Extraction of zinc from zinc or lead-zinc oxide ores often involves using acidic reagents, such as sulfuric acid and hydrochloric acid solutions, which have been shown effective in various studies. Sulfuric acid solutions readily dissolve zinc in its oxide, silicate, and carbonate forms, while lead typically does not form insoluble lead sulfate during acid leaching [13-17].

Numerous investigations have aimed to enhance the recovery and quality of zinc ores through flotation or gravity methods, yet these techniques have shown limited effectiveness in altering the inherent properties of the ores [18-19]. The focus of research has shifted towards hydrometallurgical processing of zinc oxide ores using alkaline (such as sodium hydroxide and ammonium) or sulfuric acid solutions. Several studies have explored the alkaline leaching of zinc oxide ores, with findings suggesting the superiority of alkaline media over acidic ones [20]. Optimal conditions for leaching zinc from oxide ores were achieved at 5% NaOH concentration, temperatures of 90-95 °C, and a duration of 90 minutes. In column leaching experiments conducted with low-grade zinc oxide ore containing 5.2% zinc, the utilization of ammonium sulfate resulted in the dissolution of 92.2% of the zinc [21]. Further research revealed that under specific conditions, such as leaching for 120 minutes at 358 K with a sodium hydroxide concentration of 5 mol/L and a liquid-to-solid ratio of 10/1, zinc leaching recovery exceeded 73% for zinc oxide ores sized between 65-76  $\mu m$  [22]. Notably, alkaline leaching offers advantages such as lower iron dissolution and the absence of silica gel formation. However, it is important to note that the mineral hemimorphite remains insoluble using this method.

In most industrial settings, sulfuric acid leaching stands out as the most adaptable method. However, a notable challenge arises during the leaching of zinc silicate ores, where the formation of silica gel can lead to substantial issues in liquid-solid separation. This gel-like precipitate has the potential to obstruct ore particles, resulting in poor leaching kinetics and recoveries, even in heap or dump leaching processes [5]. Additionally, sulfuric acid processing presents drawbacks such as high acid consumption and intricate purification procedures, which can hinder the efficiency of subsequent stages like solvent extraction, ion exchange, and electrolysis [23]. Various approaches, including coagulation and flocculation, have been employed to mitigate this issue. One continuous concurrent method

involves initially dissolving soluble silica and subsequently coagulating the formed colloidal silica by adjusting the solution's pH using a neutralizing agent [24]. The primary aim of this study is to explore the feasibility of selectively extracting zinc from a complex Zn-Pb ore through sulfuric acid leaching while simultaneously rejecting a significant portion of impurities (such as Fe, Cu, and Pb) in the leach residue. Several factors affecting zinc leaching were investigated, including leaching time, acid concentration, leaching temperature, and solid-to-liquid ratio.

## 2 Material and methods

The ore sample was sourced from Dh Mining Co., located in Gazipasa-Aydap, Antalya, Türkiye. Initially, the ore underwent grinding in a laboratory ball mill. The chemical composition analysis was conducted through digestion and solution analysis utilizing inductively coupled plasma optical emission spectroscopy (ICP-OES, Perkin Elmer) at Batman University, and atomic absorption spectroscopy (AAS, Perkin Elmer) at Abdullah Gül University. Additionally, the ore sample's chemical composition was analyzed using X-ray fluorescence spectrometry (XRF, Minipal 4 Panalytical) at Çukurova University. For mineralogical identification, an X-ray diffraction instrument (XRD, Bruker Discover) at Abdullah Gül University was employed, calibrated with a silicon standard for alignment within the  $2\theta=5-90^\circ$  range, utilizing Cu  $K\alpha$  radiation generated at 40 mA and 40 kV. The particle size distribution of the ore sample was determined using a laser diffraction particle size analyzer (Malvern Mastersizer 2000) at Eskişehir Osmangazi University.

The leaching experiments were conducted in 100-mL Erlenmeyer glass flasks fitted with oakum covers to minimize evaporative losses. These flasks were submerged in a temperature-controlled water bath equipped with an orbital shaker (MIPROLAB), as depicted in Figure 1 illustrating the experimental setup. The water bath temperature was regularly monitored using a glass thermometer. Each experiment involved adding 50 mL of a solution with a 2 M sulfuric acid concentration to the flask, followed by the addition of 5 g of the ground ore sample. Upon completion of the experimental sets, the flasks were filtered using Whatman 1 filter paper, and the residues were washed with deionized water. The resulting solutions (leachate) were analyzed using ICP-OES and AAS. Analytical-grade sulfuric acid (97% purity,  $H_2SO_4$ , Merck) was utilized. Throughout the tests, the solid-to-liquid ratio (w/v) and shaking rate remained constant at 1/10 and 125 rpm, respectively, unless otherwise specified. Dissolution percentages were calculated using Equation (1), and experiments were duplicated or triplicated to ensure accuracy, with dissolution percentages constructed at a 95% confidence level.

$$Dissolution (\%) = \frac{\mu \times v}{\eta \times \omega} \times 100 \quad (1)$$

$\mu$  is the metal concentration in the leachate (mg/L),  $v$  is the volume of the analysed leachate (L),  $\eta$  is the metal

concentration in ore sample (mg/kg),  $\omega$  is the mass of the ore sample (kg).



Figure 1. Experimental set-up.

### 3 Results and discussion

#### 3.1 Ore sample characterization

The chemical composition of the ore sample utilized in this study is detailed in Table 1. Predominantly, the ore sample comprises Zn, Pb, and Fe as major elements, alongside minor quantities of Cu. Figure 2 illustrates the XRD pattern of the ore sample, indicating the presence of major mineral phases such as smithsonite ( $ZnCO_3$ ), cerussite ( $PbCO_3$ ), dolomite ( $CaMg(CO_3)_2$ ), quartz ( $SiO_2$ ), and galena ( $PbS$ ). XRD analysis confirms that the ore sample can be categorized as a complex ore containing carbonate and sulfur constituents. As depicted in Figure 3, the particle size distribution analysis reveals that approximately 80% of the ore particles are smaller than 92  $\mu m$  in size.

Table 1. Chemical composition of the ore sample.

AAS/ICP-OES analyses		XRF analyses	
Element	Concentration, (%)	Compound	Concentration, (%)
Zn	16.4	SiO <sub>2</sub>	19.1
Pb	10.6	CaO	15.9
Fe	2.6	MnO	0.3
Cu	0.2	K <sub>2</sub> O	0.16
		TiO <sub>2</sub>	0.11
		Loss on ignition	20.7

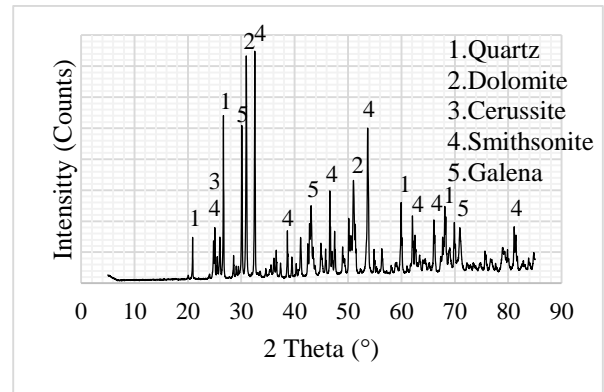
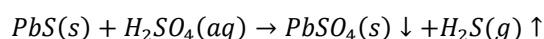
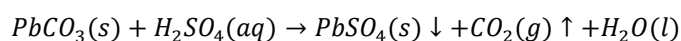
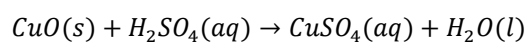
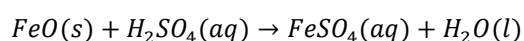
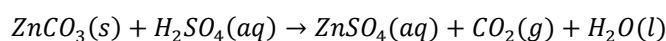
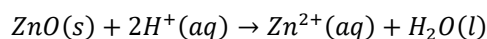
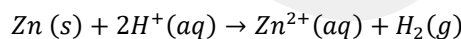


Figure 2. XRD pattern of the ore sample.

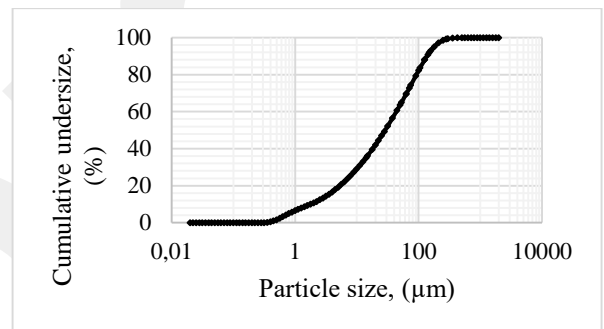


Figure 3. Particle size distribution of the ore sample.

#### 3.2 Effect of leaching time on metal dissolution

Figure 4 illustrates the impact of leaching duration on the dissolution of zinc, lead, copper, and iron from the ore sample using a 2 M sulfuric acid solution at 25 °C. Notably, zinc dissolution remained relatively constant with increasing leaching time. Similarly, copper and iron dissolutions exhibited comparable trends across the tested durations. The findings suggest that extending leaching duration did not notably enhance the dissolution of metal values. Zinc, copper, and iron were extracted into the leach solution at rates of 86.7%, 90%, and 25.2%, respectively.

The leaching conditions were as follows: a 2 M sulfuric acid concentration, a leaching temperature of 25 °C, a solid-to-liquid ratio of 1:10, a shaking rate of 125 rpm, and a leaching time of 30 minutes. This indicates that the optimal leaching time was determined to be 30 minutes. Potential chemical reactions for leaching are provided in Eqs 2-8.

$$\Delta G_{298}^{\circ} = -147.3 \text{ kJ} \quad (2)$$

$$\Delta G_{298}^{\circ} = -64 \text{ kJ} \quad (3)$$

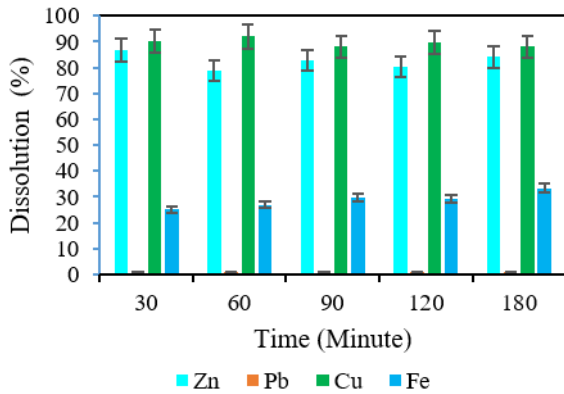
$$\Delta G_{298}^{\circ} = -106.925 \text{ kJ} \quad (4)$$

$$\Delta G_{298}^{\circ} = -116.762 \text{ kJ} \quad (5)$$

$$\Delta G_{298}^{\circ} = -90.178 \text{ kJ} \quad (6)$$

$$\Delta G_{298}^{\circ} = -90.178 \text{ kJ} \quad (7)$$

$$\Delta G_{298}^{\circ} = -90.178 \text{ kJ} \quad (8)$$

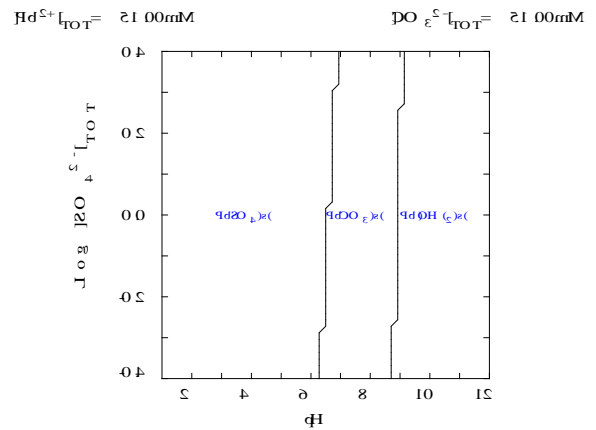


**Figure 4.** Dissolution of metal values versus of time [Conditions: 2 M sulfuric acid, 25 °C temperature, 1/10 solid-to-liquid ratio (w/v), 125 rpm shaking rate].

As demonstrated in Figure 4, no lead dissolution occurred when lead carbonate was exposed to sulfuric acid, leading to its precipitation as lead sulfate (as shown in Figure 5). The standard Gibbs free energies ( $\Delta G^\circ$ ) of these reactions at 25 °C (298 K) were computed using HSC 6 Chemistry software, revealing that the reactions occur spontaneously according to Eqs 2-6.



**Figure 5.** Collected  $PbSO_4$  rich precipitate [The precipitate collected at conditions: 2 M sulfuric acid, 30 minutes leaching time, 25 °C temperature, 2/10 solid-to-liquid ratio (w/v), 125 rpm shaking rate].

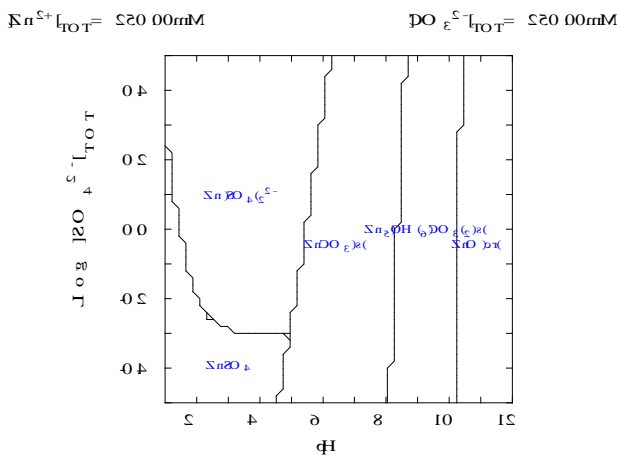


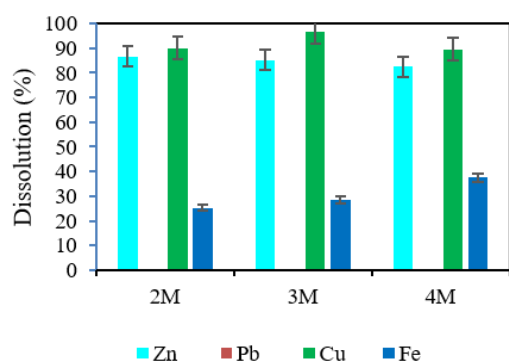
**Figure 6.** Predominance diagram of the  $Zn-CO_3^{2-}-Pb-CO_3^{2-}-SO_4^{2-}$  system (HYDRA/MEDUSA software).

Based on the ore sample's zinc and lead content added to the solution, the concentrations of  $Zn^{2+}$  and  $Pb^{2+}$  were determined to be approximately 16.4 g/L (~250 mM) and 10.6 g/L (~51 mM), respectively. Given the high affinity of  $Zn^{2+}$  and  $SO_4^{2-}$  for forming zinc sulfate, the leaching of zinc carbonate intensifies with increasing sulfuric acid concentration (Eq. 4). Based on experimental findings, a sulfuric acid concentration of 2 M was chosen, resulting in an  $SO_4^{2-}$  concentration of approximately 0.301 on a logarithmic scale (as depicted in Figure 6). According to the predominance diagrams illustrated in Figure 6,  $Zn(SO_4)_2^{2-}$  remains stable within the pH range of approximately 0-6 when the sulfuric acid concentration is 2 M. At pH levels above 6,  $ZnCO_3$  is the stable insoluble form of zinc. Conversely, the stability pH range for  $PbSO_4$  is greater than 0-7, while the pH range for  $PbCO_3$  stability spans approximately 7-9.

### 3.3 Effect of sulfuric acid concentration on metal dissolution

Figure 7 depicts the influence of sulfuric acid concentration on the dissolution of zinc, lead, copper, and iron from the Zn-Pb complex ore sample at 25 °C, with a solid-to-liquid ratio of 1/10, and a shaking rate of 125 rpm for a leaching time of 30 minutes. Interestingly, as sulfuric acid concentrations increased, zinc dissolution remained relatively stable. Approximately 85.2% of zinc was extracted into the leach solution using a 3 M sulfuric acid concentration within the 30-minute leaching duration. Under the specified conditions, over 90% of copper was leached into the solution. Fluctuations in copper dissolution were attributed to acid-consuming mineral phases like calcium and iron, low copper concentration in the ore sample, and interference of other metal ions during instrumental analysis of iron or copper concentration. Iron dissolution notably increased as sulfuric acid concentration rose from 3 M to 4 M. With a 4 M sulfuric acid concentration at 25 °C for 30 minutes of leaching time, the dissolution percentage of iron reached 37.4%. Ultimately, the optimal sulfuric acid concentration was determined to be 2 M.

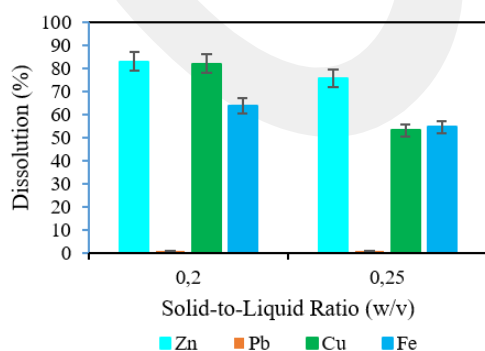




**Figure 7.** Dissolution of metal values as a function of sulfuric acid concentration [Conditions: 25 °C leaching temperature, 30 minutes leaching time, 1/10 (w/v) solid-to-liquid ratio, 125 rpm shaking rate].

### 3.4 Effect of solid-to-liquid ratio on metal dissolution

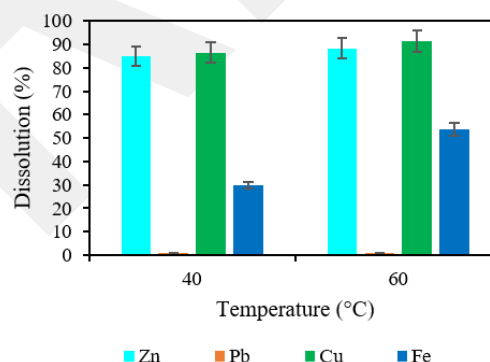
Figure 8 illustrates the impact of the solid-to-liquid ratio on the dissolution of zinc, lead, copper, and iron from the ore using a 2 M sulfuric acid solution at 80 °C for 180 minutes. Notably, the metal dissolution percentages in the leach solution exhibited a decreasing trend as the solid ratio increased. Among the experiments conducted, the highest zinc dissolution achieved was 83% at a solid-to-liquid ratio of 2/10. Additionally, 82% of copper and 63.9% of iron were co-extracted under these conditions. As the solid-to-liquid ratio increased to 2.5/10, zinc, copper, and iron dissolutions decreased to 75.6%, 53.1%, and 54.6%, respectively. The reduction in metal dissolution at higher solid-to-liquid ratios can be attributed to the limited availability of acid per unit of solid, potentially hindering dissolution reactions. Moreover, higher solid ratios may lead to stirring difficulties due to increased pulp viscosity. Conversely, higher liquid ratios result in a diluted solution, increased consumption of leaching reagents, and lower pulp viscosity, promoting better mixing of the slurry and facilitating mass transfer at the solid-liquid interface and diffusion to the leaching action. Based on these observations, a solid-to-liquid ratio of 2/10 was determined to be the optimal condition for the leaching experiments.



**Figure 8.** Dissolution of metal values as a function of solid-to-liquid ratio [Conditions: 2 M sulfuric acid, 180 minutes leaching time, 80 °C temperature, 125 rpm shaking rate].

### 3.5 Effect of leaching temperature on metal dissolution

Figure 9 presents the impact of temperature on the dissolution of zinc, lead, copper, and iron from the ore. Notably, at 40 °C and a solid-to-liquid ratio of 10% for 30 minutes, the zinc dissolution rate reached 85%, while copper and iron dissolutions were 86% and 30%, respectively. Upon increasing the leaching temperature from 40 to 60 °C, there were slight increases in zinc and copper dissolution percentages, reaching 88% and 91%, respectively. However, the solubility of iron exhibited a significant increase, with the dissolution rate reaching 54% at higher temperatures. These findings suggest that while the leaching temperature had little effect on zinc dissolution, it strongly influenced iron dissolution. Higher temperatures led to increased reaction rates. Since no acid is added to maintain a constant pH in the batch leaching system, the leaching reactions consumed acid, leading to an increase in pH that reduced zinc and copper dissolution rates. However, at higher temperatures, zinc and copper recoveries slightly improved, possibly due to polymerization and hydrolysis effects. These results emphasize the importance of performing the leaching process at lower temperatures. Additionally, impurities such as iron and copper should be minimized in the leach solution to prevent significant challenges during subsequent purification stages, including partial neutralization, precipitation, solvent extraction, and electrowinning.



**Figure 9.** Dissolution of metal values as a function of leaching temperature [Conditions: 2 M sulfuric acid, 30 minutes leaching time, 1/10 (w/v) solid-to-liquid ratio, 125 rpm shaking rate].

## 4 Conclusions

The investigation focused on the dissolution behavior of zinc, lead, copper, and iron from a complex Zn-Pb ore in sulfuric acid solution under atmospheric pressure acidic leaching conditions. The study examined the effects of leaching time, acid concentration, solid-to-liquid ratio, and leaching temperature on metal dissolution. Based on the experimental findings, the following conclusions were drawn:

1. The ore sample comprises carbonate and sulfide-type mineral phases, suggesting potential concentration via flotation before the hydrometallurgical process.

2. XRD analysis identified carbonate and oxide mineral phases as predominant in the ore sample. Direct dissolution

of zinc from the zinc carbonate mineral phase in sulfuric acid solution appears feasible.

3. The optimal conditions for zinc dissolution from the ore sample include a short leaching time (30 minutes), a low leaching temperature (25 °C), a relatively low sulfuric acid concentration (2 M), and a high solid-to-liquid ratio (2:10).

4. Selective dissolution of zinc using sulfuric acid proved challenging. More than 90% of copper and 20% of iron were co-extracted along with zinc from the leach solution, posing significant challenges for downstream processing stages such as precipitation and separation/purification.

These conclusions underscore the complexities and considerations involved in optimizing the hydrometallurgical extraction of metals from complex ore samples, highlighting the need for careful process design and management to mitigate challenges in downstream processing.

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#### Conflicts of Interest

No conflict of interest was declared by the authors.

Similarity Rate (iThenticate): 18%

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