INFORMATICS JOURNALS

Critical Minerals Metals and Materials
eISSN: xxxx-xxxx; DOI: 10.18311/c3m/2026/55153
Volume 1, Issue 2, June 2026, pp. 00-00

Recovery of Bismuth and Other Metals from Copper Sulfide Ores via Flotation Followed by Ferric Chloride Leaching

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Abstract

Copper sulfide ores subjected to this experimental study contained considerable amounts of Bi, Te, Ag, and Au, along with base metals such as Cu, Zn, and Pb. The experimental study includes flotation and ferric chloride leaching tests. Chalcopyrite and complex (Cu-Zn-Pb) concentrates resulted from flotation experiments. Ferric chloride leaching tests were carried out with complex concentrate, and the effects of leaching time, ferric and HCl concentrations, solid/liquid ratio, and temperature were investigated in relation to metal leaching recoveries. At optimum leaching conditions, 78.7% Cu, 93.2% Pb, 81.1% Zn, 88.2% Te, 87.1% Bi, and 81.2% Ag dissolved at optimum leaching conditions of 75 g/L ferric ion concentration, 20 g/L HCl concentration, 1/10 S/L ratio, 90°C temperature, and 8 hours leaching time, while Au remained in the leach residue. Additionally, low Fe dissolution was favourable in the metal recoveries from the leachate step. Kinetic studies revealed that apparent activation energies were calculated as 68.76 kJ/mol for Cu, 40.45 kJ/mol for Zn, and 9.67 kJ/mol for Bi, which are in the range of literature findings. These results indicate that the rate-controlling mechanism varies among the metals. The relatively high activation energy obtained for Cu suggests that the overall leaching rate is predominantly controlled by surface chemical reactions, whereas the intermediate value obtained for Zn indicates a mixed control regime involving both surface reaction and diffusion through the product layer. In contrast, the low activation energy determined for Bi is characteristic of diffusion-controlled processes, which shows the tendency of Bi dissolution to approach a plateau at shorter leaching times.

Major Findings: *Ferric chloride leaching enabled the simultaneous dissolution of minor valuable elements, particularly Bi and Te, together with the main metals Cu, Zn, Pb, and Ag from the Cu-Pb-Zn complex concentrate. In contrast, Fe dissolution remained relatively low, which is advantageous for downstream hydrometallurgical processing by reducing the burden on solution purification and metal recovery steps. These findings demonstrate the potential of the proposed route for the selective valorization of polymetallic copper sulfide ores.*

Keywords: *Bismuth, Tellurium, Ferric Chloride Leaching, Flotation, Polymetallic Copper Sulfide Ore*

1. Introduction

Bismuth (Bi) is a rare, strategic, and valuable metal used in numerous applications, including metallurgy, alloys, semiconductors, medicine, cosmetics, and other industrial sectors[1,2]. Bismuth resource reserves

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in nature are scarce, and they mainly occur as native Bi, Bi_2S_3, Bi_2O_3, $(BiO)_2CO_3$, and $3Cu_2S$-$4Bi_2S_3$[3]. Bismuth is not rich in the earth's crust, and Bi deposits are decreasing dramatically over the years; therefore, it is important to recover Bi along with other base metals from complex/poly metallic ores, concentrates, mineral and metallurgical plant wastes, and electronic waste, etc[4]. Bi reserves are very rare in nature, and Bi minerals are mostly associated with W, Cu, Mo, Pb, and Sn; thus, it can be separated during the beneficiation of other sulfide metals. Pyrometallurgical, hydrometallurgical, and pyro-hydrometallurgical processes are used in Bi production. Pyrometallurgical processes include precipitation, alkaline, bath, and reduction smelting techniques, where hydrometallurgical processes are mostly chloride-based leaching[3].

Major rich copper ore deposits with lead-zinc ores in Türkiye lie between Samsun and the Hopa-Artvin district of the Eastern Black Sea region[5]. Murgul copper ore deposits are rich in pyrite and chalcopyrite minerals and are one of the biggest and most important mining areas in that region. During earlier studies in our laboratory, we found that copper and pyrite concentrates contained Ge, In, and Bi. Mineralogical studies in another research indicated Se, Te, and Bi in Murgul copper ores[6]. Therefore, it was worthwhile to carry out a study on that metal's recovery.

In the flotation of copper ores containing Bi minerals, Bi passes into a copper concentrate[7]. Thus, Bi cannot be separated as an individual concentrate in the beneficiation of copper ores containing Bi. Cu-Bi concentrates should be processed hydrometallurgically and/or by combined extraction methods. Most of the hydrometallurgical methods for the processing of these concentrates propose the use of hydrochloric acid with different oxidisers, such as HNO_3, H_2O_2, Cl_2, $FeCl_2$, etc., and ferric chloride leaching of chalcopyrite, sphalerite, and galena concentrates have been extensively studied by various researchers[7-12]. The high chloride concentration in the leach medium increases the solubility of metallic sulfides such as Cu, Zn, Pb, Bi, Ag, etc. during the leaching process. These minerals react with ferric chloride and dissolve metals according to the following reactions[8]:

$$CuFeS_2 + 4\ FeCl_3 \rightarrow CuCl_2 + 5\ FeCl_2 + 2\ S° \quad (1)$$

$$ZnS + 2\ FeCl_3 \rightarrow ZnCl_2 + 2\ FeCl_2 + S° \quad (2)$$

$$PbS + 2\ FeCl_3 \rightarrow PbCl_2 + 2\ FeCl_2 + S° \quad (3)$$

$$Ag_2S + 2\ FeCl_3 \rightarrow 2\ AgCl + 2\ FeCl_2 + S° \quad (4)$$

$$Bi_2S_3 + 6\ FeCl_3 \rightarrow 2\ BiCl_3 + 6\ FeCl_2 + 3\ S° \quad (5)$$

Although much of the early work was carried out on the ferric chloride leaching of copper concentrates, the most promising application of this technology may be for lead concentrates and complex pyritic ores or concentrates. The rapid reaction of galena with ferric chloride media makes this route especially attractive for lead-bearing feeds, as the possibility of selective leaching exists. Ferric chloride leaching of chalcopyrite, sphalerite, and galena concentrates has been extensively studied by various researchers[9,12-15].

There are also some investigations on improving chloride-based leaching processes[9,16-21]. The use of microwave fields during ferric chloride leaching has been studied, and the main advantages of this method are explained as high leaching temperature under normal conditions, fast leaching and pulp heating, faster leaching time, and intense agitation without mechanical tools[16]. Additional advantages are mentioned as the existence of the commercial microwave facilities that are usable for leaching[22-24]. Mechanical activation before ferric chloride leaching of chalcopyrite and galena has also been studied[19,20,25,26]. Significant improvements in terms of leaching time and metal leaching recoveries were observed. Some reagents, such as carbon tetrachloride and methane sulfonic acid, may also be added during ferric chloride leaching to improve metal dissolution rates[9,27]. To

enhance the chloride recovery of silver and lead to a satisfactory level, it was necessary to apply the alkaline pretreatment of the feed using NaOH at a temperature of 90°C[28]. Bismuth products could be effectively separated and recovered from bismuth-containing leachates by neutralisation hydrolysis, solvent extraction, replacement, and electrowinning[4].

2. Experimental Studies

This experimental study aims to determine the distribution of base metals along with rare and precious metals in Murgul-Damar region copper sulfide ores and to investigate their beneficiation possibilities. Experimental work covers sampling, determination of physical and chemical properties, flotation, and ferric chloride leaching studies.

3. Material

In this research, selective ore samples collected from rich zones of the Murgul-Damar region (Artvin-Türkiye) were used. According to chemical analysis, the ore sample contained 14.4% Cu, 2.84% Zn, 0.18% Pb, 25.51% Fe, 417 g/t Bi, 118 g/t Te, <100 g/t Se, <50 g/t In, 77 g/t Ag, and 1.2 g/t Au. Mineralogical examinations of Murgul copper sulfide ores and concentrates showed that pyrite (FeS_2) and chalcopyrite ($CuFeS_2$) were the main minerals with the small amounts of sphalerite (ZnS), galena (PbS), melnikovite ($Zn_3(PO_4)_2{\cdot}4H_2O$), covellite (CuS), chalcocite (Cu_2S), tetrahedrite ($(Cu, Fe, Zn, Ag)_{12}SbS_{13}$), tennantite ($(Cu, Fe, Zn, Ag)_{12}As_4S_{13}$), altaite (PbTe), clausthalite (PbSe), tetradymite (Bi_2Te_2S), wittichenite (Cu_3BiS_3), bournonite ($PbCuSbS_3$), ankerite ($Ca(Fe, Mg, Mn)(CO_3)_2$), barite ($BaSO_4$), calcite, and quartz. According to the microscopic analysis, the liberation size of major minerals was found to be 74 microns. The ore sample subjected to this experimental study was crushed to a size of 2 mm by using laboratory-type jaw, cone, and roll crushers. Therefore, an ore sample under 2 mm in size was ground for 5, 10, 15, 20, and 25 minutes to find the optimum grinding time for flotation. As a result of this, 25 minutes of grinding were sufficient.

4. Flotation Experiments

The flotation experiments were carried out with the ore sample ground to 74 microns in size. Two steps of flotation tests were performed to determine metal distributions and concentration possibilities (Figure 1). A laboratory-scale Denver-type flotation cell with mechanical stirring and aeration was utilised in the flotation tests. Aerophine 3418A, which is selective for sulfide minerals other than pyrite as a collector, Dowfroth 250 as a frother, and $CuSO_4$ as an activator for sphalerite were the reagents used in the flotation tests.

As seen from Figure 1, bulk flotation experiments were performed at the natural pH with Aerophine 3418A and Dowfroth 250. Selective flotation tests before bulk flotation were carried out at a pH of 11.5 stabilised with the addition of lime (CaO) without the use of a collector and frother. In the first group of these flotation experiments for determining metal distributions, chalcopyrite, Cu-Pb-Zn complex concentrates were produced separately. Chemical analysis of flotation products shown in Table 1 and Table 2 revealed that a chalcopyrite concentrate containing 24.19% Cu content was produced with 94.4% recovery, and a Cu-Pb-Zn complex concentrate containing 6.13% Zn, 0.41% Pb, 4.3% Cu, 22.86% Fe,

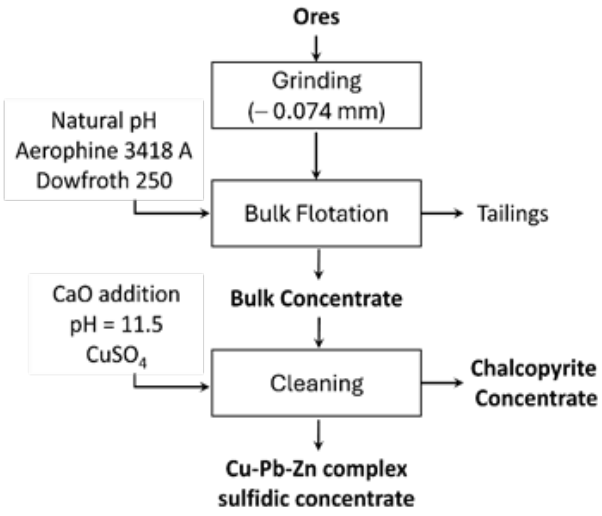


Figure 1. Flotation experiment flow diagram.

Table 1. Flotation test results

Products	Weight %	Cu		Zn		Pb	
		%	%R	%	%R	%	%R
Chalcopyrite concentrate	58.84	24.19	94.40	3.06	61.20	0.12	46.54
Cu-Pb-Zn concentrate	18.39	4.30	5.25	6.13	38.31	0.41	49.71
Tailings	22.77	0.23	0.35	0.06	0.49	0.03	3.75
Total	100.0	15.08	100.0	2.94	100.0	0.15	100.0

Table 2. Chemical analysis of flotation products

Element	Chalcopyrite concentrate	Cu-Pb-Zn complex concentrate	Tailings
Cu (%)	24.19	4.3	0.35
Zn (%)	3.06	6.13	0.06
Pb (%)	0.12	0.41	0.03
Fe (%)	33.40	22.86	4.21
S (%)	35.48	26.75	1.50
SiO_2 (%)	0.72	24.86	69.8
Bi (g/t)	512	1250	69
Au (g/t)	1.6	1.4	--
Ag (g/t)	114	53	--
Se (g/t)	<100	<100	<100
Te (g/t)	112	250	27

1250 g/t Bi, and 250 g/t Te was produced and a tailing containing 0.23% Cu was discarded.

5. Ferric Chloride Leaching Experiments

Ferric chloride leaching experiments were carried out with the Cu-Pb-Zn complex sulfide flotation concentrate to recover Cu, Zn, Pb, Bi, Te, and Ag. In the experiments, the effects of ferric ion (Fe^{3+}) and HCl concentrations, S/L ratio, temperature, and leaching time on the metal leaching efficiencies were investigated.

5.1 Effect of Leaching Time

Constant experimental conditions for investigating the leaching time effect were 75 g/L ferric ion

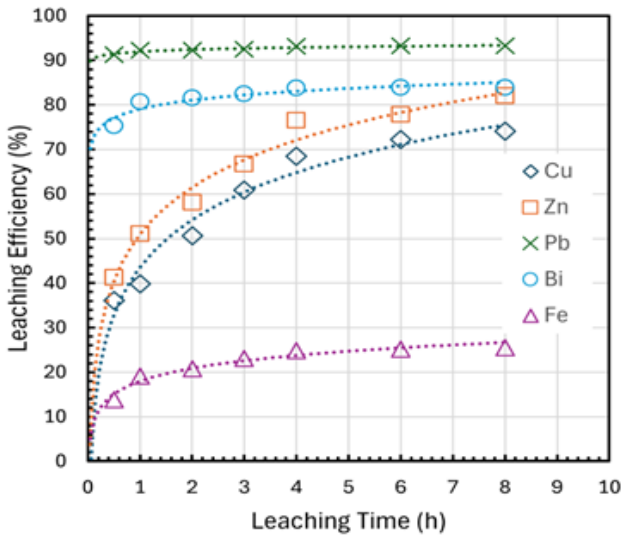


Figure 2. Effect of leaching time on metal leaching efficiencies.

concentration, 20 g/L HCl concentration, 1/10 S/L ratio, and 80°C temperature. Results given in Figure 2 show that metal leaching recoveries increased with increasing leaching time. Pb and Bi dissolutions were faster than the others; Pb and Bi recoveries reached 92.2% and 81%, respectively, in one hour and stabilised after that. On the other hand, Cu and Zn dissolution curves versus time showed parabolic behaviour. 74.1% of Cu and 82% of Zn were leached at the end of an 8-hour leaching period.

5.2 Effect of Ferric Ion Concentration

Constant experiments to study the ferric ion concentration effect are 20 g/L HCl concentration, 1/10 S/L ratio, 80°C temperature, and 8 hours of leaching time. Results are summarised in Table 3, and changing ferric ion concentration showed almost no effect on the leaching recoveries of Pb, Bi, and Fe but had an increasing effect on Cu and Zn recoveries. Significant changes in copper leaching recoveries were also observed with increasing ferric ion concentration in another study[12]. Considering the dissolution of all metals mentioned here, the optimum Fe^{3+} ion concentration was found to be 75 g/L.

5.3 Effect of HCl Concentration

Table 3 shows the effect of HCl concentration at constant experimental conditions of 75 g/L ferric ion concentration, 1/10 S/L ratio, 80°C temperature, and 8 hours leaching time. According to the results given in Table 4, leaching recoveries of Pb, Zn, Bi, and Fe were not affected by increasing HCl concentration, while Cu recoveries declined, where 20 g/L HCl concentration was found to be an optimum value. However, in earlier studies, it was found that the high chloride concentration leaching medium is aimed at increasing the solubility of metal chlorides[9].

5.4 Effect of Solid/Liquid Ratio

The effect of S/L ratio was investigated at constant experimental conditions of 75 g/L ferric ion concentration, 20 g/L HCl concentration, 80°C temperature, and 8 hours leaching time. Leaching recoveries of all metals (except Zn) did not change very much with the increasing S/L ratio, as seen from Table 5, while Zn showed an increasing trend with increasing S/L ratio. An optimum S/L ratio was about 1/10, and 74.1% Cu, 82 Zn, 93.2% Pb, and 83.9% Bi dissolution

Table 3. Effect of ferric ion addition on metal leaching efficiencies

Fe^{3+} (g/L)	Weight Loss (%)	Leaching Efficiency (%)				
		Cu	Zn	Pb	Bi	Fe
---	16.52	27.8	38.6	91.3	58.1	12.6
37.5	23.46	66.7	73.2	92.0	83.9	20.5
75	24.22	74.1	82.0	93.2	83.9	25.5
150	24.54	86.1	87.9	94.2	83.9	25.9

Table 4. Effect of HCl addition on metal leaching efficiencies

HCl (g/L)	Weight Loss (%)	Leaching Efficiency (%)				
		Cu	Zn	Pb	Bi	Fe
none	25.78	75.0	80.4	93.2	83.9	27.1
10	24.47	74.2	81.9	93.3	84.4	25.7
20	24.22	74.1	82.0	93.2	83.9	26.5
30	24.11	64.8	82.0	92.2	83.9	26.7
40	23.84	63.0	83.7	92.2	80.7	29.0

Table 5. Effect of S/L ratio on metal leaching efficiencies

S/L Ratio	Weight Loss (%)	Leaching Efficiency (%)				
		Cu	Zn	Pb	Bi	Fe
1/2.5	16.66	77.5	48.0	91.0	83.2	21.3
1/5	24.14	85.6	72.6	93.3	84.1	27.9
1/10	24.22	74.1	82.0	93.2	83.9	25.5

efficiencies were observed at the optimum S/L ratio, while Fe dissolution efficiency did not change much with S/L ratio increase.

5.5 Effect of Temperature

Constant experimental conditions for the experiments to examine the effect of temperature are 75 g/L ferric ion concentration, 20 g/L HCl concentration, 1/10 S/L ratio, and a leaching time varying from 1 to 8 hours. The effect of temperature on Cu leaching efficiencies is presented in Figure 3. As seen from the graph, copper leaching efficiency versus leaching time showed a parabolic trend, and curves shifted to higher values with increasing temperature. Cu leaching efficiency increased from 20.4% at 40°C to 78.7% at 90°C at the end of 8-hour leaching. Similar to our results, as a result of the recovery of copper from a flotation concentrate using ferric chloride leaching, it was found that 95%

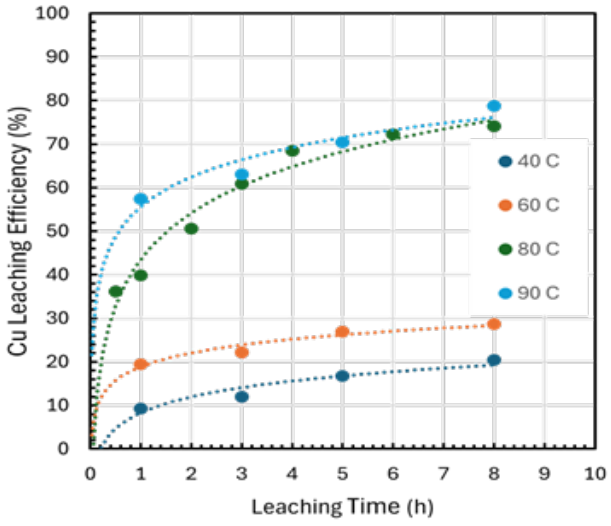


Figure 3. Effect of temperature on Cu leaching efficiency.

Cu recovery was observed in one hour at 70°C, whereas pyrite and gangue minerals did not react as in our case[29]. In the study by Arslan *et al.*[12], it was also found that increasing temperature shifted Cu dissolution efficiency-time curves to higher values, and 76.2% Cu was extracted in 8 h of leaching at 90°C. In another study, about 90% of copper was leached out from copper concentrates under optimum conditions of -100 mesh, 6 hours leaching time, 6 M $FeCl_3$ concentration, pH 2, 95°C, and 200 rpm agitation[30]. In a similar study on the leaching of natural chalcopyrite in ferric chloride, the morphology of leached chalcopyrite crystal particles showed a porous elemental sulfur layer, which supports a continuous increase in Cu leaching efficiency with time and temperature[31].

Zn leaching efficiencies with leaching time showed a similar trend as seen in Figure 4. At the end of 8 hours of leaching, Zn leaching efficiency increased from 36.6% at 40°C to 81.1% at 90°C. In another study on ferric chloride leaching of copper concentrate produced from Küre copper ores (Türkiye), it was also found that increasing temperature shifted Zn dissolution efficiency-time curves to higher values, and 91.37% Zn was extracted in 8 h at 90°C[12].

Sokić *et al.*[32] studied the possibility of obtaining zinc from complex polymetallic ores, and they focused on the leaching of sphalerite in acidic solutions and the phenomena that accompany the dissolution process. Special attention was given to research on the kinetics and mechanisms of sphalerite oxidation in acidic media influenced by various oxidants, including hydrogen peroxide, oxygen, dichromate ions, nitrate and nitrite ions, ferric and cupric ions, and others. All these results enable the development of new

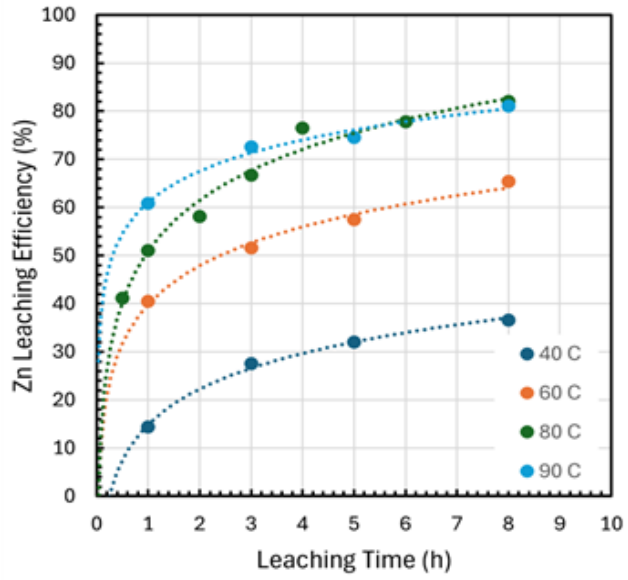


Figure 4. Effect of temperature on Zn leaching efficiency.

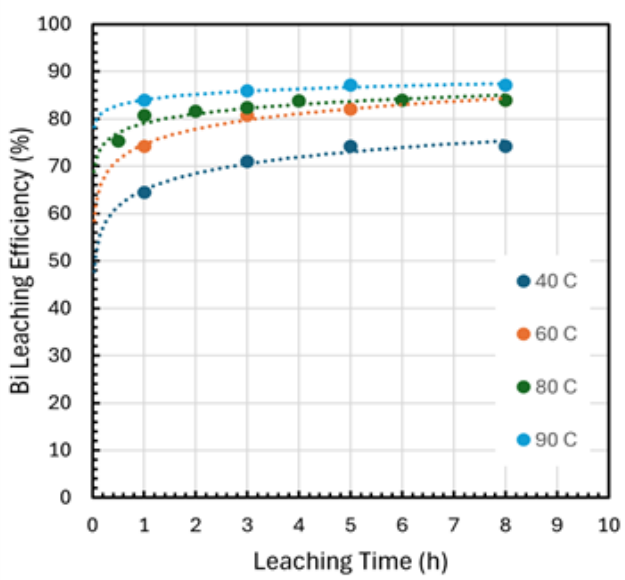


Figure 5. Effect of temperature on Bi leaching efficiency.

technological procedures to produce zinc from low-grade and complex ores while meeting increasingly rigorous environmental requirements.

Bi leaching efficiency versus leaching time is illustrated in Figure 5. Bi dissolution increased rapidly with time and reached maximum values in 3 hours at all temperatures. Increasing temperatures moved efficiency-time curves to higher values. In a study on extracting bismuth from bismutite ore containing bismutite and zinnwaldite using an acidic chloride medium, followed by the hydrolytic separation of bismuth as bismuth oxychloride (BiOCl), it was also found that bismuth was selectively leached with a high efficiency of 94.9%, while orthoclase remained in the residue, as verified by XRD analysis[33]. Sodium hydroxide (NaOH) treatment enabled the quantitative precipitation of BiOCl, which has potential as a precursor for pharmaceutical and supercapacitor applications.

Table 6 presents the effect of temperature on Fe and Pb leaching efficiencies, and the results indicate a slight temperature influence on these efficiencies. Pb dissolved quickly, and Pb dissolution was not affected much by temperature and leaching time. Pb leaching

Table 6. Effect of temperature on Fe and Pb leaching efficiencies

Time (h)	Metal Leaching Efficiency (%)							
	40°C		60°C		80°C		90°C	
	Fe	Pb	Fe	Pb	Fe	Pb	Fe	Pb
1	8.4	79.6	11.4	90.3	19.1	92.2	19.6	92.2
3	9.5	88.4	13.0	91.3	23.1	92.5	22.9	93.2
5	9.8	90.3	14.1	92.2	24.8	93.1	24.5	93.2
8	10.0	90.3	15.0	92.2	25.5	93.1	26.8	93.2

efficiency reached over 90% at all temperatures after 60°C in one hour. The rapid reaction of galena with ferric chloride media makes this route especially attractive for lead-bearing feeds, as the possibility of selective leaching exists. Similar results were also observed in the literature on ferric chloride leaching of lead sulfide and complex concentrates[13,34,35]. In an earlier study with Küre copper ores, it was found that temperature and dissolution time did not affect Pb leaching and Pb could easily be leached as compared to other metals, and almost all Pb (99%) was extracted at all temperatures in 1 h of leaching[12]. Another process consists of leaching lead sulfide concentrate with a ferric chloride-sodium chloride solution to produce lead chloride and fused-salt electrolysis of lead chloride to produce lead metal and chlorine[36,37]. Wong *et al.*[38] also studied a ferric chloride-brine leach in conjunction with electrolysis and found that over 99% of the lead in galena concentrate can be extracted in minutes. Valuable byproducts, such as Ag, Cu, and Zn, can be recovered from the leach solution by simple methods such as cementation and ion exchange, and the chief product, $PbCl_2$, is pure enough to feed directly to an electrolytic cell to obtain corroding-grade metal. Nikkhou *et al.*[39] found that in the leaching of galena with $FeCl_3$, galena particles shrank in size, and a layer of porous elemental sulfur forms. They suggest that the diffusion through the product layer dominates the process of galena leaching in chloride.

On the other hand, Fe dissolution efficiencies remained at very low values, which are favourable in nonferrous hydrometallurgy. During the leaching tests, pyrite showed a little dissolution (about 10% at 40°C), and most of the iron in the solution comes from the dissolved chalcopyrite. Gold dissolution was not studied since Au does not dissolve in the ferric chloride and stays in the leach residues from which it can be recovered.

The effect of temperature on all metal leaching efficiencies, including Ag and Te at constant experimental conditions of 75 g/L ferric ion concentration, 20 g/L HCl concentration, 1/10 S/L ratio, and 8-hour leaching

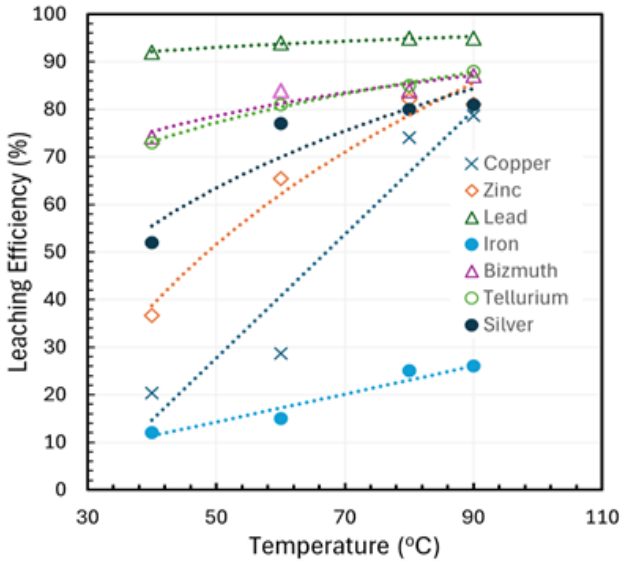


Figure 6. Effect of temperature on metal leaching efficiencies.

time, is summarised in Figure 6. As a result, increasing temperature had an increasing effect on leaching recoveries of all metals except iron. Similar to other metals mentioned above, graphs of Ag and Te also showed an increasing trend with increasing temperature. Maximum Ag and Te efficiencies reached 81% and 88.2% at 90°C, respectively. Leaching temperature of 90°C resulted in the highest metal leaching recoveries. Therefore, considering the extraction of all metals, we can choose 90°C as an optimum leaching temperature.

Leaching of Pb and Ag from copper concentrate in chloride media was also studied, and chloride concentration was less effective where temperature influenced the rate and recovery of metals[28]. The presence of oxidants, which are Fe(III) and oxygen, was favourable for Pb and Ag leaching because of the presence of sulfides of both metals in the feed. Applying alkali pretreatment to the feed for chloride leaching resulted in the most effective 90°C temperature, and under these conditions, the recovery of Pb reached almost 100% after 3 hours of leaching, and almost all of the Ag was recovered after 8 hours of leaching. Correia and Carvalho[14] also investigated ferric chloride leaching of polymetallic concentrates, and it was

possible to leach 100% of Pb in 15 minutes and 80–90% of Ag, Cu, and Zn after four hours of leaching.

Investigation on ferric chloride leaching of tellurium deposit revealed that 99.17% of Te and 92.82% of Bi were extracted at 60 g/L ferric ion concentration at 90°C[40]. They also found that the sulfur element in ore was reduced into an elementary substance, and there was no indication of H_2S production, which is a positive indicator for environmental considerations.

The solubility of silver chloride in various $FeCl_3$-$FeCl_2$-HCl solutions has been measured at the temperature range of 20-100 °C, and it was found that the solubility increased systematically with either rising temperature or increasing concentrations of the chlorides[41]. It was also concluded that silver chloride solubilities at elevated temperatures are sufficiently high that silver solubility limitations should not be a problem in most commercial ferric chloride leaching processes.

The leaching of synthetic Ag_2S in $FeCl_3$-HCl media was also investigated using the rotating disk technique[42]. The Ag_2S dissolved relatively rapidly in $FeCl_3$-HCl media in the temperature range 40-95°C with predominantly elemental sulfur formation. The dissolution kinetics consistently obeyed the parabolic rate law. The addition of chlorides such as NaCl, HCl, or $FeCl_2$, which enhances the solubility of AgCl, also increases the rate of Ag_2S dissolution. The presence of AgCI, however, sharply reduces the leaching rate. Hence, it is concluded that the dissolution rate of Ag_2S is controlled by the outward diffusion of the AgCl reaction product, as various Ag-Cl complexes, through the solution trapped in the pores of the constantly thickening layer of elemental sulfur formed on the surface of the sulfide.

6. Leaching Kinetics

To evaluate the rate-controlling step of the dissolution reaction, the experimental data were analysed according to various model equations[43]. The leaching kinetics of Cu, Zn, and Bi were evaluated using several commonly employed reaction kinetics models, including chemical reaction-controlled (shrinking core) and diffusion-controlled formulations, as well as a homogeneous first-order kinetic approach. Among

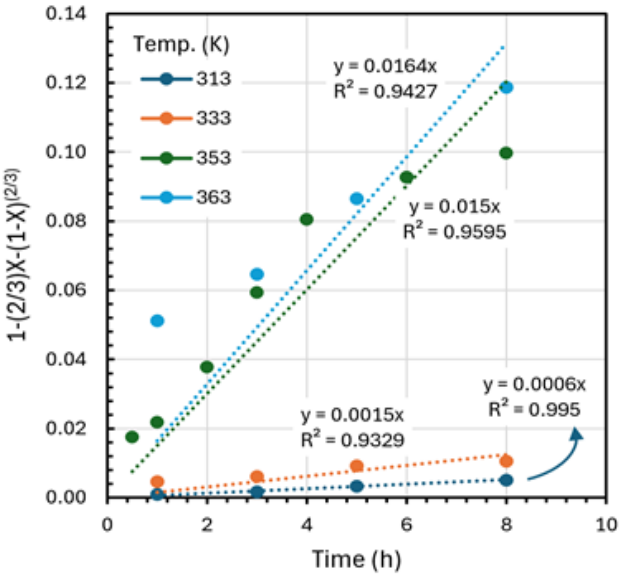


Figure 7. Linearised LSE versus time plots for Cu leaching at different temperatures based on the diffusion-controlled model.

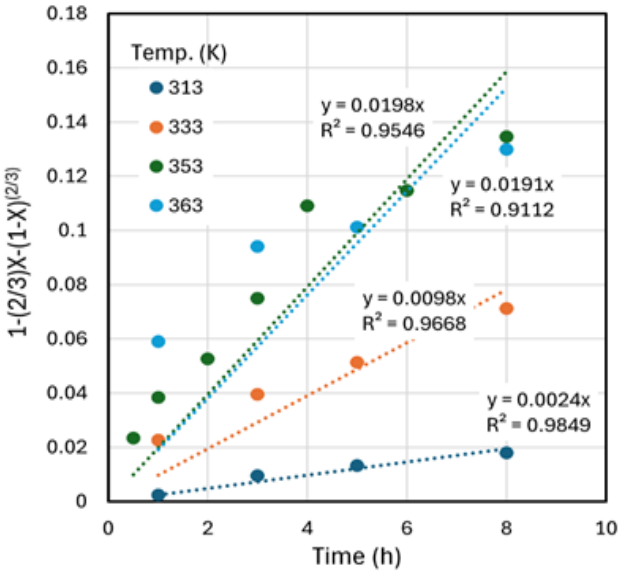


Figure 8. Linearised LSE versus time plots for Zn leaching at different temperatures, based on the diffusion-controlled model.

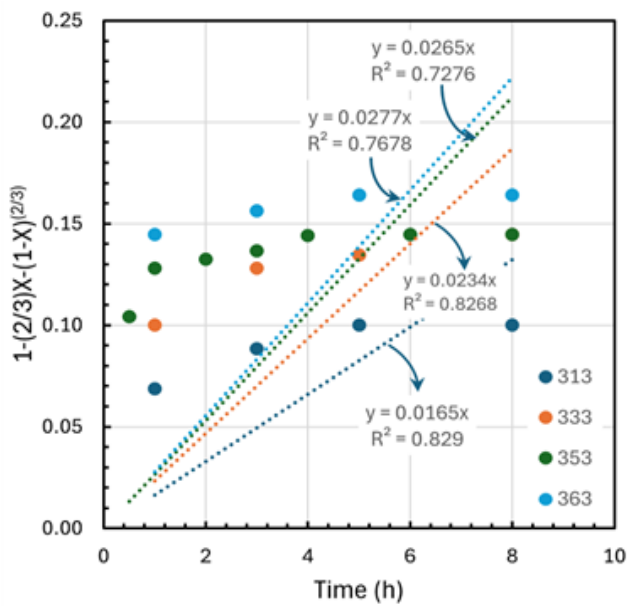


Figure 9. Linearised LSE versus time plots for Bi leaching at different temperatures, based on the diffusion-controlled model.

the tested models, the best linear correlation (highest R^2 values) was obtained for all three metals:

$$1 - (2/3)X - (1 - X)^{(2/3)} = k\,t \quad \text{(Diffusion-Controlled Model)} \tag{6}$$

where X: % dissolution efficiency of metal/100, k: reaction rate constant, and t: time (h).

Accordingly, the kinetic parameter k was determined from the slopes of linear regressions of the Left Side of Equation (LSE) versus time at each temperature. The regression lines were constrained to pass through the origin, where the extent of reaction is zero at zero time. The resulting graphs for Cu, Zn, and Bi at different temperatures are presented in Figures 7, 8, and 9, respectively.

The temperature dependence of the rate constants was subsequently analysed using the Arrhenius equation given as:

$$k = A\,e^{-Ea/RT} \tag{7}$$

where k is the rate constant, A is the pre-exponential factor, R is the gas constant (8.314 J/mol K), T is the temperature in Kelvin, and E_a is the activation energy (kJ/mole).

The Arrhenius plots of ln k versus 1/T exhibited good linearity for all three metals ($R^2 > 0.92$), allowing reliable estimation of the apparent activation energies. The slopes of the regression lines were used to calculate Ea values according to Ea = −(slope)·R. Linear regression of ln k versus 1/T yielded apparent activation energies of 68.76 kJ/mole for Cu, 40.45 kJ/mole for Zn, and 9.67 kJ/mole for Bi from the graph given in Figure 10. These results indicate that the rate-controlling mechanism varies among the metals. The relatively high activation energy obtained for Cu suggests that the overall leaching rate is predominantly controlled by surface chemical reactions, whereas the intermediate value obtained for Zn indicates a mixed control regime involving both surface reaction and diffusion through the product layer. In contrast, the low activation energy determined for Bi is characteristic of diffusion-controlled processes, which shows the tendency of Bi dissolution to approach a plateau at shorter leaching

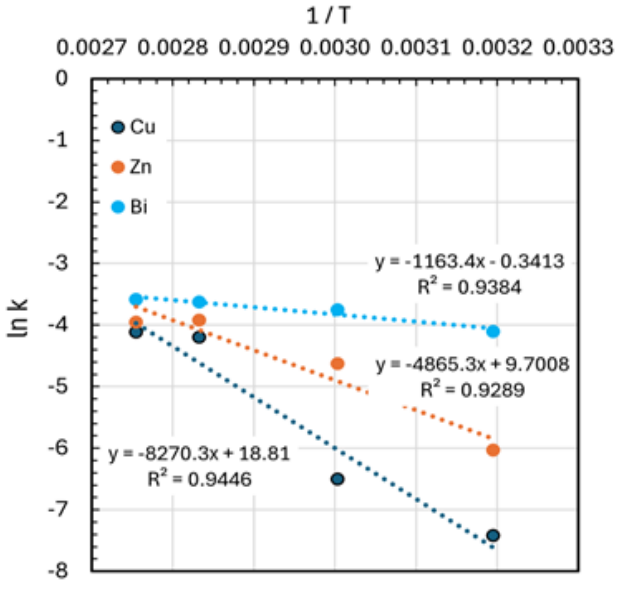


Figure 10. Arrhenius plot of all three metals (Cu, Zn, Bi) investigated.

Table 7. Some examples of apparent activation energies for different metal dissolutions in ferric chloride solutions are found in the literature

Metal Leached	Temperature range (°C)	Activation energy (kJ/mole)	References
Cu	70-100	92.8	[50]
	45-100	42	[51]
	3.5-80	55	[9]
	52-85	69	[52]
	75-96	83	[53]
	55-94	69	[31]
	60-90	43.8	[12]
	25-70	53.9	[29]
	25-106	49.63	[54]
	1-50	24.9	[55]
Zn	40-80	45.30	[56]
	45-90	46.9	[57]
	25-100	42	[58]
	70-90	69	[59]
	50-90	51.3-47.7	[60]
	---	22-38	[61]
Pb	25-50	14.96	[62]
	20-95	62.1	[63]
	28-95	40-45	[65]
Bi	40-95	56	[46]

times. Arslan *et al.*[12] also found an activation energy for copper dissolution of 43.8 kJ/mole in the study with copper ores taken from another region of Türkiye (Küre), and explained that the overall rate of reaction is controlled by the diffusion through the product layer according to the shrinking core model.

Similar leaching studies in the literature report that when several metals are leached from the same ore under identical conditions, the apparent activation energies differ substantially among the metals, reflecting differences in the controlling mechanisms, as shown in Table 7. For copper, the activation energy changes from 15 to 92.8 kJ/mole at temperatures between 3.5 and 100°C, whereas for zinc from 10 to 58.4 kJ/mol at temperatures between 25 and 100°C. In the literature studies, the rate-controlling step for Cu dissolution was found to be the diffusion of ferric ions into the sulfur layer formed on the surface of partially leached ore particles[44]. The literature data given in Table 7 also show activation energies of 14.96 to 60.82 kJ/mole (60-80 °C) for Pb, and 56 kJ/mole for Bi, which we did not calculate in this study.

The value of activation energy determined for copper dissolution in the present study is in the range of literature findings and indicates that the process is diffusion-controlled. The rate-controlling step for copper dissolution was found to be the diffusion of ferric ions into the solution, filling the pores of the sulfur

layer formed on the surface of partially leached ore particles. The scattering results observed on the values of apparent activation energy, as shown in Table 7, can be mainly attributed to admixtures and impurities, as well as the influence of the structure of the material investigated. Significant differences were also observed in the leaching rates of chalcopyrites from different localities under various leaching conditions[45].

Bismuth extraction from mixed molybdenite and bismuthinite ore by leaching in a chloride medium was investigated by Xu *et al.*[46], and various parameters influencing leaching efficiency, such as stirring speed, particle size, acid concentration, and reaction temperature, were examined. Experimental and computational results have revealed that the apparent activation energy was 56 kJ/mole, as shown in Table 7, and the Bi leaching efficiency was over 98%. A reaction control mechanism was found that explains the leaching process, which is explained by the product layer diffusion model. Bismuth ions can combine with chloride ions in solution to form stable ions under high chloride concentrations, and the use of hydrochloric acid in leaching the mixed ore is shown to alter the surface structure by increasing the effective reaction area to improve leaching efficiency and enhance the overall leaching process. In our study, apparent activation energy was found to be 9.67 kJ/mole, lower than the literature findings given in Table 7. The reason for that may come from the ore type difference; in our case, Bi was leached from polymetallic concentrate (Cu-Pb-Zn) compared to mixed molybdenite and bismuthinite ore. In another study, on recovery of bismuth from mechanically activated fine lead concentrates in chloride media, Bi was recovered by 99%, whereas it was 38% without mechanical activation[47]. Physicochemical changes in the complex sulfide concentrate due to mechanical activation accelerate the dissolution of Bi.

A novel lead and bismuth deep-separation extraction-stripping system is proposed for the leaching solution of molybdenite-bismuthinite mixed ore, which creates perfect conditions for the preparation of high-purity bismuth-based materials[48]. The results showed that N235 + 2-octyl alcohol + kerosene formed a synergistic extraction system, which realised the synergistic extraction of Bi, Fe, and Pb with solvent extraction ratios of 98.74%, 9.34%, and 46.24%, respectively. When stripping with water, the stripping ratios of iron and lead were 99.87% and 99.74%, respectively, while the loss ratio of Bi was only 0.01%. In another study, the pilot tests also indicated that greater than 95% of the bismuth was captured from the feed by using SuperLig® 83 and SuperLig® 240 resins[49]. These studies may be good example studies for the recycling process of bi-resources.

7. Conclusion

This experimental study included the distribution of base metals together with rare and precious metals, flotation tests, and the possibilities of their recoveries (Cu, Zn, Pb, Fe, Bi, Te, Ag) from Cu-Pb-Zn complex concentrates with ferric chloride leaching.

Selected ore samples from rich zones of the Murgul-Damar region (Artvin-Türkiye) contained 14.4% Cu, 2.84% Zn, 0.18% Pb, 25.51% Fe, 417 g/t Bi, 118 g/t Te, <100 g/t Se, <50 g/t In, 77 g/t Ag, and 1.2 g/t Au.

Flotation studies with ore sample revealed that a chalcopyrite concentrate containing 24.19% Cu content was produced with 94.4% recovery and a Cu-Pb-Zn complex concentrate containing 6.13% Zn, 0.41% Pb, 4.3% Cu, 22.86% Fe, 1250 g/t Bi, 250 g/t Te, 53 g/t Ag, and 1.4 g/t Au was produced and a tailing containing 0.23% Cu was discarded.

As a result of ferric chloride leaching of Cu-Pb-Zn complex concentrate, the optimum leaching conditions were found as Fe^{3+} ion concentration 75 g/L, HCl concentration 10 g/L, S/L ratio 1/10, time 8 h, and temperature 90°C. Approximately 78.7% of Cu, 81.1% of Zn, 93.2% of Pb, 88.2% Te, 87.1% of Bi, and 81.2% of Ag were extracted under the optimum leaching conditions. Additionally, low Fe dissolution (26.8%) was favourable in metal recoveries from the leachate step. Gold dissolution was not studied since Au does

not dissolve in the ferric chloride and stays in the leach residues from which it can be recovered.

Kinetic studies revealed that the apparent activation energies were calculated as 68.76 kJ/mole for Cu, 40.45 kJ/mole for Zn, and 9.67 kJ/mole for Bi, which are in the range of literature findings. These results indicate that the rate-controlling mechanism varies among the metals. The relatively high activation energy obtained for Cu suggests that the overall leaching rate is predominantly controlled by surface chemical reactions, whereas the intermediate value obtained for Zn indicates a mixed control regime involving both surface reaction and diffusion through the product layer. In contrast, the low activation energy determined for Bi compared to the literature finding is characteristic of diffusion-controlled processes, which shows the tendency of Bi dissolution to approach a plateau at shorter leaching times. Significant differences in activation energies may come from ore taken from different localities, ore mineralogy, accompanying metals and impurities, and various leaching conditions.

These findings showed that Cu, Zn, Pb, Fe, Bi, Te, and Ag can be recovered from polymetallic ores with high efficiencies and promise applicability. Beneficiation and leaching processes used in this study would be applied to similar types of ores from other regions in Türkiye and similar reserves of polymetallic ores in other countries, and also for recycling purposes. Metals dissolved during the ferric chloride leaching could be selectively recovered from leachates by precipitation, cementation, solvent extraction, ion exchange, and electrolysis processes, while Au can be extracted from leach residue by cyanide or thiosulfate leaching.

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Author Contribution

Fatma Arslan: Conceptualization, methodology, data evaluation, writing original draft preparation, review, editing, supervision. Hasan Ahmet Uzunoğlu: Methodology, experimental studies, reporting Cüneyt Arslan: Conceptualization, data evaluation, writing, review, editing, supervision. All authors have read and agreed to the published version of the manuscript.