



Chalcopyrite Leaching in a Dimethyl Sulfoxide Solution Containing Copper Chloride

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Abstract

Chalcopyrite (CuFeS_2) is the most refractory copper mineral when treated in conventional sulfate media leaching systems. This is the first study to examine the use of a dimethyl sulfoxide (CH_3SOCH_3 , DMSO) solution containing copper chloride (CuCl_2) for the leaching of chalcopyrite. Leaching experiments for a copper concentrate composed mainly of chalcopyrite were conducted at ambient pressure at 313–413 K. The leaching fractions of Cu, Fe, S, Au, and As were investigated. It was found that 90% of the Cu was extracted in 2 h, and 94% was extracted in 4 h at 373 K, which is competitive with other conventional processes. A DMSO solution containing CuCl_2 could selectively dissolve the valuable metals Cu and Au from chalcopyrite, but leave minerals with little economic value such as pyrite (FeS_2) and As in the residue. Chalcopyrite is oxidized by cupric ion in proportion to the stoichiometric ratio of Cu in the concentrate to the initial cupric ion in the DMSO solution, which is enhanced by the presence of oxygen below 373 K.

Keywords Arsenic · Copper · Gold · Leaching · Passivation · Pyrite

1 Introduction

Chalcopyrite (CuFeS_2), as one of the most copper-abundant minerals, is an important copper resource. However, chalcopyrite is the most refractory copper mineral when treated using conventional sulfate media leaching systems. The main reason for the slow rate of chalcopyrite dissolution is the formation of a layer on the surface of the mineral that hinders dissolution, termed “passivation” [1]. The slow dissolution of chalcopyrite at ambient pressure in sulfate media has hindered its industrial application. Nevertheless, due to the continuous depletion of high-grade ores, it is expected that future hydrometallurgical alternatives for chalcopyrite treatment will increase in importance and application [2]. Dreisinger reviewed hydrometallurgical options, including Activox, Albion, Anglo American-University of British Columbia, Bactech, BIOCOPTM, CESL copper, Dynatec, and total pressure oxidation processes, to prevent the passivation of chalcopyrite, and compared the advantages and disadvantages of the selected process options [3]. Li

et al. reviewed the structure, fundamental mechanisms, and kinetics of chalcopyrite leaching [4].

Although extensive research on chalcopyrite leaching has been conducted, most studies have focused on using aqueous solutions, and few studies have investigated the use of organic solvents for the leaching of chalcopyrite. Solis-Marcial and Lapidus investigated the use of polar organic solvents in combination with sulfuric acid (H_2SO_4) media for chalcopyrite leaching [5, 6]. They found that the use of acetone (CH_3COCH_3), ethylene glycol ($\text{CH}_2\text{OHCH}_2\text{OH}$), 2-propanol ($\text{CH}_3\text{CH}(\text{OH})\text{CH}_3$), or methanol (CH_3OH) together with sulfuric acid solutions favors the oxidative leaching of chalcopyrite, which results in more copper extraction in a relatively short time under ambient conditions. Ruiz-Sánchez and Lapidus further investigated the role of ethylene glycol in leaching chalcopyrite concentrates with hydrogen peroxide (H_2O_2) in aqueous sulfuric acid solutions under ambient conditions [7]. They concluded that ethylene glycol inhibits H_2O_2 decomposition catalyzed by the copper and iron ions present in the solutions, leading to an effective dissolution of chalcopyrite at room temperature.

Water exists abundantly in nature and has many excellent solvent properties. If water is appropriate for a given purpose, it should be used [8]. The use of nonaqueous solvents instead of water may increase fire, health, and environmental toxicity risks, leading to complicated processes and high risk management

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Table 1 Chemical compositions of Cu conc

Elements	Concentrate, C (wt%)
Cu	24.7
Fe	24.6
S	27.8
Au	0.007
As	0.018

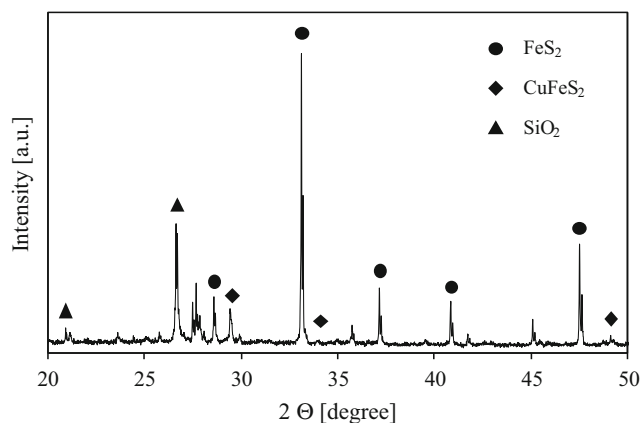
costs. However, it is of interest to employ nonaqueous solvents if they work more efficiently for a certain purpose. Dimethyl sulfide (CH_3SOCH_3 , DMSO) is an aprotic polar solvent that is widely used in chemical industries. DMSO has a number of advantages, including the ability to dissolve many inorganic salts, low toxicity, low vapor pressure at ambient temperature, and resistance to oxidation and reduction. The authors found that a DMSO solution containing copper halide can dissolve precious metals more quickly than other leaching methods under mild conditions. Cu^{2+} in DMSO acts as a strong oxidant that dissolves many metal elements [9].

In this work, the use of a DMSO solution containing copper chloride was investigated for leaching a copper concentrate composed mainly of chalcopyrite. Leaching experiments were conducted to determine the copper dissolution fractions under various conditions. Chalcopyrite is also frequently associated with valuable minerals, such as precious metals, as well as minerals with little economic value, such as pyrite (FeS_2). This necessitates costly separation processes such as leaching and flotation. Moreover, chalcopyrite may also contain “penalty” elements, such as As [10]. Therefore, the dissolution of Au and As in the copper concentrate was also investigated.

2 Materials and Methods

2.1 Materials

Indonesian copper concentrate, which is composed primarily of chalcopyrite, was used in this study. Table 1 shows the core chemical composition of the copper concentrate, which was analyzed using inductively coupled plasma optical emission

**Fig. 1** XRD pattern of residue from Cu concentrate leached by a DMSO solution containing CuCl_2

spectrometry (ICP-OES, Varian VISTA-MPX). The Cu content was 24.7 wt.%; therefore, the approximate composition of chalcopyrite is 70 wt.%. This concentrate contained 0.007 wt.% Au and 0.018 wt.% As. The XRD spectrum (Fig. S1) shows that the copper concentrate contains chalcopyrite, pyrite, and silica (SiO_2). The copper concentrate was dried for 24 h at temperatures above 373 K prior to use in the experiments.

The solution used in this experiment was prepared using reagent-grade DMSO, anhydrous cupric chloride (CuCl_2), and sodium chloride (NaCl), all of which were purchased from FUJIFILM Wako Pure Chemicals. Sulfur (first-grade reagent) was purchased from Junsei Chemical Co., Ltd.

2.2 Leaching of Chalcopyrite

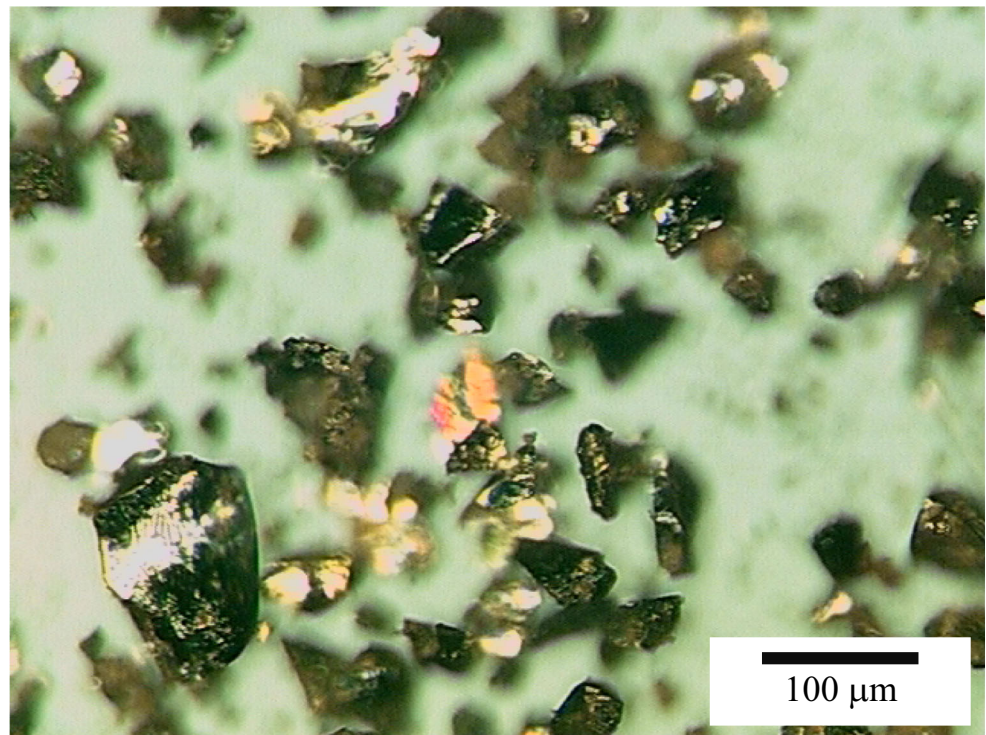
Leaching experiments were conducted according to the following procedure, unless otherwise noted. First, 10 g of concentrate were added to a 1 L eggplant flask for the rotary evaporator, and then the DMSO solution containing copper chloride was added. The solution was prepared by dissolving two equivalents of CuCl_2 and NaCl (78.7 mmol) in 200 mL of DMSO. The stoichiometric ratio of Cu in the concentrate to the initial cupric ion in the DMSO solution was 1:2. Adding NaCl ensures that CuCl_2 dissolves and forms $\text{Cu}(\text{DMSO})_3\text{Cl}^+$, $\text{Cu}(\text{DMSO})\text{Cl}_3^-$, or CuCl_4^{2-} in DMSO

Table 2 The results of the leaching of the copper concentrate for 4 h at 373 K

	Cu conc. (mmol)	Leachate (mmol)	Precipitate (mmol)	Residue (mmol)
Cu	38.9	36.6*	–	2.31
Fe	44.1	20.4*	–	23.7
S	86.8	47.8*	41.1	39.1
Au	4×10^{-3}	3×10^{-3} *	–	1×10^{-3}
As	2.4×10^{-2}	3.9×10^{-3} *	–	2.0×10^{-2}

*calculated value

Fig. 2 Optical microscope image of residue after leaching from Cu concentrate



[11]. Therefore, NaCl enhances the dissolution of metals in DMSO solutions containing copper halides [9].

The evaporator was kept at ambient pressure, and the oil bath temperature was set to 373 K. The eggplant flask was

then immersed in an oil bath and stirred for 4 h at 140 rpm. After leaching, the ore residue in the solution was immediately removed by vacuum filtration to terminate the reactions. The obtained residue was dried in a dryer heated to 383 K for 24 h,

Fig. 3 SEM images of residue after leaching from Cu concentrate. (a) original image, (b) detection for “Fe,” (c) detection for “S,” (d) mapped image by (b) and (c)

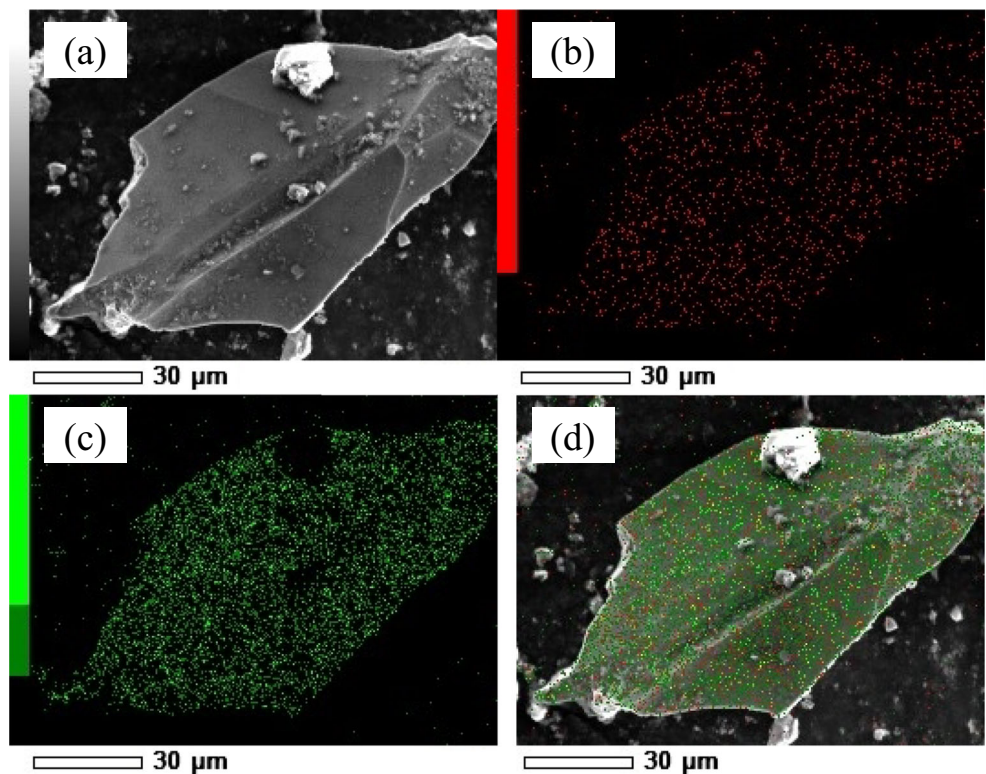
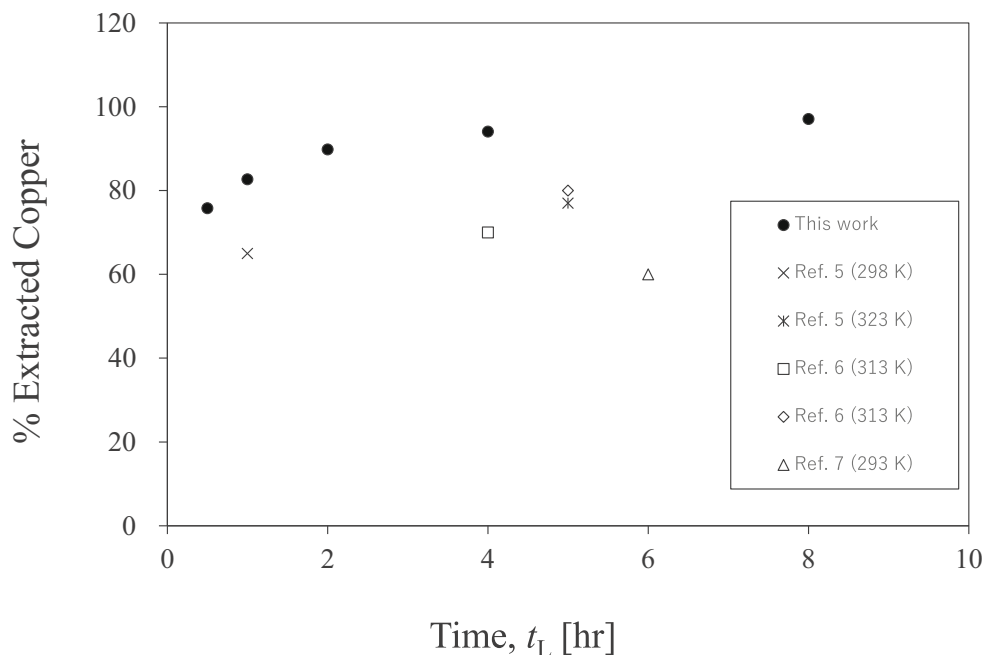


Table 3 Comparison of leaching processes for chalcopyrite

Process	Fundamental research	Fundamental research	Fundamental research	Fundamental research	Fundamental research	Galvanox	The FLSmith® rapid oxidative leach (ROL)
Reference	This work	[5]	[6]	[7]	[13]	[14, 15]	[16]
Media	DMSO + NaCl solutions	H ₂ SO ₄ +acetone solutions, or H ₂ SO ₄ +ethylene glycol solutions	H ₂ SO ₄ +alcohol solutions, or H ₂ SO ₄ +ethylene glycol solutions	H ₂ SO ₄ +ethylene glycol solutions	H ₂ SO ₄ +NaCl solutions	H ₂ SO ₄ +ferrous and ferric salts + silver enhanced pyrite solutions	H ₂ SO ₄ +ferric salts solutions
Oxidant	CuCl ₂	H ₂ O ₂ or CuSO ₄	H ₂ O ₂ or CuSO ₄ and ozone	H ₂ O ₂	Ferric ion + O ₂	Ferric ion + O ₂	Ferric ion (ferric sulfate)+O ₂
Temperature (K)	313–413	298–323	298–323	293	373	353	353
Pressure	Ambient	Ambient	Ambient	Ambient	Ambient	Ambient	Ambient
Extracted Cu within a given length of time	90% in 2 h 99% in 4 h	65% in 1 h 77% in 5 h	70% in 4 h 80% in 5 h 12% in 4 h	60% in 6 h	90% in 3 h	100% in 10 h	97–99% in 6 h
Extracted Fe within a given length of time	53% in 4 h			20–34% in 4 h			

Fig. 4 Cu leaching fractions versus time at 373 K



and the leachate was gradually cooled at 293–298 K for 24 h. A yellow precipitate was formed in the leachate (Fig. S2), collected by filtration under reduced pressure, washed using DMSO, and dried at 383 K for 24 h. The obtained precipitate was measured and analyzed using a scanning electron microscope energy dispersive X-ray spectrometer, SEM-EDS (JEOL JSM-6510A). The results confirmed that the precipitate was sulfur.

The ore residue was placed in aqua regis, a mixture of concentrated nitric acid and hydrochloric acid in a molar ratio of 1:3 and kept at 433 K for 4 h to dissolve all the contained Cu, Fe, S, Au, and As. Then, the Cu, Fe, S, Au, and As contents were quantitatively analyzed by ICP-OES. The

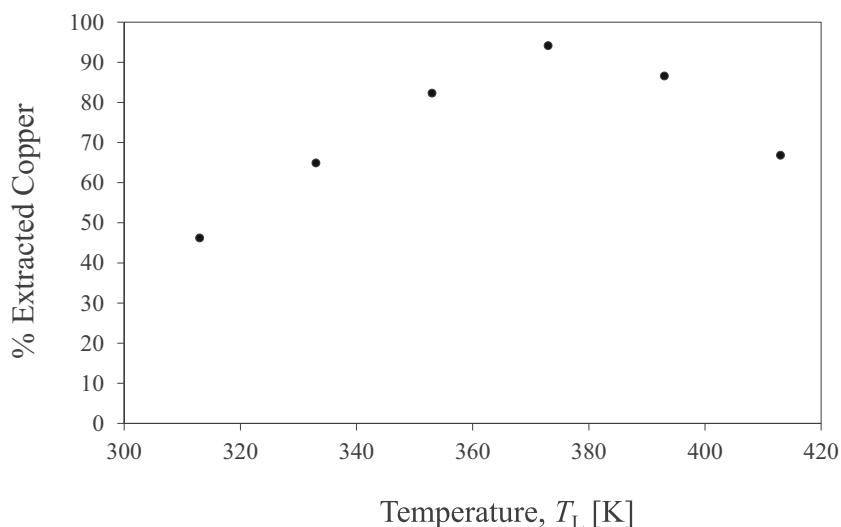
extracted fraction for each element was calculated using the following equation:

$$\begin{aligned} \text{Extracted fraction of } M (\%) & \\ &= 100 - \frac{W_{\text{residue}} \times C_{M1}}{W_{\text{ore}} \times C_{M0}} \times 100 \end{aligned} \tag{1}$$

where M is each element, that is, Cu, Fe, S, Au, or As, W_{ore} is the weight of ore (g), C_{M0} is the concentration of M in the ore (wt.%), W_{residue} is the weight of residue (g), and C_{M1} is the concentration of M in the residue (wt.%).

In addition, the principal components of the obtained residue and their morphology were analyzed and estimated using

Fig. 5 Cu leaching fractions dissolved in 4 h at various temperatures



X-ray diffraction, (XRD, Bruker AXS D8ADVANCE), and SEM-EDS.

2.3 Time and Temperature Dependence of Leaching of Chalcopyrite

To investigate the effect of time on the leaching fraction of copper concentrate, leaching experiments were conducted for 0.5, 1.0, 2.0, 4.0, and 8.0 h. The temperature was set to 373 K. The leaching rate was calculated using the chemical composition of the residue analyzed by ICP-OES.

To investigate the effect of temperature on the leaching fraction of the copper concentrate, leaching experiments were conducted at temperatures of 313, 333, 353, 373, 393, and 413 K. The leaching time was fixed at 4 h. The leaching rate was calculated using the chemical composition of the residue analyzed by ICP-OES.

3 Results and Discussions

3.1 Leaching of Chalcopyrite Using DMSO Solution Containing Copper Chloride

Table 2 summarizes the results of leaching of the copper concentrate for 4 h at 373 K.

The extracted fraction of Cu from the copper concentrate was determined to be 94.1%, as 36.6 mmol of Cu was extracted from the 38.9 mmol present in the copper concentrate. The extracted fraction of Fe was determined to be 46.3%, as 20.4 mmol of Fe was extracted from 44.1 mmol present in the concentrate.

As for S, 47.8 mmol of S was extracted from the 86.8 mmol in the concentrate. The amount of S precipitate in the leachate was 41.1 mmol. As a preliminary experiment, the solubility of S in DMSO was measured to be approximately 20 g (625 mmol)/L-DMSO at 373 K. The solubility of S in DMSO at ambient temperature (298 K) is 0.126 g (3.94 mmol)/L [12]. Therefore, 47.0 mmol of the dissolved S in the leachate would have been expected to precipitate with the decrease in temperature. However, 6.68 mmol of S remained in the leachate even if the recovered precipitate was removed. This is because the generated sulfur crystals were too small to be completely recovered by filtration.

The extracted fraction of Au was determined to be 75% as 3 μmol of Au were extracted from 4 μmol of Au in the concentrate. ICP-OES analysis did not detect Au in the leachate because the concentration of Au was lower than the lower detection limit for liquids, that is, 50 ppm. The calculated Au concentration in the leachate was 2 ppm on a mass basis, which is lower than the lower detection limit of ICP-OES. Although Au was not confirmed to be present in the leachate,

it is expected that the Au contained in the Cu concentrate could be extracted into the leachate.

Examination of the residue showed that 20 μmol of As out of the initial 24 μmol of As remained. The calculated As concentration in the leachate was 1 ppm on a mass basis. Therefore, ICP-OES analysis did not detect As in the leachate. These results suggest that most of the As did not dissolve in the DMSO solution containing CuCl_2 and remained in the residue. This suggests that As could be separated from other valuable metals present in the concentrate.

XRD analysis was performed to identify the components of the residue after leaching, and the results are shown in Fig. 1. According to the XRD spectrum, the main component of the residue is pyrite (FeS_2). An optical microscope image of the residue is shown in Fig. 2, which indicates the presence of pyrite crystals. The residue was further analyzed by SEM-EDS, and the mapping results for each element are shown in Fig. 3. It can be seen that the surface of the residue is a compound of Fe and S. Because the concentrate contains pyrite in addition to chalcopyrite, this suggests that chalcopyrite dissolves preferentially over pyrite when leaching using a DMSO solution containing CuCl_2 .

From the stoichiometric results shown in Table 2, the contents of Fe and S in the residue are 23.7 and 39.1 mmol, respectively. The Fe/S ratio is 0.59, which is larger than the value of 0.5 for pure pyrite (FeS_2). The contents of Cu and Fe in the leachate were 36.6 and 20.4 mmol, respectively. The Cu/Fe ratio is 1.8, which is much larger than the value of 1.0 for pure chalcopyrite (CuFeS_2). Solis-Marcial and Lapidus investigated the reaction mechanism for chalcopyrite leaching when methanol was used in combination with sulfuric acid media and CuSO_4 and O_3 as oxidants at 313 K [6]. Their results showed that iron dissolved in a lesser proportion relative to the copper extraction throughout the leaching process. They suggested that the chalcopyritic iron remained in the residue as FeS. Regarding iron extraction, the same behavior was observed in this study.

The mechanism of leaching the concentrate will be further discussed in the next section.

3.2 Time and Temperature Dependence of Chalcopyrite Leaching

The extracted fraction of Cu versus time is presented in Fig. 4, where the results obtained by previous studies are also shown for comparison. It was found that 90% of the Cu was extracted in 2 h, and 94% was extracted in 4 h at 373 K. Table 3 shows a detailed comparison of the leaching processes for chalcopyrite at ambient pressure. Although the system using DMSO solution is still in a fundamental research stage, the results for extracted Cu and Fe within a given length of time are competitive with other conventional processes. One of the advantages of using DMSO as a solvent is its high boiling point, which

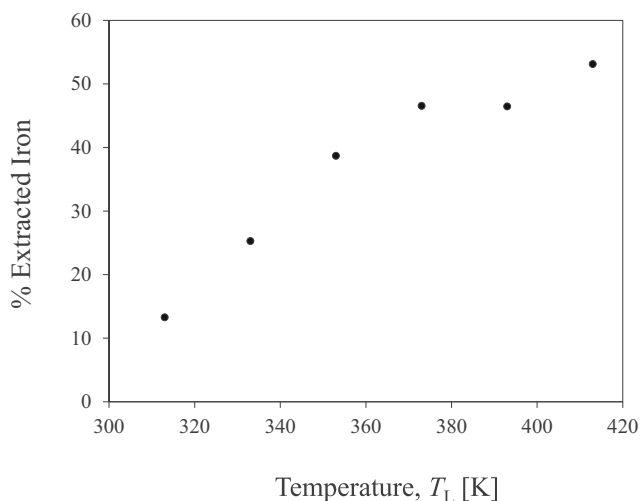


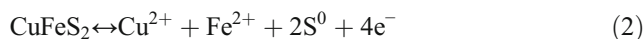
Fig. 6 Fe leaching fractions dissolved in 4 h at various temperatures

allows a high operating temperature at ambient pressure, leading to a high dissolution rate.

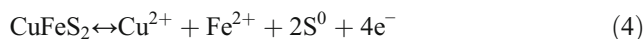
The extracted fraction of Cu in 4 h at various temperatures is shown in Fig. 5. The extracted fraction of Cu increased with an increase in temperature, reaching 94% at 373 K, but decreased with an increase in temperature from 373 to 413 K.

The extracted fraction of Fe in 4 h at a range of temperatures is shown in Fig. 6. The extracted fraction of Fe increased with an increase in temperature up to 373 K. However, unlike Cu, the extracted fraction of Fe did not decrease with an increase in temperature between 373 K and 413 K.

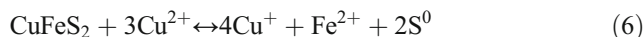
The main reaction for chalcopyrite dissolution in ferric/ferrous sulfate (aqueous) systems consists of the following anodic and cathodic half-cell reactions [2]:



Therefore, the reaction for chalcopyrite dissolution in cupric/cuprous DMSO systems may consist of the following anodic and cathodic half-cell reactions:



Then, the overall reaction is:



If oxygen exists in DMSO solutions, the following anodic and cathodic half-cell reactions take place [17, 18]:

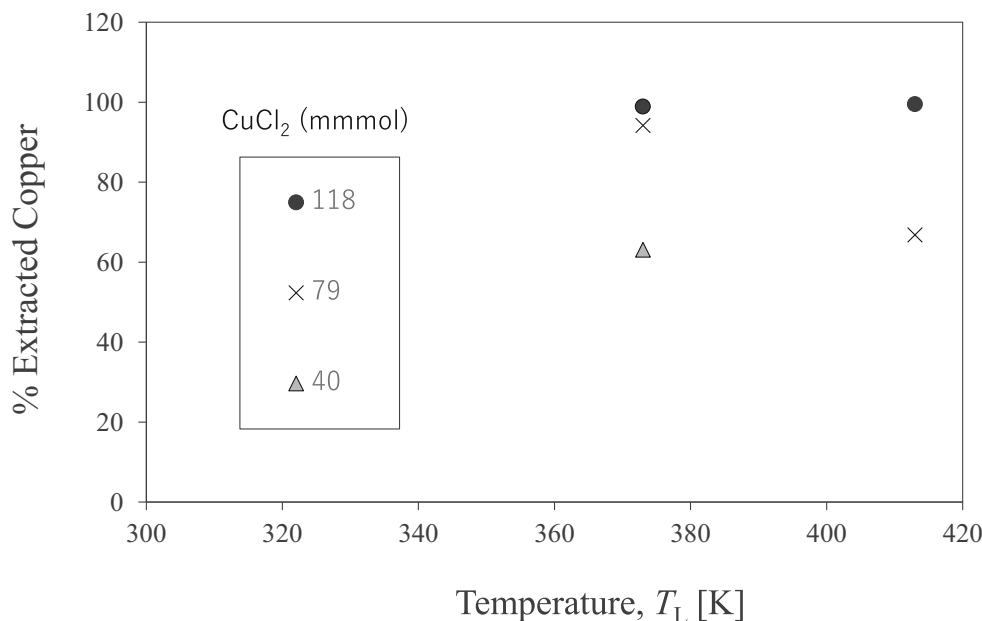


Then, the overall reaction is:



The cupric ion is regenerated by this reaction and acts as an oxidant, as shown in Eq. (6). The ferrous ion may also be oxidized to a ferric ion and act as an oxidant. Therefore, the dissolution of chalcopyrite is enhanced by the presence of oxygen in the DMSO solution. An increase in the leaching temperature enhanced the dissolution of chalcopyrite (Eq. (6)) but decreased the dissolution of oxygen in the DMSO solution, inhibiting the dissolution of chalcopyrite. These are

Fig. 7 Cu leaching fractions dissolved in 4 h at various initial CuCl_2 concentrations in DMSO solution



likely the reasons for the drop in the extracted fraction of copper above 373 K (Fig. 5).

Leaching experiments were conducted for different concentrations of CuCl_2 (40 mmol and 118 mmol) with equivalent NaCl in DMSO for 4 h at 373 K and 413 K. The stoichiometric ratio of Cu in the concentrate to the initial cupric ion in the DMSO solution was 1:1 and 1:3, respectively. The results are shown in Fig. 7. It was observed that the dissolution of chalcopyrite was enhanced with an increase in the concentration of CuCl_2 in the DMSO solution. When the concentration of CuCl_2 in the DMSO solution decreased from 79 to 40 mmol, by approximately 50%, the extracted fraction of Cu at 373 K decreased from 94% to 63%, which was larger than the expected value of 47% based on the stoichiometric decrease in CuCl_2 in the DMSO solution. When the concentration of CuCl_2 in the DMSO solution increased from 79 to 118 mmol, the extracted fraction of Cu at 413 K increased proportionally from 63% to 99%. This result indicates that the leaching of chalcopyrite only takes place based on Eq. (6) at 413 K because of the low dissolution of oxygen in the DMSO solution.

In addition, preliminary leaching experiments for pyrite using the DMSO solution for 4 h at 373 K and 413 K were conducted. No dissolution of pyrite was observed at 373 K, whereas a small fraction of pyrite was dissolved at 413 K (Fig. S3). The increase in the extracted fraction of Fe with an increase in temperature from 393 K to 413 K is likely attributable to the dissolution of pyrite in the copper concentrate. The mechanism of dissolution of copper concentrate should be investigated further.

The recovery of Cu and Au from the leachate should also be investigated in future work. Previous work has shown that the precipitation of dissolved Au and Cu in DMSO solutions can be achieved by the addition of water [9]. Au and Cu from the leachate can be selectively precipitated by controlling the pH of the water. Electrowinning of Au and Cu from the leachate is also one possible method for recovery [19]. These methods should be integrated and optimized.

4 Conclusion

This study showed that a DMSO solution containing CuCl_2 could dissolve chalcopyrite under mild conditions, while not dissolving pyrite. In addition, a DMSO solution containing CuCl_2 dissolves precious metals, such as Au, but does not dissolve As contained in chalcopyrite ore. Therefore, a DMSO solution containing CuCl_2 can selectively dissolve valuable metals from chalcopyrite and leave minerals with little economic value or penalty elements in the residue. The extraction of Cu within a given length of time in this system was shown to be faster than that of conventional methods. Chalcopyrite has been recognized as the most refractory copper mineral when treated using conventional sulfate aqueous leaching systems. One of the

advantages of using DMSO as a solvent is its high boiling point, which allows for a high operating temperature at ambient pressure, leading to a high dissolution rate.

The mechanism of chalcopyrite leaching using a DMSO solution was investigated. Chalcopyrite is oxidized by the cupric ion, which is enhanced by the presence of oxygen below 373 K. The leaching of chalcopyrite takes place depending on the stoichiometric ratio of Cu in the concentrate to the initial cupric ion in the DMSO solution at 413 K because of the low dissolution of oxygen in the DMSO solution.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s42461-021-00400-3>.

Authors' Contributions Conceptualization, Hidekazu Kato and Yasunari Matsuno; Methodology, Kota Takatori, Hidekazu Kato and Yasunari Matsuno; Software, Kota Takatori; Validation, Kota Takatori, Hidekazu Kato, Akihiro Yoshimura and Yasunari Matsuno; Formal Analysis, Kota Takatori and Akihiro Yoshimura; Investigation, Kota Takatori, A.Y. and Y.M.; Resources, Hidekazu Kato; Data Curation, Kota Takatori; Writing – Original Draft Preparation, Kota Takatori, Akihiro Yoshimura and Yasunari Matsuno; Writing – Review & Editing, Kota Takatori, Akihiro Yoshimura and Yasunari Matsuno; Visualization, Kota Takatori and Akihiro Yoshimura; Supervision, Hidekazu Kato, Akihiro Yoshimura and Yasunari Matsuno; Project Administration, Yasunari Matsuno; Funding Acquisition, Yasunari Matsuno.

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Data Availability The data that support the findings of this study are available from the corresponding author, Y.M., upon reasonable request.

Declaration

Conflict of Interest The authors declare that they have no conflict of interest.

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