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Copper leaching from the chalcopyrite-bearing MoS₂ concentrate by mixed chlorides solution

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Abstract: In this study, the dissolution of copper sulfide minerals by the ferric (FeCl₃) and ferrous (FeCl₂) chloride leaching for upgrading the content of molybdenum disulfide (MoS₂) in a molybdenite concentrate was investigated. The effect of various parameters was studied on the copper dissolution behaviour from the concentrate. In this matter, the copper dissolution was reached 94.84 % under the optimized leaching conditions. The kinetics of copper dissolution from the concentrate was established using a shrinking core model (SCM), and the process was controlled by diffusion, with a corresponding activation energy of 18.63 kJ mol⁻¹ at the temperature range of 343–373 K. The amount of copper in the leachate was tested by the inductively coupled plasma-optical emission spectrometer (ICP-OES) and the solid phase was studied by X-ray diffraction (XRD) and scanning electron microscope (SEM). Results of the experiments show that the content of MoS₂ in the solid residue was increased up to 88.59 % after the leaching.

Keywords: molybdenum disulfide; ferric/ferrous leaching; thermodynamic; kinetic models.

INTRODUCTION

The molybdenite concentrate contains the leading mineral of molybdenum disulfide (with formula MoS₂) <90 %, as well as numerous metals sulfide minerals, gangues in the form of oxides, and silicates.¹ The naturally occurring form of MoS₂ is a crucial solid lubricant for additives the purpose of which is to support withstanding of antiwear at extreme pressure, and it is used primarily for reducing friction in heavy conditions. The content of MoS₂ in the concentrate can be upgraded by a chemical refining approach that will be the background in order to obtain MoS₂ with a high purity of 98 %.² Furthermore, MoS₂ is a critical source of metal molybdenum and its other products manufacturing depends on its

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purity.^{3,4} Copper (Cu) commonly occurs in the form of the most abundant copper minerals such as chalcopyrite (CuFeS_2), covellite (CuS), and chalcocite (Cu_2S) in molybdenite concentrate.⁵ The dissolution of undesired copper minerals in the concentrate, particularly CuFeS_2 , which is one of the most complicated phases to dissolve in any liquid medium, requires a suitable technique within the field of the refining process.

We processed the molybdenite concentrate with sodium nitrate solution in a sulfuric acid medium to dissolve copper and iron from the concentrate by selectivity.⁶ Due to this result, the MoS_2 content of 81.33–90.73 wt. % was reached after the leaching. The effectivity of the copper dissolution from the molybdenite concentrate using some oxidizing agents such as sulfuric acid and oxygen pressure,⁷ and the leaching followed the sulfidation process has been reported by several researchers.⁸ Moreover, Jennings *et al.* investigated the leaching mechanism of metals from the molybdenite concentrate with mixed chlorides CaCl_2 – CuCl_2 – FeCl_3 solution.⁹ On this occasion, the extraction rate of Cu, Pb and Ca was up to 98, 98 and 79 %, respectively. In addition, the feasibility of refining the molybdenite concentrate has been demonstrated by the metal impurities removal from the concentrate *via* NaNO_3 – HCl – HNO_3 solution.¹⁰ Under the optimum conditions, molybdenum (Mo) content upgraded up to 59 % for 10 h of leaching.

For many years chloride-based techniques, particularly FeCl_3 , have been applied to treat and extract various metals from sulfide minerals.¹¹ Commonly, ferric (Fe^{3+}) ion is a more effective oxidant with a redox potential of 0.77 mV in the acidic media, high metal solubility, and can be extracted more quickly than other oxidants.¹² FeCl_3 is often used as an oxidant for the oxidation of sulfur in copper sulfides by the leaching approach, and the efficiency is lower when an additional reagent is not included.^{13,14} Therefore, the FeCl_3 solution in the presence of FeCl_2 was selected to dissolve copper from the molybdenite concentrate in this study.

The advantage of operating under FeCl_2 leaching is the significant increase in the boiling point of the lixiviant and H^+ activity. As well, the consumed leaching solution is an enhancement of the revival process where the FeCl_2 can be replaced by FeCl_3 by reaction with liquid chlorine, thus reprocessed for the treatment of unleached copper impurities. Even though the ferric and ferrous (Fe^{2+}) ions oxidation method is used widely for chalcopyrite dissolution,^{15,16} this method has not been used for the leaching of copper from the molybdenite concentrate, particularly the combined process of them.

The present study experimentally investigated the novel process for the dissolution of copper from the concentrate using FeCl_3 – FeCl_2 leaching to upgrade the MoS_2 content in the molybdenite concentrate, without breaking down its structure. The dissolution behaviour of copper from the concentrate was investigated at various intervals of leaching time and the shrinking core models were

used for the kinetic studies. Moreover, the value of Gibbs energy changes of copper dissolution has been estimated by thermodynamic analysis.

EXPERIMENTAL

Materials

In this study, the molybdenite concentrate from the “Erdenet” mining plant in Mongolia was used, with a particle size of less than 0.074 mm of about 91.03 % distribution. According to the chemical composition analyzed by ICP-OES, the main elements Mo, Cu, and iron (Fe) in the concentrate were 49.98, 1.89 and 2.69 %, respectively. Quartz (SiO₂) and sulfur (S) contents in the concentrate defined by chemical methods were 4.62 and 34.86 %, respectively. A peak with high intensity corresponding to Mo and S dominated, and low-intensity peaks of Cu, Fe, and silicon (Si) were exhibited in the initial sample of the molybdenite concentrate by SEM-EDS analysis (Fig. 1). Lixiviants as reagents of analytical grade containing FeCl₃ and FeCl₂, which were used, were prepared with distilled water in this experiment.

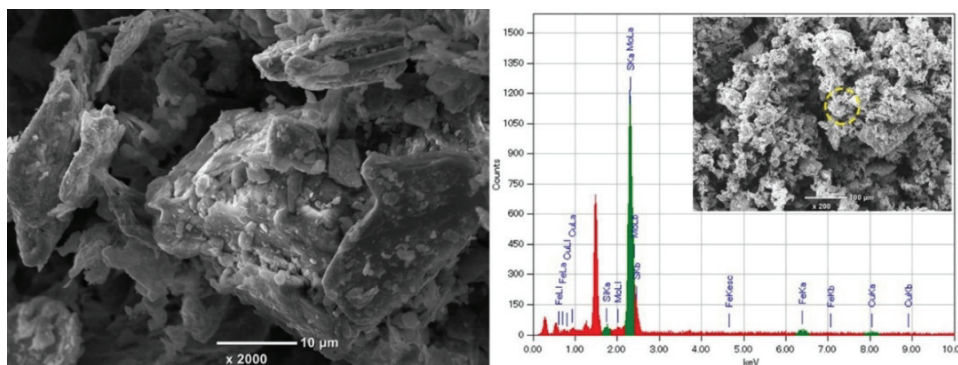


Fig. 1. SEM micrograph of the molybdenite concentrate.

Characterization

The chemical composition of the concentrate, obtained solid products, and solutions were identified by an inductively coupled plasma-optical emission spectrometer (ICP 7200, ICP-OES). The content of SiO₂ and S was determined by chemical gravimetric analysis. The X-ray diffractometer (XRD-7000, Shimadzu) and scanning electron microscopy with energy dispersive spectroscopy (SEM6000-EDS2300, JEOL) were performed to analyse the mineral and elemental composition of the concentrate and obtained solid products.

Experimental procedure

The leaching experiments were carried out in a round bottom glass reactor under solid to liquid ratio varied from 1:10 to 1:2.5, at the temperature range of 343–373 K for 30–240 min with a constant stirring speed of 300 rpm. 10 g of the concentrate was added into 100 mL leach solution containing FeCl₃ (10–30 %) in the presence of FeCl₂ (10–30 %). After completing the experiment, the leach liquor and solid residue were separated by filtration to determine the dissolution efficiency of the copper by ICP-OES analysis. Then, the characterization of solid residue was subjected to observation in XRD and SEM-EDS techniques.

Leaching kinetics

The copper leaching reaction is a heterogeneous process that occurs at the interface of solid and liquid phases. Therefore, the behaviour and mechanism of the dissolution can be explained by the SCM. According to SCM, the leaching process is considered generally to be controlled by the following steps:¹⁷ surface chemical reaction (Eq. (1)), diffusion through the product layer, Eq. (2), or mixed models (Eq. (3)):

$$1 - (1 - \alpha)^{1/3} = k_c t \quad (1)$$

$$1 - \frac{2}{3} \alpha - (1 - \alpha)^3 = k_d t \quad (2)$$

$$[(1 - \alpha)^{-1/3} - 1] + \frac{1}{3} \ln(1 - \alpha) = k_m t \quad (3)$$

where α is the fraction of copper dissolution; t is the leaching time, min; k_c , k_d and k_m are the apparent rate constants, min^{-1} .

Afterward, based on the Arrhenius equation, the activation energy (E_a) could be determined by plotting the $\ln k$ versus T^{-1} :

RESULTS AND DISCUSSION

Thermodynamic analysis

The thermodynamic analysis for defining the probability of reactions between the feed sample and leaching agent was performed using Gibbs energy change calculation (TABLE I). HSC Chemistry 5 software was used to calculate the thermodynamic standard values for all compounds in the reaction at temperatures of 298.15–373.15 K.

TABLE I. The Gibbs energy change (kJ mol^{-1}) of the main chemical reactions¹⁸ for copper dissolution at various temperatures

No.	Main chemical reaction	T / K				
		298.15	343.15	353.15	363.15	373.15
1	$\text{CuFeS}_2 + 3\text{FeCl}_3 = \text{CuCl} + 4\text{FeCl}_2 + 2\text{S}$	-130.90	-131.96	-132.20	-132.44	-132.67
2	$\text{CuFeS}_2 + 4\text{FeCl}_3 = \text{CuCl}_2 + 5\text{FeCl}_2 + 2\text{S}$	-154.29	-154.93	-155.08	-155.22	-155.36
3	$\text{Cu}_2\text{S} + 2\text{FeCl}_3 = 2\text{CuCl} + 2\text{FeCl}_2 + \text{S}$	-93.21	-94.43	-94.70	-94.98	-95.25
4	$\text{CuS} + 2\text{FeCl}_3 = \text{CuCl}_2 + 2\text{FeCl}_2 + \text{S}$	-56.50	-57.11	-57.24	-57.37	-57.51

In addition, the Eh -pH diagrams for Cu-Fe-S-H₂O and Cu-Fe-S-Cl-H₂O systems at 373 K within the pH range from 0 to 6 were constructed using HSC Chemistry 5 software data source, as shown in Fig. 2.

Copper in the molybdenite concentrate existed as copper sulfides and was dissolved by chloride leaching. The Eh -pH diagrams of the Cu-Fe-S-H₂O and Cu-Fe-S-Cl-H₂O systems are presented in Fig. 2a and b. As shown in Fig. 2a,

copper exists at a high potential (over 0.5 V_{SHE}) in the form of Cu²⁺. Moreover, the pH range of the Cu²⁺ stable region is lower than 2.0. Hence, the dissolution of copper from the concentrate without the oxidizing agents is very complicated. Compared with the condition of the Cu–Fe–S–H₂O system,¹⁹ the oxidation potential of Cu²⁺ is enhanced and copper can be transferred in the form of copper chloride into the solution with the presence of chloride at the pH value of less than 2.5 for the Cu–F–C–H₂O system, when the oxidation potential is between 0.5 and 1.5 V_{SHE}.

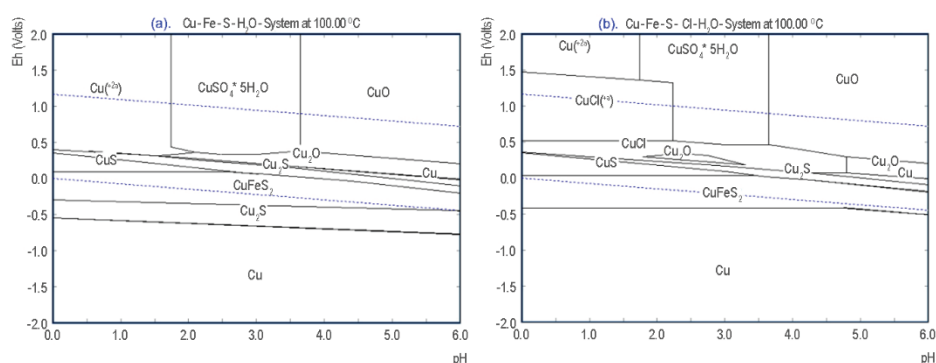


Fig. 2. *Eh*–pH diagram for the investigated systems.

Selection of leaching parameters on Cu dissolution

Effect of Fe(III) chloride concentration. The effect of the FeCl₃ concentration on the copper dissolution from the concentrate was examined, and the result is shown in Fig. 3. When the FeCl₃ concentration increased by 10 to 30 %, the copper dissolution was enhanced from 80.09 to 86.38 % after 60 min. With a further increase in leaching time, the dissolution level tends to be stable during the processes. Even though a suitable leaching time seems like a high yield in a short time of 60 min, it was selected for 240 min in the subsequent experiments to get more dissolution efficiency of copper. According to the experimental results, the FeCl₃ concentration was selected at 10 % in the following experiments.

In the reported work,²⁰ the efficiency of copper dissolution from the chalcopyrite concentrate was about 92 % when the FeCl₃ concentration was 100 g L⁻¹ and the leaching time of 480 min at 95 °C in an acidic medium. However, when the FeCl₃ concentration increased higher, the leaching rate of copper gradually decreased during the process. Some researchers^{21,22} have reported that the existing elemental sulfur at the surface of chalcopyrite, especially its concentrate, is due to the sulfur in the sulfides converted within the elementary form (S²⁻ → S). That is because the oxidation product of sulfides is more probably the formation of elemental sulfur as stated in all reactions (1–4, Table I) in chloride media than sulfate.

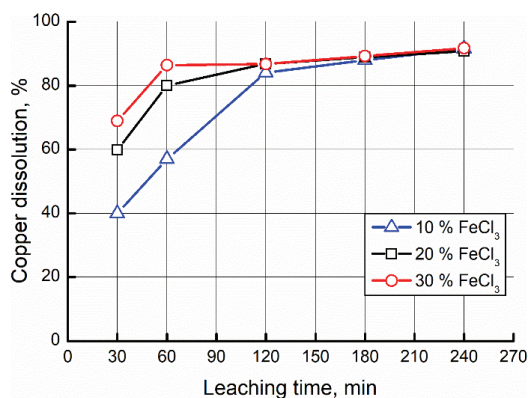


Fig. 3. Effect of the FeCl₃ concentration on the copper dissolution (1:10, 373 K, 20 % FeCl₂).

Effect of Fe(II) chloride concentration. The influence of FeCl₂ concentration was examined between 10 and 30 %, as shown in Fig. 4. The experimental results showed that the copper dissolution increased with an enhanced leaching time. Increasing the Fe²⁺ concentration in the solution affected significantly the copper dissolution. The copper dissolution yield increased from 72.35 to 94.38 % after 240 min, while the FeCl₂ concentration was increased from 10 to 30 %. Otherwise stated, it showed that the enhancement of FeCl₂ concentration positively affected the chalcopyrite leaching. Consequently, a FeCl₂ concentration of 30 % was chosen for the following experiments to enhance the copper leaching rate.

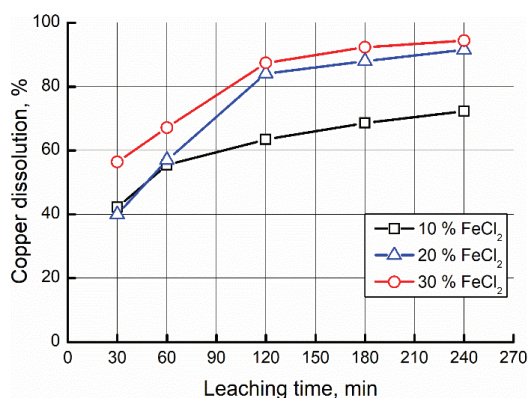


Fig. 4. Effect of the FeCl₂ concentration on the copper dissolution (1:10, 373 K, 10 % FeCl₃).

The FeCl₃ with additional FeCl₂ was used as an oxidant to improve the activity of H⁺ and the boiling point of the leaching solution. At the same time, FeCl₂ can be oxidized to FeCl₃, which could be used for recycling to further leaching of each copper compound from the concentrate. These results indicate that Fe²⁺ is closely related to the copper dissolution. Moreover, Fe²⁺ enhances the kinetics of copper dissolution substantially compared to Fe³⁺.¹⁵

Effect of solid to liquid (S:L) ratio. The leaching experiments were carried out using S:L ratios ranging from 1:2.5 to 1:10. The dependence between the Cu dissolution and phase ratio is represented in Fig. 6. As can be seen, the dissolution of copper was approximately 94.38 % at the S:L ratio of 1:10, when the S:L ratio decreased from 1:10 to 1:2.5, the copper dissolution gradually enhanced after leaching of 240 min. It was shown that a lower S:L ratio had a lesser probability of the reaction for copper dissolution in the short time between the concentrate and oxidants. Moreover, the another reason possible was that the increase in the S:L ratio enhances the medium diffusion of the solution and made the reaction more sufficient. Based on these results, the ratio of 1:10 was used for determining the other parameter.

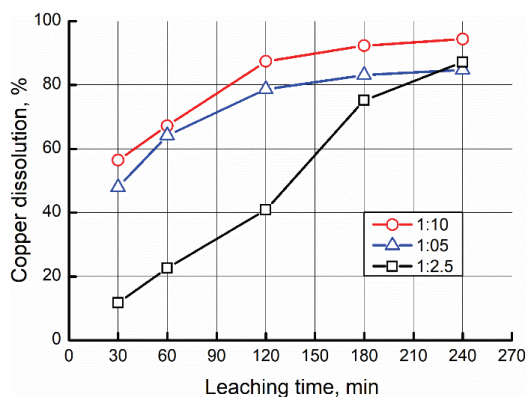


Fig. 6. Effect of the S:L ratio on the copper dissolution (373 K, 10 % FeCl₃, 30 % FeCl₂).

Effect of leaching temperature. The effect of temperature on the copper dissolution was studied at different temperatures of 343–373 K. As can be seen in Fig. 5, the copper dissolution rapidly proceeded up to 63.76–87.43 % at 120 min, and then enhanced remarkably to 78.70–94.38 % when the leaching time was over 120 min. In other words, it was observed that the temperature influence on

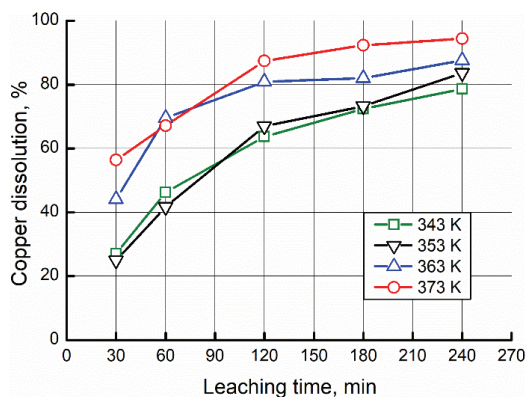


Fig. 5. Effect of the temperature on the copper dissolution (1:10, 10 % FeCl₃, 30 % FeCl₂).

the dissolution of copper diminished after 240 min. It indicates that the breakdown of chalcopyrite structure goes effectively at high-temperature values by $\text{FeCl}_3\text{-FeCl}_2$ for 240 min leaching. Therefore, it was concluded that the appropriate temperature was 373 K, which can be positively influenced by improving the Cu leaching kinetics.

Kinetics analysis

The dissolution efficiency of copper at varied temperatures was fitted by the SCM. The parameters of kinetic models (correlation coefficient, R^2 and rate constant, k) were obtained from the results plotted in a linear relationship according to Eqs. (1)–(3) as shown in Fig. 7. The results could be evaluated by the highest values of R^2 at various temperatures (Fig. 6). As shown in Fig. 7a–c, even though all correlation coefficients are over 0.93, the highest value is 0.99, it was confirmed that the leaching process of chalcopyrite from the concentrate in $\text{FeCl}_3\text{-FeCl}_2$ solution was controlled *via* the diffusion model (Fig. 7d) at the studied various temperatures.

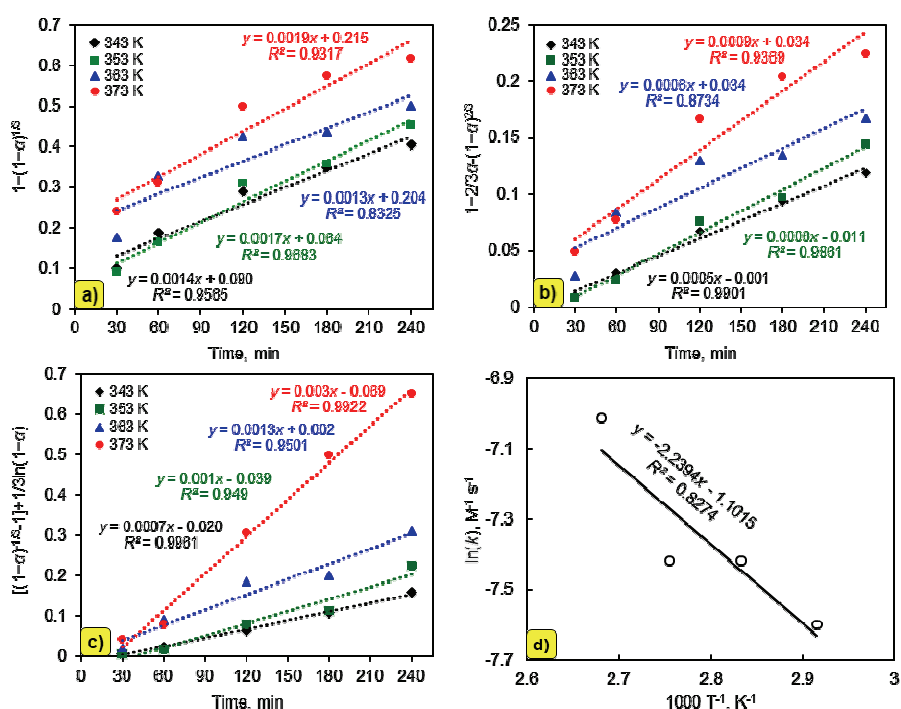


Fig. 7. a)–c) Plots of the models vs leaching time for Cu dissolution at different temperatures; d) Arrhenius plot based on the apparent rate constants calculated from a)–c).

The values of the activation energy of a reaction were calculated as 6.78, 18.62, and 48.93 kJ mol^{-1} for surface chemical reaction controlled, diffusion-

-controlled and mixed-controlled, respectively. The activation energy of the diffusion-controlled model is considered as lower than 20 kJ mol⁻¹ because the temperature was a lower influence on the dissolution of copper. When the value of activation energy is generally more than 42 kJ mol⁻¹, the apparent constant rate is controlled by a chemical reaction. While the activation energy is between 20 and 42 kJ mol⁻¹, the rate-controlling step is expected for a mixed controlled system.²³ Thus, the activation energy obtained confirms that the copper leaching from the concentrate was followed by a diffusion-controlled model.

Leaching residue characterization

The leached residue was obtained with an experiment performed under the optimum conditions (leaching temperature of 373 K, FeCl₃ concentration of 10 %, FeCl₂ concentration of 30 %, solid to liquid ratio of 1:10, leaching time of 240 min, and kept the constant stirring speed of 300 rpm) were examined by XRD and SEM-EDS methods. The content of the elements in the residue was determined by ICP-OES and chemical methods where the main elements as Mo, S and undissolved Si were increased to 52.93, 35.66 and 2.48 %, respectively. In contrast, the amount of Cu and Fe were diminished to 0.12 and 1.77 %, respectively, which suggested that unreacted some chalcopyrite and pyrite particles remained in the residue during the chloride leaching. Moreover, the dissolution process's standard deviation (*SD*) was calculated by statistical analysis (TABLE) constructed within a 95 % confidence interval of repeated measurement (*n* = 3).

TABLE II. Statistical analysis of experimental results obtained under optimal conditions

<i>n</i>	Content of elements in the residue, %			Cu content in solution, %
	Mo	S	Fe	
1	52.93	35.66	1.76	1.80
2	52.99	35.62	1.77	1.78
3	52.86	35.71	1.79	1.80
Mean	52.93	35.66	1.77	1.79
<i>SD</i>	0.065	0.045	0.015	0.009

According to the results of the XRD pattern shown in Fig. 8, the main minerals in the molybdenite concentrate are MoS₂, CuFeS₂ and FeS₂ (Fig. 8a). But some copper minerals, CuS and Cu₂S with minor amounts are not detected due to the detection limit of the equipment. As it can be seen from the X-ray pattern of the residue (Fig. 8b), the main phase was MoS₂ except for that FeS₂ and SiO₂ particles have remained undissolved compounds for the FeCl₃-FeCl₂ leaching system.

The SEM-EDS analysis of the leaching residue after the FeCl₃-FeCl₂ leaching system is shown in Fig. . The intensity of energy peak corresponding to Cu in the residue was eliminated and the intensity of energy peaks related to Mo, S, Fe, and Si was increased after leaching. Fig. displays the EDS spectrum with

highlighted dominant peaks of Mo and S elements in the leaching residue, and it is obvious that chalcopyrite surfaces were effectively dissolved by FeCl_3 – FeCl_2 leaching process. The results suggest that the high-grade molybdenite concentrate with a low amount of Cu could be produced by the combined chloride leaching.

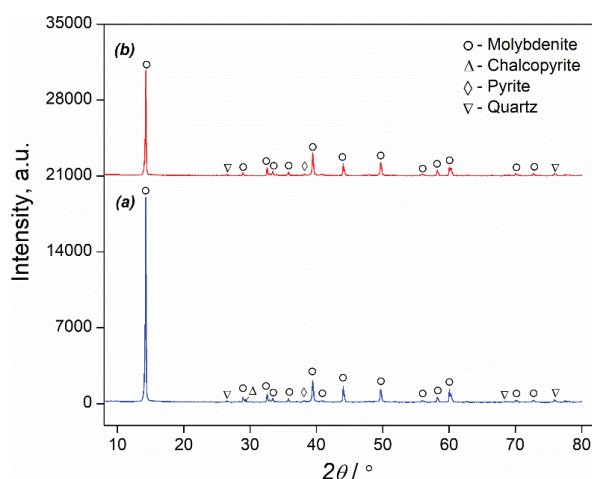


Fig. 8. X-ray diffraction analysis of the molybdenite concentrate (a) and solid residue (b).

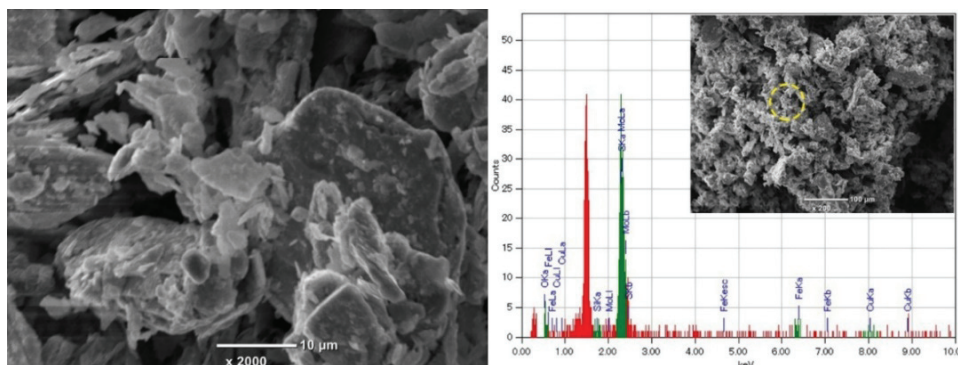


Fig. 9. SEM micrograph of the solid residue.

CONCLUSIONS

A novelty process to dissolve copper from the molybdenite concentrate has been effectively demonstrated by the combined Fe^{3+} and Fe^{2+} leaching methods. It was found that the copper dissolution increased with the FeCl_2 concentration, and the optimum leaching temperature was near the boiling point of the solution. When the amount of solid phase was comparatively lower, which made the diffusion more easy, thus an S:L ratio became superior for the copper leaching from the concentrate. The most efficient yield of copper was 94.83 %, obtained under

the following conditions: the temperature of 373 K, leaching time of 240 min, FeCl₃ concentration of 10 %, FeCl₂ concentration of 30 %, solid to liquid phase ratio at 1:10, and kept the constant stirring speed of 300 rpm. The result of the kinetic study using a shrinking core model suggested that the copper leaching process was controlled by the diffusion model with an activation energy of 18.62 kJ mol⁻¹. The characterization of the leached residues indicated that the copper sulfide minerals were almost completely dissolved in the FeCl₃-FeCl₂ combined solution and remained at 88.59 % MoS₂ aside from SiO₂ and FeS₂ in the residue. Therefore, it can be concluded that a Fe³⁺/Fe²⁺ leaching system is an alternative approach for copper removal from the molybdenite concentrate. However, the novel process can solve the issue of copper leaching from the concentrate, but there are some obstacles to the recovery of iron and silica from leached residue.

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ИЗВОД

ИЗЛУЖИВАЊЕ БАКРА ИЗ КОНЦЕНТРАТА MoS₂ КОЈИ САДРЖИ ХАЛКОПИРИТ
МЕШАНИМ РАСТВОРОМ ХЛОРИДА

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У овом раду испитивано је излуживање бакар сулфида испирањем фери- и феро-хлоридом ради повећања садржаја молибден-дисулфида (MoS₂) у концентрату молибденита. Проучаван је утицај различитих параметара на понашање растварања бакара из концентрата. У овом случају, под оптималним условима лужења постигнуто је излуживање бакара од 94,84 %. Кинетика растварања бакара из концентрата је утврђена коришћењем модела скупљајућег језгра (SCM), а процес је контролисан дифузијом, са енергијом активације од 18,63 kJ mol⁻¹ у температурном опсегу 343–373 K. Процент бакара у процедурној води је тестиран индуктивно спрегнутим плазма-оптичким емисионим спектрометром (ICP-OES), а чврста фаза је проучавана рендгенском дифракцијом (KSRD) и скенирајућим електронским микроскопом (SEM). Резултати експеримента показују да је садржај MoS₂ у чврстом остатку повећан до 88,59 % након испирања.

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