Particle Size Dependence of the Flotation Kinetics and Recovery for Copper-Molybdenum Ore in Seawater

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バージョン：

Particle Size Dependence of the Flotation Kinetics and Recovery for Copper-Molybdenum Ore in Seawater

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Abstract: Copper and molybdenum are often contained within porphyry copper ore, and their efficient recovery can be achieved by flotation, but the detailed flotation behavior and effects of various parameters must be elucidated. In this paper, the effect of different particle sizes on the flotation kinetics and recovery of copper-molybdenum ore is investigated by employing flotation experiments and mineral liberation analysis in seawater. The results indicate that liberation and flotation recovery increase with decreasing particle size and that the gangue mineral ratio is much higher after 35 s of flotation. Moreover, the flotation data are analyzed by fitting them with a first-order kinetic equation, confirming that the kinetics and recovery decrease with increasing particle size.

Keywords: copper; flotation kinetics; Mineral liberation analysis; molybdenum; particle size

1. Introduction

Flotation is a mineral concentration method[1)] that is widely applied in copper ore processing because of its low cost and environmentally friendly nature[2)]. Flotation relies on surface wettability and a high surface area to separate valuable minerals from the useless host material (i.e., gangue); therefore, it is necessary to grind the ore before flotation. Given that crushing and grinding constitute most of the mineral processing cost, it is desirable to minimize the processing time[1)]. However, insufficient grinding decreases the separation efficiency during flotation because many valuable minerals and gangue are combined in middling particles. Hence, it is essential to obtain the optimum particle size to supply the flotation circuit[3,4,5)]. Furthermore, it is helpful to estimate the flotation results based on the flotation kinetics and expected recovery for various flotation times and mineral compositions, to determine the optimum flotation conditions. Figure 1 illustrates the typical flotation results, showing the change in flotation recovery with time[1)]. Most of the recovery occurs in the early stage, and then it takes more time to recover the remaining flotation products, before reaching saturation for the given conditions. In other words, the recovery rate starts high and gradually decreases to zero over time [1,3)]. Figure 2 illustrates the relationship between the particle size and the flotation rate constant for a given condition. Notably, there is an optimum particle size for maximizing the flotation rate[1,3,4)].

Various particle analyses, such as particle liberation and mineralogical distributions, are necessary to determine the optimum particle size for efficient flotation. This information is generally considered to be difficult to obtain; however, mineral liberation analysis (MLA) can be

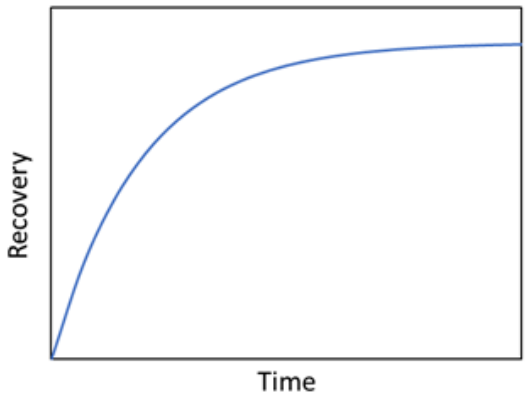

Fig. 1: Schematic Fig. of the relationship between flotation recovery and time[1)]

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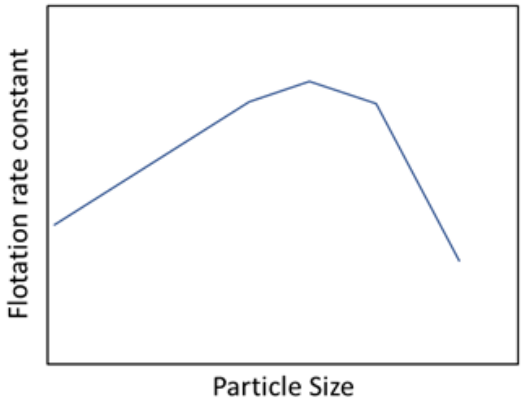

Fig. 2: Schematic Fig. of the relationship between the flotation rate constant and the particle size[4)]

utilized for this purpose. MLA can be employed to determine mineralogical information from the results obtained by scanning electron microscopy and energy-dispersive X-ray spectroscopy (SEM-EDX) results, where each particle is characterized and the statistical data is analyzed[6,7,8)].

In this study, to estimate the effect of particle size on flotation kinetics and recovery, MLA was conducted. Flotation experiments were carried out using various particle sizes, and the flotation kinetics were calculated by fitting the recovery data. Herein, copper-molybdenum ore was investigated with seawater flotation because the kinetic behavior of this important system remains unclarified[9,10)]. Copper ore often associates molybdenum[11,12)] and these efficient recovery is important issue since copper and molybdenum minerals surface are different and flotation characteristics are also quite different[13,14)]. Various treatments are suggested for selective flotation of copper and molybdenum such as oxidation with plasma[15)], electrolysis[16)], hydrogen peroxide[17,18,19)] and reduction[20,21)] in seawater[22,23,24)]. Improvements in copper mineral processing contribute to not only copper production[25,26,27)] but also wastewater[28,29,30)] treatment and environmental remediation[31,32)].

2. Materials and Methods

2.1. Materials

The copper-molybdenum ore sample was mined from a porphyry copper deposit in Chile. From the results of the chemical analysis, the total copper and molybdenum grades were 0.4% and 0.06%, respectively. The mineral composition is presented in Table 1, showing that the total copper and molybdenum mineral assay is 1.1%. The composition of the copper and molybdenum minerals is shown in Table 2. The main copper mineral is chalcopyrite ($CuFeS_2$, 89.0%), and the other copper minerals are bornite (Cu_5FeS_4, 4.1%), chalcocite (Cu_2S, 2.8%), covellite (CuS, 1.6%), atacamite ($Cu_2(OH)_3Cl$, 2.5%), and native copper (Cu, 0.02%). Molybdenite (MoS_2, 95.8%) is the main molybdenum mineral. This mineral composition was determined by MLA, and the method is described in the following subsection.

The sample ore was crushed to less than 6 mm at the mine and then crushed to 1.4 mm or less using a roll-mill-crusher in the laboratory. Then, 0.875 kg of ore sample was mixed with 0.5 L of seawater (65 w/w% pulp density) and ground in a stainless steel rod and ball mill until the determined particle sizes were reached (i.e., 100, 170, 210, 240, and 320 μm P_{80}). Seawater was taken from Niihama in Japan. Table 3 shows the result of chemical analysis of the seawater. The electrical conductivity and pH of the seawater were 48 mS/cm and 7.9, respectively.

Table 1: Mineral composition of the ore sample

Mineral	Assay, wt%
Feldspar	40.4
Quartz	20.7
Mica	21.4
Chlorite	6.3
Oxide minerals	3.1
Clay	0.8
Other minerals	3.1
Pyrite	3.0
Copper and molybdenum minerals	1.1

Table 2: Composition of the Cu and Mo minerals

Copper mineral	Assay, wt%
Chalcopyrite	89.0
Bornite	4.1
Chalcocite	2.8
Atacamite	2.5
Covellite	1.6
Native Copper	0.02
Molybdenum mineral	Assay, wt%
Molybdenite	95.8
Molybdenum oxide	4.2

2.2. Mineral Liberation Analysis (MLA)

MLA was conducted using an automated mineral analysis system based on SEM-EDX (MLA650F, FEI, Hillsboro, USA). After the flotation test, the recovered products were cemented with bakelite resin (sumilite resin PR-50252, Sumitomo Bakelite Co., Ltd.) and then mixed with phenol resin (MultiFast Black, Struers Co., Ltd.) for thermal curing. The cemented sample were polished using abrasive papers followed by SiC paper. Surface is mirror finished with diamond suspension buffing. The resulting specimens were used for MLA. The MLA measurements were conducted with two modes, SPL: sparse phase liberation

mode and XBSE: extended back-scattered electron mode. SPL mode was used to analyze the composition of the copper and molybdenum minerals, and XBSE mode was used to analyze all minerals in the specimens.

2.3. Flotation Tests

The flotation tests were performed with a Denver flotation instrument (D12, Metso Minerals, Inc., Helsinki, Finland) with an impeller speed of 1150 rpm. Thionocarbamate was used as a collector of copper, diesel oil as a collector of molybdenum, MIBC: methyl isobutyl carbinol as a frother, and lime ($Ca(OH)_2$) as a pH control. Figure 3 shows the flotation test process flowchart. The ground ore samples were placed in a flotation cell with a capacity of 2.4 L. The slurry was adjusted to 33% solid with seawater addition. Next, MIBC (8.57 mg/L), thionocarbamate (30 g/t), diesel oil (15 g/t) were added to the slurry. The slurry pH was adjusted with addition of lime (pH 7.6), followed by 1 min of conditioning, and then flotation was carried out. This pH was chosen since Cu-Mo recovery is maximum in seawater due to lower precipitation concentrations according to our previous study[9]. The floated products were recovered after 35, 85, 254, 508, and 1270 s, and the total flotation time was 21 min. This time interval is selected to plot each point, around 2–2.4 times the value of the previous point.

3. Results and Discussion

Flotation tests were conducted on a laboratory scale using samples composed of various particle sizes for 21 min, and the float recovery results of copper and molybdenum are shown in Figure 4A and B, respectively. The recovery of copper and molybdenum decreased with increasing particle size. Specifically, the recovery of copper decreased by 6.6% with each 100 μm increase in particle size, whereas the recovery of molybdenum decreased by 7.5% with the same increments. This decrease in recovery may be because of an increase in middling ore content with the increase in particle size. To investigate this, experiments were carried out using ground ore samples with particle sizes of 170 and 240 μm, and the liberation of each copper mineral was measured. The liberation % was calculated as follows: If the particle exists as only one mineral, it is considered a liberated particle; if the mineral

Table 3: Result of the chemical analysis of the seawater sample

Concentration, mg/L								
Na	Mg	Cl	Ca	Cu	Fe	S	Mo	TOC*
9,800	1,100	17,000	360	< 0.1	< 0.1	790	< 0.1	1

*TOC: total organic carbon

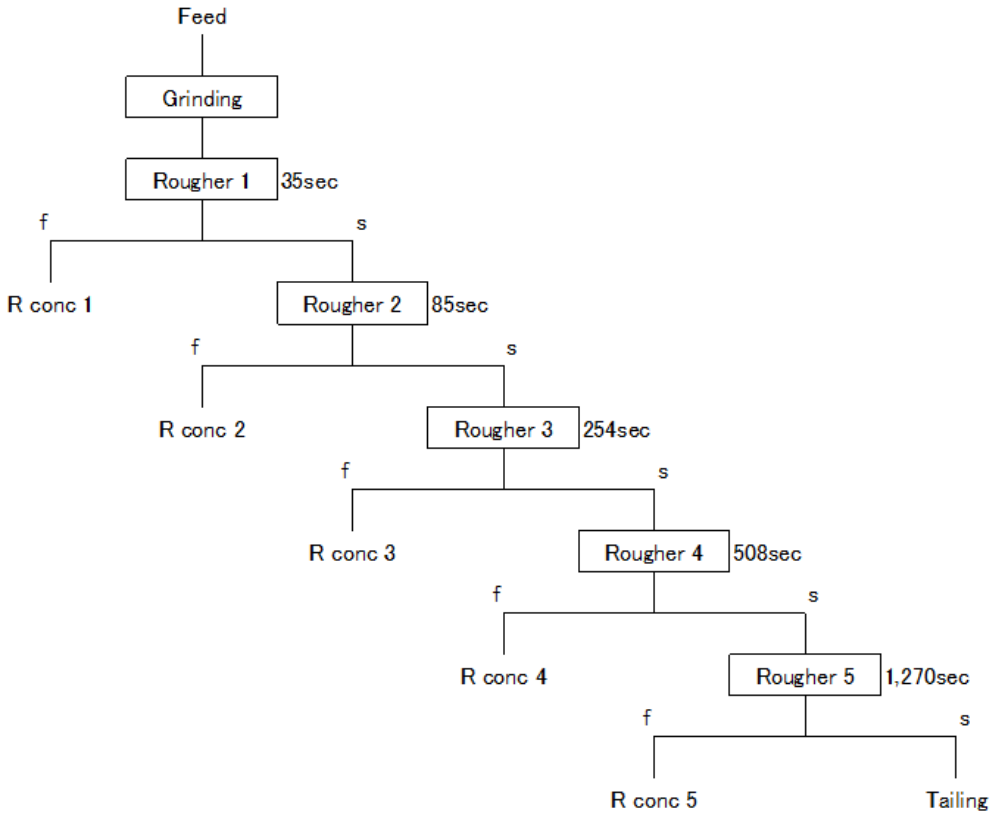

Fig. 3: Flowchart of the batch flotation test process

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is middling, it is not considered a liberated particle, and the liberation ratio (liberated particles/all particles) is multiplied by 100. As shown in Figure 5, the liberation % of chalcopyrite, bornite, covellite, and chalcocite is higher for the 170 μm particles than for the 240 μm particles.

To examine the kinetics, flotation recovery was examined over time using ore samples with different particle sizes, and the results are shown in Figure 6. The float recovery of copper and molybdenum both increase with the decrease in particle size, consistent with the previous results. Additionally, flotation recovery exhibited a higher rate in the early stage of flotation, and then decreased in the later stage.

To further investigate this phenomenon, MLA was conducted for early-stage float products (before 35 s) and late-stage float products (after 35 s), using ore samples with a 170 μm particle size. Figure 7A shows the results obtained before 35 s, and Figure 7B shows the results obtained after 35 s. Before 35 s, the float product contains high concentrations of copper minerals and molybdenite, but a relatively low concentration of gangue. After 35 s, the float product contains considerably lower concentrations of copper and molybdenite, with a relatively high concentration of gangue. The addition of a copper collector (thionocarbamate) and molybdenum collector (diesel oil) promotes the preferential flotation of copper minerals and molybdenite, and their concentrations are higher before 35 s. After 35 s, the middling particles composed of these copper, molybdenum, and gangue minerals can begin to float, and thus the gangue mineral concentration increases. The same analysis was conducted using ore samples with a 240 μm particle size, and the results are shown in Figure 8A (before 35 s) and 8B (after 35 s). A similar trend to that in Figure 7 is observed. However, before 35 s, the chalcopyrite concentration is

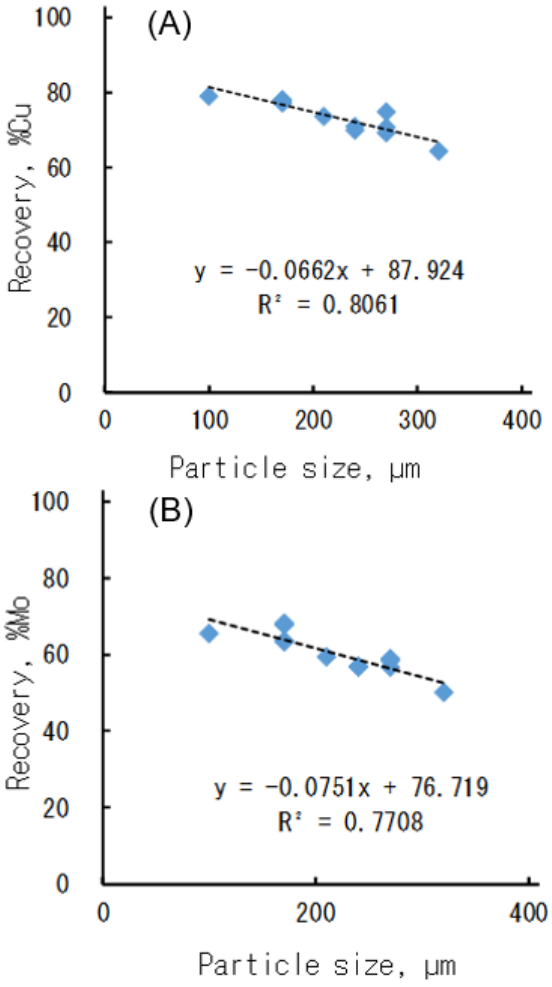

Fig. 4: Flotation recovery of (A) copper and (B) molybdenum from seawater slurries as a function of the ore particle size

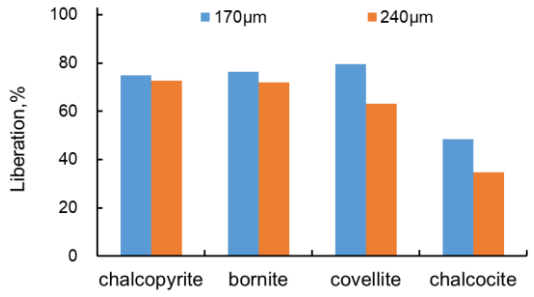

Fig. 5: Liberation % of the chalcopyrite, bornite, covellite, and chalcocite from ore samples composed of 170 and 240 μm particles

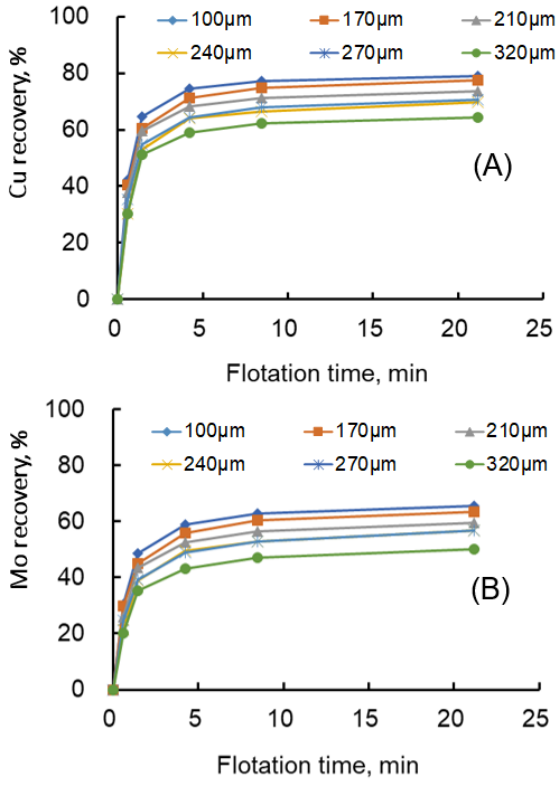

Fig. 6: Flotation recovery of (A) copper and (B) molybdenum from seawater slurries as a function of the flotation time, for samples with different ore particle sizes

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lower for 240 µm particles than for 170 particles, but after 35 s, the chalcopyrite concentration is higher for 240 µm particles than for 170 µm particles. This indicates that when the particle size is larger, more minerals exist as middling particles than liberated particles, and liberated chalcopyrite floats first, followed by middling chalcopyrite. As a result, smaller particles (170 µm) contain more chalcopyrite than larger particles (240 µm) before 35 s, but after 35 s, the larger particles (240 µm) contain more chalcopyrite than the smaller particles (170 µm).

To confirm this, the liberation % was determined for the float products. Figure 9A shows the results for float products obtained with a flotation time of less than 35 s, and Figure 9B shows the results for products obtained with a flotation time of more than 35 s. As shown in Figure 9A, the early stage of flotation (before 35 s) exhibited a high liberation % for copper minerals (chalcopyrite, chalcocite, covellite, bornite) and molybdenite, and the liberation % is higher for samples with smaller particle sizes. Conversely, the later stage of flotation (after 35 s) exhibited a lower liberation % for chalcopyrite and chalcocite. Bornite, covellite, and molybdenite exhibited high liberation, but from Figure 8B, this sample contains these minerals in very low concentrations, and the MLA was not consistent. Nonetheless, these results confirm that liberated copper and molybdenum minerals can float in the early stage of flotation, whereas the middling minerals become liberated in the later stage of flotation. Moreover, particle size can influence liberation and flotation recovery; smaller particle samples float earlier owing to higher liberation.

To estimate the kinetics and recovery of flotation, the results were fitted using a kinetic model that is commonly

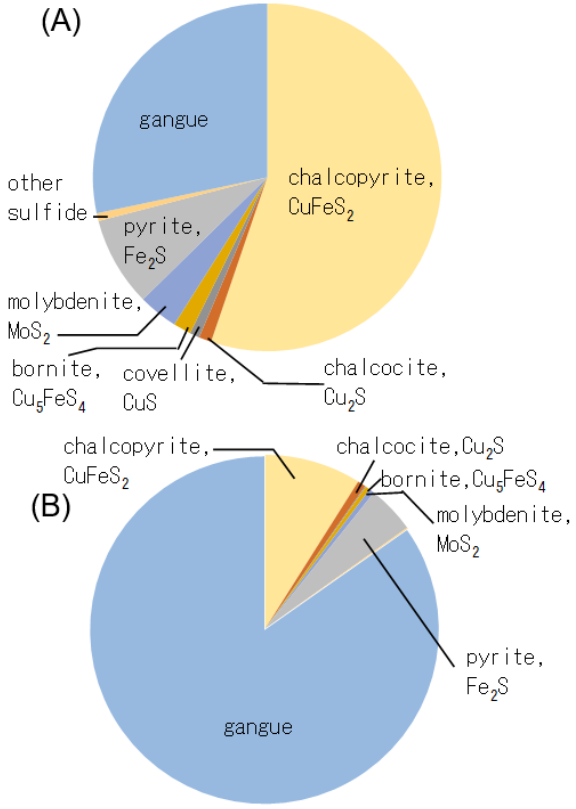

Fig. 7: Mineral composition of float products obtained (A) before 35 s and (B) after 35 s, for ore samples with 170 µm particle sizes

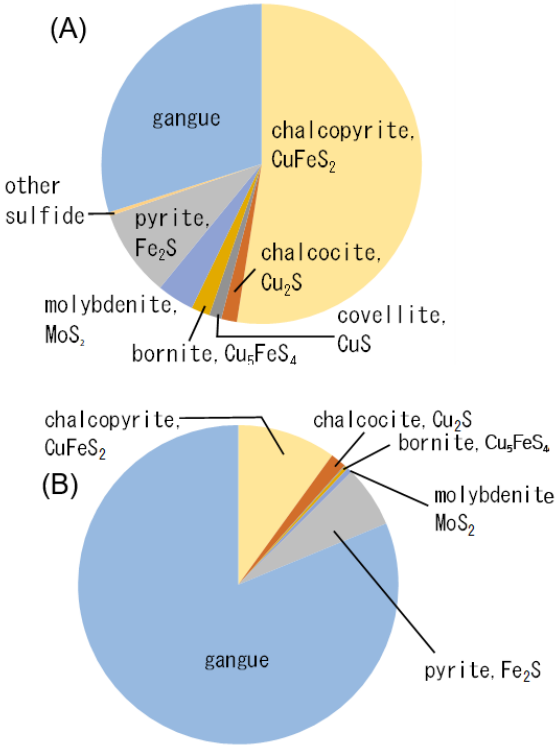

Fig. 8: Mineral composition of float products obtained (A) before 35 s and (B) after 35 s, for ore samples with 240 µm particle sizes

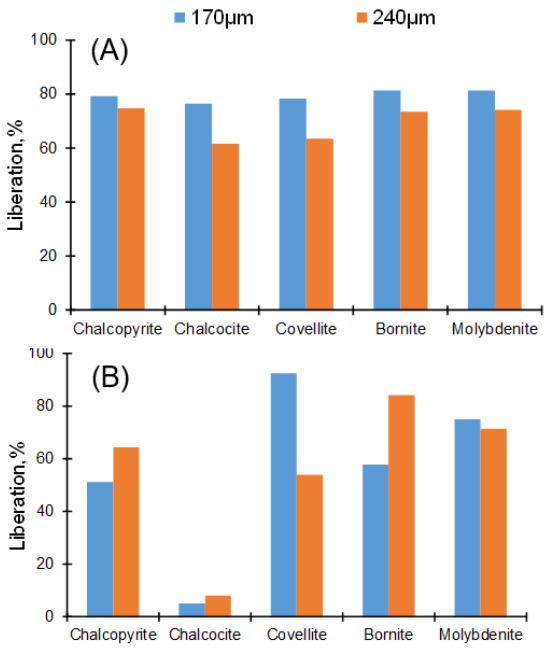

Fig. 9: Liberation % of the float product obtained (A) before 35s and (B) after 35s, for ore samples with 170 and 240µm particle sizes

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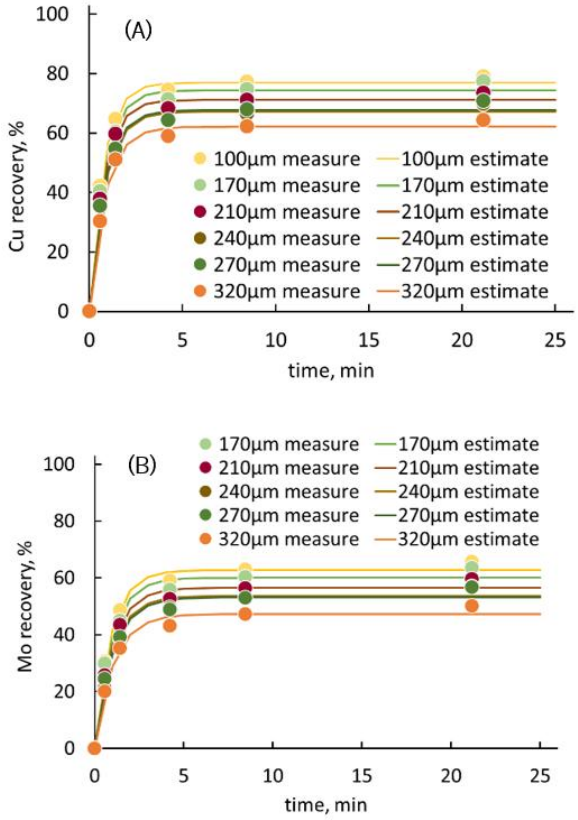

Fig. 10: Various particle size of flotation recovery of Cu (A) and Mo (B) with time and fitted value based on Eq. 1

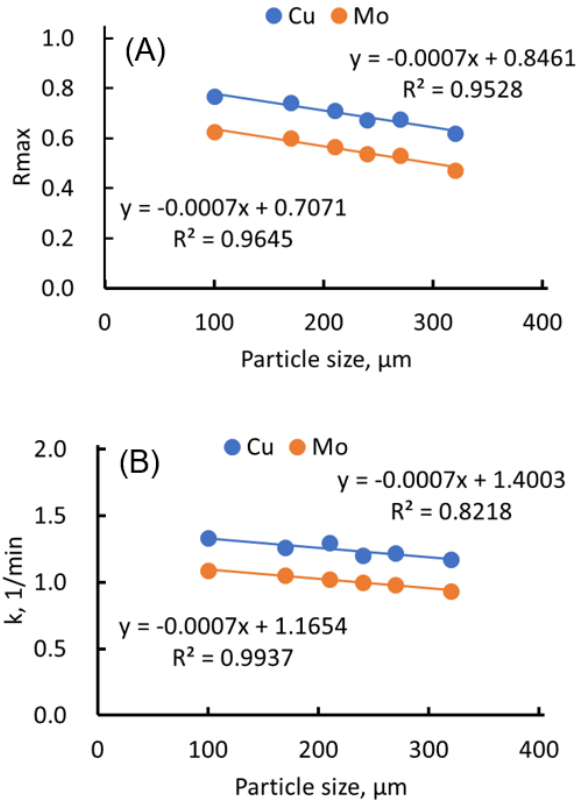

Fig. 11: Effects of sample particle size on the (A) maximum recovery and (B) kinetic constant

applied to mineral flotation[33,34,35].

$$R = R_{max}(1 - e^{-kt}) \quad (1)$$

This equation contains the flotation time (t), recovery (R), maximum recovery (R_{max}), and kinetic constant (k). All flotation results (particle sizes of 100–300 μm, considering Cu and Mo recovery) were fitted using Eq. 1, and the results are shown in Figure 10. Overall, the experimentally measured values are well fitted.

Given that the flotation results are accurately fitted using Eq. 1, flotation results can be estimated based on the maximum recovery (R_{max}) and kinetic constant (k). To evaluate the effects of particle size on these parameters, the relationships between them were examined, and the results are summarized in Figure 11A and B. Both the maximum flotation recovery and kinetic constant decreased with increasing particle size, and their correlations can be calculated. Under this condition, flotation recovery and kinetics are influenced by particle size, and this effect can be quantitatively evaluated.

4. Conclusion

It is important to investigate the effect of particle size on flotation because the crushing and grinding costs can be high, and thus detailed estimation is necessary to minimize the required crushing and grinding time. In this study, a first-order kinetics model was used to estimate the effects of particle size on kinetics and recovery, demonstrating high consistency between the model and the experimental results.

To investigate flotation behavior, mineralogical information and liberation-middling information were investigated by MLA. The flotation experiments indicated that the recovery of copper and molybdenum increased with decreasing particle sizes of the starting samples. This may be because of the higher liberation of minerals with smaller particle sizes. Furthermore, most of the liberated copper minerals float before 35 s, and after 35 s, the float copper minerals are associated with gangue minerals. From the flotation results, a conventional first-order kinetic model can be used in combination with the kinetic constant and maximum recovery to estimate the particle size dependence: With increasing particle size, the flotation kinetics and maximum recovery decrease.

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