Investigation of a Flotation Process with De-Sliming and Attrition to Upgrade and Recover Cu and Mo from a Cu-Mo Flotation Tailing

Jan Pana Rabatho^{1,*1}, William Tongamp¹, Atsushi Shibayama^{1,*2},
Yasushi Takasaki², Sachihito Nitta³ and Tetsuo Imai⁴

¹Faculty of Engineering and Resource Science, Akita University, Akita 010-8502, Japan

²International Center for Research and Education on Mineral and Energy Resources, Akita University, Akita 010-8502, Japan

³Nippon Eirich Co., Ltd., Tower Mill Business Department, Narita 287-0225, Japan

⁴Consultant Tetsuo Imai PE Office, Funabashi 273-0037, Japan

In some (Cu-Mo) mines around the world, Mo recovery is low due to poor liberation, fine dissemination in gangue minerals, and losses of Cu and Mo to tailing streams. Studies to recover these materials as a new resource will increasingly become an attractive option. In the current study, a flotation tailing sample obtained from a mine recovering only 40% Mo was re-floated to upgrade Cu and Mo. The sample containing 0.07% Cu and 0.01% Mo, was used in the study and the goal was to improve the grades to over 2.0% Cu and 0.5% Mo, to obtain an additional feed for the flotation process. Sodium aluminium silicate ($\text{NaAlSi}_3\text{O}_8$) and quartz (SiO_2) were observed as the dominant mineral phases. The original samples were milled and screened to pass 105 μm screen to be used in the subsequent tests. To evaluate the efficiency of the process, both single and multi-stage flotation steps including de-sliming for the removal of fines and attrition for surface activation, together with other parameters (pH, PAX concentration, time) were performed. Based on the experimental results, a four-stage flotation process allowed the improvement of Cu and Mo grades to 1.63% and 0.24% respectively. A single-stage flotation process with de-sliming and attrition stages before flotation with PAX and MIBC concentrations of 40 g/t improved Cu and Mo grades to 2.07% and 0.64%, with a recovery of 48.60% and 65.97% respectively. [doi:10.2320/matertrans.M-M2011803]

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1. Introduction

Copper (Cu) and molybdenum (Mo) usually occur as chalcopyrite (CuFeS_2) and molybdenite (MoS_2) minerals in complex sulphide ores and are mostly associated with pyrite (FeS_2) and silicate gangue minerals. These Cu and Mo sulphide minerals are usually recovered by flotation to improve their grades from below 1.0% to over 25%, and then sent to smelters to recover Cu and Mo.¹⁻⁸⁾ During flotation, some amount of Cu and Mo report to tailing streams as these metals are entrapped in gangue materials and remain unliberated, and are contained in fine slimes, or report to tailings due to process upsets. Huge amounts of such tailings are generated in many (Cu-Mo) mines around the world.

Due to the increased utilization of Cu and Mo in many industries and the rapid depletion of existing ore deposits, research on process development to economically treat low grade deposits, mine and process tailings, resources with high impurities and industrial wastes, will continue to demand high attention.⁹⁻¹¹⁾ Moreover, the treatment of such wastes constitutes reduction on environmental load.¹²⁻¹⁴⁾

The application of flotation to concentrate valuable minerals or the treatment of flotation tailings is environmental-friendly and economically feasible since the materials involved have been reduced to size suitable for direct treatment, and also less volume of material resulting in a reduction of energy requirement for the down stream processes (metal recovery) or containment facility for environmental control. However, the fine materials also pose other challenges, such as; surface oxidation, agglomeration

during exposure to air and moisture, high fine particle contents, and others that will affect flotation conditions, including collector/frother selections and concentrations, extend of surface modification by attrition or addition of promoters and depressants among others.¹⁵⁻²⁴⁾

In some Cu-Mo mines around the world, the recovery of Mo is lower than that of Cu indicating that more Mo is being lost into tailings together with some Cu as unliberated mineral, and finely disseminated in gangue fraction. These tailings could be reprocessed for the recovery of Cu and Mo. In the current study, Cu and Mo contained in sulphide ore flotation tailings and associated with sodium aluminium silicate ($\text{NaAlSi}_3\text{O}_8$) and quartz (SiO_2) as dominant mineral phases, was used as the starting materials to develop a treatment option for their subsequent recovery. Single-stage and multi-stage flotation was performed with or without de-sliming steps. Parameters such as reagent concentrations and slurry pH, were investigated to evaluate best conditions for Cu and Mo recovery.

The main objective of this study was to investigate a process or combination of processes to improve the grades of Cu and Mo in the rougher tailings obtained from Cu-Mo flotation operation, from 0.07% Cu and 0.01% Mo to >2.0% Cu and >0.5% Mo. This upgraded material can be returned as additional resources to the flotation process at stages such as rougher feed stream.

2. Experimental

2.1 Samples

The samples used in this study were obtained from the rougher tails of two different sulphide ores (sample A and sample B). Characterization of the samples by powder X-ray

*1Graduate Student, Akita University

*2Corresponding author, E-mail: sibayama@ipc.akita-u.ac.jp

Fig. 1 The X-ray diffraction patterns of samples A and B.

Table 1 Chemical composition of sample A and sample B (mass%).

Element	Sample A	Sample B
Mo	0.01	0.01
Cu	0.07	0.07
Fe	2.26	1.84

diffraction (XRD) showed similar peaks of sodium aluminium silicate complex ($\text{NaAlSi}_3\text{O}_8$) and quartz (SiO_2) as the dominant phases (Fig. 1). Chemical analysis by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES, SPS 3000, Seiko instrument Inc., Japan) showed that both samples contained similar amount of Cu and Mo (0.07% and 0.01%) as indicated in Table 1. The original samples were first prepared by drying and screening to obtain $-105\ \mu\text{m}$ and size fractions larger than $105\ \mu\text{m}$ were milled to pass $105\ \mu\text{m}$ for use in subsequent tests.

2.2 Flotation

A 0.50 l (500 mL) modified MS agitation-froth flotation machine type laboratory flotation equipment with mixing and froth formation sections separated by a wall with opening at the bottom for the supply of air when needed, was used for all flotation runs (Fig. 2). The sample charge for all tests was fixed at 100 g and was added into 500 mL water to obtain a pulp density of 20%. The slurry pH was controlled or adjusted with the addition of $\text{Ca}(\text{OH})_2$ and HCl. Potassium amyl xanthate or PAX ($\text{C}_5\text{H}_{11}\text{OCSSK}$) and Methyl isobutyl carbinol or MIBC ($\text{C}_6\text{H}_{14}\text{O}$) were used as flotation reagents.

In all flotation tests, MIBC addition was fixed at 40 g/t while the addition of PAX was varied from 40–800 g/t. Three types of flotation experiments were performed to evaluate Cu and Mo recovery; (1) a single-stage flotation, (2) a multi-stage flotation involving addition of flotation reagents to compensate for the loss after froth collection for Cu and Mo analysis, and (3) a multi-stage flotation where after the first-stage flotation, the first tailings was de-slimed to remove fines ($-44\ \mu\text{m}$) followed by flotation with the addition of reagents to collect more froth at each stage.

Fig. 2 Illustration of the 0.50 l laboratory MS agitation-froth flotation machine used in this work.

Multi-stage flotation was performed at four stages with addition of PAX gradually reduced from 200 g/t (first-stage) to 100 g/t (second-stage), 50 g/t (third-stage) and 25 g/t (fourth-stage). The addition of flotation reagents in each stage was to compensate for the loss of flotation reagents after froth collection for Cu and Mo evaluation.

Attrition milling of the samples before flotation was also performed for 10 or 30 min. The pulp density was fixed at 20% solids, while the slurry pH at a range of pH 9.5–11.0. The stirring rate was kept constant (1500 rpm) for all tests.

2.3 De-sliming

After milling the ores, fines in the milled products and in secondary sources, such as tailings, cause slime problems during flotation, and the removal of such slimes is necessary to decrease reagent consumption and improve metal recovery in flotation. In the current study, de-sliming was performed with the tailings from the first-stage of the multi-stage flotation system using a $44\ \mu\text{m}$ screen to remove the $-44\ \mu\text{m}$ slime fractions. The flotation was continued from second to fourth-stage using the $+44\ \mu\text{m}$ fraction.

2.4 Characterization and chemical analyses

Characterization of all solid samples for phase determination was performed using an X-ray diffraction (XRD) analysis equipment with Cu $K\alpha$ radiation source (Rigaku Co., Japan) at a standard scanning speed of $5^\circ/\text{min}$. All solid samples were dissolved in aqua-regia and the solutions were analyzed for chemical composition using an Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES, SPS 3000, Seiko instrument Inc., Japan).

3. Results and Discussion

3.1 Flotation of sample A and sample B

The floatability of samples A and B was investigated in a three-stage flotation process and three different concentrates were obtained to evaluate the recovery of Cu and Mo. In the first-stage, PAX addition was at 40 g/t and after removing froth 1, 20 g/t of PAX was added for the second-stage. After removing froth 2, again 20 g/t PAX was added for the third-stage to collect froth 3. The MIBC addition was fixed

Table 2 Flotation behaviour of samples A and B by monitoring Cu, Mo and Fe, grades in the froth and tailings fractions in a three-stage flotation study. PAX concentration was 40 g/t in the first-stage and 20 g/t in the second and third-stages. MIBC concentration was fixed at 40 g/t and flotation performed for 10 min.

	Sample A (%)			Sample B (%)		
	Cu	Mo	Fe	Cu	Mo	Fe
Feed	0.07	0.01	2.26	0.07	0.01	1.48
Froth 1	0.48	0.06	4.45	0.60	0.04	18.10
Froth 2	0.44	0.09	7.15	0.47	0.03	13.50
Froth 3	0.28	0.06	7.90	0.33	0.04	8.67
Tail	0.04	0.01	1.93	0.04	0.01	0.99

Fig. 3 Cumulative recovery of Cu, Mo and Fe for samples A and B from a three-stage flotation study. PAX concentration was 40 g/t for the first-stage and 20 g/t in the second and third-stages, respectively. MIBC concentration was fixed at 40 g/t and flotation performed for 10 min.

at 40 g/t, and flotation time at 10 min for each stage. The results obtained from the analysis of all solid samples at each stage are shown in Table 2 and the cumulative recovery of Cu, Mo and Fe is shown in Fig. 3.

The grade of Cu for sample A reached 0.48% in the first-stage flotation after which it gradually decreased. The grade of Mo became maximum in the second stage (0.09%) and decreased in the third-stage. Figure 3 indicates that the cumulative recoveries for sample A in the three-stage flotation test, were 49% for Cu and 38% for Mo. Similarly, both Cu and Mo grades in sample B increased using the same three-stage flotation test (Table 2), with cumulative recoveries of 45% for Cu and over 50% for Mo. Compared with sample A, Cu grade in sample B was a little higher, while Mo grade was lower, with Cu a little higher but high Fe recovery in sample A resulted in a slight increase in Cu recovery and this could be due to the association of Cu with pyrite (FeS_2) and chalcopyrite (CuFeS_2), which reported to concentrate during flotation. In sample B, lower Fe recovery slightly decreased the recovery of Cu but increased the recovery of Mo (Fig. 3). The XRD patterns of the concentrate from third-stage flotation for both sample A and B (Fig. 4), indicate the presence of FeS_2 which was not detected in the initial samples (Fig. 1). The tails obtained from the third-stage flotation do not show peaks of FeS_2 (Fig. 5).

Both grades and recoveries of samples A and B were very similar. Therefore, in the subsequent tests, we used only sample A.

Fig. 4 The X-ray diffraction patterns of the combined froth or concentrate from the three-stage flotation for comparison of samples A and B.

Fig. 5 The X-ray diffraction patterns of the final tails from the three-stage flotation for comparison of samples A and B.

Table 3 Effect of different PAX concentrations on the recovery of Cu and Mo in a single-stage flotation. MIBC was kept constant at 40 g/t, slurry pH at 11.0, slurry pulp density of 20% and time for 10 min.

PAX addition (g/t)	Grade (%)		Recovery (%)	
	Cu	Mo	Cu	Mo
40	0.21	0.05	13.98	7.97
100	0.18	0.05	21.60	13.65
200	0.17	0.05	16.40	12.80
400	0.20	0.05	22.17	16.18
800	0.11	0.06	22.29	24.07

3.2 Effect of PAX concentration on floatability of sample A

The effect of PAX concentration on the flotation of sample A was investigated at different concentrations ranging from 40 to 800 g/t in a single-stage process. Other conditions were kept constant (slurry pH 11, flotation time 10 min, MIBC addition 40 g/t and pulp density 20%). Table 3 shows the results of the recovery for Cu and Mo. The results indicate that increasing the PAX addition to

Fig. 6 Flotation behaviour or recovery profile of Cu and Mo of sample A as a function of PAX concentration at pH 9.5 and pH 11. Pulp density of the slurry, MIBC concentration and flotation time were fixed at 20% solids, 40 g/t and 10 min, respectively.

above 40 g/t did not give any significant effect on the recovery of Cu and Mo. Low recovery can be due to poor liberation while the slight decrease in the recovery/grade with the increase in PAX addition can be due to the increased flotation of gangue materials. Generally, the grades remained below 0.2% and 0.06% for Cu and Mo, respectively, at any PAX concentration.

3.3 Effect of pH on the floatability of sample A

Flotation experiments for sample A were performed at pH 9.5 and 11 with varied concentrations of PAX in a single-stage process. The results are shown in Fig. 6. In both pH conditions, increasing PAX concentration did not exhibit any significant increase in the recoveries of Cu and Mo. However, a higher recovery for both metals is achieved at pH 9.5. This could be due to the increased amount of FeS_2 recovered in the concentrate at pH 9.5, indicating the association of both Cu and Mo with FeS_2 . At pH 9.5, the depression effect of FeS_2 was not effective causing some FeS_2 to float and reported in the concentrate resulting in an increase in the recovery of Cu and Mo.

3.4 Effect of attrition time on the floatability of sample A

The original tailing sample containing sulphides and exposed to air and water or moisture, usually undergoes oxidation and agglomeration. The properties of the tailings change and usually undergo sulphide oxidation leading to pH decrease due to the formation of sulphuric acid. To enhance floatability of the sulphide minerals, cleaning of the surface is necessary and this could be achieved by attrition. Attrition increases the surface area of particles exposed to flotation reagents. The results shown in Fig. 7 indicate that the application of attrition for less than 10 min, has a positive effect on the recovery of Cu and Mo with Fe being the least affected. When attrition was applied for more than 10 min, the recovery of Cu and Mo decreased. This could be due to the production of fine particles which cause agglomeration by forming thin layers around particles of valuable minerals and fine particles have high surface areas that require larger amounts of flotation reagents. This means that excessive attrition is a waste of energy, while more fines means more

Fig. 7 Effect of attrition time on the recovery of Cu, Mo and Fe. (Test conditions: pH 9.5, pulp density 20%, MIBC and PAX 40 g/t, time 10 min).

Fig. 8 Distribution of metals in different size fractions of sample A after attrition.

expenditure on flotation reagents. The other reason could be that, MoS_2 is a soft mineral compared to pyrite and chalcopyrite and during longer attrition periods, fine MoS_2 particles are generated and covered pyrite and chalcopyrite particles making them hydrophobic. This may cause a loss of selectivity in the flotation and the accumulation of pyrite in the froth/concentrate, affecting Cu and Mo grades in the froth.

Both PAX and MIBC concentrations were fixed at 40 g/t and a single-stage flotation was performed for 10 min. The Cu and Mo recoveries increased from around 60% for the as received sample to around 80% for sample prepared by attrition for 10 min. This shows that attrition was necessary to expose or activate the sample surface for flotation. However, as attrition time was increased to 30 min, Cu and Mo recoveries dropped to 75 and 65%, respectively. This could be due to slime formation and agglomeration, hindering effective flotation.²⁵⁾

The distribution of Cu, Mo, Fe and gangue fraction (SiO_2) in different size fractions of sample A ground for 10 min is shown in Fig. 8. This figure indicates that higher fraction of the metals report in the $-32\ \mu\text{m}$ size fraction. However, in the fine size fraction, high amount of gangue material (SiO_2) was present, which could have a negative impact on recovery of Cu and Mo during flotation. Therefore, a cut-off size of around $44\ \mu\text{m}$ can be used to remove the $-44\ \mu\text{m}$ size

fraction as slimes. According to the data shown in Fig. 8, about 59% Mo and 54% Cu are found in the size range 53–125 μm , but with lower content of silica fines or slimes. This size fraction could be treated in the flotation process to upgrade and recover both Mo and Cu.

3.5 De-sliming of sample A

De-sliming is a common procedure performed in mineral processing primarily to eliminate fine particles (slime particles) that consume excessive amounts of collector because of their large surface areas and that coat valuable minerals hindering the bubble-mineral contact.

A single-stage flotation test was performed, with or without de-sliming to evaluate effect of de-sliming and the results obtained are shown in Fig. 9. Tests were conducted at slurry pH 9.5 with the addition of 40 g/t of PAX and MIBC. De-sliming was carried out by a screening process to remove the $-44\mu\text{m}$ fraction.

Fig. 9 Behaviour of Cu and Mo recovery during flotation from two different processes: (a) single-stage flotation without de-sliming and (b) single-stage flotation with de-sliming. (Test conditions: pH 9.5, pulp density 20%, MIBC and PAX 40 g/t, time 10 min).

For test without de-sliming, high Mo recovery was achieved (60%) since it is an easily floatable mineral and responds fast to flotation, while Cu recovery was only 30%. For tests with de-sliming or screening to remove the $-44\mu\text{m}$ fraction, the recovery of Cu increased to over 45% together with Fe (45%). The recovery of Mo decreased by over 10% and this could be attributed to the loss of Mo to slimes which were removed by de-sliming.

3.6 Single and multi-stage flotation of sample A

In the single-stage flotation, the tailing sample was first screened using a 44 μm screen to remove the $-44\mu\text{m}$ fraction as fine slimes and the $+44\mu\text{m}$ fraction was milled (attrition) for 10 min, prior to flotation to recover Cu and Mo (Fig. 10). Both PAX and MIBC additions were fixed at 40 g/t and, at the pH and pulp density of 9.5 and 20% respectively. The chemical analysis of the concentrate obtained indicated improvements in Cu and Mo grades to 2.07% and 0.62%, respectively, and their recoveries were 48.6% Cu and 66.07% Mo. The results given in Table 4 show that the removal of the $-44\mu\text{m}$ fraction constituting fines, and the surface activation of the $+44\mu\text{m}$ fraction by attrition before flotation, improved both grade and recovery. The tests performed with attrition first followed by screening (dotted line in Fig. 10) gave lower grades of both Cu ($<1.0\%$) and Mo ($\sim 0.5\%$) and this could be attributed to the

Table 4 Comparison of grade and recovery of Cu and Mo from single-stage flotation with an attrition step and a multi-stage flotation with a de-sliming step.

	Grade (%)		Recovery (%)	
	Cu	Mo	Cu	Mo
Feed	0.07	0.01		
Single-stage	2.07	0.62	48.60	65.97
Multi-stage	1.63	0.24	23.58	33.82

Fig. 10 Experimental flowsheet for the single-stage flotation process incorporating screening to remove fines and attrition. Dotted line (---) represents (attrition + screening) process.

agglomeration of the fine gangue fraction onto Cu and Mo bearing mineral surfaces during attrition.

In the multi-stage flotation, pulp density and slurry pH were the same as the single-stage flotation, and PAX and MIBC were added step-wise at four stages and froths were collected at each stage. The MIBC addition was kept constant but PAX addition was reduced gradually at each stage beginning with 200 g/t at the first-stage, 100 g/t, 50 g/t and 25 g/t for stages two to four respectively. After the first-stage flotation, the tailing fraction was screened using a 44 μ m screen to remove the fines as slimes and used the +44 μ m fraction in flotation stages two to four. Overall, the final grades of Cu and Mo in the multi-stage flotation were 1.63% Cu and 0.24% Mo (Fig. 11 and Fig. 12). According to these results and the Cu and Mo grades from the single-stage

Fig. 11 Grades of Cu, Mo and Fe from froth or concentrates obtained after the fourth-stage flotation for (a) multiple-stage without de-sliming and (b) multiple-stage with de-sliming and compared against grades in original sample A. (pH 9.5, pulp density 20%, MIBC and PAX 40 g/t, time 10 min).

flotation, (Cu, 2.07%; Mo, 0.62%) both screening to remove fines as slimes and attrition to activate mineral surfaces for subsequent flotation are necessary to improve the metal grades.

Figure 11 shows a comparison of the effect of de-sliming on final grades. It can be seen from the figure that both tests (with and without de-sliming) produced similar results. A schematic flowsheet of the multi-stage flotation process is shown in Fig. 12. From this figure, it can be seen that the grades of Cu and Mo in the first-stage were only 0.13% and 0.04%, respectively, as compared to the grades of the same metals (2.07%, Cu, 0.62%, Mo) in single-stage with the screening step to remove fines and the attrition step to activate mineral surface, (Table 4).

In the multi-stage flotation, after the de-sliming process, Cu and Mo recoveries significantly increased to 1.63% Cu and 0.24% Mo at the fourth-stage. At the same time, the recovery of Fe dropped to below 10% from over 20% in the earlier stages (first and second stage). This could be due to the depression in FeS₂ floatability at pH 9.5, resulting to the increase in Cu and Mo grades.

The Cu and Mo grades, including Fe, obtained in the fourth-stage of the multi-stage flotation were lower than Cu and Mo grades obtained in the single-stage flotation. The decrease in the recovery of Cu and Mo in the first-stage of the multi-stage flotation is thought to be due to fines or slimes in the sample (Fig. 8) which was removed before flotation in the single-stage after attrition (Fig. 9).

3.7 Process evaluation

The main objective in this study was to investigate a process or combination of processes to improve the grades of Cu and Mo in the rougher tailings obtained from a Cu-Mo flotation operation, from <0.1% to >2.0% Cu and >0.5% Mo. This material can be returned as an additional resource to the flotation process, at a certain stage such as rougher feed stream.

Fig. 12 Schematic diagram of multi-stage flotation process including a de-sliming stage performed in the current study with summarized results.

Several process options have been investigated and the results were discussed in the manuscript. The negative effect of slimes in flotation has been discussed. In this study, the removal of fines (de-sliming) followed by an attrition process to activate mineral surfaces as a sample preparation stage prior to single-stage flotation was enough to meet the test objective.

4. Conclusion

The main objective in this work was to apply conventional flotation technique to upgrade Cu and Mo contents in a Cu-Mo flotation tailings containing Cu and Mo below 0.1% to over 2% Cu and 0.5% Mo to be used as an additional resource to the flotation circuit for possible recovery.

The findings in the current study showed that a single-stage flotation process with a screening-step to remove fines to reduce slimes during flotation and an attrition-step to activate mineral surfaces prior to flotation, were observed as the best condition to improve both Cu and Mo grades. Final results showed that Cu grade has been improved from 0.07% to 2.07% and Mo from 0.01% to 0.62%, giving recoveries of 48.60% Cu and 65.97% Mo. A moderate addition of PAX and MIBC at 40 g/t, slurry pH of 9.5 and flotation time of 10 min were sufficient to achieve these results.

Such a process can be developed to treat millions of tons of flotation tailings containing both Cu and Mo and offer as an additional resource for the flotation stream for possible recovery, constituting both resource recovery and reduction of environmental load.

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