Original Paper

Effect of Flotation Reagents for Upgrading and Recovery of Cu and Mo from Mine Tailing by Flotation

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Abstract

Application of flotation for upgrading and recovery of copper (Cu) and molybdenum (Mo) contained in flotation tailings from a copper/molybdenum mine was investigated in this study. The sample contained mainly pyrite (FeS₂) and quartz (SiO₂) as dominant phases with Cu and Mo contents at 0.38% and 0.23% respectively. A flotation system employing use of collector/frother mixture [kerosene + sodium dodecyl sulphate (SDS) + polyethylene alkyl ethyl] with tannic acid and sodium silicate as FeS₂ and SiO₂ depressants, compared against potassium amyl xanthate (PAX) and methyl isobutyl carbinol (MIBC) additions were investigated to evaluate Cu and Mo recovery. According to the results, the grades of Cu and Mo in the concentrate did not increase to above 1% when PAX and MIBC were used as collector and frother, even at increased PAX addition. Also grades for tests performed at varying pH rangig from pH 3 to 11 showed no significant improvement. However, with the use of the collector/frother mixture and tannic acid, Cu and Mo grades increased to 1.4 and 2.4% respectively. The grade of Mo was further increased to 8.0% at 52% recovery when sodium silicate was used with the collector/frother mixture. These results indicate that Cu and Mo lost in such tailings can be upgraded and offered as additional resource for possible recovery.

Key words: Flotation reagents, Flotation, Mine tailings, Copper, Molybdenum, Pyrite

1. Introduction

Many copper sulphide ore deposits occur with molybdenum, mainly associated with pyrite and silicate gangue minerals. Flotation is mainly applied to recover both copper and molybdenum sulphides and their grades are usually upgraded to over 25% for feed to smelting operations. In some Cu and Mo mines around the world, during bulk flotation of Cu and Mo, recovery of Mo is usually lower than that of Cu¹. This is usually attributed to by poor liberation, slimes coating, process upsets, resulting in both Cu and Mo reporting to tailing streams and huge amounts of these wastes remain in many parts of the world. Due to increased utili-

zation of Cu and Mo metals in many industries and rapid depletion of existing ore deposits, research on process development to economically treat low grade deposits, mine and process tailings, resources with high impurities, industrial wastes will continue to demand higher attention^{2,3}.

Molybdenite (MoS₂) is highly hydrophobic and floats easily among other sulphide minerals, but its enrichment or concentration by flotation is not always simple. The enrichment by flotation of ores containing clay gangue has resulted in the decrease of the recovery of molybdenum to just below 40% recovery. Treatment of such wastes by flotation constitutes reduction on environmental load and economically feasible since material has already been reduced to size for direct treatment which is also widely discussed in literature⁴⁻⁷.

Table 1 Chemical composition of the original flotation tailing sample

Element	Cu	Mo	Fe	SiO ₂	Others
Content (wt%)	0.38	0.23	36.66	37.37	25.36

However, the materials also pose other challenges such as surface changes during exposure to environment, fines, poor liberation and so on 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. Such challenges are discussed by many other researchers^{8–16}.

In this study, Cu and Mo resources contained in sulphide ore flotation tailings associated with FeS₂ and SiO₂ was used as starting material to develop a flotation treatment option for their subsequent recovery. Flotation parameters including slurry pH, collector and frother concentrations, addition of FeS₂ and SiO₂ depressants, time were investigated to evaluate Cu and Mo recovery.

2. Experimental

2.1. Sample

Flotation tailings samples from a rougher circuit were obtained from an operating copper and molybdenum mine in Asia. The contents of Cu and Mo from chemical analysis were 0.38% and 0.23% respectively (Table 1) and characterisation by X-ray diffraction (XRD) analysis showed that the sample contained FeS₂ and SiO₂ as dominant phases (Fig. 1). The distribution of Cu and Mo in different size fractions evaluated by ICP is shown in Fig. 2.

2.2. Reagents

All reagents used where chemical grade received from Wako Chemicals Japan. PAX (Potassium amyl xanthate, C₅H₁₁OCSSK) and MIBC (Methyl Isobutyl Carbinol, C₆H₁₄O) were used as standard collector and frother respectively. SDS (Sodium Dodecyl Sulfate, C₁₂H₂₅OSO₃Na), polyethylene alkyl ethyl and kerosene were mixed together to prepare collector/frother mixture. Purity of both SDS and polyethylene alkyl ethyl were 90% and 0.11 g samples each were dispersed in 100 ml distilled water to prepare 0.1% concentration strength solution. A 3 ml of kerosene was added to the 100 ml solution containing SDS and polyethylene alkyl ethyl and mixed thoroughly before use as collector/frother mixture. 0.1% concentration strength Tannic acid (C₃₄H₂₈O₂₁) and

Fig. 1 Characteristic XRD pattern of the original flotation tailing sample. **Fig. 2** Distribution of metals in different size fractions.

sodium silicate (Na₂SiO₃·xH₂O, n = 5, 6, 8, 9) for FeS₂ and SiO₂ depression were prepared separately to be used together with collector/frother mixture in flotation.

2.3. Flotation

A 0.5 L modified fahrenheit (FW) type laboratory flotation cell with mixing and froth formation sections separated by a wall with opening at the bottom for the supply of air when needed was used in all tests. Samples were charged at 50 to 125 g into 500 ml of water to obtain pulp densities ranging between 10 and 25%. Either H₂SO₄ or NaOH were used to adjust slurry pH before addition of reagents for further conditioning and flotation.

Varying PAX, MIBC and collector/frother mixture concentrations, slurry pH, flotation time, addition of tannic acid and sodium silicate depressants and their concentrations were performed to investigate flotation behaviour of Cu and Mo for their upgrading and recovery.

2.4. Characterization and chemical analyses

Characterization of all solid samples for phase determination was performed using an X-ray dif-

Table 2 Effect of PAX concentration on Cu and Mo recovery (Flotation conditions: Pulp density at 20% and slurry pH 11, MIBC addition at 40 g/t and time at 10 minutes)

PAX addition (g/t)	Concentrate			
	Grade (%)		Recovery (%)	
	Cu	Mo	Cu	Mo
40	0.21	0.05	13.98	7.97
100	0.18	0.05	21.6	13.65
200	0.17	0.05	16.4	12.8
400	0.2	0.05	22.17	16.18
800	0.11	0.06	22.29	24.07

fraction (XRD) analysis equipment with Cu K α radiation source (Rigaku Co., Japan) and a standard scanning speed of 5°/min) was used to analyse the sample. Chemical composition of all solid samples after dissolving in aqua-regia were analyzed using an inductively Coupled Plasma-Optical emission Spectroscopy (ICP-OES, SPS 3000, Seiko instrument Inc, Japan).

3. Results and Discussion

3.1 Effect of PAX concentration on flotation

PAX as collector and MIBC as frother commonly used in flotation of sulphide minerals was first used to investigate their effect on flotation of Cu and Mo. Results from flotation experiments with PAX concentration ranging from 40 to 800 g/t and MIBC addition fixed at 40 g/t, pulp density at 20%, slurry pH 11 and time of 10 minutes are presented in Table 2. The grades of both Cu and Mo showed no improvement even at increased PAX concentration and the grades of both metals remained below 0.2% in the concentrate fraction. This result indicates that minerals hosting both Cu and Mo are contained in FeS₂ and SiO₂ where both report to the tailings fraction as FeS₂ is also depressed at pH 11.

3.2 Effect of pH on flotation

Maintaining PAX and MIBC concentrations at 100 g/t and 200 g/t, respectively, flotation behaviour of Cu and Mo was investigated at varying slurry pH ranging from pH 3 to 11 and the results obtained are presented in Table 3 and Fig. 3. Pulp density was maintained at 20% and flotation was performed for 10 minutes, slurry pH was adjusted by adding either H₂SO₄ or NaOH.

The grades of both Cu and Mo at pH 3.0 in the concentrate were extremely low at 0.18% Cu and 0.05% Mo. The grades of both metals increased as

Table 3 Results showing effect of pH on flotation of Cu and Mo (Flotation conditions: 20% pulp density, PAX and MIBC additions at 100 g/t and 200 g/t, flotation time 10 minutes)

Slurry (pH)	Concentrate			
	Grade (%)		Recovery (%)	
	Mo	Cu	Mo	Cu
3	0.18	0.05	21.6	13.65
5	0.38	0.7	5.29	5.79
7	0.72	0.94	4.52	2.88
9	0.9	0.81	6.83	6.78
11	0.66	0.74	6.25	4.35

Fig. 3 Flotation behaviour of Cu and Mo in flotation tailings sample as a function of pH. Flotation time was fixed at 10 minutes, PAX addition at 100 g/t and slurry pulp density at 20%.

pH was raised from pH 5.0 to pH 11.0, but did not show any significant increase to above 1%. Also their corresponding recoveries remained consistently low and in most cases below 10%. The decrease in the recovery could be as the results of the instability of xanthate at acid pH because at acidic conditions the stability of xanthate is not stable and decomposes thus affecting the performance of xanthate, also Cu and Mo particles interactions with gangue mineral, may cause slime coatings on the Cu and Mo particles, therefore, reducing their flotation recovery by reducing their surface contact with the collector. However, an increase in pH has a positive effect on grades of Cu and Mo. At alkaline conditions the floatability of Cu also improved compared to FeS₂ because of the depression effect on FeS₂ and other gangue minerals including silicate.

Table 4 Flotation behaviour of Cu and Mo with collector/frother mixture prepared from sodium dodecyl sulphate, polyethylene alkyl ethyl and kerosene (Flotation conditions: Pulp density = 20%, collector/frother mixture = 50 to 100 g/t, time = 10 to 30 minute, slurry pH = 11)

Reagent (g/t)		Concentrate				Flotation time (minute)
		Grade (%)		Recovery (%)		
		Cu	Mo	Cu	Mo	
SDS + Polyethylene alkyl ethyl + kerosene	50	0.55	1.27	17.67	66.53	10
		0.72	0.78	32.5	57.38	20
		1.11	1.28	32.29	60.82	30
	100	1.19	1.45	42.25	66.34	25
	Kerosene	50	0.38	2.7	2.95	34.11
SDS	50	1.21	0.5	18.8	12.71	
Polyethylene alkyl ethyl	50	0.95	3.49	4.31	25.69	
SDS + Kerosene	50	1.02	0.87	45.02	62.85	

Reagents

SDS (0.001%) + Polyethylene alkyl ethyl (0.001%) + kerosene (3% in water)

SDS (0.001%) + Kerosene (3% in water).

3.3 Effect of collector/frother mixture on flotation

The effect of kerosene (commonly used in coal flotation), sodium dodecyl sulfate (SDS) and polyethylene alkyl ethyl prepared as a collector/frother mixture and used for flotation of Cu and Mo was investigated and results are shown in Table 4. Slurry pH and pulp density were maintained at pH 11 and 20% solids, collector/frother mixture at 50 g/t while flotation time was ranged between 10 and 30 minutes.

When collector/frother mixture (kerosene + SDS + polyethylene alkyl ethyl) was used, the grades of both Cu and Mo increased as flotation time was increased from 10 to 30 minutes: At 25 and 30 minutes, the grades reached over 1% for both Cu and Mo with recovery for Cu ranging between 32 and 42% while that of Mo ranging between 61 and 66%. Flotation tests with kerosene, SDS, polyethylene alkyl ethyl separately and mixture of kerosene + SDS mixture were performed to see their effect against mixture of the three reagents also shown in the same table (Table 4). Each of them does not show any consistent grade increase and recoveries are also lower when compared with the collector/frother mixture.

3.4. Effect of depressants on flotation**3.4.1. Tannic acid**

The use of tannic acid at varying concentrations (50 to 130 g/t) to depress FeS_2 and observe flotation behaviour of Cu and Mo was investigated in this section and the results are given in Table 5.

The collector/frother mixture (kerosene + SDS + polyethylene alkyl ethyl) addition was fixed at 50 g/t for all tests, pulp density at 20%, pH 11 and flotation time ranged from 10 to 30 minutes. At all conditions investigated, the grades of both Cu and Mo have been increased to over 1%. A flotation time of 10 minutes and tannic acid concentration of between 75 to 100 g/t was sufficient to achieve these results with Mo grade reaching closer to 2%. The recovery of Cu ranged between 15 and 28% while Mo recovery reached up to 61%.

A 25 g flotation concentrate sample obtained from several tests at 150 g/t of tannic acid addition and flotation time of 30 minutes was treated using 75 g/t of tannic acid and 10 minutes flotation time. This was done as an additional step to further remove silica and other gangue minerals entrapped with the concentrate as the cleaning stage. Both Cu and Mo grades have been further increased to 2.71% Cu from 1.32% and 7.40% Mo from 1.85% respectively, and recoveries increased to 48% Cu and 85% Mo. XRD pattern of the concentrate before and after cleaning stage are shown in Fig. 4. After cleaning stage FeS_2 and SiO_2 peaks shortened as FeS_2 and SiO_2 were further removed by cleaning and at the same time MoS_2 peaks increased as the purity and grade increased. After cleaning, MoS_2 became the most dominant phase indicating that Mo in the tailings sample can be recovered during flotation by using the collector/frother mixture for flotation with tannic acid as FeS_2 depressant.

Table 5 Effect of tannic acid addition and flotation time on the recovery of Cu and Mo (Flotation conditions: 20% pulp density; flotation time 10 to 30 minute; collector and frother mixture addition fixed at (50 g/t) and pH 11)

Flotation time (minute)	Tannic acid concentration (g/t)	Concentrate			
		Grade (%)		Recovery (%)	
		Cu	Mo	Cu	Mo
10	50	1.09	1.48	26.64	58.7
	75	1.14	1.99	14.74	41.95
	100	1.23	1.9	19.84	50.35
	125	1.42	1.61	27.62	51.02
	150	1.29	2.4	17.25	53.83
20	150	1.16	1.64	26.8	61.55
30	150	1.32	1.85	26.02	59.18

Fig. 4 Characteristic XRD patterns of concentrate samples for tests performed with collector/frother mixture + tannic acid to recover Mo. (a) Concentrate 1 (collector/frother mixture + 150 g/t tannic acid for 30 minutes; (b) Concentrate 2 (cleaning/flotation of concentrate 1 with collector/frother + 75 g/t tannic acid for 10 minutes). Slurry pH, pulp density and collector/frother addition were fixed at 11, 20% and 50 g/t respectively.

The selective flotation of Mo can be explained as follows: Kerosene reacts as oil and the buoyancy of oil is the physical fact that most minerals can float. Oil (kerosene) has specific gravity less than that of water and when mixed with water and agitated with a stirrer, froth is formed. This froth rising to the surface of the liquid mixture carried particles of sulphides and this buoyancy is not

dependent only on oil, it needs bubbles to attach to the surface and carry particles to the surface of the slurry, this is where sodium dodecyl sulphate is needed for its frothing and collector reagent. Sodium dodecyl sulphate when stirred formed small bubbles which selectively attached to Mo. The selective adhesion of oil for particles having a metallic lustre is decisive factor in the process. This decisive character is mostly of sulphides. Apparently it is the metallic lustre that is the decisive factor, for this includes minerals like coal and molybdenite. The effect of this marked preference of oil for lustrous metallic surfaces is intensified by the fact that gases have a similar adhesiveness for oil. So that if present in water they will join in preventing the wetting of the metallic surfaces. This is equally important for gangue mineral for not having metallic surfaces they are weakly adhesive to films of oil and gas, while they are strongly adhesive to water that is why they are easily wetted exhibit the opposite preference.

Also in Fig. 5, a schematic flow of the experimental procedure and chemical analysis of the concentrate and tail samples when tannic acid was used with the collector/frother mixture is provided. Same data is tabulated in Table 5 (flotation time of 30 minutes, tannic acid and collector/frother mixture additions fixed at 130 g/t and 50 g/t). The best condition is at 30 minutes flotation time, a mixture of kerosene (3% in water, SDS (100 g/t, polyethylene ether (100 g/t)) and tannic acid (150 g/t) in 20% pulp density.

3.4.2. Sodium silicate

Sodium silicate as silicate gangue mineral depressor was also investigated and compared to

Fig. 5 Flowsheet of flotation test using collector/frother mixture 50 g/t, tannic acid 150 g/t with chemical analysis of froth and tailing fractions (Flotation conditions: 20% pulp density, pH 11, and flotation time of 20 minutes).

results of tannic acid as depressor for pyrite. Experimental procedure was similar to that of tannic acid as shown in Fig. 5. A 0.1% strength sodium silicate was added (8 to 40 g/t) into slurry prepared at varying pulp densities ranging from 10 to 25%. Collector/frother mixture (kerosene + SDS + polyethylene alkyl ethyl) was added at 50 or 100 g/t and results obtained are shown in Table 6.

At pulp densities below 20%, Cu grades remained above 1% but the recoveries were extremely below 10%. On the other hand, Mo grades at 10 and 15% pulp density reached 8% and recoveries above 33%. Decrease in the grades of the two metals from increased pulp density was due to decrease in collector/frother mixture concentration as addition is kept constant at 50 g/t. Maintaining pulp density at 15% and collector/frother mixture addition at 50 g/t but increasing sodium silicate addition to 8 to 40 g/t did not show any significant improvement, also increasing collector/frother mixture addition to 100 g/t. The difference in the recovery of Cu and Mo in this case could be attributed to the fact, when tannic acid was used as pyrite depressor some of the Cu minerals associated with pyrite also got depressed therefore reducing the floatability and recovery of Cu. The best condition was at 10 minutes flotation time, a mixture of kerosene (3% in water, SDS (100 g/t), polyethylene alkyl ether (100 g/t) and sodium silicate (20 g/t) in 10% pulp density.

At 10 and 15% pulp density Cu and Mo grades in the concentrate improved to above 1.0 and 8.0% respectively, when only the top of the froth was collected to obtain a concentrate rich in MoS₂. Recovery for Cu was low at below 6% but Mo recovery

Table 6 Results show effect of sodium silicate on flotation of Cu and Mo at varying slurry pulp densities. Collector/frother addition was varied between (40 to 130 g/t), Na₂SiO₃ addition ranging between (8 to 40 g/t), slurry pH 11 and flotation time at 10 minutes

Pulp density (%)	Concentrate				collector/frother mixture (g/t)	Na ₂ SiO ₃ addition (g/t)
	Grade (%)		Recovery (%)			
	Cu	Mo	Cu	Mo		
10	1.28	8.02	5.15	52.33	100	20
	0.89	1.36	22.2	42.1		
15	0.96	2.04	7.9	17	67	13
	1.45	8.06	3.73	33.59		
20	1.20	4.95	5.00	33.43	50	10
25	0.62	4.7	4.22	51.98	40	8
15	0.2	0.48	11.96	46.29	67	27
	0.27	1.27	2.25	17.02		133
	0.62	1.31	15.47	53.22		
	0.52	0.66	28.55	59.36		
	25	1.06	2.48	16.77	63.83	80

Reagents

Collector/frother mixture: (3% kerosene in water + sodium dodecyl sulphate (SDS) + polyethylene alkyl ethyl

ery reached between 33 and 53%. A second test for 10% pulp density was performed to collect whole froth during flotation time resulted in collecting more concentrate weight giving Cu and Mo grades lower at 0.89 and 1.36% respectively. Similarly for 15% pulp density giving Cu and Mo grades at 0.96 and 2.04% with recoveries of 7.90 and 17.00%. The weight of Mo in the concentrate fractions for all four results remained similar.

4. Conclusion

This study was performed to apply conventional flotation technique to upgrade Cu and Mo metals contained in a Cu and Mo flotation tailings and return to process for subsequent recovery. It was observed that performing normal flotation with only PAX and MIBC showed no significant impact on the recovery of both Cu and Mo. The use of a mixture of collector and frother reagents, depressants of FeS₂ and silica allowed improvement of grade and recovery of both Cu and Mo.

- (1) At 30 minutes flotation time, a mixture of kerosene (3% in water), sodium dodecyl sulfate SDS (50 g/t), and polyethylene alkyl ethyl (50 g/t) as flotation collector and frother and tannic acid (150 g/t) in 20% pulp density for pyrite depression improved grade of Cu in the concentrate to 1.3% and Mo at 1.9%. Recovery of the two metals reached 25% and 59% respectively.
- (2) At 10 minutes flotation time, a mixture of kerosene (3% in water), sodium dodecyl sulfate SDS, (100 g/t) and polyethylene alkyl ethyl (100 g/t) as flotation collector and frother mixture and sodium silicate (20 g/t) addition in 10% pulp density for silica improved Cu and Mo grades to 1.3% and 8.0% respectively. Recoveries of the two metals are 5% and 52%, respectively.

According to the experimental results, it is clear that the effect of flotation reagents can be improved by further liberation of particles using attrition and other separation method necessary to improve the separation efficiency.

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