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Cassiterite recovery from a sulfide ore flotation tailing by combined gravity and flotation separations

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Abstract: Cassiterite (SnO_2), which is the most important Sn-containing mineral, is extensively found in large quantities in discarded tailings. The recovery of cassiterite from discarded sulfide ore flotation tailings can reduce resource wastage and environmental pollution. The gravity separation technique can recover multiple valuable minerals, such as cassiterite, whose densities considerably differ from those of their associated gangue minerals. However, its recovery efficiency rapidly decreases as the mineral particle grain size decreases. To recover the finer valuable mineral particles from gravity separation tailings, flotation separation can be used as a supplementary method. In this study, the gravity and flotation separation techniques are combined to recover cassiterite from a sulfide ore tailing. The Sn grade and recovery of the final concentrate is 31.40% and 88.05%, respectively, thus indicating a highly efficient recovery of cassiterite by using the combined gravity and flotation separation technique. This study can be an important reference for recovering cassiterite from low-Sn-grade tailings.

Keywords: cassiterite, Falcon concentrator, shaking table, flotation, gravity separation

1. Introduction

Tin (Sn) is an indispensable rare metal used in modern industry, and is known as the "monosodium glutamate in the industry." Sn and its compounds have been extensively used in many applications such as alloy fabrication, catalysis, and food packing. Cassiterite (SnO_2) is the most important Sn-containing mineral from which Sn may be economically extracted (Tan et al., 2016). Although its associated gangue minerals, such as quartz, sercite and clay minerals, have low densities, cassiterite is a highly dense mineral. Owing to the considerable difference in their densities, cassiterite can be efficiently recovered using the gravity separation technique. Gravity separation offers multiple advantages such as low recovery cost and minimal environmental impact; moreover, products recovered using the technique exhibit excellent dehydration characteristics. Consequently, gravity separation has been extensively used to recover cassiterite (Angadi et al., 2015).

A commonly used gravity separation equipment is the shaking table (Qin et al., 2012). In this machine, the asymmetrical vibration of the table surface, water flow flush, and turbulent flow between adjacent riffles is combined to separate minerals with different specific gravities. The table surface vibration is generated using a mechanism that superimposes a gradual forward stroke and rapid return; this vibration moves the mineral particles on the table. In this equipment, flush water is longitudinally introduced along the feed side. The mineral particles diagonally move across the table from the feed end, spread over the table, and eventually reach various exit positions in the concentrate, middling and tailing edges depending on their specific densities. The shaking table can concentrate minerals with a particle size in the range of 0.6-8.0 mm (Angadi et al., 2015). The efficiency of the shaking table in separating coarse particles is high; however, it is low in separating fine particles because of the low settling velocities of smaller particles. To increase the recovery efficiency of fine particles, enhanced

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gravity separators use centrifugal or other forces. One of these separators is the Falcon concentrator (Su et al., 2017).

The Falcon concentrator comprises a conical bowl that can spin at high rotation speeds (Fig. 1d), generating an artificial enhanced gravity field several hundred times greater than that of Earth (Su et al., 2017). The feed is introduced from the top of the Falcon concentrator unit through a central vertical feed pipe and accelerated using an impeller. The enhanced artificial gravity field causes the rapid deposition and stratification of mineral particles within the smooth bowl. Because of the enhanced gravity field generated by the high rotation speed of the spinning bowl, fine mineral particles are separated based on their differential settling velocities. The Falcon concentrator can treat mineral particle sizes as small as 20 µm (Oruç et al., 2010). However, the gravity separation tailings produced by the shaking table and the Falcon concentrators still contain a certain amount of valuable mineral particles. To recover these finer mineral particles from the tailings, the flotation method is adopted. This method is a supplementary technique that increases the comprehensive recovery of Sn.

The flotation method facilitates the recovery of fine particles of valuable minerals from gravity tailings. It is an extensively used separation technology that offers multiple advantages such as low production cost, simple implementation and high fine particle separation efficiency (Ren et al., 2014). The method uses the hydrophobic difference among various mineral surfaces to separate valuable minerals from their associated gangue minerals. The pure cassiterite surface has a compact hydration layer, thus exhibiting strong hydrophilicity. The flotation collector adsorption can increase the hydrophobicity of the cassiterite surface. Oleic acid is the most extensively used collector that is inexpensive and exhibits a strong collecting ability. However, it shows limited selectivity. Recently, hydroxamic acids have been extensively used in oxide mineral flotation (Tian et al., 2018a; Tian et al., 2019). Among these acids, benzohydroxamic acid (BHA) exhibits excellent selectivity but limited collecting ability in the cassiterite flotation (Tian et al., 2017a; Tian et al., 2017b). To enhance BHA adsorption on the cassiterite surface, activators are commonly used (Tian et al., 2018c; Tian et al., 2018e). Generally in the form of nitrate, Pb^{2+} is the most extensively used activator for improving the BHA collecting ability in cassiterite flotation (Tian et al., 2018b; Tian et al., 2018d). However, Pb^{2+} is expensive and poses a considerable threat to human health. In this study, oleic acid and BHA are used in cassiterite flotation to combine their merits and decrease the use of Pb^{2+} ion.

In this study, gravity and flotation separation methods are combined to recover cassiterite from sulfide ore tailings. Herein, the performance of the combined process is investigated, and the effects of the operational parameters of gravity separation and flotation reagent scheme on the cassiterite grade and the recovery of the final concentrate are investigated. The results of this study can provide an important reference for the recovery of cassiterite from low-Sn-grade tailings.

2. Materials and methods

2.1. Materials and reagents

The tailing used in this study was obtained from a deposit in Canada. The major valuable minerals of the raw ore include chalcopyrite, sphalerite, and stannite. In a previous study, the flotation method was adopted to recover these valuable minerals (Zhang et al., 2020). After their recovery, most Sn-containing minerals in the raw ore were lost in the flotation tailings. Note that the flotation tailings have a Sn grade of 0.70%; moreover, ~92% and ~8% of the Sn quantities lost in the flotation tailings occur as oxides (cassiterite and wood tin) and hydroxides (colloidal tin) (Table 1), respectively. The primary gangue minerals are the typical silicate minerals such as quartz, clay minerals, and feldspar (Table 2). The liberation degree of cassiterite in the flotation tailings is considerable (Zhang et al., 2020); hence, grinding need not be implemented to liberate cassiterite.

Table 1. Existing forms of Sn in the flotation tailings (%)

Existing form	Mineral	Sn grade	Distribution
Oxide	Cassiterite, Wood tin	0.64	91.89
Hydroxide	Colloidal tin	0.06	8.11
Total	-	0.70	100.00

Table 2. Relative contents of major minerals in the raw ore (%)

Mineral	Chemical formula	Content	Mineral	Chemical formula	Content
Cassiterite	SnO_2	0.85	Clay minerals	-	13.65
Colloidal tin	-	0.51	Limonite	$FeO(OH)\cdot nH_2O$	9.41
Hemimorphite	$Zn_4[Si_2O_7](OH)_2$	0.76	Feldspar	-	7.68
Gahnite	$ZnAl_2O_4$	0.27	Dolomite	$CaMg(CO_3)_2$	2.40
Anglesite	$PbSO_4$	0.73	Chlorite	-	2.35
Quartz	SiO_2	49.60	Fluorite	CaF_2	3.80
Sericite	-	5.30	Others	-	2.69

In flotation experiments, $Pb(NO_3)_2$ was used as the activator and BHA ($C_7H_7NO_2$) and oleic acid ($C_{18}H_{34}O_2$) were combined to be used as collectors. Sodium silicate ($Na_2SiO_3\cdot 9H_2O$) and sodium carboxymethyl cellulose (CMC; degree of substitution=1.2; molecular weight=250000; viscosity=1500~3100 mPa·s) were combined to be used as depressants. Methyl isobutyl carbinol (MIBC, $C_6H_{14}O$) and sulfuric acid (H_2SO_4) were used as the frother and the pH regulator, respectively. In this study, all reagents were of industrial grade, and the water used in flotation experiments was tap water.

2.2. Gravity separation experiments

The concentrator used in this study was the Falcon L40 laboratory model (Sepro Mineral Systems, Canada). It operated as a semi-batch unit equipped with a 4-in diameter ultrafine smooth-walled bowl. The maximum processing capacity of Falcon L40 can reach up to 300 kg/h of feeding, with high mine concentration and centrifugal force field of up to 75 wt% and 300 G, respectively. The feed capacity of each test was 150 g and the partical size compositions of the feed are listed in Table 3. The shaking table model type was LY-1100×500×430 (Hengchang Mining Machinery, China). The particles of size +75-μm were recovered on a fine-sand shaking table, whereas the particles of -75+38 and 38-μm were recovered on a slime-type shaking table. For particles of all sizes, the deck angle was in the range of 5°-7°, a stroke was 10 mm, frequency was 300 r/m and pulp density was 25 wt%. The concentrate and tailings produced by the Falcon concentrator and the shaking table were settled, dried, and weighed. Moreover, the Sn grades of two products were determined via chemical analysis. The Sn recoveries of the concentrate and tailings were calculated based on the weight distribution and the Sn grades of two products.

2.3. Flotation experiments

All flotation experiments were performed in a mechanical agitation flotation machine (XFG, Hengchang Mining Machinery, China) using a 3.0-L flotation cell for roughing and scavenging flotation experiments and a 0.5-L flotation cell for cleaning flotation experiments at an impeller speed of 1800 rpm. In each roughing flotation experiment, the mineral suspension was prepared by adding raw ores (1000 g) to a certain amount of tap water. After each flotation experiment, the concentrate and tailings were separately filtered, dried, and weighed. Then, the Sn grades of the two products were determined via chemical analysis. The Sn recoveries of the concentrate and tailings were calculated based on the weight distribution and the Sn grades of two products.

3. Results and discussion

3.1. Gravity separation

Cassiterite is the primary valuable mineral in the flotation tailings, and quartz, clay minerals and feldspar are the main gangue minerals associated with it. To separate cassiterite from gangue minerals, the gravity separation method was adopted. The gravity separation experiments include the use of Falcon concentrator and shaking table.

3.1.1. Falcon concentrator separation

According to a previous study, the separating effect of Falcon concentrator mainly depends on the

Table 3. Partical size compositions of Falcon concentrator separation feed (%)

Product	Yield	Sn grade	Sn recovery
+75-μm	28.02	0.71	27.62
-75+38-μm	30.69	0.54	28.00
-38-μm	41.29	0.82	44.38
Feed	100.00	0.70	10.00

centrifugal force, feed pressure and pulp density (Abaka-Wood et al., 2019). Figs. 1a and 1c show that when the centrifugal force and the pulp density were increased to 175 G and 25%, respectively, the Sn recovery of the concentrate gradually increased to 89.63%. This high recovery rate is ascribed to the improved recycling effect of fine sized cassiterite in the large centrifugal force field. At high centrifugal forces, the growth in Sn recovery was halted by the poor recovery efficiency of the Falcon concentrator for -3-μm particles (Foucaud et al., 2019). Interestingly, the trajectory of large particles in the composite stress field will change when the feed pressure changes (Foucaud et al., 2019). As the feed pressure increased from 0.10 to 0.15 MPa, the Sn recovery of the concentrate gradually decreased from 90.05% to 89.63%, while the Sn grade rapidly increased from 1.49% to 1.63% (Fig. 1b). After a comprehensive study, the optimal experimental conditions were determined: centrifugal force, feed pressure and pulp density of 175 G, 0.15 MPa and 25%, respectively. Table 4 summarizes the Falcon concentrator separation results under the optimum experimental conditions. The Sn grade of 1.63% of the concentrate was more than twice that of the feed. The tailings with a Sn grade of 0.11% and a yield of 61.22% were directly discarded. The separation results demonstrate that the Falcon concentrator exhibit an excellent tail-discarding effect.

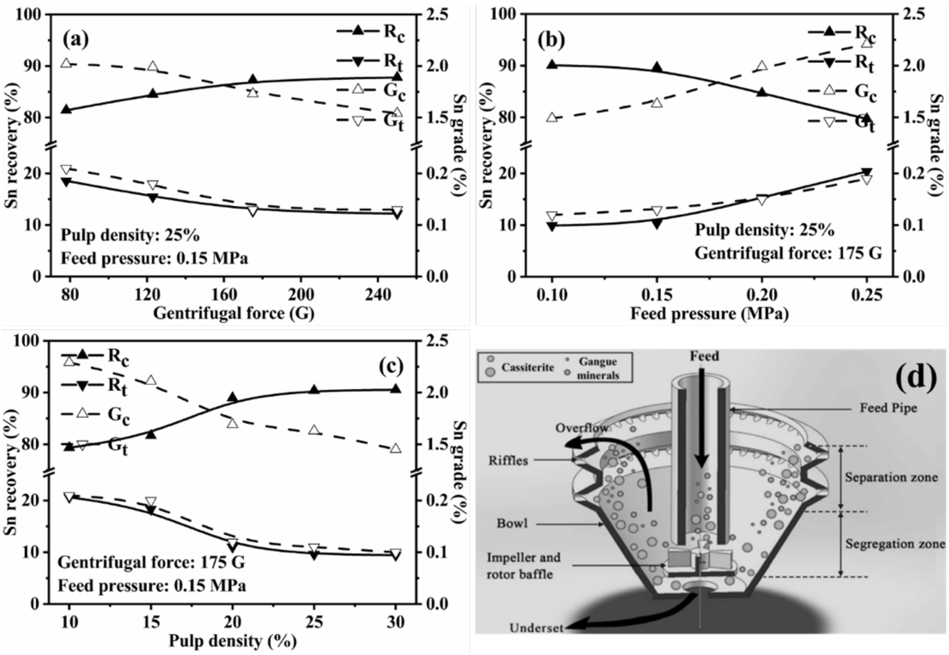

Fig. 1. Sn recoveries and grades of Falcon concentrator separation concentrate and tailings as functions of (a) centrifugal force, (b) feed pressure, and (c) pulp density (R_c and R_t refer to the Sn recoveries of the concentrate and tailings, respectively, and Gc and Gt refer to the Sn grades of concentrate and tailings, respectively); (d) schematic of the separation process in the Falcon series centrifugal concentrator (Zhang et al., 2018)

Table 4. Falcon concentrator separation result (%) under the optimum experimental conditions (centrifugal force: 175 G, feed pressure: 0.15 MPa and pulp density: 25%)

Product	Yield	Sn grade	Sn recovery
Concentrate	38.78	1.63	90.37
Tailings	61.22	0.11	9.63
Feed	100.00	0.70	10.00

3.1.2. Shaking table separation

The shaking table was used to enrich cassiterite from the Falcon concentrator separation concentrate. Before the shaking table experiments, the feed sample (i.e., the Falcon concentrator separation concentrate) was classified into different particle-size fractions: +75, -75+38 and -38 µm. The Sn grade and recovery of the shaking table separation concentrate were 54.31% and 78.03%, respectively. The experimental results reveal the excellent separation and enrichment performance of the shaking table. However, without further mixture treatment of the shaking table separation middlings and tailings, a small but significant amount (21.97%) of cassiterite will be lost.

Table 5. Shaking table test results (%)

Product		Yield		Sn grade		Sn recovery	
	Concentrate*	2.37		54.31		78.03	
Middlings + Tailings	+75-µm Middling	97.63	12.70	0.35	0.81	21.97	6.24
	+75-µm Tailings		38.34		0.11		2.56
	(−75+38)-µm Middling		7.30		1.66		7.35
	(−75+38)-µm Tailings		25.96		0.08		1.28
	−38-µm Middling		3.51		1.35		2.87
	−38-µm Tailings		9.82		0.28		1.67
	Feed	100.00		1.63		100.00	

*Mixture of +75, (−75+38) and −38-µm concentrates

3.2. Flotation experiments

The Sn grade of -38-µm tailings (0.28%) was considerably higher than that of the (-75+38)-µm tailings (0.08%) and +75-µm tailings (0.11%) (Table 5). This result indicates that the cassiterite recovery using the shaking table separation gradually becomes difficult as the size of the particles decreased, and the recovery of finer mineral particles using the flotation method is better than that using the shaking table separation technique. Therefore, the flotation method was used to recover cassiterite from the shaking table separation middlings and tailings.

3.2.1. Single-factor experiments

The primary gangue minerals associated with cassiterite in the flotation tailings include quartz, clay minerals, and feldspar, which are typical silicate minerals (Table 2). For the flotation of silicate minerals, Na_2SiO_3 is extensively used as an efficient depressant; accordingly, it was used in the flotation experiments. To increase the cassiterite recovery, the roughing flotation time was set to 2. Profiting from the strong collect capacity of oleic acid and the better selectivity of BHA, their combination was used as the collector (Wu and Zhu, 2006; Zhou et al., 2014). The effects of pH and the dosages of Na_2SiO_3, $Pb(NO3)_2$, oleic acid and BHA were investigated following the processes shown in Fig. 2a. All reagent dosages used in roughing flotation 2 were half of those used in roughing flotation 1. Moreover, all reagent dosages shown in Fig. 2(c-g) refer to those used in roughing flotation 1. Fig. 2c shows that the Sn recovery of the flotation concentrate considerably increased from ~50% to 61.2% as the pH level increased from 4.5 to 5.5 and then remained stable despite a further increase in pH. The high recovery is ascribed to the chemical adsorption of BHA on the cassiterite surface (Qin et al., 2012). However, the Sn grade of the flotation concentrate gradually decreased from ~3% to 2.58% as the pH level increased from 4.5 to 5.5 and significantly decreased to ~1.9%with a further increase in pH. This phenomenon is related to the extent of ionization and hydrolysis of the collector and the good adsorption of oleic acid on the surfaces of the gangue minerals, such as quartz, clay minerals and feldspar (Qin et al., 2011). Fig. 2d shows that as the dosage of Na_2SiO_3 increased from 125 to 150 $g \cdot t^{-1}$, the Sn grade of the concentrate significantly increased from ~2% to 2.58%. With further increments in the dosage of Na_2SiO_3, the Sn grade of the flotation concentrate gradually increased, while the Sn recovery considerably decreased. The Sn recovery of the concentrate exhibited a considerable improvement with an increase in the dosage of $Pb(NO_3)_2$ from 40 to 80 $g \cdot t^{-1}$ and showed a gradual increase with further increase in the dosage (Fig. 2e). The Sn recovery of the flotation concentrate rapidly increased with an increase in the dosages of oleic acid and BHA. This rapid increase stopped when the dosages of oleic acid and BHA were 400 and 200 g·t-1, respectively (Figs. 2f and 2g). The Sn recovery reached a peak value of 61.2% as the dosages of oleic acid and BHA were adjusted to 400 and 200 g·t-1, respectively. The optimal experimental conditions were determined: pH level of 5.5 and Na_2SiO_3, $Pb(NO_3)_2$, oleic acid and BHA dosages of 150, 80, 400 and 200 g/t, respectively.

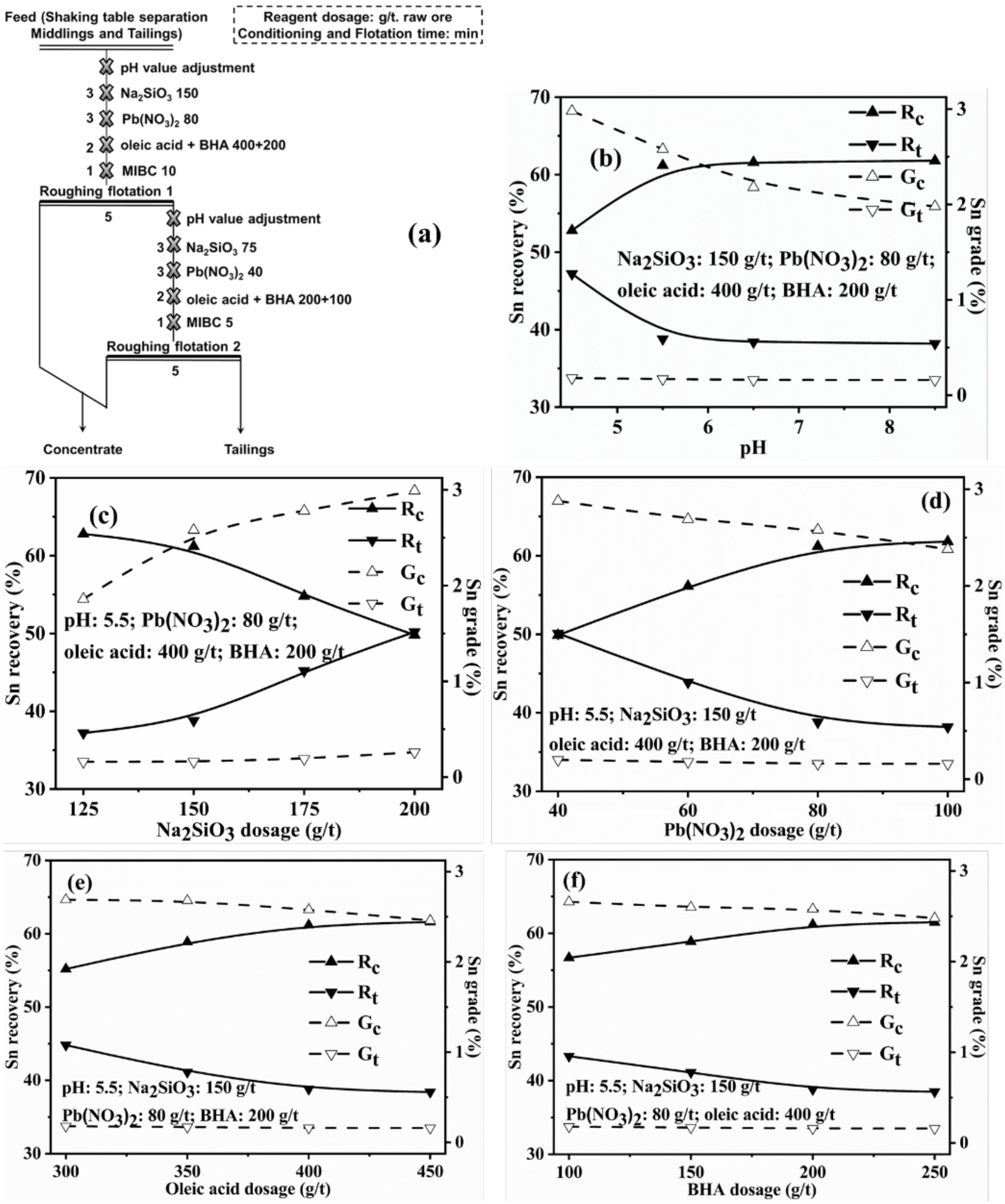

Fig. 2. (a) Technological process showing the effects of pH and each reagent dosage; (b) Sn recoveries and grades of the flotation concentrate and tailing as a function of pH; dosages of (c) Na_2SiO_3, (d) $Pb(NO_3)_2$, (e) oleic acid and (f) BHA (all reagent dosages shown in Fig. 5(b–f), refer to those used in roughing flotation 1; R_c and R_t refer to the Sn recoveries of the concentrate and tailing, respectively; G_c and G_t refer to the Sn grades of the concentrate and tailing, respectively)

3.2.2. Open-circuit flotation test

Fig. 3 shows the open-circuit flotation flowchart. The flotation results summarized in Table 6 were obtained using the flotation process with two-stage roughing flotation, three-stage cleaning and three-stage scavenging. The open-circuit flotation concentrate has a Sn grade and recovery of 12.01% and 40.59%, respectively, indicating the excellent flotation separation of cassiterite from its associated gangue minerals.

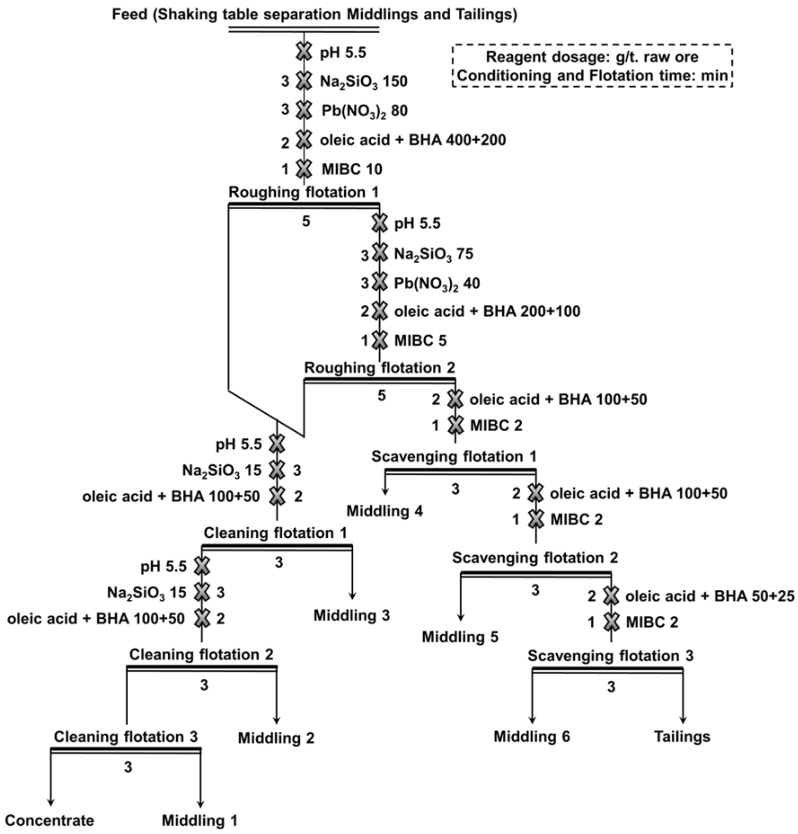

Fig. 3. Open circuit flotation flowchart

Table 6. Open-circuit Sn flotation (from shaking table separation middlings and tailings) experimental results (%)

Product	Yield	Sn grade	Sn recovery
Concentrate	1.17	12.01	40.59
Middling 1	0.62	1.58	2.84
Middling 2	3.23	1.03	9.57
Middling 3	4.44	0.68	8.69
Middling 4	9.93	0.71	20.32
Middling 5	4.14	0.38	4.53
Middling 6	3.15	0.18	1.63
Tailings	73.32	0.06	11.83
Feed	100.00	0.35	100.00

3.2.3. Closed-circuit flotation test

The closed-circuit flotation experiment was performed on the basis of the results of the single-factor and open-circuit experiments. Fig. 4 shows the closed-circuit flotation flowchart, and Table 7 summarizes the closed-circuit flotation experimental results. The Sn grade of the closed-circuit flotation experiment feed was only 0.35%, and the final flotation concentrate has a Sn grade and recovery of 11.22% and 86.36%, respectively. The flotation tailings has a Sn grade of 0.05%, which is considerably lower than that of the Falcon concentrator separation tailings (0.11%; Table 2). Note that the flotation tailings can be directly discarded. The flotation reagent scheme using oleic acid and BHA as the collectors, and Pb^{2+} as the activator combined with Na_2SiO_3 as the depressant, can efficiently recover cassiterite from its associated gangue minerals.

3.3. Final experimental results of the combined gravity and flotation separation method

Table 8 summarizes the final experimental results of the combined implementation of the gravity and flotation separation methods. The final concentrate, which is the mixture of the shaking table separation and flotation concentrates, has a Sn grade and recovery of 31.40% and 88.05%, respectively. The final experimental results demonstrate highly efficient cassiterite recovery from the sulfide ore flotation tailings through the combined use of gravity and flotation separation technologies.

Fig. 4. Closed-circuit flotation flowchart

Table 7. Closed-circuit Sn flotation experimental results (%)

Product	Yield	Sn grade	Sn recovery
Concentrate	2.75	11.22	86.36
Tailings	97.25	0.05	13.64
Feed	100.00	0.35	100.00

Table 8. Final experimental results (%) of the combined gravity and flotation separation method

Product		Yield		Grade		Recovery	
Concentrate	Shaking table separation concentrate	1.96	0.92	31.40	54.31	88.05	71.38
	Flotation concentrate		1.04		11.22		16.67
Tailings	Falcon concentrator separation tailing	98.04	61.22	0.08	0.11	11.95	9.32
	Flotation tailings		36.82		0.05		2.63
Feed		100.00		0.70		100.00	

4. Conclusions

The sulfide ore flotation tailing obtained from a deposit in Canada has a Sn grade of only 0.70%, of which cassiterite is the major valuable mineral. The primary gangue minerals include typical silicate minerals such as quartz, clay minerals, and feldspar. To reduce resource wastage and recover cassiterite with fine fractions, the combined technological process of gravity and flotation separation methods are adopted.

In this study, the gravity separation methods involve the use of the Falcon concentrator followed by the shaking table. Under the optimal experimental conditions of the Falcon concentrator, a tailing with a Sn grade of 0.11% and a yield of 61.22%, respectively, is discarded. To further enrich cassiterite from the Falcon concentrator separation concentrate, the shaking table was employed. The shaking table separation concentrate has a Sn grade and recovery of 54.31% and 78.03%, respectively. Notably, the Sn recovery of the table separation tailings is 21.97%. To achieve comprehensive Sn recovery, the flotation method is employed using Na_2SiO_3 as the depressant, $Pb(NO_3)_2$ as the activator, and oleic acid and BHA as the collectors. The Sn grade and recovery of the flotation concentrate are 11.22% and 86.36%, respectively.

The final concentrate produced using the combined gravity and flotation separation methods delivers a comprehensive Sn grade and recovery of up to 31.40% and 88.05%, respectively. The final tailing exhibits a Sn grade of only 0.08%. These experimental results demonstrate highly efficient cassiterite recovery from the low-Sn-grade tailing by the combined implementation of the gravity and flotation separation technologies.

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