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Experimental research on mineral processing for a stibnite ore in Shaanxi

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Abstract. The refractory stibnite used in this research is from Shan yang, Shaanxi. The ore characteristics are coarser grain size of the stibnite, which is favorable for flotation. But the clay minerals (kaolinite and illite), pyrite and arsenopyrite in the ore will bring some difficulties to flotation. Through on the research of the slurry regulator, stibnite collector and gangue mineral inhibitor, better flotation index was obtained. Under the condition of the Sb raw ore grade 2.25%, the flotation index of the concentrate contains Sb 65.12% with the recovery of 91.56%, and the arsenic content of 0.20 was obtained.

1. Introduction

With the rapid development of the national economy, the demand for antimony resources continues to increase. As an important strategic metal for national production, it is widely used in the fields of flame retardants, catalysts, plastic stabilizers, etc. [1]. China is the world's largest antimony resource country, with reserves and production ranking first in the world. However, due to years of over-exploitation and unreasonable development, there is a serious shortage of reserve resources [2,3]. In order to achieve sustainable development of antimony resources, reasonable development and utilization must be carried out [4].

In the natural world, antimony is mainly in the form of sulphide ore and oxidized ore. Sulphide ore is mainly stibnite, which has good floatability and is mainly recovered by flotation [5,6,7]. The stibnite often contains associated elements such as As, Cu, Fe, Au, and Ag [8]. Among them, arsenic is a harmful impurity, which is difficult to separate from stibnite, and it will pollute the environment during the smelting process. Therefore, it is necessary to effectively suppress the stibnite ore during the flotation process [9]. The presence of clay minerals in gangue minerals can also lead to pulp muddy, increase the dosage of reagent, increase the difficulty of flotation, and reduce the flotation index.

The refractory stibnite used in this research is from Shan yang, Shaanxi Province. The existing problems in the mineral processing process are as follows: The ore contains a large amount of clay minerals (kaolinite and illite), resulting in a grade of stibnite concentrate <50%, which does not meet industry requirements. Due to the presence of ore pyrite and arsenic pyrite, the arsenic content of antimony concentrate is >1%, exceeding industry requirements.

In order to solve the above problems, the experiment will start from the aspects of flotation reagents and processes to simplify production flow and improve the flotation index.

2. The property of ore

The results of multi-element analysis of ore are shown in Table 1. The results of X-ray diffraction are shown in Table 2.

Table 1. The multi-element analysis results of the ore (%).

Element	Sb	Fe	S	$BaSO_4$	K_2O	MgO	CaF_2
Wt	2.18	3.06	2.85	0.35	1.10	0.26	1.72
Element	CaO	SiO_2	AL_2O_3	As	Cu	Pb	Zn
Wt	6.09	68.90	13.24	0.27	0.003	0.002	0.007

Table 2. X-ray diffraction results of the ore (%).

Mineral	Stibnite	Pyrite	Arsenopyrite	Ankerite
Content	4.8	0.6	1.2	12.4
Mineral	Quartz	Potassium feldspar	Kaolinite	Illite
Content	68	2	3	8

The results show that stibnite is the main recoverable metal mineral. Pyrite and arsenopyrite should be discarded as harmful impurities during flotation. The gangue minerals are mainly composed of quartz, followed by ankerite, kaolinite and illite. Clay minerals (kaolinite and illite) tend to be over grinded and muddy during the grinding process, resulting in excessive pulp viscosity and affecting the selection index. Most stibnite has a coarse dissemination size of 0.06mm-0.2mm.

3. The principle flowsheet

Based on the properties of the ore and the problems in the flotation, three measures were adopted in the flowsheet. (1) Due to the coarse grain size of the stibnite disseminated (0.06-0.2 mm), coarser grinding fineness can be used to reduce slurry muddy. And use a reasonable regulator to reduce the viscosity of the slurry. (2) Highly selective collector for stibnite should be used. (3) Reasonable pyrite and arsenopyrite inhibitors are selected to reduce arsenic content in the concentrate.

Through the condition test, the final flowsheet conditions are as follows: -0.074 mm60% grinding fineness; TZ-10 as regulator; lead nitrate as activator of stibnite; 5[#] oil as collector; potassium permanganate as inhibitor. The principle flowsheet is shown in Figure 1.
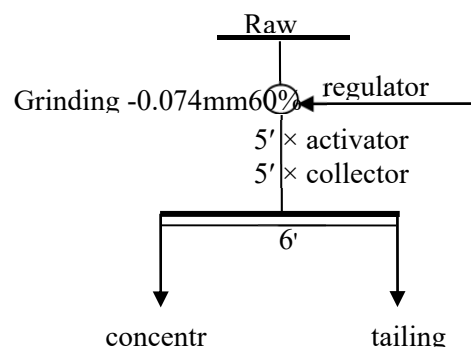

Figure 1. The principle flowsheet.

4. Result and discussion

4.1. Effect of regulator dosage on flotation

Usually, sodium silicate, sodium hexametaphosphate, lime, etc. are used as the regulator [10,11,12], however, it was found through the test that the flotation index was not satisfactory when using the above regulator (concentrate grade was less than 50%). Finally, TZ-10 was used as regulator, which was a compounded inorganic solid salt, a non-volatile, water-soluble agent. The anion produced by the hydrolysis of the inhibitor TZ-10 in the slurry forms a hydrophilic film on the surface of the mineral, which increases the wettability of the non-target mineral surface and thus forms an inhibitory effect. The addition amounts of lead nitrate and $5^{\#}$ oil are 1000g/t and 50g/t, respectively. The flowsheet is showed in Figure 1 and the results are shown in Figure 2. As the amount of TZ-10 increases, the concentrate grade increases, but the recovery does not change much. Therefore, the optimum dosage of TZ-10 is 500g/t, and the flotation index of 44.00% of concentrate grade and 91.40% recovery is obtained.
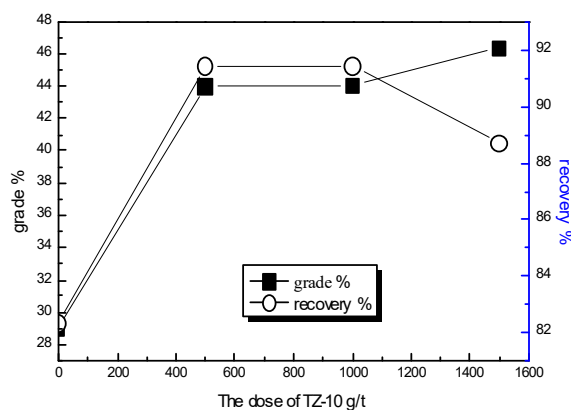

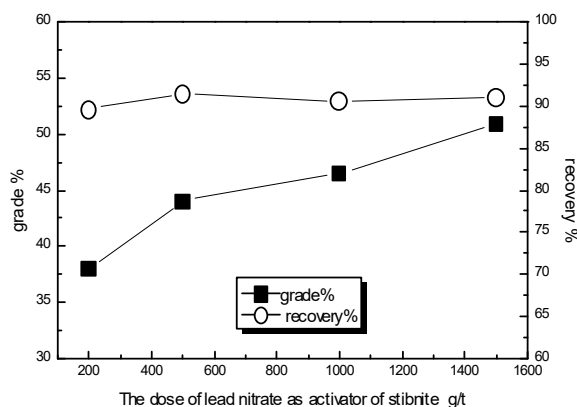**Figure 2.** Effect of regulator dosage on flotation.   **Figure 3.** Effect of activator dosage on flotation.

4.2. Effect of activator dosage on flotation

Lead ions have an activation effect on stibnite, and lead nitrate is usually used as a stibnite activator [13]. The flotation flowsheet is showed in Figure 1 and the results are listed in Figure 3. As the dose of lead nitrate increasing, the concentrate grade was increased but the recovery was decreases slightly. Final determination of lead nitrate dosage of 1000g/, concentrate grade is 45.51%, recovery rate is 90.50%.

4.3. Effect of collector dosage on flotation

In comparison of different collectors, xanthates, DDC, and aerofloats showed high collecting effects not only on stibnite, but also on pyrite and arsenopyrite [14]. Finally, the self-prepared $5^{\#}$ oil was selected as the special collector of stibnite. The lead ions produced by lead nitrate in the slurry tend to adhere to the surface of the stibnite to form a cationic coating surface. The functional group of the new agent $5^{\#}$ oil greatly enhances the binding ability to lead ions, and the polar group is more easily selectively adsorbed. On the surface of the stibnite, a layer of hydrophobic film is formed to enhance the floatability of the stibnite. At the same time, the functional group of $5^{\#}$ oil can weaken the hydrophobic ability of non-polar groups, reduce the concentration of the mud cover on the surface of the bubble, and reduce the surface energy of the muddy minerals (illite, kaolinite), Thereby reducing the influence of muddy minerals on the bubble layer.

The results show that $5^{\#}$ oil has a better collecting effect on stibnite, but the effect on pyrite and arsenopyrite is poor. Therefore, $5^{\#}$ oil was effective for improving concentrate grade and reducing

arsenic content. The $5^{\#}$ oil was yellow oil-like liquid. Due to the foamability effect of $5^{\#}$ oil, the $2^{\#}$ oil may not be used during the flotation, which would prevent the mud from mixing into the mineralized bubbles and greatly improve concentrate grade. The flowsheet is showed in Figure 1 and the results are listed in Figure 4. As the dose of $5^{\#}$ oil was increased, the concentrate grade was apparently reduced but the recovery was greatly improved. The optimal dose of $5^{\#}$ oil was 50 g/t, which resulted in a concentrate grade of 45.52% and recovery of 91.40%. Due to its foamability effect, the dose of $5^{\#}$ oil should not be large.
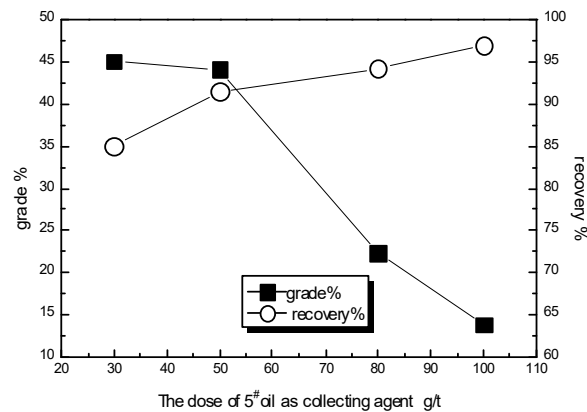

Figure 4. Effect of collector dosage on flotation.

5. Close-circuit flotation tests.

The close-circuit flotation test under the optimum parameters was proceeded to enhance the Sb grade and recovery of concentrate. The flow is "one roughing- one scavenging - twice cleaning, the middling order returns".

The closed-circuit flotation experiment flowsheet is showed in Figure5 and the results are listed in Table. 3. From the Table 3, the concentration with Sb grade of 65.12% and Cu recovery of 91.56% was obtained. And the arsenic content was 0.20%, which completely met the quality requirement.
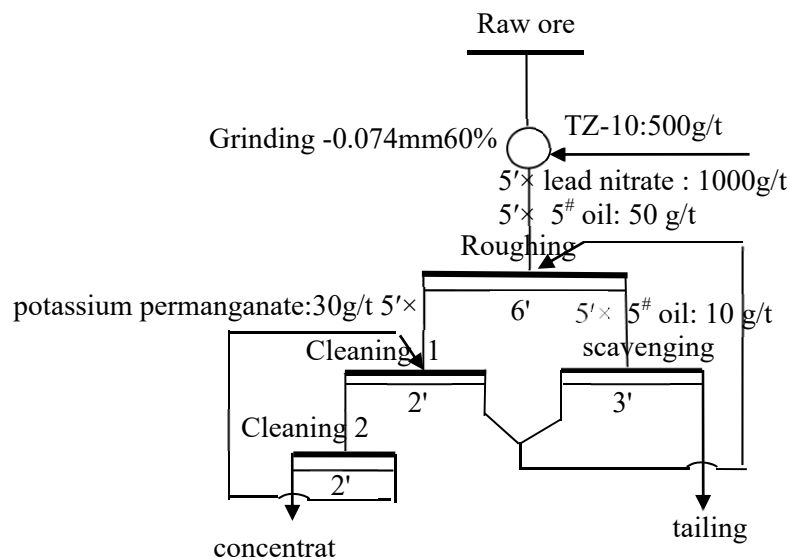

Figure 5. Flowsheet of close-circuit flotation.

Table 3. Results of the close-circuit flotation (%).

Product	Yield	Grade (Sb)	Recovery (Sb)
Concentrate	3.16	65.12	91.56

6. Conclusions

It can be seen from the results and discussion above that:

1)The stibnite in this experiment was in coarse dissemination size (0.06 mm-0.2 mm), so the grinding fineness was increased utmost to reduce the mudding of the clay minerals, which was proved feasible. The final grinding fineness was -0.074 mm60%. the flotation index of the concentrate contains Sb 65.12% with the recovery of 91.56%, and the arsenic content of 0.20 was obtained.

2) In this experiment, the new collector $5^{\#}$ oil and the new regulator TZ-10 was adopted. In the first cleaning, potassium permanganate was added to depress pyrite and arsenopyrite. Due to the foamability effect of $5^{\#}$ oil, the $2^{\#}$ oil may not be used during the flotation, which would prevent the mud from mixing into the mineralized bubbles and greatly improve concentrate grade. In summary, this study simplifies the process flowsheet, reduces the cost of the flotation, and improves flotation index.

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