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Enhanced flotation of refractory gold ore by using sulfur-oil agglomeration with $(NH_4)_2S_2O_3$ as regulator in weak acidic pulp

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ABSTRACT

In this study, a new approach "sulfur-oil agglomeration" for gold flotation was put forward to recover gold particles in laboratory and industrial scale experiments. The optimum recovery of gold was obtained by adding $(NH_4)_2S_2O_3$, oil emulsions and sulfur into the mill, together with butyl xanthate as the collector in weak acidic pulp adjusted by H_2SO_4.

Fundamental concepts of gold flotation were considered, including the sulfur-oil-gold agglomeration mechanisms and the role of $(NH_4)_2S_2O_3$. The possible mechanism had been put forward: (1) nature of $(NH_4)_2S_2O_3$ is complex, it renders the oxidized gold surface cleaned, the exposed fresh surface shows strong natural oleophilicity; (2) in weak acidic pulp, S decomposed from $(NH_4)_2S_2O_3$, a highly hydrophobic/oleophilic material, agglomerated with gold; (3) then the oil droplets overspread the gold surfaces and the collision of these oil wetted particles caused formation of "liquid bridges" each upon them. As a result, the fine gold particles in the beginning were converted to the masses in the form of sulfur-gold-oil which were bonded together by liquid bridges. Meanwhile, the sulfur could strengthen the adsorption of collectors on gold surface, as well as the floating size range of gold is widen, and the recovery of gold is significantly improved.

Industrial trials conducted at Yunnan, China showed that an average gold recovery of 87.39% into a concentrate assaying 25.84 g/t Au from a feed of 1.8–3.0 g/t could be achieved by the aid of sulfur-oil agglomeration method.

1. Introduction

Investigations of new technologies to recover fine/ultrafine gold particles from various types of ores, with the increasing depletion of resources, have been a preoccupation of high-efficiency flotation-metallurgy process for metallurgists and mineral processing engineering for several decades. Many techniques were applied to process gold ores, such as gravity separation, amalgamation, flotation, cyanidation, or the combination of these processes (Torres et al., 1999). In the past few decades, may efforts have been made to make it possible for the economical recovery of gold from low and marginal grade ores (Alim Gül et al., 2012; Allan and Woodcock, 2001; Bulatovic, 2010; Chryssoulis and Dimov, 2004; Coetzee et al., 2011; Curreli et al., 2005; Forrest et al., 2001; Hendriks and Chevalier, 2004; O'Connor and Dunne, 1994; Sun et al., 2014).

Froth flotation, which is receiving increasing industrial attention, is an effective process for gold recovery, sometimes the results is not satisfied. However, recovery of fine (5–37 μm), especially the ultrafine (<5 μm) gold particles with high slime and/or high carbon using flotation separation is still not satisfied, and improvement of production target continues to be a challenge. Glembotski et al. (1974) and Sen et al. (2005) pointed out that the decrease of the selectivity for fine/ultrafine gold particles was mainly due to the co-flotation of gangue components. That is, the presence of excess amount of slimes and an excess amount of collector were considered to be detrimental to the recovery (Yalcin and Kelebek, 2011). Conventional gravity separation and flotation method alone cannot recover these kinds of ores effectively.

On account of above problems, the coal-oil agglomeration (CGA) method, whereby hydrophobic gold particles are recovered from

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ore slurries into coal-oil agglomerates, and a subsequent recovery through smelting or co-smelting of these agglomerates, has been generally recognized by the researchers and companies (Cilingir and Sen, 2000; Marciano et al., 1994; Moses and Petersen, 2000; Wu et al., 2004). The CGA for free or native gold recovery has been a research subject since a patent which was authorized to BP Australia Ltd in 1986 (Sen et al., 2005). Moses and Petersen (2000) revealed that the CGA process was an alternative to the mercury amalgamation process. CGA process takes advantage of the greater hydrophobicity and oleophilicity of coal and gold compared to that of most gangue materials. Results of Shi et al. (2000) demonstrated that it shows higher efficiency for the recovery of 5–300 μm gold particles. Furthermore, CGA method is well-adapted for the recovery of oxidized zone of gold deposit or quartz vein gold deposit: on the one hand, coal can help to produce agglomerates with oil and gold by "liquid bridges" (Ünal and Aktaş, 2001), the apparent size of gold particle is lengthened; on the other hand, in CGA system, the mineralized froth is more steady which is benefit for flotation operation. As for the operation, the amount and size of carbonaceous material, type and amount of oil, coal/oil ratio (Calvez et al., 1998), time of agitation, (Wu et al., 2004) contact time (Bravo et al., 2005) and number of recycling of the agglomerates play a decisive role on the CGA (Gaidarjiev, 1996; Sen, 2000).

Based on the CGA, a new approach "coal-oil assisted gold flotation" was used to recover gold particles, only small amount of coal and agglomerating agents were added to the pulp to assist gold flotation (Sen et al., 2005). It improved the flotation performance for coarse (~300 μm) and fine (<53 μm) gold particles, and the process was not affected by the initial number of gold particles. However, our study found that the oil/coal ratio exerted a tremendous influence on gold recovery, increase of coal/oil ratio gave rise to the recovery, whereas the grade of the concentrates decreased for both synthetic and natural gold ore samples. Thus, a appropriate coal/oil ratio is very important.

To sum up, for the recovery of refractory gold ore with low grade, fine or ultrafine particles, complex sulfide type gold deposit, further developments of excellent regulator, new carrier materials and depressants are extremely urgent. In this study, laboratory and industrial scale tests are conducted to determine the effect of sulfur-oil agglomeration with $(NH_4)_2S_2O_3$ as the regulator on the flotation recovery of gold. Aim of this work is to compare the conventional flotation process (CGA method) with the new techniques, and two different types of ores are tested at laboratory scale. Comparisons were made through the separation parameters: grade and recovery of gold.

2. Experimental

2.1. Materials

2.1.1. Ore samples

(1) The first gold bearing ore (Sample I) used in this study is obtained from Wenshan, located in Yunnan region, Southwest of China. The ores (65% passing 74 μm) are seriously oxidized, and extremely fine disseminated. The ore body contains 2.02 g/t Au, 7.2 g/t Ag, 4.35% S on average. Gold is identified in its native form as well as associated with silver ores, which is the only mineral with economic value in the ore. Phase analysis shows that: quartz and feldspar, pyrite, muscovite contain Au 0.74 g/t, 34.5 g/t and 3.66 g/t, which account for 25.8%, 45.3% and 28.9% of the total content of gold in the ore, respectively. More than 50% of native gold particles are liberated under 1 μm in various shapes.

(2) The second gold bearing ore (Sample II) is taken from Honghe, also located in Yunnan region. The refractory gold ore sample mainly showed micro disseminated structures, which contains 1.68 g/t Au, 1.4% carbon and 0.59% S on average. Gold is mainly present as submicroscopic gold and more than 93.84% of the gold particles are liberated under 5 μm. Gold is identified associated with sulfides (86.26%) and quartz (7.58%), only 6.16% of gold is present in its native form.

2.1.2. Reagents

Kerosene by 99% (by weight) and alkyl phenol polyoxyethylene ether by 1% were emulsified in a four baffled emulsifying cell to produce oil emulsions, agitated at 10,000 rpm for 25 min. Copper sulphate and OP-20 are used as activator and emulsifier of kerosene. Butyl xanthate was employed as the collector, and the frother is MIBC. Sulfuric acid is applied to adjust pH to a proper value. The reagents used in our test were of analytically pure. Carbon is purchased from Shanxi, China, used in the CGA is the high quality bituminous coal with the ash content is <7%.

2.2. Methods

2.2.1. Conventional flotation

After being crushed and screened, the ore samples (−3 mm) were wet-milled and transferred directly to the 3.0 L XFG single-trough flotation cell. Then the flotation reagents were added into the pulp, and the pH was adjusted to 6.5. The flowsheet of closed-cycle flotation with single-stage gold roughing, two-stage gold cleaning, three-stage gold scavenging was conducted in the laboratory tests. Then the concentrate and tailing samples were collected, dried and chemical analyzed.

2.2.2. CGA method

Coal (<7% ash quality bituminous coal) particles (12% by weight) were agglomerated using emulsified kerosene before added to the flotation pulp. ESJ high-speed stirrer with speed control was used for agglomeration. The mixture was agitated at 2500 rpm for 10 min. Then the pre-agglomerated coal particles-oil-water suspension was added to the flotation pulp and agitated at 2500 rpm for 10 min. After that, stirring speed was decreased to 2000 rpm, then the same kinds of flotation reagents were used in conventional flotation experiments. pH of the pulp was adjusted to 6.5.

2.2.3. Sulfur-oil agglomeration

The ammonium thiosulfate, sulfur and emulsified kerosene were added into the mill simultaneously and mixed with the gold bearing pulp under the natural pH condition (about 7.5). After then the ore samples were transferred directly into the flotation cell. Pulp pH should be adjusted to 6.5 by H_2SO_4. Flotation procedure was the same as the conventional process.

2.2.4. SEM-EDS analysis

Scanning Electron Microscope and Energy Dispersive Spectrometer (SEM-EDS) tests were conducted by JSM-6360LV with the acceleration voltage 20 kV. Meanwhile, X ray energy dispersive spectrometer was configured with the version DAX.LTD, EDX-GENESIS 60S. Gold powder (0.32 g) was added into the solution made by a mass of $(NH_4)_2S_2O_3$ (2.325 g) mixed with 30 mL water. The pH value of the solution was regulated to 4 by sulfuric acid. Thereafter, the solution was stirred for 10 min, and then stirring for another 10 min with addition of 30 mL emulsified kerosene solution ($c = 0.1\%$). After that, the solution was filtered, washed by distilled water for 20 times and dried in the vacuum drying box under 30 °C. Finally, the solid sample was test by SEM-EDS.