



## Improved thiosulfate leaching of a refractory gold concentrate calcine with additives



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### ABSTRACT

One of the major problems of thiosulfate leaching was the high consumption of thiosulfate. In this work, additive was used to reduce thiosulfate consumption and simultaneously increase gold leaching. Thiosulfate leaching without additive was not effective to treat a refractory gold concentrate calcine with high iron, sulfur and arsenic due to its low gold extraction (72.7%) and high thiosulfate consumption (42.4%). Thus, several additives including sodium chloride, sodium sulfite, CMC (sodium carboxymethyl cellulose), CMS (sodium carboxymethyl starch) and HA (sodium humic acid) were used to improve the leaching. HA as a new kind of additive had the best performance, and it could improve gold extraction to 81.4%, which was obviously higher than the extraction by cyanidation (78.2%), and reduce thiosulfate consumption to 13.2%. As the XPS and SEM/EDS results indicated, there were less passivation species including copper sulfides, sulfur, iron hydroxide, etc. on the surface of calcine leached with the addition of HA. The role of the HA in the thiosulfate leaching was also preliminarily analyzed in this paper, and was proposed as follows: HA weakened the interaction between cupric ions and thiosulfate, and also eliminated the catalytic effects of iron minerals on thiosulfate decomposition; on the other hand, HA prevented passivation species from coating the surfaces of gold and gold-bearing minerals, and allowed the extraction of some encapsulated gold.

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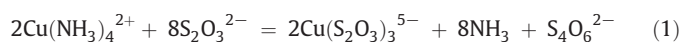
### 1. Introduction

The gold concentrate calcine used in this study was the product of oxidative roasting of a high-sulfur and high-arsenic gold concentrate produced in Hunan Province of China. Oxidative roasting technology is used extensively for the desulfidation and dearsenization of gold concentrate in China because this technology has certain advantages such as modest investment, high efficiency, and technical maturity. (De Michelis et al., 2013; Xu et al., 2014). To date, cyanide leaching has always been the predominant method for gold extraction due to its simple process and low cost, but now there are increasing worries regarding the toxicity of cyanide from government and public (Muir, 2011). Therefore, it was necessary to develop an appropriate non-cyanide method to extract gold from the gold concentrate calcine for the sake of environmental protection.

Among non-cyanide methods, chloride leaching, thiourea leaching and thiosulfate leaching have been the most widely studied in the past few decades. Gold can usually be efficiently extracted by chloride leaching. However, this method requires the use of hazardous chlorine and suitable corrosion resistant equipment. Furthermore, its reaction selectivity to impurities is poor (Hasab et al., 2014). Compared with cyanidation, thiourea leaching is an expensive technique due to its high price and high consumption (Örgül and Atalay, 2002). Compared

with the above-mentioned methods, thiosulfate leaching is attractive in terms of its reduced environmental risks, good reaction selectivity, mild investment, etc. (Jeffrey et al., 2003).

At present, the industrial application of thiosulfate leaching is still rare. The primary reason for that is the high consumption of thiosulfate during the leaching, particularly for the copper-ammonia oxidant system (Tanriverdi et al., 2005). It is widely considered that thiosulfate consumption takes place mainly via the reaction between Cu(II) and  $S_2O_3^{2-}$  ions (Breuer and Jeffrey, 2003a; Chu et al., 2003; Feng and van Deventer, 2007c). This reaction is shown as Eq. (1), and it can cause a lower Cu(II) concentration in leachate and thus diminishing gold leaching rate. Moreover, the tetrathionate generated by Eq. (1) will undergo further reactions of decomposition and hydrolysis to produce polythionates, sulfate, sulfite, etc. (Feng and van Deventer, 2002, 2007b; Senanayake, 2004). These sulfur species deriving from thiosulfate decomposition can form a compact coating layer consisting of sulfur, copper sulfide, etc. on gold surface, and hence lead to the passivation of gold leaching (Jeffrey et al., 2008). The ubiquitous iron species, including iron hydroxides and iron oxides, originating from mild steel grinding media, iron sulfides and iron bearing non-sulfides can also coat on the gold surface and cause the passivation (Feng and van Deventer, 2007a).



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A lot of works in the field of additive have been dedicated to the stabilization of thiosulfate. As has been reported (Chandra and Jeffrey, 2004; Senanayake, 2012; Senanayake and Zhang, 2012), the presence of anions such as sulfate, chloride, phosphate, etc. and organics such as EDTA (ethylene diamine tetraacetic acid), CMC (sodium carboxymethyl cellulose), and amino acids can depress the oxidation of thiosulfate by cupric ions and leave intact the catalytic capacity of cupric ions for gold leaching. The roles of these additives in thiosulfate leaching of gold are widely believed to be that they can compete with thiosulfate anions to complex with cupric ions at axial coordinate sites and thus reduce the interaction between cupric ions and thiosulfate (Breuer and Jeffrey, 2003b; Feng and van Deventer, 2011; Senanayake, 2012). In addition, CMC has highly negatively charged functional groups including carboxyl and hydroxyl, and renders all of passivation species, gold and mineral particles negatively charged due to its non-selective adsorption (Feng and van Deventer, 2011). So, CMC can prevent passivation species from coating the surfaces of gold and gold-bearing minerals, and thus hinders the passivation of gold leaching.

Base on the above, the thiosulfate leaching of a refractory gold concentrate calcine with several additives including sodium chloride, sodium sulfite, CMC, CMS (sodium carboxymethyl starch) and HA (sodium humic acid) has been studied in this paper to investigate a possible increase in gold extraction and/or decrease in thiosulfate consumption. The molecular structure of CMS is similar with that of CMC (Mihaela Friciu et al., 2013). Humic acid is an extremely complex mixture of large molecular organics and mainly occurs in peat, lignite, etc. Humic acid also has plentiful negatively charged functional groups such as carboxyl and hydroxyl groups (Allard, 2006), and is widely applied in agriculture, chemical industry and environmental protection. The role of HA during thiosulfate leaching was also studied by XPS and SEM/EDS analyses for the purpose of understanding its action mechanism.

## 2. Experimental work

### 2.1. Material and reagents

The gold concentrate calcine used in this study had particle sizes of 72% less than 44  $\mu\text{m}$ , and its chemical and mineralogical compositions are shown in Table 1. The gold content in this calcine was 84.27 g/t, and the contents of iron, sulfur and arsenic were as high as 31.25%, 1.03% and 0.55%, respectively. The mineralogical phases of the calcine were determined by quantitative X-ray diffraction analysis, and the analysis result showed that the main minerals were hematite, quartz and magnetite. Table 2 shows the result of chemical phase analysis of gold in the calcine ground to 90% finer than 44  $\mu\text{m}$ . As the result showed, 78.8% of gold was exposed (i.e. cyanide leachable), and the others were mainly encapsulated in iron oxides except that 6.6% of gold occurred in sulfide minerals including arsenopyrite and pyrite.

The reagents, such as sodium thiosulfate, sodium cyanide, cupric sulfate, ammonia solution, sodium hydroxide, sulfuric acid, sodium chloride, sodium sulfite, CMC, CMS, and HA, which were used in this study were all analytically pure. Redistilled water was used throughout all experiments. The HA derived from lignite was provided by Guangzhou Luyuan Chemical and Glass instrument Co, Ltd., China. Its chemical

composition is 55.6% C, 2.5% H, 21.7% O, 1.0% N, 0.53% S, 18.1% Na, 0.28% Al, 0.18% Fe and 0.12% Ti.

### 2.2. Analytical techniques

Au, Cu and Fe were all determined using an atomic absorption spectrometer (AA-6800, SHIMADZU). Cupric concentration was monitored at 618 nm using a UV-Vis spectrophotometer (721N, INESA). Sulfate ion concentration was measured by precipitation with barium chloride. For the purpose of detecting free cyanide, the cyanide leachate added with tartaric acid and zinc nitrate was first heated and then the released hydrogen cyanide was absorbed by sodium hydroxide solution, and the cyanide in the lye was measured by silver nitrate titration with a rhodanine indicator in the end. Thiosulfate concentrations were analyzed by an iodometric method with the indicator Vitex. In order to eliminate the effects of cupric tetra-amine complex and sulfite on the iodine titration, a certain amount of EDTA-2Na and formaldehyde was added prior to the titration. Ammonia concentration was tested by  $\text{H}_2\text{SO}_4$  titration with the indicator methyl red.

Chemical phase analysis was widely utilized in China to determine the distribution ratio of gold for its every phase in gold-bearing material, and the analysis procedure was as follows: at first, the phases where Au occurred were ascertained by optical microscope identification and electron microprobe analysis; and then selective dissolution was performed to determine Au content in each phase with certain chemical reagents.

All calcine samples were adequately rinsed with distilled water and then dried under vacuum overnight before XPS and SEM/EDS analyses. XPS was employed to detect the surface species of calcine samples by an X-ray photoelectron spectrometer (ESCALAB 250Xi, Thermo Fisher). Calcine samples were subjected to SEM/EDS analysis in order to study their surface topographies and compositions, which was implemented using a scanning electron microscope (MIA3, TESCAN) coupled with an energy dispersive X-ray analyzer (Oxford).

### 2.3. Leaching tests

All leaching tests were performed in 1 L baffled PVC reactor using an overhead stirrer with a flat-bladed impeller, which was open to air through a sampling port. A rotary splitter was used to obtain representative samples from the gold concentrate calcine blended uniformly by four times conings. These samples were further ground to a particle size of 90% less than 44  $\mu\text{m}$  by using a ball mill before they were leached. For each leaching test, 0.5 L of distilled water containing desired quantities of requisite reagents was firstly transferred into the reactor, and then a given mass of calcine was added and the pulp formed was simultaneously agitated at a constant speed of 200 rpm. Pulp pH was adjusted to a preset value with the addition of NaOH (3 mol/L solution) or  $\text{H}_2\text{SO}_4$  (10 vol.% solution), and was kept steady by readjustment every 0.5 h. Pulp temperature was kept at  $25 \pm 0.5$  °C with a water bath.

When leaching was completed, the pulp was filtrated and the obtained filtrate was immediately subjected to iodine titration. The leaching percentage of Au was calculated based on the analysis of the leach residue, which was washed adequately. Duplicate leaching tests were implemented with only average results reported because the standard deviations of all the tests were within 2%. The mass balance for

**Table 1**  
Chemical and mineralogical compositions of calcine sample (mass %).

|                            | Au                | Ag                | $\text{SiO}_2$ | $\text{Al}_2\text{O}_3$ | CaO          | MgO    |
|----------------------------|-------------------|-------------------|----------------|-------------------------|--------------|--------|
| Chemical compositions      | 84.3 <sup>a</sup> | 13.6 <sup>a</sup> | 37.96          | 5.97                    | 1.55         | 0.72   |
|                            | As                | Cu                | Pb             | S                       | Sb           | Fe     |
|                            | 0.55              | 0.10              | 0.24           | 1.03                    | 0.25         | 31.25  |
| Mineralogical compositions | Hematite          | Magnetite         | Arsenopyrite   | Pyrite                  | Chalcopyrite | Quartz |
|                            | 46.3              | 6.2               | 1.3            | 0.9                     | 0.18         | 37.2   |

<sup>a</sup> Unit g / t.

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