Minerals Engineering 24 (2011) 1703–1709

Contents lists available at SciVerse ScienceDirect

Minerals Engineering

journal homepage: www.elsevier.com/locate/mineng

Diagnostic pre-treatment procedure for simultaneous cyanide leaching of gold and silver from a refractory gold/silver ore

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ARTICLE INFO

Article history:
Received 16 May 2011
Accepted 22 September 2011
Available online 22 October 2011

Keywords:
Gold ores
Precious metal ores
Sulfide ores
Cyanidation
Leaching

ABSTRACT

This study investigates the optimization of simultaneous dissolution of gold and silver from a refractory gold ore through determination of pre-treatment stages. Based on the mineralogical studies (thin layer and polished section) and chemical analysis on the ore sample, a "diagnostic leaching" procedure was designed. Results from diagnostic leaching suggest that the most effective pre-treatment agents for gold and silver are ferric chloride and sulfuric acid media, respectively. Optimum conditions for the simultaneous dissolution of gold and silver were determined using a two factorial design technique. Pre-treatments with sulfuric acid and ferric chloride reagents increased the efficiency of the dissolution of gold from 54.7% to 82% and silver from 37.4% to 81.6%.

1. Introduction

One of the most important problems facing the gold and silver industry is that the placer and free milling gold and silver ores are almost terminating. Hence, the use of refractory ores has been increased during the recent years. In general, gold refractory ores are classified into carbon, sulfide and tellurium ores. Among the refractory ores, the sulfide type is the most common (Lunt and Briggs, 2005). Each of these ores, in turn, requires special flow sheet for gold and silver extraction (Gupta, 2003).

To extract gold and silver by hydrometallurgical route, cyanide leaching has been the dominant process in gold mining industry in the past two centuries. To reduce environmental and toxicity risks associated with the use of cyanide, many efforts have been made to replace cyanide media with other reagents such as thiosulfate (Aylmore and Muir, 2001), thiourea (Groenewald, 1977), halide (Tran et al., 2001), oxidative chloride (Sparrow and Woodcock, 1995), bisulfide (Hunter et al., 1998), ammonia (Han, 2001), thiocyanate (Monhemius and Ball, 1995) and bacterial leaching (Sparrow and Woodcock, 1995). Cui and Zhang (2008) compared with different leaching methods for recovering precious metals. Cyanidation is still the dominant method for extracting gold and silver from the ores. However, cyanidation is not suitable for refractory ores; therefore, according to the ore characterization, a variety of pre-treatments are used to increase dissolution efficiency. The methods used for increasing the efficiency of conventional cyanidation performance applied to sulfide ores are bio-oxidation, pressure oxidation and roasting (Miller and Brown, 2005; Thomas, 2005; Thomas and Cole, 2005; Hammerschmidt et al., 2005). In cases, where gold sulfide ores are presented in the form of fine particles inside the matrix of gangue minerals, ultra fine grinding to $-10\ \mu m$ is also applicable as a pre-treatment method.

In addition to the above-mentioned methods, *diagnostic leaching* based on oxidative leaching has also been used as pre-treatment for the refractory ores (Lorenzen and Van Deventer, 1992; Henley et al., 2000; Torres et al., 1999). The basis of this method is, first, to identify the host phase(s) for the precious metals using a series of oxidative leaching reagents; then, determining the required pre-treatments to liberate the metals. The oxidizing media do not have the ability to bring gold into the solution; therefore, cyanidation steps are also required. Most of the researches in this case are focused on gold (Lorenzen and Tumilty, 1992; Torres et al., 1999) and a few on silver (Celep et al., 2009; Rohde et al., 2011) and lead them (Greet and Smart, 2002).

The main problem facing the silver diagnostic leaching is the silver affinity to form complexes with some of the oxidizing reagents. In the presence of nitric acid as an oxidizing reagent, silver nitrate ($AgNO_3$) is formed. The nitrogen in nitrate ion is reduced from 5 to 4 and nitrogen dioxide gas forms. Also black precipitates of silver oxide (Ag_2O) are produced (Cotton, 1997). In the presence of ferric chloride AgCl precipitates is formed which is dissolved later in the presence of excessive chloride ions to form different silver chloride complexes of $AgCl_2^-$ and $AgCl_3^{2-}$ (Griffith, 1990). According to the following reactions, the AgCl precipitates dissolve in the presence of excessive chloride ions to form different silver chloride complexes:

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doi:10.1016/j.mineng.2011.09.013

$$AgCl + Cl^- \rightarrow [AgCl_2]^-_{(aq)} \quad (1)$$

$$[AgCl_2]^-_{(aq)} + Cl^- \rightarrow [AgCl_3]^{2-}_{(aq)} \quad (2)$$

$$[AgCl_3]^{2-}_{(aq)} + Cl^- \rightarrow [AgCl_4]^{3-}_{(aq)} \quad (3)$$

However, the previous researchers have not considered this large amount of silver which could be as high as 1 g/L, as suggested by Dinardo and Dutrizac (1985).

In this study, a diagnostic leaching process was designed for the simultaneous dissolution of gold and silver from a refractory sulfide ore. Accordingly, a suitable pre-treatment procedure for increasing the efficiency of cyanide dissolution of gold and silver was proposed. Two factorial design of experimental technique and ANOVA were used to optimize the pre-treatment procedure for simultaneous cyanide dissolution of gold and silver.

2. Design of experiments

The leaching procedure used in the diagnostic leaching procedure in this study is shown in Table 1. This table was constructed based on the pre-treatment leaching stages suggested by Lorenzen (1995). According to the mineralogical studies in this work, some of the stages suggested by Lorenzen (1995) were eliminated, as it is clear from Table 1 that cyanide washing by sodium cyanide (NaCN) was done to destroy the precipitated gold and silver. In the leaching pre-treatment stage, sodium cyanide, hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) were used to leach gold, calcite and the labile base metal sulfides, respectively. To destroy sphalerite, galena and tetrahedrite, ferric chloride ($FeCl_3$) was added and nitric acid (HNO_3) was the leaching reagent for the pyrites mineral.

Optimization of the pre-treatment stages was carried out using a two factorial technique. Optimization parameters were considered as categorical parameters to determine their value at which maximum dissolution of gold and silver were reached. By using two factorial technique, oxidizing reagents in diagnostic leaching were divided into two categories. The first group (Factor A) consisted of ferric chloride and hydrochloric acid and the second group (Factor B) consisted of sulfuric acid and nitric acid, are illustrated in Table 2.

3. Experiments

3.1. Materials

The sulfide ore used in this study was obtained from Sardasht area in Northwestern of Iran. Elemental analysis of the sample is presented in Table 3. It is shown that the ore sample contained ca. 5.4 ppm Au and 325 ppm Ag. Inductively coupled plasma (ICP) model VISTA-PRO was used to measure gold and silver reported to the solution. The leach residue was also analyzed by fire assay and atomic absorption spectroscopy (AA) to determine mass balance for both gold and silver and calculate the metals recovery.

The ore sample was divided into two homogeneous fractions. For the mineralogical tests, one fraction was divided into six size fractions; 100% passing coarser than 300 μm (+50 mesh), 212–300 μm (50–70 mesh), 150–212 μm (70–100 mesh), 75–150 μm (100–200 mesh), 63–75 μm (200–230 mesh) and finer than 63 μm (−230 mesh). For the diagnostic leaching experiments, another fraction of the homogenized ore sample was ground into 80% passing 37 μm (400 mesh) using a laboratory ball mill for 45 min. All of the reagents used in this study were analytical grade obtained from Merck.

3.2. Mineralogical analysis

To design a diagnostic leaching procedure efficiently, a complete mineralogical examination of the ore sample is required (Lorenzen, 1995). Mineralogical analysis was done using X-ray diffraction (XRD), polished sections and thin sections microscopy to study all the phases presented in the ore sample. For this purpose, first, the sample was divided into six size fractions (Section 3.1) and then, thin and polished sections were prepared from each size fraction. Thin sections were prepared by cementing a thin slice of the tailing to the glass. A cover slip was cemented on top of the sample. Transmitted light microscopy was used to examine minerals that transmitted light in the thin sections. These minerals included non-metallic minerals. A polished section of the sample was also prepared and reflected light microscopy was used to examine minerals that did not transmit light in a thin section, but reflected light to varying degrees when polished (Espiari et al., 2006). The XRD results are given in Fig. 3a and b. Fig. 3a suggests that the ore sample consisted of the major phase of quartz and the minor phases of barite, pyrite and calcite. Fig. 3b illustrates

Table 1
Mineralogical analysis and selective pre-treatment leaching stages and the minerals destroyed (adopted from Lorenzen, 1995).

Metallic phase (10%)		Non-metallic phase (90%)		Pre-treatment stages	Minerals likely to be destroyed
	Content		Content		
Pyrite	65–70	Silica	60	NaCN washing	Precipitated gold
Galena	15–23	Barite	25	NaCN	Gold
Sphalerite	3–7	Calcite	15	HCl	Calcite, calcium carbonate
Tetrahedrite	<2			H_2SO_4	Sphalerite, labile copper sulfides, labile base metal sulfides, labile pyrite
				$FeCl_3$	Sphalerite, galena, tetrahedrite
				HNO_3	Pyrite, arsenopyrite, marcasitef

Table 2
Experimental design using two factorial technique.

Std	Run	Factor A 1 = $FeCl_3$, −1 = HCl	Factor B 1 = H_2SO_4, −1 = HNO_3	Ag Rec (%)	Au Rec (%)
2	1	1	−1	50.9	78.7
3	2	−1	1	86.5	74.5
1	3	−1	−1	55.8	70.9
4	4	1	1	81.6	82.0