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Characterization of an iron oxy/hydroxide (gossan type) bearing refractory gold and silver ore by diagnostic leaching

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Abstract: A detailed characterization of an iron oxy/hydroxide (gossan type) bearing refractory gold/silver ore was performed with a new diagnostic approach for the development of a pretreatment process prior to cyanide leaching. Gold was observed to be present as native and electrum (6−24 μm in size) and associated with limonite, goethite and lepidocrocite within calcite and quartz matrix. Mineral liberation analysis (MLA) showed that electrum is found as free grains and in association with beudantite, limonite/goethite and quartz. Silver was mainly present as acanthite (Ag_2S) and electrum and as inclusions within beudantite phase in the ore. The cyanide leaching tests showed that the extractions of gold and silver from the ore (d_{80}: 50 μm) were limited to 76% and 23%, respectively, over a leaching period of 24 h. Diagnostic leaching tests coupled with the detailed mineralogical analysis of the ore suggest that the refractory gold and silver are mainly associated within iron oxide mineral phases such as limonite/goethite and jarosite-beudantite, which can be decomposed in alkaline solutions. Based on these characterizations, alkaline pretreatment of ore in potassium hydroxide solution was performed prior to cyanidation, which improved significantly the extraction of silver and gold up to 87% Ag and 90% Au. These findings suggest that alkaline leaching can be used as a new diagnostic approach to characterize the refractoriness of iron oxy/hydroxide bearing gold/silver ore and as a pretreatment method to overcome the refractoriness.

Key words: refractory gold and silver ore; characterization; diagnostic leaching; mineral liberation analysis (MLA); alkaline pretreatment

1 Introduction

Refractory gold and silver ores yield low gold and silver extractions in cyanide leaching [1]. A suitable pre-treatment process such as roasting [2], pressure oxidation [3], bio-oxidation [4], alkaline [5,6] or alkaline sulfide leaching [7] and, to a limited extent, ultrafine grinding [8−10], is often required to overcome refractoriness and render the encapsulated gold and silver accessible to the lixiviant action of cyanide and oxygen.

The gossan ores are intensely oxidized, weathered or decomposed rock, usually the upper and exposed part of an ore deposit or mineral vein. The gossan (or iron cap) ores mainly consist of quartz and iron oxides, limonite, goethite, and jarosites. It has been reported that the natural jarosite-type minerals such as beudantite [$PbFe_3(SO_4)(AsO_4)(OH)_6$], argentojarosite [$AgFe_3(SO_4)_2(OH)_6$] and the silver-bearing plumbojarosite [$(Ag,Pb)Fe_3(SO_4)_2(OH)_6$] are the major silver (230 g/t Ag in average) carriers in the gossan ores from Rio Tinto (Spain) [11−14]. Gold and silver extractions from these types of ores are often limited to 70%−80% Au and 40% Ag since jarosite-type phases are not readily dissolved during the conventional cyanidation process [15].

The diagnostic leaching procedure developed by LORENZEN [16] is a characterization method to determine the reasons of refractory behavior of gold and silver ores. It involves cyanide leaching to extract unlocked gold or silver after each stage of acid treatment using a variety of mineral acids selected to decompose gold/silver-bearing target mineral(s) [17−19]. The decomposition of some mineral phases in different acid leaching stages may be represented by the following chemical reactions [20−22]:

$$CaCO_3+2HCl\rightarrow CaCl_2+H_2O+CO_2 \quad (1)$$

$$MS+H_2SO_4\rightarrow MSO_4+H_2S\ [M=Zn,\ Cu,\ Pb,\ etc.] \quad (2)$$

$$2FeS_2+10HNO_3\rightarrow 2Fe^{3+}+2H^{+}+4SO_4^{2-}+10NO+4H_2O \quad (3)$$

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$$SiO_2+6HF\rightarrow H_2SiF_6+2H_2O \tag{4}$$

LORENZEN and van DEVENTER [18] used diagnostic leaching to identify the refractory nature of gold ores (only 1.3%−2.4% leachable gold with direct cyanidation). It was found that approximately 95% of Au in ore was extracted by means of selective destruction of gold-bearing minerals by nitric and sulphuric acids within diagnostic leaching procedure. TEAGUE et al [23] studied diagnostic leaching of a refractory gold ore and a bulk flotation concentrates. HENLEY et al [24] studied the characterization of a refractory telluride ore by a two-stage diagnostic approach. CELEP et al [22] concluded that the refractoriness of a refractory gold ore was associated with the dissemination and encapsulation of the very fine gold particles largely within carbonates, oxides, sulfides and, to a small extent, silicates present in the ore matrix. Although the diagnostic leaching procedure includes different acid leaching stages and the subsequent cyanidation, it is inefficient to decompose jarosite type minerals. In this work, an alkaline leaching was adopted as a diagnostic leaching stage for decomposition of jarosite type minerals.

Detailed characterization such as mineral liberation size, mineral associations and modal mineralogical analysis is pre-requisite for plant design and mineral processing operations [25]. The distribution of precious metal, grain size and mode of occurrence (liberation, exposure, and mineral association) and their gangue components were quantitatively determined by means of automated scanning electron microscopic techniques (QEMSCAN/MLA). The mineral liberation analysis (MLA) system consists of a specially developed software package and a scanning electron microscopy (SEM) equipped with an energy dispersive spectrum (EDS) analyzer. The analysis was operated on particles mounted in 30 mm epoxy resin blocks to provide automated quantitative mineral liberation characterization [26]. The system utilized stable back-scattered electron (BSE) signals defined by its average atomic number to generate images of mineral grains from a scanning electron microscope (SEM). Mineral identification was verified by using of a single X-ray analysis at a point within the particle. Parameters such as liberation, mineral grain size, mineral locking and mineral associations were then measured by the system. In this system, X-ray mapping was also used when similar BSE brightness of minerals was identified [27]. There are seven different operating modes available for the MLA system: standard BSE liberation analysis (BSE), extended BSE liberation analysis (XBSE), sparse phase liberation analysis (SPL), particle X-ray mapping (PXMAP), selected particle X-ray mapping (SXMAP), X-ray modal analysis (XMOD), and rare phase search (RPS) [28].

In this work, the detailed characterization of a gold- and silver-bearing gossan type ore was performed to provide an insight into the refractory behaviour of the ore. The mineral liberation analysis was undertaken to determine the occurrence and liberation of gold and silver, and to identify refractory silver-bearing phases. A new, modified diagnostic approach was developed for gossan type ore so as to identify the refractions of silver-bearing minerals as well as gold-bearing phases. Based on the results of these characterizations, an alkaline pretreatment process was proposed and demonstrated on the ore sample.

2 Experimental

2.1 Materials

The ore sample (~150 kg) was obtained from Bolkardağ–Niğde (Turkey) gold and silver deposit. It was crushed to <1 mm in a jaw crusher and then ground in a rod mill. The chemical composition of the ore sample (Table 1 and Table 2) was determined using inductively coupled plasma-emission spectroscopy (ICP-ES) after four kinds of acid digestion and X-ray fluoressans (XRF). The gold and silver contents of the ore were determined by lead collection fire assay fusion−gravimetric finish method. The ore sample was found to contain 8.4 g/t Au, 167.0 g/t Ag, 2.6% Pb, 4.8% Zn and 1.6% As mass fraction. Quartz, goethite, calcite and dolomite were identified as the major mineral phases by X-ray diffraction analysis (Fig. 1). The optical mineralogical analysis of the ore sample was also performed under an ore microscope (Leitz Wetzlar 1432) for which the polished sections of a number of hand-picked pieces were prepared from the as-received ore.

Table 1 Chemical analysis of ore sample (mass fraction, %)

SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O
23.9	4.31	36.9	1.3	6.18	0.06	0.87
TiO_2	P_2O_5	MnO	Cr_2O_3	C_{Total}	S_{Total}	LOI
0.17	0.02	1.44	0.15	1.72	0.55	15.3

Table 2 Element analysis of ore sample (mass fraction, %)

Ag	Au	Ni	Ba	Sn	
1.67×10^{-2}	8.4×10^{-4}	5.16×10^{-2}	1.97×10^{-2}	1.72×10^{-2}	
Cd	Sb	Cu	Pb	Zn	As
3.92×10^{-2}	2.63×10^{-2}	0.15	2.60	4.80	1.59

2.2 Methods

2.2.1 Sample preparation

The crushed ore sample was ground by a rod mill for 3−50 min at 50% pulp density. Ultra-fine grinding of the ore was carried out using 2 mm ceramic ball at 800 r/min stirring rate in a laboratory scale pin-type vertical