



Implementation of sodium hydroxide pretreatment for refractory antimonial gold and silver ores

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ABSTRACT

Alkaline pretreatment of a refractory gold–silver ore containing antimony minerals such as stibnite, andorite ($\text{Sb}_3\text{PbAgS}_6$) and zinkenite ($\text{Pb}_9\text{Sb}_{22}\text{S}_{42}$) was tested using sodium hydroxide in order to determine its effectiveness in improving the recovery of gold and silver. Mineralogical investigations show that silver was present as andorite and Au/Ag alloy. Gold particles have been observed as associated with quartz and inclusions within the antimony minerals. Increasing the sodium hydroxide concentrations from 0.5 to 5 mol/L, increasing the temperature from 20 to 80 °C, and reducing the particle size from 50 to 5 μm enhanced the removal of antimony from the ore. Up to about 75.5% Sb removal was achieved by alkaline pretreatment, which in turn remarkably improved the extraction of silver from levels of less than 18.7% to 90% and gold from less than 49.3% to 85.4% during subsequent cyanidation. These findings, consistent with mineralogical results, suggest that alkaline leaching can effectively be used as chemical pretreatment method as an alternate to the alkaline sulfide leaching in the processing of refractory antimonial gold–silver ores.

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1. Introduction

The Akoluk ore deposit, hosted by volcanic–sedimentary rocks of the Eastern Pontides, is situated near Ordu in northeastern Turkey. The ore deposit contains a variety of sulfide and oxide minerals, as well as native gold (Tüysüz and Akçay, 2000; Yaylılı-Abanuz and Tüysüz, 2010). Total reserves are estimated to be about 1 million tonnes (Anon, 1993). The most significant trace and minor minerals are associated with stibnite and zinkenite (Pb–Sb sulfosalt), which along with sphalerite are the most widespread ore minerals in the deposit (Ciftci, 2000). According to Aslaner and Ottemann (1972), gold occurs as inclusions in zinkenite (PbSb_2S_4). Recently, Celep et al. (2009) reported that the ore consisted predominantly of quartz, the illite/kaolinite group of clay minerals and barite with lesser amounts of pyrite, stibnite, sphalerite, zinkenite and andorite. Gold occurs as small particles ranging from 1 to 88 μm in association with sulfide minerals and quartz. Cyanide leaching tests of 24-h duration showed that metal extractions were consistently low, at less than 47% for gold and less than 19.2% for silver. Diagnostic leaching tests suggested that the decomposition of the sulfide could improve the extraction of gold and silver by about 29.5% and 56.7%, respectively. Detailed mineralogical characterization of the Akoluk gold–silver ore has indicated that the ore contains antimony sulfides, including andorite ($\text{Sb}_3\text{PbAgS}_6$) and zinkenite ($\text{Pb}_9\text{Sb}_{22}\text{S}_{42}$), as the main silver and gold

carriers (Alp et al., 2010; Celep et al., 2011). Ultrafine grinding and roasting of the ore were ineffective as pretreatment for improving the recovery of gold and silver by cyanidation (Celep et al., 2010). Cyanidation tests also showed that lead nitrate addition had a limited effect on gold and silver extractions. The low gold and silver extractions from the ore indicated its refractory nature to cyanide leaching and the need for a suitable chemical pretreatment process to improve metal dissolution.

Refractory gold ores do not respond to direct cyanidation; therefore, such ores have to be pretreated prior to cyanidation to liberate the contained gold and silver so that they are readily amenable to extraction (La Brooy et al., 1994). Roasting, pressure oxidation, bio-oxidation and, to a limited extent, ultrafine grinding have been commercially applied to increase gold recoveries from refractory ores (Corrans and Angove, 1991; Iglesias and Carranza, 1994; Gunyanga et al., 1999). Alkaline sulfide leaching is a suitable pretreatment process for antimonial ores and concentrates (Ubaldini et al., 2000; Baláž and Achimovičová, 2006; Curreli et al., 2009; Awe and Sandström, 2010; Awe et al., 2010) by making the silver available to cyanide solutions or removing the hazardous or penalty elements such as As and Sb from the ores and concentrates. Alp et al. (2010) and Celep et al. (2011) have already shown that alkaline sulfide leaching is an effective pretreatment method ahead of cyanide leaching for the extraction of gold and silver from antimonial refractory ores. Recoveries as high as 90% Ag and 82.6% Au were achieved after the removal of 95% Sb in an alkaline pre-treatment stage under the conditions of 4 mol/L Na_2S and NaOH, $\leq 15 \mu\text{m}$ particle size, and 80 °C (Alp et al., 2010). In addition, increasing NaOH concentrations

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enhanced the dissolution of Sb-bearing minerals. This finding was consistent with reports on leaching of antimonial sulfides such as stibnite (Anderson and Kryš, 1993; Ubaldini et al., 2000; Smincáková and Komorová, 2005; Smincáková, 2009). Smincáková (2009) showed that the leaching of stibnite by sodium hydroxide was possible. Baláž (2000) showed that proustite (Ag_3AsS_3) and pyrargrite (Ag_3SbS_3) decompose during the alkaline leach, which makes high silver recovery possible during subsequent cyanidation. No such studies on alkaline pretreatment of antimonial ores containing andorite ($\text{Sb}_3\text{PbAgS}_6$) and zinkenite as the main gold/silver bearing minerals have been reported in the literature.

Recent studies (Alp et al., 2010; Celep et al., 2011) indicated that gold losses (up to 13%) occur during the alkaline sulfide leaching and the losses increase at high temperatures and high concentrations of sulfide. Furthermore, considering the problematic health issues associated with the sulfide leaching, this study was designed to evaluate alkaline leaching using sodium hydroxide as a potential pretreatment process alternative to alkaline sulfide leaching.

2. Experimental

2.1. Materials

The ore used in this study is an antimony-rich refractory gold-silver ore from Akoluk (Ordu-Turkey). The ore samples were reduced in size by crushing and grinding. A laboratory-scale stirred media mill was used for fine grinding (80% passing size, $d_{80} = 50, 15$ and $5 \mu\text{m}$) prior to the leaching tests. Particle size analysis of the ore samples was performed by a Malvern Mastersizer 2000 model laser analyzer. The earlier chemical and mineralogical studies (Celep et al., 2009) indicated that it is a high grade gold and silver ore assaying at 220 g/t Ag and 20 g/t Au. In addition, the ore contained 52.2% SiO_2 , 17.1% Ba, 4.7% Al_2O_3 , 6.9% S, 1.6% Sb, 1.5% Zn, 1.3% Fe_2O_3 , 0.4% Pb, 0.02% As and 0.04% Cu (all in weight percent). Quartz, the illite/kaolinite group clay minerals and barite are the predominant phases in the ore. Pyrite, stibnite, sphalerite, zinkenite and andorite are the main sulfide minerals identified in the ore (Celep et al., 2011).

2.2. Mineralogical characterization of the ore

Mineralogical analysis of the ore sample was performed to determine gold and silver bearing phases. Characterization studies were carried out using a FEI Quanta 400MK2 Scanning electron microscopy (SEM) equipped with EDAX Genesis 4XMI at the Mineral Research and Exploration Institute of Turkey and a HITACHI variable-pressure SEM with a Link microanalysis system at CANMET. Microanalysis of the mineral grains was performed by a JEOL JXA 8900 electron probe X-ray microanalyzer (EPMA) at CANMET utilizing five wavelength dispersive spectrometers (WDS) operated at 20 kV with a probe current of 20 to 30 nA.

2.3. Experimental work

Tests were designed to evaluate the effects of sodium hydroxide concentrations, temperature and particle size (d_{80} : 5–50 μm). The ground samples (d_{80} : $\leq 50 \mu\text{m}$) were leached in a 1-L glass reactor immersed in a water bath to control the leaching temperature (20–80 °C) within ± 2 °C. The vessel with 200-mL leach solution (NaOH) and 70 g ore sample (solids 35% w/vol) was continuously stirred at 750 rpm. Sodium hydroxide (NaOH, assay 99.9% Merck) in the range of 0.5–5 mol/L NaOH was used to maintain the alkalinity. The leach solution was sampled (10 mL) at regular time intervals for the analysis of antimony. At the end of leaching after 120 min, solid and liquid phases were separated by filtration and the filtrates were analyzed for Sb, Au, Ag, Pb, Zn, Cu and Fe. The residues were air-dried, and sampled for analysis to determine the metal recoveries.

Cyanide leaching of the residues was then carried out to determine the effects of alkaline leaching pretreatment on the extraction of gold and silver. Glass reactors (1 L) were used for cyanide leaching of the residues. The reactors were mechanically agitated with pitched-blade turbine impellers and aerated at a flow rate of 0.3 L/min (Alp et al., 2010; Celep et al., 2011). NaCN (Merck) and NaOH were used to adjust the pH at 10.5 during cyanidation. In all tests, 10-mL samples were taken from the leach pulp at pre-determined time intervals and then centrifuged to obtain clear aliquots for the determination of Au, Ag and free cyanide in solution. Silver nitrate titration in the presence of p-dimethylamino-benzal-rhodanine (0.02% w/w in acetone) as the indicator was used to determine the concentration of free CN^- in samples (Celep et al., 2011). If required, concentrated cyanide solution (5% NaCN) was added to maintain free CN^- concentration at the initial level of 1.5 g/L NaCN, and consumption of NaCN was recorded (Celep et al., 2011). On the termination of cyanide leaching tests, the residues were digested in acid (HCl, HNO_3 , HClO_4 and HF) to determine the undissolved metal content. Analysis of gold, silver and antimony from the solutions was carried out using an atomic absorption spectrometer (AAS-Perkin Elmer Analyst 400). The extraction of metals was calculated based on the metal content of leaching residues.

3. Results and discussion

3.1. Alkali pretreatment

Alkaline leaching has a significant advantage over the alkaline sulfide leaching due to lower reagent costs. In addition, a small amount of gold can be dissolved in alkaline sulfide solutions (Anderson, 2001; Jeffrey and Anderson, 2003; Alp et al., 2010; Celep et al., 2011). This situation will bring additional costs in the course of gold recovery from solution. The alkaline sulfide leaching has also potential environmental issues related to the formation of H_2S gasses.

3.1.1. Dissolution of metals during pretreatment

Alkali pretreatment of the ore at 15 μm (d_{80} particle size) using 3 mol/L NaOH at 80 °C in sodium hydroxide solution caused the dissolution of elements in the following proportions: 64.4% Sb, 0.5% Ag, 0.06% Fe, 1.5% Cu, 0.05% Zn and 0.03% Pb. The pretreatment had no effect on the dissolution of gold. The results indicate that sodium hydroxide leaching is highly selective for the removal of antimony from the ore.

Although alkaline leaching using alkali metal hydroxides is potentially applicable to elements that form anionic complexes such as Al, Sb, As, Cu, Fe and Pb, the higher metal extractions would require high pressure or temperatures under oxidizing conditions (Gupta and Mukherjee, 1990; Filippou et al., 2007). The alkaline leaching (3 mol/L NaOH, 80 °C, d_{80} : 15 μm) had no important effect on the dissolution of metals with the exception of antimony. For this reason, the effects on the removal of antimony of NaOH concentration, temperature and particle size were investigated.

3.1.2. Effect of NaOH concentration

The effect of leaching time on antimony removal from the ore was investigated at different reagent concentrations (0.5–5 mol/L NaOH) at the fixed slurry temperature of 80 °C. The experimental results are presented in Fig. 1. Antimony dissolution varies between 10.4 and 70.1%. Increasing leaching time resulted in the increase of antimony extraction. Most of antimony dissolution had occurred within the first 5 min at high reagent concentrations. When the leaching time was increased to 2 h, antimony removal was improved only slightly, even at 5 mol/L NaOH.

Fig. 1 shows the effect of NaOH concentration (0.5–5 mol/L) on the removal of Sb from the ore ($d_{80} = \leq 15 \mu\text{m}$) at 35% w/v solids and 80 °C. The dissolution of Sb improved with increasing the

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