Effect of Na₂S+NaOH pre-identification leaching on oxidized refractory Au-Ag ores

Oksitlenmiş refrakter Au-Ag cevherleri üzerinde Na₂S+NaOH ön tanımlama liçinin etkisi

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Abstract

This study was conducted to investigate the effect of the alkaline sulfide (Na₂S+NaOH) pre-identification process on cyanide leaching performance in oxidized refractory gold-silver ores. The ore has intensive oxide, carbonate, and silicate content, and contains Au/Ag minerals containing electrum, acanthite, and silver-bearing FePbZn Sulphate-Arsenate/Hydroxide minerals together with Fe, Pb, and Zn minerals. Niğde Ulukışla refractory Au/Ag ore has 8-9 g/T gold and 170-180 g/T silver grades. In mineralogical examinations, it was found that these metals were closed in minerals such as iron oxy/oxyhydroxide, calcite, and quartz or neighboring these minerals. In the cyanide soluble process after alkali sulfide preliminary procedure at room temperature, metal extraction yields were obtained as 62% for gold and 7% for silver. It is seen that silver is adversely affected by this process compared to the direct cyanidation process. However, after the hot alkali treatment process, metal extractions improved by approximately 25% of gold and 74% of silver. This increase in yield can be considered as converting Ag-oxyhydroxide into soluble Ag forms as a result of the effect of hot alkaline sulfur leaching on the cyanide process. The most important advantage of this decomposition process is to achieve high metal extraction efficiency in lower alkali concentrations.

Anahtar kelimeler: Na₂S, NaOH, Gold, Silver, Refractory ore

Öz

Bu çalışma, oksitli refrakter altın-gümüş cevherlerinde alkali sülfür ön tanımlama işleminin siyanür liç performansına etkisini araştırmak amacıyla yapılmıştır. Yoğun oksit, karbonat ve silikat içeriğine sahip olan cevherde Fe, Pb ve Zn minerallerinin yanı sıra elektrum, akantit ve gümüş içeren FePbZn Sülfat-Arsenat/Hidroksit minerallerini içeren Au/Ag mineralleri de bulunmaktadır. Niğde Ulukışla refrakter Au/Ag cevheri 8-9 g/t altın ve 170-180g/t gümüş tenörlerine sahiptir. Mineralojik incelemelerde, bu metallerin demir oksi/oksi hidroksit, kalsit, kuvars gibi minerallerin içerisinde kapanım halinde bulunduğu ya da bu minerallerle komşu olduğu tespit edilmiştir. Oda sıcaklığında gerçekleştirilen alkali sülfür ön işlemi sonrası siyanür çözündürme prosesinde metal kazanım verimleri altın için %62 ve gümüş için %7 olarak elde edilmiştir. Direkt siyanürleme işlemi ile kıyaslandığında gümüşün bu işlemden olumsuz etkilendiği görülmektedir. Bununla beraber, sıcak alkali işlem prosesinden sonra, metal ekstraksiyonlarında yaklaşık olarak %25 oranında altında ve %74 oranında gümüşte iyileşme meydana gelmiştir. Bu verim artışı sıcak alkali sülfür liçinin siyanür prosesine etkisi sonucu Ag-Oksihidroksitleri çözünebilir Ag formlarına dönüştürmesi şeklinde değerlendirilebilir. Bu bozundurma prosesinin en önemli avantajı ise daha düşük alkali konsantrasyonunda yüksek metal kazanım verimlerinin ulaşılabilmesidir.

Keywords: Na₂S, NaOH, Altın, Gümüş, Refrakter cevher

1 Introduction

General extraction methods of gold and silver ores are carried out by physical, physicochemical, and chemical methods, depending on the formation and liberation of the ore [1-2]. Among these methods, hydrometallurgical processes, which are among the chemical methods, are carried out by leaching processes in which these metals are dissolved with the addition of appropriate solvents. The reagents used to obtain these precious metals as a result of dissolution processes are cyanide (CN), thiourea [CS(NH₂)₂], thiosulfate [S₂O₃], thiocyanate [SCN], sulfite [Na₂S, (NH₄)₂S, NaHS], ammonia [NH₃], and solvent reagents such as chloride-bromide-iodide [2-4]. Among

these reagents, the place of cyanide reagent is of great importance in obtaining 84% of the gold produced in the world. For this reason, the performance of the cyanide leaching to be applied is considered the main criterion in classifying these ores according to their evaluability [5]. This leaching process must be carried out at pHs higher than 9.4 to prevent excessive cyanide loss due to hydrolysis [2]. In the extraction of Au/Ag from ores, ores are divided into two depending on the mineral structure and the ability of the cyanide reagent to reach the metals. Free gold-silver ores are those that have a size of $d_{80}<75$ μm, and more than 90% Au/Ag can be extracted by dissolution at pH=10-11, with sufficient cyanide leaching concentration for 20-30 hours [6-

7]. On the other hand, the sensitivity of the ore structure and composition to cyanide reagent may be faster than Au/Ag. However, by adding more reagents such as cyanide and oxygen, it is possible to extraction gold and silver metals economically. In addition, all ores where these reagents cannot directly reach the mineral structure of the ore, that is, where the conventional cyanidation process fails, are called refractory ores [8-11]. Although it is claimed that around 30-35% of world gold production today is made from refractory ores, this rate of production from the refractory deposits will continue to increase with the consumption of oxidized gold deposits near the surface. Therefore, economical and environmentally friendly extraction methods with physical, biological, chemical, pressure, and thermal pretreatments need to be evaluated in the extraction of these ores [8, 11].

Antimony and arsenic minerals are dissolved from refractory Au/Ag ores containing minerals such as proustite, pyrargyrite, tennantite, tetrahedrite, stibnite, enargite, and jamesonite by alkaline sulfur leaching carried out with sodium-sulfur and sodium hydroxide. During the dissolution process, elements such as Sb and As are dissolved from the decomposed structure, and after this process, precious metals such as Au-Ag are obtained with cyanide and alternative reagents. In the alkali sulfur leaching process, Na_2S decomposes in the aqueous environment as follows (1) [12-15]. In this leaching process, temperature has an important role in metal extraction [16].

$$Na_2S \rightarrow 2Na^+ + S^{2-} \quad (1)$$
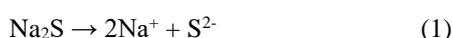
While S^{2-} ion hydrolysis occurs during alkaline sulfur leaching (2), NaOH is added to the medium to prevent the hydrolysis of this ion (3) [12].

$$S_2 + 2H_2O \leftrightarrow 2HS + 2OH \quad (2)$$

$$HS + H_2O \leftrightarrow H_2S + OH \quad (3)$$
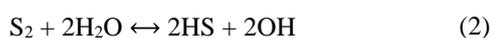
Dissolution of gold in alkaline sulfide systems occurs by sulfides and polysulphides (4) [17, 18]. To extract gold dissolved in alkaline sulfur solutions from the solution, methods such as electrolysis, cementation, precipitation, solvent extraction, and ion exchange are needed [5].

$$2Au + S_2^{2-} + 2S^{2-} \rightarrow 2AuS^- + 2S^{2-} \quad (4)$$

Similarly, silver can dissolve in alkaline sulfur solutions [19]. The reaction is as follows (5):

$$2Ag^+ + S_4O_6^{2-} + H_2O \rightarrow Ag_2S + 2SO_4^{2-} + S^0 + 4H^+ \quad (5)$$

According to the anodic and cathodic half-reactions that took place in alkaline cyanide solutions, the general dissolution reactions of Au are as follows (6) [20-21]:

$$2Au + 4CN^- + O_2 + 2H_2O \rightleftharpoons 2Au(CN)_2^- + H_2O_2 + 2OH^- \quad (6)$$

The dissolution reaction in the degradation of silver-bearing antimony (tetrahedrite) (7-9) [14-15] and arsenic

(enargite) (8) [22] sulphosalts by alkaline leaching is as follows:

$$Cu_{12}Sb_4S_{13(s)} + 2Na_2S_{(aq)} \rightarrow 5Cu_2S_{(s)} + 2CuS_{(s)} + 4NaSbS_{2(aq)} \quad (7)$$

$$NaSbS_{2(aq)} + Na_2S_{(aq)} \rightarrow Na_3SbS_{3(aq)} \quad (8)$$

$$2Cu_3AsS_{4(s)} + 3Na_2S_{(aq)} \rightarrow 3Cu_2S_{(s)} + 2Na_3AsS_{4(aq)} \quad (9)$$
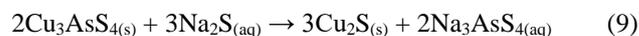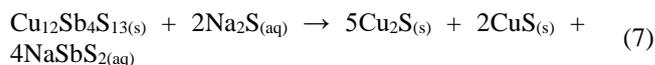
Jarosite structures, especially argentojarosite, react with alkaline pretreatment. After pre-treatment of argentojarosite, silver reacts as AgOH. Many studies have been carried out on the degradation of synthetic argentojarosite in the alkaline environment [23-27]. By alkaline decomposition of this mineral, sulfate ions are removed from the crystal lattice structure, leaving behind a solid gel containing Fe-Ag hydroxide (10) [23, 28].

$$AgFe_3(SO_4)_2(OH)_{6\ (s)} + 4OH^- \rightarrow Ag(OH)_{\ (s)} + 3Fe(OH)_{3\ (s)} + 2SO_4^{-2}_{\ (aq)} \quad (10)$$
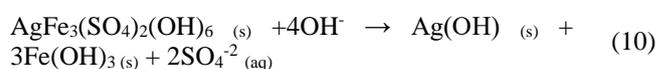
By cyanidation of AgOH, $2Ag(CN)_2$ is obtained. In the alkaline degradation process, the silver in the structure of argento-natrojarosite remains in the solid part [Ag(OH) crystal], and can then be dissolved in cyanide leaching as $[Ag(CN)_2]^-$ complex [24-25, 28-29]. In this process, silver is preserved in its degradation products. During the cyanidation process, silver passes into solution as $[Ag(CN)_2]^-$. The KOH/KCN medium reaction rate is the same as NaOH/NaCN. Sodium sulfate or sodium chloride increases the reaction rate of this reaction [24]. Metallic silver is obtained by partial decomposition of silver ferritin at 100°C (12, 13) [28].

$$Ag(OH)_{\ (s)} + 2CN^- \rightarrow Ag(CN)_2^-_{\ (aq)} + OH_{\ (aq)} \quad (11)$$

$$1/2Ag_2O.Fe_2O_{3(s)} \rightarrow Fe_2O_{3(s)} + 2Ag_{(s)} + ½O_2 \quad (12)$$

$$Ag(OH)_{(s)} + 3Fe(OH)_{3(s)} \rightarrow ½Ag_2O.Fe_2O_{3(s)} + 2H_2O \quad (13)$$
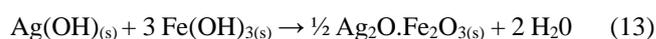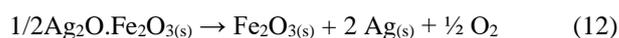
It is an important finding that acanthite is produced as S_2O_3 in the cyanide reaction (14,15) [30].

$$4S^0 + 6NaOH \rightarrow 2Na_2S + Na_2S_2O_3 + 3H_2O \quad (14)$$

$$(X-1)S^0 + Na_2S \rightarrow Na_2S_X \ (X= 2 - 5) \quad (15)$$
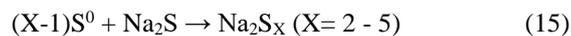
Along with the deterioration of the refractoriness of sulfide antimony minerals from refractory ores in alkaline environments, gold losses of up to 13% have been observed in alkaline sulfide leaching. It has been determined that these losses increase even more at high temperatures and high sulfide concentrations. When alkaline leaching in NaOH medium was developed as an alternative to alkaline sulfite leaching, NaOH leaching made it possible to apply it because it consumes less reagent than alkaline sulfite leaching. It has been concluded that in alkaline sulfide leaching, additional investment costs may arise to extract gold losses, and there

may be environmental limitations since alkaline sulfide leaching carries the potential for H_2S gas formation [31].

Niğde Ulukışla Bolkardağ ore is a refractory gold-silver ore that has undergone oxidation [32]. When the reasons for the refractoriness of this ore are investigated, in addition to the presence of precious metals as electrum, the unique structures of silver and gold attract attention [33-34]. It has been determined in mineralogical investigations that gold and silver are found together with these minerals and/or as inclusions within the minerals in the ore, which generally contain oxide, carbonate, and silicate structures as gangue minerals [35]. Iron oxy/oxyhydroxide-containing minerals in the structure were identified as limonite, goethite, and lepidocrosite minerals [36]. It has been observed that especially silver mineral is found together with jarosite/beudantite minerals in the ore structure [37-38].

In this study, the evaluation of alkaline sulfur leaching together with sodium hydroxide in the structures of refractory gold-silver ores that have undergone oxidation was investigated.

2   Material and methods

In the mineralogical investigations of the Niğde-Ulukışla/Bolkardağ Au/Ag ore, it was determined that it is an oxidized ore containing calcite, barite, quartz, and clay minerals, and mostly limonite, goethite, cerussite, smithsonite, hydrozincite, hemimorphite, and jarosite minerals. In addition, in these investigations, gold was found in the form of native and electrum in small sizes (<24 µm), mostly together with Fe-oxide minerals, while silver was found in acanthite-argentite (Ag_2S) and Ag-containing beudantite ($PbFe_3(AsO_4)(SO_4)(OH)_6$) [39-40]. It was determined that it was found in the form of jarosite minerals.

The following grains were detected in MLA (modal liberation analysis) analysis determined by XBSE (single X-ray per grain identified in the backscattered electron image). As seen in the legends, gold and silver grains, electrum, acanthite, and silver-bearing FePbZn sulfate-arsenate/hydroxide structures are observed. Some of the classified images of the particles in the plus (Fig. 1-b) and minus (Fig. 1-c) 200µm fractions along with the legends (1-a) are given [5].

Gold and silver were detected from the grains within these determined particles. Figure 2 shows the image of acanthite in +200 micron size, adjacent to silicate and as inclusions in iron oxy-hydroxide.

In Figure 3, electrum and Ag-bearing sulfate-arsenate/hydroxide grains were determined within the particle containing pyrite and iron oxy-hydroxide in size -200 microns.
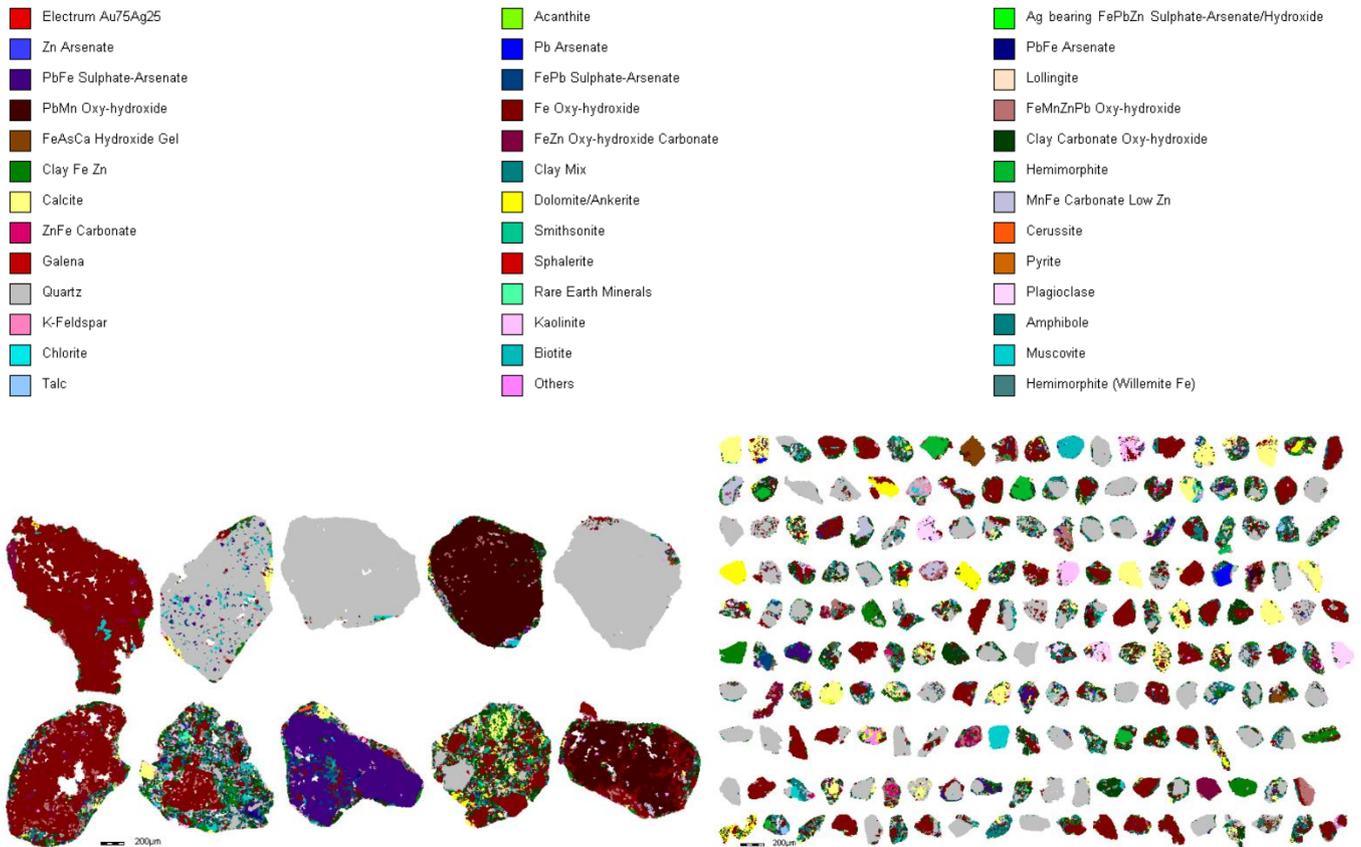
Figure 1. MLA analysis of the ore determined by XBSE (1-a legend (image of above), 1-b individuals classified at +200 µm (image of below-left), 1-c features classified at -200 µm (image of below-right)) [5]
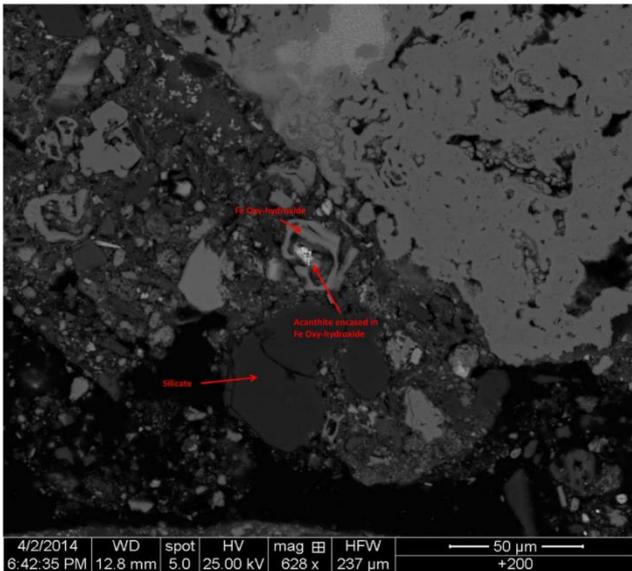
Figure 2. BSE picture displaying acanthite encased in Fe oxy-hydroxide in the +200 micron fraction [5]
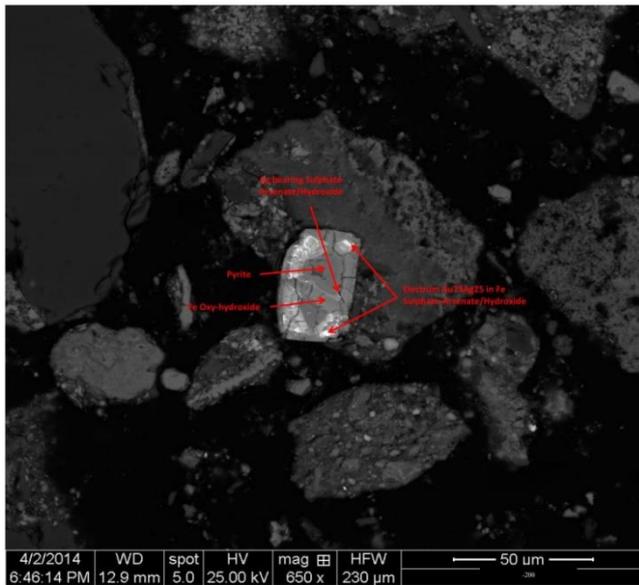
Figure 3. BSE image displaying electrum mixed in Fe oxy-hydroxide around pyrite and Ag-bearing sulfate-arsenate/hydroxide in the vicinity of these two minerals, in the -200 micron fraction [5]

The sieve analysis distribution of the ore obtained after the sample obtained from Niğde Ulukışla Gümüştaş Mining was reduced to -1 mm by drying and crushing (jaw crusher) processes is given in Figure 4.

After 3 minutes of grinding, the raw ore was packaged to be used in experiments. The particle size distribution after grinding is given in Table 1.
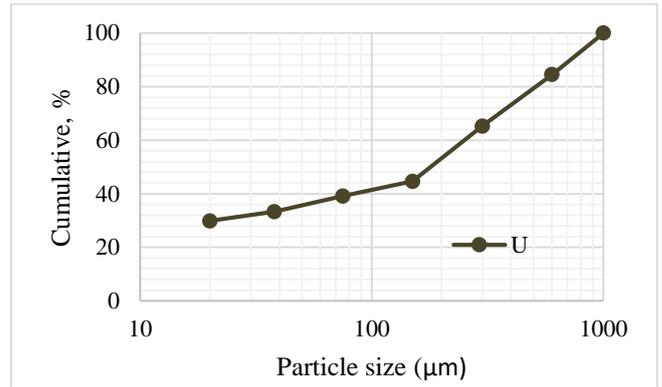
Figure 4. Sieve analysis of the main sample (U: Undersize).

Table 1. 3-minute grinding results

Grinding time	d_{10}	d_{50}	d_{90}
min		μm	
3	1.68	18.77	91.82

Chemical analysis of the ore was obtained by fire assay, ICP-ES (4 acid dissolution), and XRF analyses at the ACME (Canada) Laboratory. In this analysis, it can be seen in Table 2 that there are 2.59% Pb, 4.78% Zn on an element, and 34-35% Fe_2O_3, 22-23% SiO_2, and 5-6% CaO on a compound.

Table 2. Chemical analysis of the test sample

Compound	(%)	Element	(%)	Element	g/T
SiO_2	22.80	Pb	2.59	Au	8.6
Al_2O_3	4.07	Zn	4.78	Ag	176
Fe_2O_3	34.77	As	1.60	Cu	130
CaO	5.79			Sb	500
MnO	1.35			Ba	300
MgO	1.32				
SO_3	0.18				

Alkali experiment conditions (Table 3) were determined as 0.5 M Na_2S and 0.5 M NaOH concentrations (pH=9-10), 8 hours leaching time, 1.15 g/cm³ (w/v) pulp density (PD), 25 and 80°C leaching temperatures. The cyanidation process (pH>10.5) was carried out at 1.5 g/L NaCN concentration, 24 hours, 1.30 g/cm³ (w/v) pulp density, and at room temperature, by controlling cyanide and pH.

Table 3. Experimental conditions

Stage	Agent	Time (h)	PD g/cm³	Temp. (ºC)	Conc.
Alkali	Na_2S	8	1.15	25/80	0.5M
	NaOH				0.5M
Cyanidation	NaCN	24	1.30	25	1.5g/L

Following dissolution and solid-liquid separation by flocculation, cyanide leaching was applied. After the alkali treatment and cyanide treatments, metal extractions were determined in Perkin Elmer-AA 400 AAS (Atomic Adsorption Spectroscopy) for gold and silver solubility, and

the yields were calculated. In Gümüştaş Mining's Tepeköy R&D Plants, in addition to measurements from leaching liquids, fire assay analysis was also performed on the ore/waste sample. According to the fire assay results of the test sample, gold was found as 9.02 ppm and silver was as 169.9 ppm.

3 Results and discussions

In the study where a hot lime and cyanidation process was applied to Bolkardağ ore, grinding efficiency (0-1-3-5-7-10-30 min) was examined. Following the solid-liquid separation in which alkaline treatment was applied at 80℃, 8 hours, 40kg/t, cyanidation was carried out at 25℃ for 24 hours. According to the experimental results, while silver yields remained at lower levels after 5-minute grinding, gold and silver extraction were obtained 88-80% as a result of the experiments [32]. In a different study where grinding was examined (3-10-20-30 min) and there was no pre-treatment application, the cyanidation process was carried out with 1.5g/L NaCN, 24 hours of leaching time, at room temperature, and 25% solids by weight. As a result of the experiments, it was determined that the gold yield was between 77-88% and the silver yield was between 23-43% [36]. Different parameters were studied in the study where the (simultaneous) effect of the potassium hydroxide process on cyanidation was investigated. These parameters are KOH concentration (0.32-2-3.68M), time (38-240-442 min), and temperature (20-57.5-95℃), especially with the increase of parameter values, gold (80-96%) and silver (80-99%) yields were also positively affected [33].

Similar to this study, when the effectiveness of lime performance was examined, the temperature was determined to be in the range of 20-95℃ and the duration was 0-16 hours. Especially silver performance started to decrease after 4 hours and 60℃ temperature. While gold was obtained at around 98%, silver could be obtained at around 80% [34]. In the study investigating the cyanidation performance after the lime process, the cyanidation time was determined as 0.13-84 hours and the cyanide concentration was 0.13-4g/L. Alkali pretreatment conditions: 8 hours, 80℃, 10% solids, 40 kg/t CaO; and simultaneous cyanidation was carried out at 60℃ leaching temperature. It was observed that gold reached up to 97% but silver remained at 76% [35]. In another alkali study, NaOH concentration was examined. Sodium hydroxide process concentrations were also examined in the range of 0.38-3.68 M and temperature in the range of 25-95℃. It was determined that silver was at its highest level in direct cyanidation efficiencies around 0.5h. Although simultaneous NaOH + cyanidation leaching yielded 80% silver and 85% gold in alkaline concentration, it reached up to 99% Au and 90% Ag with increasing temperature [37].

In this study, solid-liquid separation was performed after sodium sulfur alkali treatment. After the alkaline leach liquid was separated for analysis, the washed and dried solid was subjected to cyanidation. After the 24-hour leaching process with cyanide and pH control, the liquid solution was read directly in AAS together with the alkali treatment solution. Like the main sample, the waste sample after the cyanide

treatment was analysed for gold and silver after the fire assay. As a result, the pregnant solution after alkaline treatment, the pregnant leach solution after cyanide treatment, and total metal extractions (confirmed with waste) were obtained.

Silver yields obtained from experiments carried out at 25℃ and 80℃ leaching temperatures are given in Figure 5. While the dissolution was 1.58% after alkaline sulfur leaching at room conditions, it only increased to 6.52% after cyanidation. While low dissolution efficiencies (≤2%) were observed at 80℃ leaching temperature, the total efficiency after cyanidation increased up to 80.86%. The direct cyanidation efficiency carried out under the same conditions was obtained as 25-28%. As a result of the cyanidation experiment carried out after the alkaline leaching process under room conditions, silver extraction efficiency was negatively affected.

It was observed that the silver extraction efficiency in 0.0004M $[S_2O_3^{2-}]$ thiosulfate leaching from jarosite type silver compounds in 0.01 M NaOH environment at 30℃ was low, but the yield increased with increasing temperature, sodium hydroxide concentration or $[S_2O_3^{2-}]$ concentration. However, in alkaline cyanide leaching, increasing the temperature or increasing the amount of alkali increased the silver metal extraction performance [41]. This situation was also observed in simultaneous sodium hydroxide leaching with low silver extraction at low alkali concentrations and low temperatures [37]. It has been determined that silver extraction in refractory gold ores containing arsenopyrite remains at low levels at low NaOH values, but improvements occur at high concentrations, low solid rates, and high times [42].
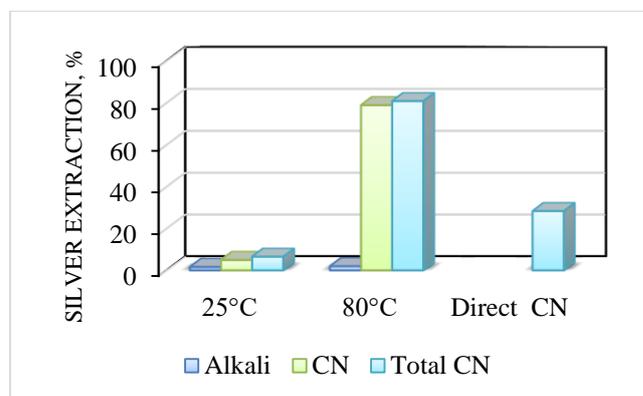
Figure 5. Ag extraction by alkali leaching, cyanide leaching, and total CN leaching (25/80℃)

Gold yields of alkaline sulfur leaching at room conditions and 80℃ are given in Figure 6. While Au solubilities after alkaline leaching were around 5-8% at both temperatures, gold extraction efficiencies from the pregnant solutions after cyanidation reached 56-57% in the 25℃ experiment and 62% in total. Hot alkaline sulfur leaching cyanidation the pregnant solution increased by 78-79% and up to 87% in total. In addition, the gold performance of direct cyanidation tests was found to be 52.41%. As a result, it is seen that alkaline sulfur leaching at room conditions causes around 9-

10% more gold extraction than direct leaching, and hot leaching has a positive effect on gold performance.
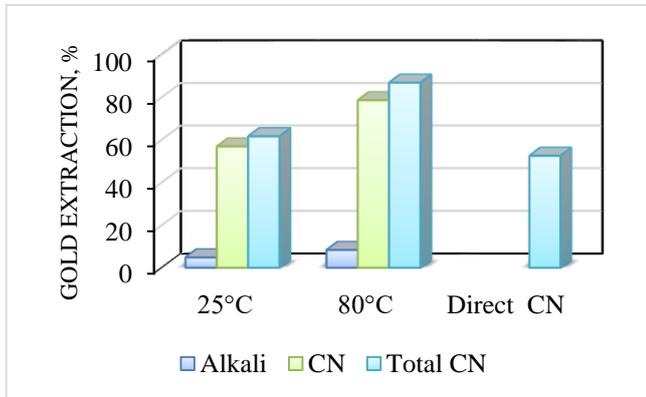
Figure 6. Au extraction by alkaline leaching, cyanide leaching, and total CN leaching (25/80°C)

Sodium hydroxide followed by cyanide and thiosulfate leaching were applied as preliminary treatment of refractory gold ores rich in arsenopyrite. In this sulfur-containing ore, the alkaline environment positively affected the gold extraction [42]. In addition to the different alkaline environments applied to this ore (such as NaOH, KOH, and CaO) being important factors, the effectiveness of hot alkaline pretreatment also had a high impact on gold extraction [33-35, 37].

The experiment performed at 25°C was not effective for either metal. In fact, alkaline sulfur leaching (especially in terms of silver) had a lower efficiency than direct cyanidation. In experiments, it was measured that pH (8.6-9.4) increased with temperature (25-80°C). Sodium sulfur chemical also breaks down into its ions more easily when pH increases. On the contrary, at low temperatures such as room conditions, it is thought that this low-concentration chemical may act as a suppressor in the ore structure at high concentration values such as in flotation [43].

Although the temperature is effective, in a different study, similar values (60-80%) of Au/Ag extractions were obtained from refractory antimony ores where Na_2S and NaOH concentrations were 1-2 M and 3.75 M or 2-4 M and 2.5 M, respectively. Again, in this study, it was observed that temperature increases the extraction efficiency [31].

In the cyanidation process after hot alkaline leaching, the yield increased for both metals. These metal extractions, especially those obtained at higher alkali concentrations in the literature, have also been achieved in alkaline sulfur leaching. This result is also seen as an important advantage in terms of CN leaching. Therefore, the main reason for this change in efficiency can be explained as the further increase in the effectiveness of solvent reagents due to the effect of ambient temperature.

The important reason for the low silver yield can be explained by the fact that the silver mineral content of the ore consists of three different types and the electrum of these minerals is less than the others, but it is more easily liberated (dissolved), and the others are more difficult to liberate (dissolve) [5, 35, 39-40]. Therefore, it is seen that other Ag

minerals are also positively affected by the solubility as the temperature is increased to 80°C.

4 Conclusions

This study was conducted to examine the cyanide process efficiency of the alkali sulfur leaching (Na_2S and NaOH) in metal extraction from oxidized refractory Au/Ag ores. In the mineralogical examinations of the ore, it was determined that the gold and silver minerals consisted of electrum, acanthite, and Ag-bearing FePbZn Sulphate-Arsenate/Hydroxide minerals. These minerals are observed in BSE images in which they are located in many minerals such as iron oxy/oxyhydroxide, quartz, and calcite. The ore also consists of oxide/carbonated content of metals such as lead, zinc, and iron.

In the alkaline pretreatment experiments carried out on the ore with Na_2S and NaOH, 8-hour dissolution was carried out at 25°C and 80°C, and cyanidation was carried out after the solid-liquid separation process. Although the dissolution of silver was low at 25°C, it was found that the achievement efficiency at high temperature reached 81 %. While the dissolution of gold at room temperature was higher than direct leaching, it was determined that the efficiency of metal extractions with the hot sulfur leaching process increased further.

As a result, it was determined that lower alkali sulfide concentrations, especially silver, were significantly effective in metal extractions by increasing cyanide performance with high temperatures.

Conflict of interest

The authors declare that there is no conflict of interest.

Similarity rate (iThenticate): 9%

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