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Cyanide leaching of high-grade silver ore and determination of its refractoriness degree: A Preliminary Study

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Yüksek Tenörlü Gümüş Cevherinin Siyanür Liçi ve Refrakterlik Derecesinin Belirlenmesi: Bir Ön Çalışma

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Abstract
This study investigates the beneficiability of high-grade silver ore obtained from Kütahya province by cyanide leaching. First, chemical and mineralogical analyses were carried out to characterise the silver ore. Then, leaching tests are conducted. In leaching experiments, the conditions at Eti Gümüş plant were maintained, and the optimum NaCN amount and time-dependent dissolution behaviour of the silver ore were investigated. As a result of the leaching experiments, it was observed that silver recovery increased with increasing NaCN concentration, with highest recovery of 66% achieved at 2 g/L. It was also found that silver dissolution was largely complete within 5 hours, but continued for 24 hours, albeit at a low rate. According to the study, the experimental conditions were optimized as follows: 2 g/L of NaCN, a solid-to-liquid ratio of 30%, a particle size of -15 µm, a temperature of 24.5 °C, an agitation speed of 600 rpm, and a leaching time of 24 hours. In addition, it was determined that the silver ore studied was moderately refractory, considering that the maximum silver recovery obtained as a result of cyanide tests was 66%.

***Keywords:** Silver; Cyanide Leaching; Refractoriness; Hydrometallurgy.*

Öz
Bu çalışmada, Kütahya ili Gümüşköy bölgesinden temin edilen yüksek tenörlü gümüş cevherinin siyanür liçi ile zenginleştirilebilirliği incelenmiştir. Öncelikle gümüş cevherinin karakterizasyonuna yönelik olarak kimyasal ve minerolojik analizler yapılmıştır. Ardından liç testlerine geçilmiştir. Liç deneylerinde, Eti Gümüş tesisindeki liç koşulları korunmuş olup, optimum NaCN miktarı ve zamana bağlı gümüş cevherinin çözünme davranışı araştırılmıştır. Yapılan liç deneyleri sonucunda, NaCN miktarının artışına bağlı gümüş kazanımının arttığı görülmüş olup, en yüksek gümüş kazanımına %66 ile 2 g/L'lik NaCN konsantrasyonunun kullanıldığı deneyde ulaşılmıştır. Aynı zamanda, gümüş çözünmesinin 5 saatlik bir zaman diliminde büyük ölçüde tamamlandığı ancak çözünmenin az miktarda da olsa 24 saat boyunca devam ettiği ortaya koyulmuştur. Yürütülen çalışmaya göre, 2 g/L NaCN, katı oranı %30, tane boyutu -15 µm, sıcaklık 24.5 °C, karıştırma hızı 600 rpm ve liç süresi 24 saat olarak deney şartları optimize edilmiştir. İlave olarak, siyanür testleri sonucunda, elde edilen maksimum gümüş kazanımın %66 olduğu dikkate alındığında, üzerinde çalışılan gümüş cevherinin orta düzeyde refrakter olduğu tespit edilmiştir.

***Anahtar Kelimeler:** Gümüş; Siyanür Liçi; Refrakterlik; Hidrometalurji.*

1. Introduction

In nature, silver is often found in combination with gold, copper, lead, and zinc. It is well known that silver is more challenging to beneficiate due to its mineralization. This is thought to be due to silver ore usually occurring as more than 200 different minerals or bound to other minerals (Altınkaya, 2016). Silver metal has properties such as high thermal and electrical conductivity, malleability, brittleness, and optical reflectivity. For this reason, it is utilized in various technological fields, ranging from batteries to industrial-scale solar energy systems.

In hydrometallurgy terms, silver ores are classified under two groups, namely free milling ores and refractory ores (Nazari et al. 2017). Generally, ores containing free and coarse silver particles are recovered by direct cyanide leaching as part of hydrometallurgy. However, methods are also used in which pre-concentrates are obtained prior to cyanide leaching with the aid of gravimetric separators such as shaking tables and chutes. Knelson and Falcon concentrators, which utilize centrifugal force in addition to gravity force, are also preferred as pre-beneficiators for the recovery of silver ores (Celep et al. 2006). Cyanide leaching, in particular, is the most common hydrometallurgical method used for the recovery of primary silver ores due to its economic cost. In addition, potential alternatives to cyanide leaching include thiourea, thiosulphate, and halogen leaching (McNulty, 2001).

The cyanide leaching method involves dissolving the precious metal by adjusting the solution pH to 10.5-11 in

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the presence of O_2 using sodium or potassium cyanide (Celep et al. 2015). The solid fraction is then removed using a thickener and filtration, while the silver is recovered from the solution by precipitation with zinc powder or by adsorption with activated carbon. The reaction showing the dissolution of elemental silver in cyanide solution is given in Equation (1) (Habashi, 1997).

$$4Ag + 8NaCN + O_2 + 2H_2O \rightleftharpoons 4Na[Ag(CN)_2] + 4NaOH \quad (1)$$

The dissolution reactions of sulphur-containing silver and cyanide occur according to Equation (2) and Equation (3).

$$2Ag_2S + 8NaCN + 2O_2 + H_2O \rightleftharpoons 4Na[Ag(CN)_2] + Na_2S_2O_3 + 2NaOH \quad (2)$$

$$2Ag_2S + 10NaCN + O_2 + 2H_2O \rightleftharpoons 4Na[Ag(CN)_2] + 2NaSCN + 4NaOH \quad (3)$$

When silver ore is beneficiated by cyanide leaching, the term ‘refractory’ is used for the ore if the metal recovery efficiency is below 80%. The term refractoriness is preferred to indicate that hydrometallurgical recovery of this type of ore is complex. As for the causes of refractoriness, various mineral encapsulations, particularly sulphurous and preg-robbing ores, as well as other reagent-consuming metals, can be mentioned (Ahlatçı et al. 2017). The degree of refractoriness of an ore varies depending on its mineralogy and metal recovery. If gold and silver metals are recovered from an ore at a rate of 90% or more, this ore is not refractory. However, when the recovery of gold and silver metal from an ore is less than 50%, the ore is defined as ‘high refractory’. In addition, if metal recovery is in the range of 50-80%, it is referred to as ‘moderately refractory’, and if metal recovery is in the range of 80-90%, it is referred to as ‘low refractory’ (La brooy et al. 1994).

To increase metal recovery from low- or high-refractory ores, pre-treatments such as flotation, roasting, pressure oxidation, bio oxidation, chlorination, and combinations of these are employed (Ahlatçı et al., 2017; Adams, 2016). Additionally, metal recovery can be enhanced by increasing the metal's contact with the solution through ultra-fine grinding (Celep et al., 2015; Celep and Yazıcı, 2013). Global events are increasing the interest in gold, a popular investment instrument. However, it will not be possible to meet future global demand for gold due to the daily decline in gold reserves and the resulting excessive price. In this respect, silver metal stands out as an alternative investment instrument. From this point of view, silver metal has become critical for the prosperity of the country and the regional economy. On the other hand, considering that new silver deposits are being discovered every day in our country, more research on the beneficiability of silver ores is also needed. The expansion of research and development activities for silver mining will increase the existing silver production capacity in our country, providing relief to the regional and national economies in the coming years.

In this study, the beneficiability of a high-grade refractory silver ore in cyanide solution was investigated. In this context, the silver ore was characterized chemically and mineralogically. The cyanide concentration- and time-dependent dissolution behaviors of silver metal were then investigated. Finally, the refractoriness of the studied silver ore was determined, and some solutions were proposed.

2. Material and Method

2.1 Material

In this study, silver ore obtained from the Gümüşköy region of Kütahya was used. Silver ore was reduced to a particle size of -15 µm as a result of size reduction processes consisting of crushing (below 4 mm in the primary jaw crusher) and grinding (agate mill). At this stage, the samples were reduced by applying coning-quartering and gridding methods. Some of the reduced samples were reserved for use in experimental studies, while the remaining portion was stockpiled. During these operations, representative samples were collected for chemical and mineralogical analyses to characterize the ore. For the cyanide leaching experiments, Merck brand analytical-grade sodium cyanide (NaCN) was used to prepare the leaching solution. In addition, a basic solution was prepared for pH adjustment using Merck-brand sodium hydroxide (NaOH). Chemical and mineralogical analyses were performed on previously collected representative samples to characterize the ore. In this context, chemical analyses of the ore sample were performed by X-ray fluorescence spectrometry (XRF) and Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-OES). XRF results of silver ore are given in Table 1.

Table 1. Chemical analysis results of Gümüşköy ore

Oxide (XRF)	%	Element (ICP)	ppm
SiO_2	28,15	Ag	176,6
CaO	7,56	Cu	153
Al_2O_3	2,68	Pb	5366
MgO	3,5	Zn	28833
Na_2O	0,92	Fe	13000
BaO	27,6	Mn	1841
K_2O	0,69	Sb	1864
		As	8351

The XRF analysis results in Table 1 show that the silver ore contains 28.15% quartz and an abundant amount of barite at 27.6%. At the same time, 7.56% calcium oxide was detected, suggesting that the silver sample may potentially contain minerals such as calcite and dolomite. Finally, the fact that the ore contains 2.68% aluminum oxide suggests that it also includes some clay minerals.

The chemical analyses of the ore were continued by ICP Spectroscopy, and the elements present in the representative ore sample at the ppm level are given in Table 1. As a result of ICP Spectroscopy analysis, it was determined that the feed sample contained a high level of silver at 176.6 ppm. It is also understood from these results that the ore is rich in iron, zinc, lead and arsenic.

Mineralogical analyses of the silver ore were carried out using X-Ray Diffraction (XRD) spectroscopy equipment available at the mineralogy and petrographic research laboratory of the General Directorate of Mineral Research and Exploration (MTA). XRD patterns of the minerals detected in the representative silver ore are given in Figure 1.

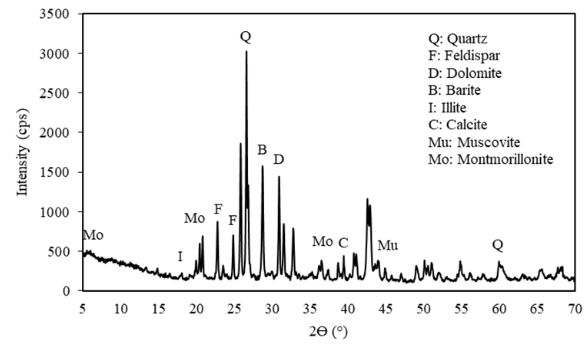

Figure 1. XRD analysis result of silver ore.

According to the XRD results given in Figure 1, the main minerals found in the sample are quartz, dolomite, and barite. XRD analysis also reveals the presence of clay minerals, including illite, muscovite, and montmorillonite, alongside these minerals.

2.2. Method

Cyanide leaching experiments were conducted on silver ore in a tank under atmospheric conditions using the agitation method. In leaching experiments, pulp samples were collected at different time intervals and filtered. Following the separation of the solid and liquid phases after the leaching process, the resulting solid waste was analyzed for Ag by atomic absorption spectrometry (AAS). Thus, leaching efficiencies were calculated using the silver content of the solid residual product and the feed sample. Figure 2 shows the experimental setup used in cyanide leaching experiments. As shown, the experimental setup consists of a three-necked dissolution vessel equipped with an anti-evaporation spiral, a magnetic stirrer, a pH meter, and an air pump that supplies oxygen to the system.

Figure 2. Experimental setup for cyanide leaching experiments

On the other hand, in the leaching experiments, the solid ratio was maintained at 30%, the particle size was set at minus 15 micrometers, the temperature was 24.5 °C, the mixing speed was 600 rpm, the air volume was 0.5 bar, and the pH was greater than 10.5. In these experiments, the effect of cyanide concentration on silver recovery efficiency was systematically investigated as a function of time. During all dissolution tests, pH was measured with a Hanna HI-9125 pH meter.

3. Results and Discussion

The effect of cyanide content on silver recovery was investigated in experiments using solutions with three different cyanide concentrations. In the experiments in question, the solid ratio was fixed at 30%, the particle size was at -15 micrometers, the temperature was 24.5°C, the mixing speed was 600 rpm, the air pressure was 0.5 bar, and the pH was greater than 10.5. During the dissolution tests applied to silver samples, samples were taken from the pulp at different time points, and the tests were completed after 24 hours. The results of the periodically taken samples and the calculated Ag recoveries are shown in Figure 3.

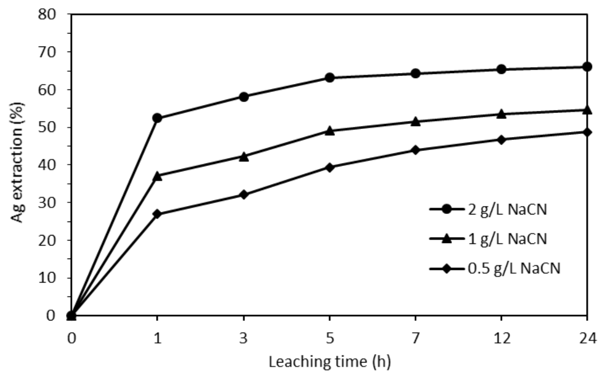

Figure 3. Cyanide concentration and time-dependent silver recoveries

From Figure 3, it is clear that silver recovery efficiency increases with increasing cyanide concentration. Comparing the cyanide concentrations, 66% of silver was recovered from refractory silver ore as a result of the dissolution test using 2 g/L NaCN at the end of 24 hours. When lower cyanide concentrations of 1 and 0.5 g/L and NaCN chemicals were used, silver recoveries were 54.69% and 48.75%, respectively. When the silver recoveries realized in the dissolution tests performed with all three cyanide concentrations were interpreted as a function of time, it was found that the dissolution was largely completed at the end of 5 hours in a similar manner for all cyanide concentrations. After this point, silver metal continued to dissolve, albeit in small amounts, at all cyanide concentrations. Based on all results, it can be concluded that a NaCN concentration of 2 g/L and 24 hours of leaching time are optimal for this refractory ore, recovering the highest silver recovery rate of 66%. These results are confirmed by previous studies conducted by Celep et al. (2011) and Celep et al. (2015) on selective silver recovery from refractory ore using cyanide leaching.

On the other hand, it is also important whether this ore is refractory, and, accordingly, the degree of refractoriness is also important for this ore, for which dissolution tests were carried out in cyanide solution. Whether an ore is refractory or not is decided according to the results of leaching tests on that metal. As a result of these leaching tests to be carried out under optimum conditions, if the recovery efficiency of the precious metal remains below 90%, there is hydrometallurgical complexity with that ore, i.e., refractoriness. After the refractoriness of the ore is finalized according to the leaching results, the degrees of refractoriness of the ore in question are discussed. The refractoriness of ores containing precious metals such as gold and silver can be determined using the classification of La Brooy et al. (1994). The degree of refractoriness of an ore can be determined using the information in Table 2.

Table 2. Classification of degrees of refractoriness of ores (La Brooy et al. 1994).

Metal recovery (%)	Refractoriness degree
<50	Highly refractory
50-80	Moderately refractory
80-90	Low refractory
90-100	Not refractory (Free milling)

As mentioned above, 66% silver metal was recovered directly from the silver ore used in the study via cyanide leaching under optimum conditions. Silver recovery from this ore ranged from 50 to 80, according to the refractoriness classification of La Brooy et al. (1994). In this case, it can be stated that the ore used in the study has moderate refractoriness. After all results are evaluated, the studied ore is considered less complex than other ores and achieves a relatively satisfactory silver recovery. It is possible to achieve much higher values in leaching efficiencies by means of an ultra-fine grinding process for the ore in question. At the same time, this ore is suitable for blending with more complex ore types that exhibit low recovery efficiency encountered in different parts of the quarry. For this reason, it is recommended to use the ore by blending it with other complex ores during processing, as this results in a high-grade and relatively less complex product.

4. Conclusions

Since precious metals, such as gold and silver, are reliable investment instruments, interest in them is increasing daily. In particular, due to the adverse developments on a global scale, the price of gold has been breaking records day after day. Like gold, silver—another precious metal—has also attracted significant interest and demand due to its role as an investment asset. So much so that, in just the past three years, the price of silver per ounce has nearly tripled. In this respect, silver ores have gained in importance in our country's mining. In this regard, additional research and development are needed to advance mining technology for silver ores in Türkiye.

In this study, the chemical beneficiability in cyanide solution of high-grade refractory silver ore obtained from the Gümüşköy region of Kütahya province was investigated. Dissolution tests were carried out under the same conditions as at the Eti Gümüş A.Ş. plant. In these tests, certain parameters, such as solid ratio, temperature, mixing speed, and particle size, were kept constant. For cyanide concentration, an important parameter, the amounts of 0.5, 1, and 2 g/L NaCN were tested separately. From these tests, it was determined that the optimal amount of cyanide was 2 g/L of NaCN, with a silver recovery efficiency of 66%. On the other hand, as a result of the dissolution tests, it was observed that most of the silver in the ore was dissolved by the end of 5 hours, but dissolution continued for 24 hours, albeit at a low rate. Moreover, because the maximum silver recovery from this ore was 66%, it was classified as moderately refractory according to the refractoriness classification of La Brooy et al. (1994).

According to mineralogical and chemical analyses, it can be stated that silver metal is found in or with oxide structures, such as quartz, barite, and other carbonated minerals, including dolomite and calcite, rather than in sulphide structures. Considering this situation, it seems possible to achieve high leaching efficiencies by ultra-fine grinding at finer sizes. In this respect, diagnostic leaching is recommended to obtain a more comprehensive mineralogical characterization of the ore. Thus, it will be clarified which mineral plays which role in the moderate dissolution of silver metal and the intricate and complicate structures in the mineralisation process. Once the main minerals that prevent the dissolution of silver metal have been identified, it is possible to get rid of the minerals that prevent dissolution with the help of pre-chemical leaching processes (acid and base leaching). In this way, much higher silver recoveries can be obtained from silver ores with a similar structure by cyanide leaching.

Declaration of Ethical Standards
The authors declare that they comply with all ethical standards.

Credit Authorship Contribution Statement Author
Author 1: Research, Methodology, Experiment, Writing
Author 2: Research, Methodology, Writing

Declaration of Competing Interest
The authors have no conflict of interest to declare regarding the content of this article.

Data Availability
All data generated or analyzed during this study are included in this published article.

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