

Evaluation of Refractory Behaviour of Kaletaş (Turkey) Gold Ore

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ABSTRACT: In this study, the refractory behavior of Kaletaş ore was investigated within a diagnostic approach. Pretreatment of the ore via roasting and ultrafine grinding prior to cyanide leaching was also examined to enhance the gold recoveries. No significant improvement in the extraction of gold by roasting of the ore at 450-600 °C was observed despite the decrease in the cyanide consumption. Two-stage cyanidation of ultrafinely ground ore was found to improve the extraction of gold to 93% with the implication of presence of readily recoverable gold and slowly dissolving gold in the ore. Diagnostic leaching provided an insight into the refractoriness of the ore, which could be attributed to the very fine gold particles locked up largely within the carbonates, oxides and sulfides and, to a small extent, within silicates present in the ore matrix.

1 INTRODUCTION

Gold ores can be broadly categorized as "free milling" and "refractory" depending on their response to cyanide leaching (La Broy, 1994, Gunyanga et al, 1999). A gold recovery of >90% can be readily achieved with a conventional cyanide leaching of free milling ores (Rubisov et al, 1996). However, refractory gold ores are often characterized by the low gold extractions (<50-80%) in cyanide leaching. The refractoriness of gold ores and concentrates stems primarily from the inherent mineralogical features with reference to the mode of presence and association of gold, and carbonaceous matter present. In practice, the refractoriness has been reported to be caused by (Petruk, 1989, Dunn and Chamberlain, 1997, Browner and Lee, 1998)

- Very fine dissemination (<10 µm) or presence of gold particles as solid solution within mostly pyrite and arsenopyrite
- Association and presence of gold with tellurides and base metal sulfides of lead, copper and zinc
- Carbonaceous matter and silicate minerals present
- Ultrafine gold locked up in the gangue matrix (mostly quartz) or complexes with manganese oxides

A suitable pre-treatment process is often required to overcome the refractoriness and render the gold accessible to the oxidative action of cyanide and oxygen (Gunyanga et al, 1999, Ubaldini et al, 1994, Lehman et al, 2000). Ultrafine grinding, modified cyanidation, roasting, pressure oxidation and bacterial oxidation are the pretreatment methods currently practiced for refractory gold ores and concentrates (Smadmovic et al, 1999, Costa, 1997, Iglesias and Carranza, 1994). Roasting, simple stage process using fluidized bed roaster at around 650°C, is one of the most common methods for the treatment of gold bearing pyrite/arsenopyrite and pyrrhotite concentrates to produce porous calcine and hence to increase the amenability to cyanidation (Roshan, 1990, Long, 2000, La Broy, 1994). Pressure oxidation and biooxidation methods have gained importance in recent years mainly due to the environmental problems (e.g. SO₂ emissions) associated with the roasting process (Smadmovic et al, 1999, La Broy, 1994, Long, 2000, Costa, 1997).

The selection of a particular pretreatment method is based primarily on the economics of gold recovery. However, the determination of the mode of occurrence and association of gold is of fundamental importance to identify the nature of refractoriness and then to manipulate and modulate the process variables to maximize gold recovery within a given pretreatment method. Diagnostic

leaching can provide an insight into the deportment of gold within ore (Lorenzen and TumiSty, 1992; Henley et al., 2000). It is essentially an analytical tool based on the selective removal of the phases at stages with each stage followed by cyanide leaching step to extract the gold exposed (Lorenzen, 1995; Lorenzen and Van Devender, 1993).

Kaletaş gold ore offers an estimated resource of 362 000 tonnes ore with an average grade of 6.8 g/t Au (Tüysüz et al., 1994). The Kaletaş disseminated gold occurrence, hosted by thin-bedded, silty to sandy limestones, consists of siliceous lenses developed along permeable zones such as fault, fracture and bedding planes. Gold is enriched in silicified limestones, especially along zones of extensive carbonate removal. The gangue minerals are composed of calcedonic quartz, calcite, dolomite, illite and halloysite (Tüysüz et al., 1994, Çubukçu & Tüysüz, 2000).

In previous studies (Alp et al., 2003; Gönen, 1999), in cyanide leaching tests, the gold recovery from Kaletaş ore has been reported to be limited to 50-67%. Roasting (at 550°C) as a pretreatment step before cyanidation was shown to improve the recovery of gold only to 80% (Oktay, 2001).

In this study, the application of roasting, ultrafine grinding and flotation to increase gold recovery from the ore was investigated. Diagnostic leaching procedure was also employed to identify the reason for the low gold recoveries and hence refractory behavior of the ore.

2 MATERIAL AND METHOD

2.1 Material

A total amount of 150 kg ore sample was obtained from Kaletaş (Gümüşhane) gold deposit. A number of hand-picked pieces of the ore were separated for the mineralogical analysis. The polished sections prepared from the hand-picked pieces for mineralogical examination were examined under an ore microscope (Leitz Wetzlar).

The remaining bulk of the samples was reduced to -4 trim, using jaw and roll crushers, and riffled to obtain 2 kg representative sub-samples. These were then ground in a laboratory rod mill for experiments at different grinding time (Table 1).

Table 1. Changing of particle size of ore versus grinding time.

Grind Time, min	10	20	30	40	50,60,70,80
Passing 38/tm,%	25	40	60	98	100

The chemical composition of the ore sample is presented in Table 2. The ore sample was determined to contain 6.8 g/t Au and 1.2 g/t Ag.

Table 2. Chemical composition of the ore sample

Compound	Content (%)	Element	Content (ppm)
SiO ₂	54.89	Au	6.8
Al ₂ O ₃	4.88	Ag	1.2
Fe ₂ O ₃	2.38	Cu	281.0
CaO	17.86	Zn	242.0
As	3.88	Pb	359.0
MgO	0.30	Zn	242.0
Na ₂ CO ₃	0.09	Sr	223.7
K ₂ O	0.14	Sb	101.9
TiO ₂	0.09	Ni	46.0
P ₂ O ₅	0.52	V	42.0
MnO	0.03	Co	16.1
Cr ₂ O ₃	0.02	Zr	17.9
LOI	16.30	U	6.6
Tot. C	3.75	Cd	2.1
Org. C	0.18	Ga	3.3
		Mo	7.0

The chemical and mineralogical analysis of the samples used in this study indicated that the ore consisted of predominantly quartz, calcite and, to a less extent, silicates and sulphides of Pb, Fe, Zn, As and Cu. Pyrite, realgar, orpiment, marcasite and native sulphur were identified as sulphide phases (Fig. 1-2). Quartz and calcite were the most abundant non-sulphide phase (Fig. 1-3). Pyrite were commonly present as particles of 3-75 µm in size and as finely disseminated within quartz (Fig. 3). Gold were present as native and electrum form (Fig. 1-2). Organic matter was also identified to be present in the ore.

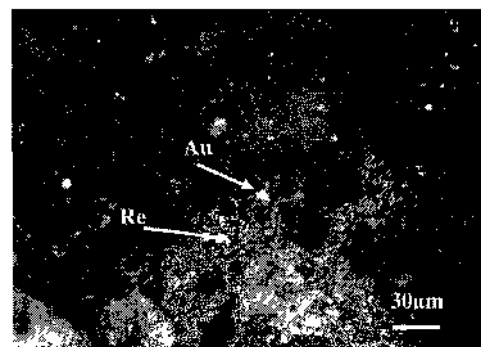


Figure 1. A view of a Au particle (9 µm) as electrum and realgar gram (Re) in quartz matrix.

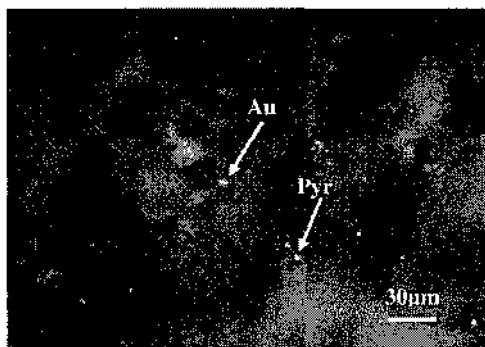


Figure 2. A view of Au (3,5/ım) and pyrite (Pyr) grains in quartz matrix.

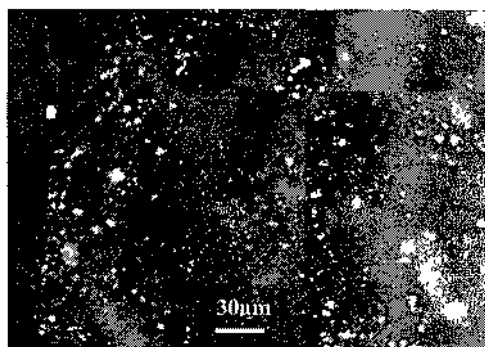


Figure 3. A view of pyrite grains (1-15fıra) disseminated within quartz matrix.

2.2 Method

The cyanide leaching tests (24 h) were performed in a glass reactor equipped with a pitched blade turbine impeller rotating at 1400 rpm (Figure 4). NaCN concentration was maintained at 1.5 g/l over the leaching period and the consumption of cyanide was recorded. Roasting of the ore samples (1 h) in 250 g batches were carried out in a furnace set to the predetermined temperature. The calcines produced at different temperatures (450-600 °C) were subjected to cyanide leaching. The experimental conditions for cyanide leaching and flotation tests are shown in Table 3-4.

Diagnostic leach tests involved a series of acid leaching stages aimed to destroy specific minerals, followed by cyanidation of the residue from each stage. Table 5 shows the sequence and conditions of acid leaching steps in diagnostic leach tests. Conditions for two-stage cyanide leaching of ultrafinely ground ore to determine the gold available to cyanidation are presented in Table 6.

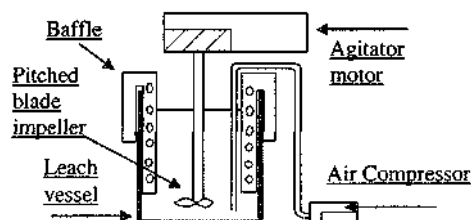


Figure 4. Experimental set-up for cyanide and diagnostic leaching of the ore.

Table 3. Experimental conditions for cyanide leaching of the ore and calcine.

Sample weight, g	250
Water	Tap water
Girind time, min.	10-20-30-40-50-60-70-80
Pulp density, w/w%	25
pH (NaOH)	10-10,5
Agitation, rpm	1450
NaCN, g/l	1,5
Leach time, h	24
Temperature, °C	20±3

Table 4. Experimental conditions for the flotation of the ore

Sample Weight, g	= 1000
Pulp density, w/w %	36
pH	7-7.4
Grind time, min.	10-15-20-30
Aerofloat 208, g/t	100
MIBC, g/t	20
CuSO ₄ , g/t	1000
Na ₂ SiO ₃ , g/t	1000
Agitation, rpm	1250
Flotation time, min.	5

Table 5. Sequence and conditions of diagnostic leaching of the ore sample (80% passing 37 /ım) (Lorenzen and Van Devanter, 1993; Lorenzen, 1994; Henley et al. 2000).

Treatment Stage	Leach Parameter	Minerals likely to be destroyed
NaCN, 1.5 g/l	24 h, pH 10.5	Gold
HCl (12%)	L/S=2:1, 8h 60°C	Calcite, Dolomite, Galena, Pyrrhotite, Hematite
H ₂ SO ₄ (48%)	L/S=2:1, 5h 80°C	Cu-Zn sulphides, Labile pyrite
HNO ₃ (33%)	L/S=2:1, 6h 60°C	Pyrite, Marcasite Arsenopyrite,
HF (20%)	L/S=2:1, 6h	Silicates
Acetonitrile elution (40%)	L/S=2:1, 6h 10 g/l NaCN	Gold adsorbed on carbon

Table 6 Conditions for two-stage cyanidation of ultrafinely ground (6 h) ore

Conditions	Stage	Gold
NaCN (1.5 g/l), 24h, pH 10 >	Stage I	Native, electrum and metastable gold
NaCN (2 g/l), 96 h, pH 12, 1kg/tPb(NO ₃) ₂	Stage II	Gold tellurides
Remaining gold	Invisible gold, Au selenide-metalloid-sulfides	

Analysis of gold in the samples removed at predetermined intervals was carried out using an AAS. Leach residues at the end of each stage were also analyzed for gold to establish mass balance and determine the gold recovery. CN concentration was determined by titration with silver nitrate using rhodamine as the indicator.

3 RESULTS AND DISCUSSIONS

3.1 Cyanide Leaching of the Ore

Figure 5 shows the extraction of gold and cyanide consumption at different grinding times. Decreasing the particle size of the ore appeared not to produce the desired effect. This could indicate the refractory nature of the ore in agreement with the findings of mineralogical analysis. These low gold recoveries suggested the presence of encapsulated gold particles which did not come into contact with cyanide solution. The cyanide consumption was observed to increase with decreasing particle size as illustrated in Figure 5.

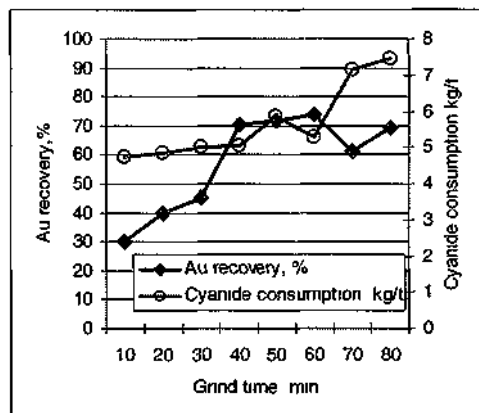


Figure 5 Gold recovery and cyanide consumption versus grind time

3.2 Cyanide Leaching after Roasting of the Ore

In an attempt to decompose the sulfide phases such as pyrite and As-sulfides and hence to expose the locked-up gold in these minerals, roasting of the ore as a pretreatment step prior to cyanidation was carried out at different temperatures. As the temperature increased, only a limited improvement in the extraction of gold was noted to occur as shown in Figure 6. Highest gold recovery was obtained in the cyanide leaching of the calcine produced at 550°C.

Compared with the untreated ore, there was no beneficial effect of roasting (Figure 7) apart from the decrease in the cyanide consumption (Figure 6). It is relevant to note that over an initial leaching period of 1 h, 60% of gold was dissolved, whilst over the following 23 h, the extraction of gold was negligible.

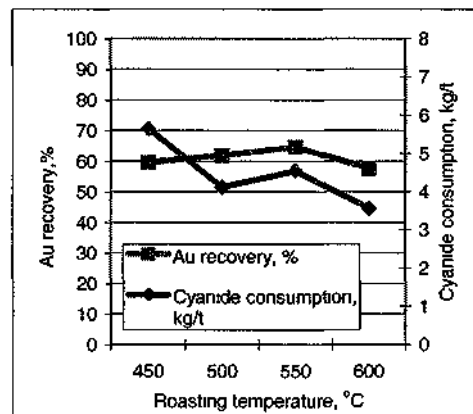


Figure 6 Au recovery and cyanide consumption versus roasting temperature

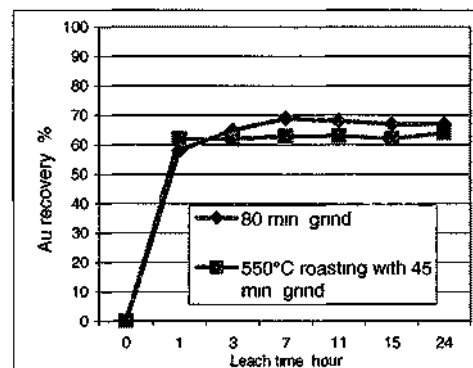


Figure 7 Au recovery leach kinetic with/without roasting

3.3 Flotation Tests

Preliminary flotation tests were undertaken to evaluate the recovery of free and sulfide-associated gold (Figure 8). Apart from zinc, consistently unsatisfactory recoveries for gold, copper and iron was obtained despite the production of concentrates with a gold content of 20-37 g/ton. Further detailed tests appeared to be required to improve the recovery of gold.

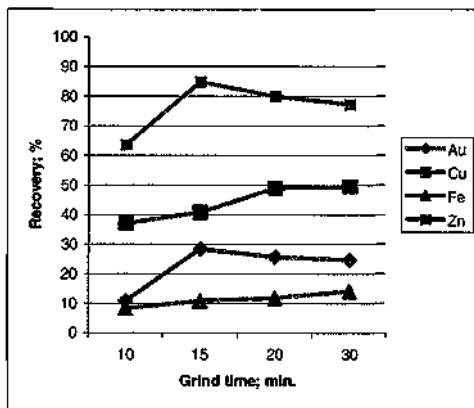


Figure 8. Gold and other metal constituents recovery versus grind time by flotation

3.4 Two-Stage Cyanide Leaching of Ultrafine Ground Ore

Figure 9 shows the results of two-stage cyanidation after ultrafine milling of the ore. These tests were designed to clarify whether the low gold extraction was due to the incomplete "liberation" of gold particles for cyanidation. The findings indicated that 74% of gold could be recovered in Stage I using standard cyanidation procedure adopted in this study. Compared with the earlier findings (Figure 5-6) only a limited enhancement with ultrafine milling was apparent.

In Stage II, the residues from Stage I was subjected to extended cyanide leaching using stronger cyanide solution over 96 h. A cumulative extraction of 93% Au was obtained in two-stage process. The gold extraction in Stage I could indicate the readily cyanidable gold content of the ore while the result of Stage II showed the presence of gold with slow dissolution characteristics.

The findings also revealed that 7% of the gold present in the ore was inaccessible gold.

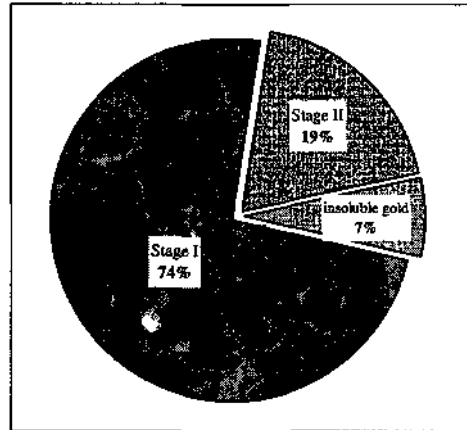


Figure 9. Extraction of gold from the ultrafine milled (6 h) ore in two-stage cyanidation.

3.5 Diagnostic Leaching Tests

Figure 10 illustrates the results of the diagnostic leaching of the ore which involves stepwise acid treatment followed by standard cyanide leaching after each stage. Initial cyanide leaching of untreated ore resulted in a gold extraction of 72%, this indicating the cyanide recoverable gold consistent with the findings reported in the previous sections. The remaining portion of the gold appeared to be refractory in character presumably due to the inaccessibility of gold particles to the action of cyanide. A sequence of acid treatment (HCl, H₂SO₄, HNO₃, HF) was shown to improve the extraction of gold to 100% (Figure 10).

It has been reported that sequential acid leaching using HCl, H₂SO₄ and HNO₃ would destroy a variety of mineral phases (Lorenzen and Van Devanter, 1993). It is extremely difficult to quantify the decomposed phases and hence draw firm conclusion from the data due to the non-selective nature of acid treatment expected i.e. partial removal of sulfides by HCl and H₂SO₄ prior to HNO₃ treatment. Notwithstanding this, in the first stage of acid leaching by HCl, the oxides and carbonates would be completely destroyed to expose the gold associated. Further acid treatment with H₂SO₄ and HNO₃ would be expected to remove the sulfides present. It could be estimated from the diagnostic leaching data that the refractoriness of the ore is caused by fine dissemination and association of gold particle within carbonates and oxides (-13%), sulfides (-13%) and silicates (-2%). The results of

acetonitrile elution stage suggests that no pre-grinding activity of carbonaceous material present

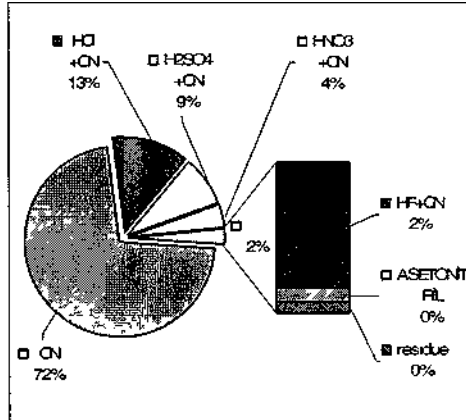


Figure 10 Extraction of gold following each stage of acid treatments with in diagnostic leaching of the ore

4 CONCLUSION

In this study, the reasons for the low gold recoveries from the refractoriness of Kaletaş ore were investigated by adopting a diagnostic procedure. Pretreatment of the ore by roasting and ultrafine grinding was also examined to overcome the refractoriness and to improve the recovery of gold. Roasting was shown to produce no positive effect on the extraction of gold. Preliminary flotation tests failed to produce a gold/sulfide concentrate at acceptable recoveries.

The findings of the diagnostic leaching tests and two-stage cyanide leaching of ultrafine milled ore provided invaluable information on the refractory behavior of the ore. It was deduced that the occlusion of very fine gold particles within mineral phases such as carbonates, oxide and sulfides appeared to be the main reason for the refractoriness. Furthermore, the presence of alloyed gold with slow dissolution character could also contribute to the refractory behavior of the ore.

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