CYANIDE LEACHING BEHAVIOR OF AN ARSENIC RICH REFRACTORY GOLD ORE

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ABSTRACT

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In this work, the possibility of gold extraction using cyanide leaching from a refractory type gold ore, particularly rich in arsenic minerals of realgar and orpiment, was assessed. In this regard, the effectiveness of the cyanide leaching process on the ore was determined using bottle-roll leaching tests with respect to cyanide amount, cyanidation period, and particle size.

In the study, Bottle Roll Tests were carried out at first in order to determine cyanide consumption tendency of the ore, with an ore with the particle size of 150 μm (P_{100}), by using solutions containing NaCN in the amounts of 500, 1000, 1500 and 2000 ppm. As a result of the tests, it was decided to proceed to the 2nd stage, at which amenability of the ore to cyanide leaching was determined with solutions containing 1500 ppm of NaCN.

At the 2nd stage, Bottle Roll Tests were conducted on samples with the particle size of 150, 106, 73, 57, 37 μm (P_{100}), and the leaching behavior of the gold ore and the total amount of NaCN that it consumed during leaching were determined. In the tests performed, it was determined that the ore has a highly refractory structure containing high amounts of cyanicide. It was determined that Au extraction yield increases as the particle size decreases; however, consumption of NaCN increases with the increasing yield efficiency. When the particle size was decreased to 37 μm from 150 μm, Au extraction yield increased to 71.1% from 37.5%, and the total consumption of NaCN per ton of ore increased to 8.8 kg from 5.6 kg.

A higher consumption of NaCN with the P_{100} of 37 µm and an Au extraction yield around 70% revealed the fact that the ore should be pre-treated before cyanide leaching. At the 3rd stage of the study, the leaching behavior of the samples which were pre-treated with roasting was examined. Through roasting, it was intended to rehabilitate refractory characteristic of the ore, increase its amenability to cyanide leaching, and decrease activity of cyanicides which lead to a higher consumption of cyanide. Within this scope, the behavior of gold ore in oxidizing thermal environment (0-1000 ºC) was determined at first through thermogravimetric analyses (TGA); and having regard to the obtained TGA profiles, it was decided to perform the ore roasting process at 400 ºC and higher temperatures. Bottle roll tests were carried out on the ore samples with a particle size of 150 µm (P_{100}) and roasted at different temperatures (550, 650 and 750 ºC) for different periods (30, 60 and 90 minutes); and after such pre-treatment, Au extraction from the ore and NaCN consumption were determined.

After the roasting process performed at 750 ºC for a period of 90 minutes, an Au extraction yield around 70% was obtained with a NaCN consumption of 3.7 kg per ton of ore. It revealed that Au extraction yield, which can be obtained only with a fine particle size of 37 µm without the roasting treatment, can be obtained with a much larger size (150 µm) and a much lower consumption of NaCN after the pre-treatment. As a result of the tests, the optimum roasting conditions for gold ore were determined as 650 ºC temperature and a roasting period of 60 minutes.

Keywords: Gold, Cyanide Leaching, Bottle Roll Test, Roasting, Sodium Cyanide (NaCN)

ÖZ

ARSENİKÇE ZENGİN REFRAKTER TİPİ ALTIN CEVHERİNİN SİYANÜR LİÇİ DAVRANIMININ BELİRLENMESİ

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Bu çalışmada arsenikli mineraller olan realgar ve orpiment bakımından zengin refrakter tipi bir altın cevherinden siyanür liçi işlemi ile altın kazanım olanakları incelenmiştir. Bu doğrultuda cevher üzerinde siyanür liçi işleminin etkinliği şişe çevirme testleri kullanılarak, siyanür miktarı, siyanürleme süresi, tane boyutu parametreleri dahilinde, altın kazanım verimi ve siyanür tüketimi bakımından değerlendirilmiştir.

İlk olarak cevherin siyanür tüketim eğiliminin belirlenmesi amacıyla tane boyutu 150 µm (P_{100}) olan cevher ile, 500, 1000, 1500 ve 2000 ppm NaCN içeren çözeltiler kullanılarak şişe çevirme testleri yapılmıştır. Testler sonucunda, cevherin siyanür liçine olan yatkınlığının belirlendiği 2. aşamaya 1500 ppm NaCN içeren çözeltilerle devam edilmesine karar verilmiştir.

2. aşamada tane boyutu 150, 106, 73, 57, 37 µm (P_{100}) olan numuneler ile şişe çevirme testleri yapılarak cevherin liç davranımı ile liç süresince tükettiği toplam NaCN miktarı belirlenmiştir. Yapılan testlerde, cevherin yüksek refrakter özellikli ve yüksek miktarda siyanisit içeren bir yapıya sahip olduğu belirlenmiştir. Tane boyu küçüldükçe Au kazanım veriminin arttığı fakat artan verimle birlikte NaCN tüketiminin de

yükseldiği tespit edilmiştir. Tane boyutu boyutu 150 µm'dan 37 µm'a küçültüldüğünde Au kazanım verimi %37.5'den % %71.1'e çıkmış, ton cevher başına toplam NaCN tüketimi ise 5.6 kg'dan 8.8 kg'a yükselmiştir.

P_{100} 37 µm tane boyutunda yüksek NaCN tüketimi ile %70'ler civarındaki Au kazanım verimi, siyanür liçinden önce cevherin bir ön işlem ile muamele edilmesi gerekliliğini ortaya çıkarmıştır. Çalışmanın 3. aşamasında bir ön muamele işlemi olarak ''Kavurma'' uygulanan numunelerin liç davranımı incelenmiştir. Kavurma ile cevherin refrakter özelliğinin rehabilite edilmesi, siyanür liçine yatkınlığının arttırılması ve yüksek siyanür tüketimine yol açan siyanisitlerin etkinliğinin azaltılması amaçlanmıştır. Bu kapsamda ilk olarak termogravimetrik analizler (TGA) ile Kaletaş cevherinin oksitleyici ısısal ortamda (0-1000 ºC) davranımı belirlenmiş, elde edilen TGA profillerinden hareketle cevher için kavurma işleminin 400 ºC ve üzeri sıcaklıklarda yapılmasına karar verilmiştir. Tane boyutu 150 µm (P_{100}) olan ve farklı sıcaklık (550, 650 ve 750 ºC) ve sürelerde (30, 60 ve 90 dk.) kavrulan cevher örnekleri ile şişe çevirme testleri yapılarak ön muamele sonrası cevherden Au kazanımı ve NaCN tüketimi belirlenmiştir.

750 0C'de 90 dk. süresince yapılan kavurma işlemi sonrasında, ton cevher başına 3.7 kg NaCN tüketimi ile %70 civarında Au kazanım verimi elde edilmiştir. Bu durum, kavurma işlemi yapılmaksızın sadece 37 µm gibi ince bir tane boyutunda elde edilebilen Au kazanım veriminin, ön muamele sonrasında çok daha iri boyutta (150 µm) ve çok daha düşük NaCN tüketimi ile elde edilebildiğini göstermiştir. Testler sonucunda 650 ºC sıcaklık ve 60 dk.'lık kavurma süresinin Kaletaş cevheri için optimum kavurma koşulları olduğu belirlenmiştir.

Anahtar Kelimeler: Altın, Siyanür Liçi, Şişe Çevirme Testi (Bottle Roll Test), Kavurma, Sodyum Siyanür (NaCN)

To My Loving Family

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LIST OF SYMBOLS AND ABBREVIATIONS

ABBREVIATIONS	DESCRIPTION
ppm	Parts Per Million
ROM	Run-of-Mine
d_{50}	50 % passing particle size, µm
d_{80}	80 % passing particle size, µm
d_{100}	100 % passing particle size, µm
SEM	Scanning Electron Microscopy
T	Temperature, °C
XRD	X-ray Diffraction
TGA	Thermogravimetric Analysis
XRF	X-ray-Fluorescence
ICP-MS	Inductively Coupled Plasma-Mass Spectroscopy
EDS	Energy-dispersive X-ray Spectroscopy
AAS	Atomic Adsorption Spectroscopy
NaCN	Sodium Cyanide
CN^-	Cyanide Anion
HCN	Hydrogen Cyanide
Au	Gold
S	Sulphur
O_2	Oxygen
AsS	Realgar
As_2S_3	Orpiment
N_2	Nitrogen
CO_2	Carbon Dioxide
SO_2	Sulphur Dioxide
NaOH	Sodium Hydroxide
CaO	Calcium Oxide

UNITS	DESCRIPTION
C	Celcius
cm	centimeter
mm	millimeter
µm	micron meter
g	gram
kg	kilogram
l	Liter
%	Percent
kg/ton	kilogram/ton
g/ton	gram/ton
min	minute
h	hour
rev/min	revolution per minute
l/h	liters/hour

CHAPTER 1

INTRODUCTION

1.1 General Remarks

Gold is important in history because of its unique properties. Such features include - but not limited to- being non-reactive with oxygen and water, being non-oxidizing and easily shapeable and being a good conductor. Gold has been used as jewelry, for decoration and exchange material. Nowadays gold is also being used as a critical material in many industries, like electronics, aerospace, medical, dentistry and engineering. The history of gold is very ancient and goes back to antiquity around 4000 B.C. (Yannopoloulos, 1991). Gold price rapidly increased in 1970s and the production also followed that rise. Between 1980 and 1990 gold production was doubled in almost all regions of the world (Marsden & House, 2006). Gold is a rare material and is getting rapidly depleted. Especially, free milling gold ores with coarser gold particles are becoming rare. Therefore, refractory gold ore reserves have become more important than ever, recently. Cyanide usage in gold mining has started about a hundred years ago and nearly 90% of the gold production depends on cyanidation (Şentürk, 2009).

1.2 Historical Development of Gold Processing

History of gold processing started at 2900 BC. It is the first metal that attracted human attention with its appearance. Even the earliest civilizations assigned high value and prestige on gold (Hammett, 1966).

Marsden & House, (2006) divided the history of gold processing into four eras:

- Pre-cyanidation era (before 1888)
- Cyanidation era (1889-1971)
- Major technological developments era (1972-2000)
- 21st century developments

In pre-cyanidation era, in 3050 B.C. gold was being using as a means of financial transactions in the form of grains and small bars (Marsden & House, 2006). Gold was firstly processed in Egypt in 2900 B.C. (Hammett, 1966). Anatolian civilizations developed advanced processing knowledge for metallic ores and mining practices. It dates back to the Neolithic period. The earliest Anatolian gold monuments were discovered at Alacahöyük, which were made in the early Bronze Age, 3,000 BC. Figure 1.1 shows the first coin produced by Lydians.

Figure 1.1. First gold coin in the world (TGMA, 2014)

Method of mining is called panning or washing for gold and placer type deposits in streams or rivers were being processed. For example, in Turkey fine native gold is often found in quartz veins in hydrothermally altered volcanic rocks near rivers. One such river, rich in alluvial gold, is Gediz and Gediz was a major source of placer deposits and native gold for early civilizations in Anatolia.

The first known gold process is amalgamation of gold with mercury (1000 B.C.). In 750 B.C. use of aqua regia was found.

After recognition of the solubility of gold in cyanide solutions (by Elsner, 1846), a new era has started. Cyanide was started being using as a leaching reagent. Initial recovery methods depended on zinc cementation and electrolytic cells. In early 19th century activated carbon was considered as an alternative to zinc cementation. The first Carbon in Pulp (CIP) plant was opened in 1949 (Marsden & House, 2006). In late 1960’s first heap leaching operation was started in USA for processing low grade ore.

In 1980’s economic climate has changed. The demand for gold dramatically increased. New technologies like carbon regeneration, electro winning cells, autoclave and reactors, roasting were developed. (Marsden & House, 2006). Also, in the 1980’s the interest in Turkish gold deposits has again rose and search for potential gold resources were accelerated (TGMA, 2014).

In 1960’s refractory gold ore deposits started to attract attention, too. Many studies were done on treatment of such deposits. First pre-treatment method was oxidation by roasting. In addition alternative methods like chlorination (1979), pressure oxidation (1985) and biooxidation (1991) were developed (Marsden & House, 2006).

In 21st century, research topics changed to (Marsden & House, 2006):

- Alternative leaching reagents like thiourea
- Combination of biooxidation with heap leaching
- Sulphidic ore treatment
- Resin adsorption systems (RIP, RIL and RIS)
- Recovering gold from secondary resources
- Mineralogical and chemical characterization of gold
- Reagent recovery and recycling
- Improving metal recovery
- Electrowinning from dilute solutions
- Using flotation as a concentration technique
- New gravity concentration techniques, etc.

1.3 Statement of the Problem

Demand for gold and gold prices follow an increasing trend over the last decades. Also, free milling ores are being depleted and processing of low grade ores have become a necessity. Based on this, processing of low-grade ores, with <5 ppm gold, has recently become a common practice (Kongolo & Mwema, 1998). Turkey has high potential of refractory gold ore reserves and processing of such ores are critical for the country.

Changing trends towards refractory gold ores forced mining companies to make processing methods feasible in both economically and environmentally. Because for such ores operating and capital costs are relatively high and treatment is difficult. There are lots of work on processing of refractory gold ores and for increasing recoveries while keeping the processing expenditures at a minimum. The main processing method used in refractory gold ores is cyanide leaching but gold extraction yields are very low in direct cyanidation. Therefore, every gold reserve has unique mineralogy and it is important to examine the mineralogy of the ore before cyanidation. With prevailing efforts several pre-treatment methods and alternative processing methods have been developed in order to overcome refractoriness.

Refractory gold ore samples should be investigated in detail and most suitable method selected to pretreat such ores, and increase the recovery.

1.4 Objective of the Study

Gold extraction potential of a specific type gold ore, -an arsenic rich reserve- by means of cyanide leaching were examined within the scope of this thesis. The response of the studied ore to cyanidation was identified through Bottle Roll Tests. Studied parameters included dosage of Sodium Cyanide (NaCN), cyanidation period and particle size. Effect of cyanidation was expressed in terms of Au extraction and NaCN consumption.

The goals of this study include determination of cyanide consumption trends of the ore, investigation of the effect of particle size on Au extraction and the effect of roasting, as a pre-treatment method on reducing refractoriness of this As-rich gold ore and improving Au extraction.

1.5 Outline of the Thesis

The thesis has five main chapters. The details of these chapters are as follows:

Chapter 1 begins with general remarks and history of gold mining. It also covers statement of the problem and objectives of the study.

Chapter 2 provides brief information on gold reserves, mineralogical properties of gold, and diagnostic leaching. In addition, information on pretreatment methods, cyanidation, challenges and opportunities of refractory gold ore processing are explained. It continues with highlights and remarks from previous studies.

In Chapter 3, information on materials and methods are given. It explains general characteristics of the sample, mineralogical and thermal analyses, bottle roll tests, roasting and chemical analysis. All the experiment steps are explained in details.

Chapter 4 includes the achieved results and discussion of bottle roll tests with and without roasting treatment.

Chapter 5 consists the points concluded from the overall work and recommendations for possible future work.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 Mineralogical Properties of Gold

Gold is known for its bright lustrous yellow color and has been frequently used as a financial asset from past to today. Gold is one of the softest precious metals (Hammett, 1966) which is mostly used in jewelry, in coinage, and for investments. The mineralogical properties of the native gold are listed in Table 2.1 (Rose & Merloc, 2008).

Table 2.1 Mineralogical Properties of Gold (Rose & Merloc, 2008)

Property	**Native Gold**
Au %	>75
Specific Gravity	16-19.3
Hardness	2.5-3
Color	Golden Yellow to brass yellow
Crystal System	Isometric, Cubical
Transparency	Opaque
Cleavage	None
Luster	Metallic

From the information in Table 2.1, native and liberated gold can be separated from the other minerals with gravity separation. Gold can only be soluble in aqua regia (Yannopoloulos, 1991).

Each deposit has a different gold mineralogy, and occurs in a variety of mineralogical forms on the basis of the following (Marsden & House, 2006):

- Occurrence of gold in the ore matrix
- Gold grain size distribution in the ore
- Host and gangue mineral
- Mineral mixtures
- Mineral variances
- Time effect

2.2 Classification of Gold Deposits

2.2.1 Placer Gold Deposits

Placer deposits are formed by weathering, erosion and transport by continuous water flow or wind. These may also be named as ‘‘Alluvial Deposits’’. Such deposits consist of sand, gravel width gold entities (Rose & Merloc, 2008). In the late 1880’s, exploitation of these easy-to-process deposits was common (Arbiter & Han, 1992). Gravity concentration techniques can easily be applied to the placer deposits (Yannopoloulos, 1991). Gold particles are liberated in different sizes (50 to 100 μm) (Arbiter & Han, 1992).

Placer deposits would be classified into two groups: 1. Shallow or recent placers and 2. Deep level or Ancient placers. First group are those which have not been covered by other deposits. Second one are those, buried deeper in rivers (Rose & Merloc, 2008).

2.2.2 Epithermal Gold Deposits

Epithermal deposits are formed at shallow depths and moderate to low temperatures (350-150°C) as veins and disseminations in volcanic sedimentary rocks. (Arbiter &

Han, 1992). This type is generally low grade deposits, yet the reserves are relatively large making their exploitation feasible.

2.3 Gold Reserves

2.3.1 World Gold Reserves

World gold mining has been increasing since late 1960s. With technological developments and increasing prices, exploration for new reserves has been accelerated. Australia holds a large share of the world's gold mine reserves, approximating 9,100 tons. Worldwide, the production of gold reached approximately 3000 tons in 2015 (Figure 2.1).

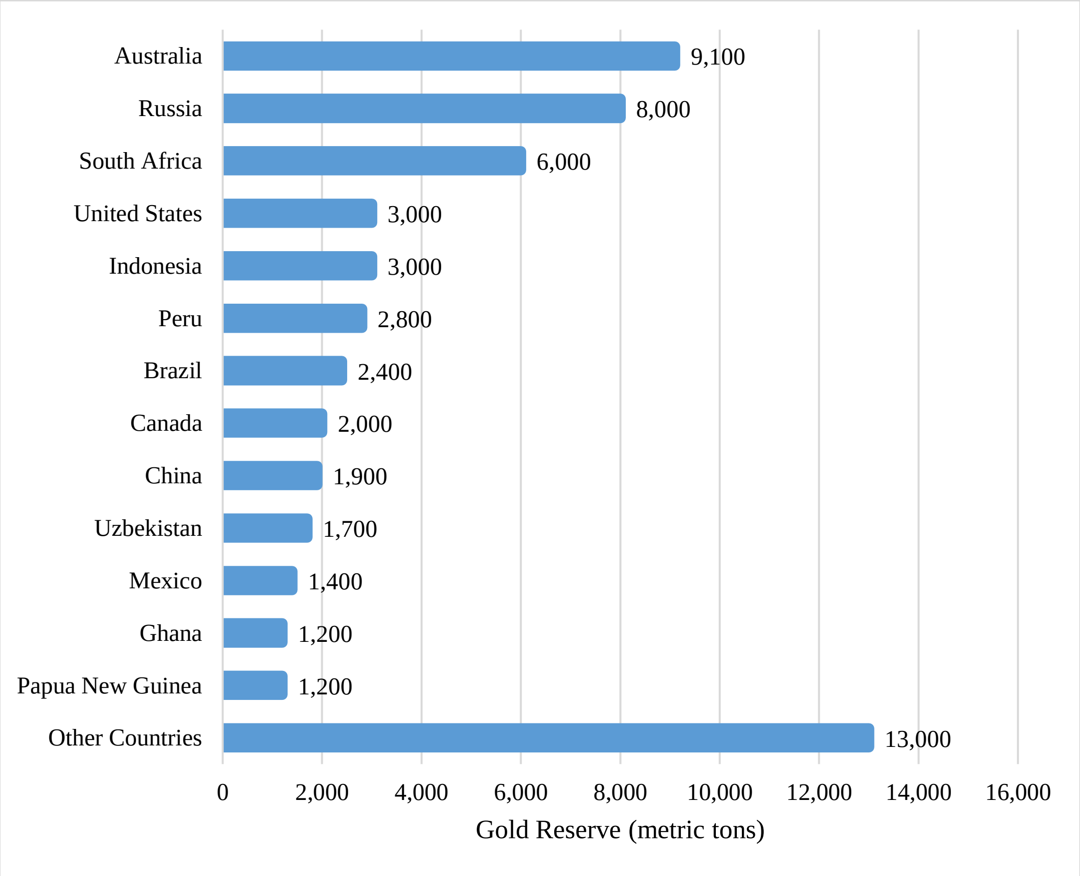

Figure 2.1. World mine reserves of gold of 2015, by country (in metric tons) (Statista, 2015)

When it comes to gold production worldwide, China was the largest producer in 2015 with around 14% of global production. In Figure 2.2, World gold mining map is shown with geographical dispersion of gold supply. Also, Table 2.2 shows gold production with respect to countries.

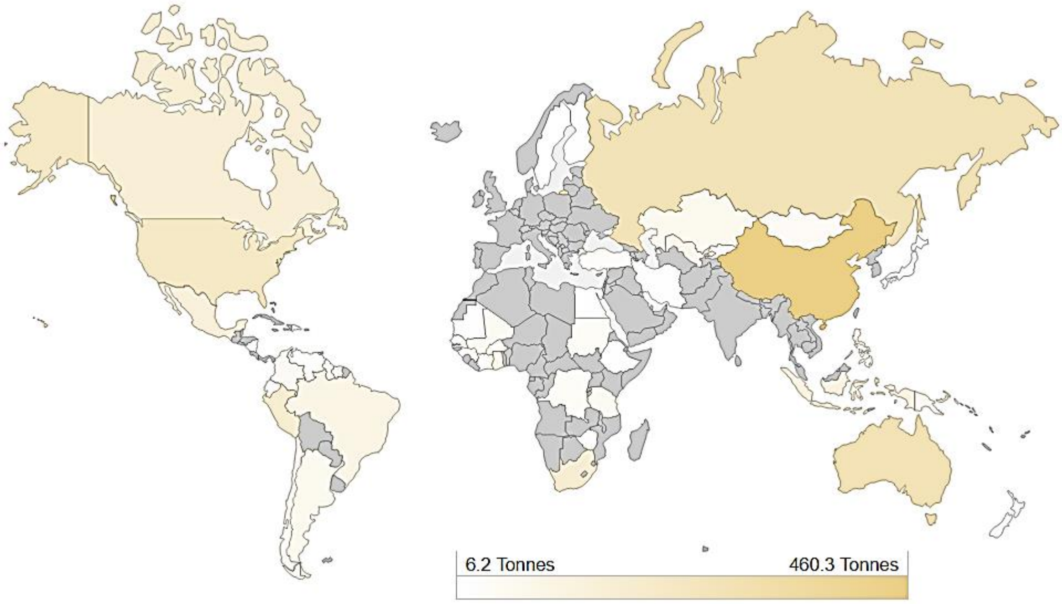

Figure 2.2. World Gold Mining Map for 2015 (World Gold Council, 2015)

Table 2.2. Gold Production for 2015 (World Gold Council, 2015)

Country	Ton	Country	Ton
China	460.3	Mongolia	32.8
Australia	273.8	Turkey	27.4
Russia	268.5	Zimbabwe	24.8
United States	214.0	Guinea	23.5
Peru	170.5	Venezuela	22.2
South Africa	167.5	Côte d'Ivoire	21.6
Canada	157.2	Kyrgyz Republic	18.7
Mexico	133.2	Suriname	17.6
Indonesia	113.0	Guyana	17.6
Brazil	95.0	Ecuador	15.2
Ghana	94.7	Egypt	13.7
Uzbekistan	85.5	Ethiopia	12.6
Kazakhstan	63.7	New Zealand	11.7

Table 2.2. (cont'd) Gold Production for 2015 (World Gold Council, 2015)

Country	Ton	Country	Ton
Argentina	63.5	Iran	11.0
Papua New Guinea	58.4	Mauritania	9.0
Tanzania	51.7	Finland	8.4
Mali	50.1	Nicaragua	8.3
Colombia	44.0	Japan	7.0
Philippines	41.1	Senegal	6.7
Chile	41.1	Sweden	6.2
Dem. Rep. of the Congo	39.7		
Burkina Faso	36.5		
Dominican Republic	33.7		

2.3.2 Turkish Gold Reserves

Türkiye hosts some of the world's largest gold deposits with proven reserves of 700 Ton (MTA, 2013) and predicted resources of 5,700 Ton (TGMA, 2014). Gold mining in the country has grown continuously over the past decade and Türkiye became the leading gold producer in Europe (MTA, 2013). Brief history on Turkish gold mining is presented below:

In 1985, Mining Law 3213 attracted first large-scale foreign investment enabling the discovery of several large gold deposits during the late 1980's and early 1990's. In 1997, Ovacık - Bergama became the first modern gold mine in Turkey, which was discovered in 1989 and started production in 1997 by Normandy Mining Company. 2001 was an active year for exploration and development of gold mines by several international mining companies. In 2002, Turkish government introduced stabilizing and supportive economic policies. In 2004, with changes in the Mining Law (Mining Law 5177) several significant and supportive aspects for the development of gold mining industry were introduced. In 2005, Koza Gold acquired Ovacık Mine from Newmont. In 2006, Kışladağ mine was started as Turkey's first heap-leach operation by Tüprag, as a subsidiary of Eldorado Gold Company (TGMA, 2014). The production increased since that time (Figure 2.3). Gold and Silver Deposits in Turkey are presented in Figure 2.4.

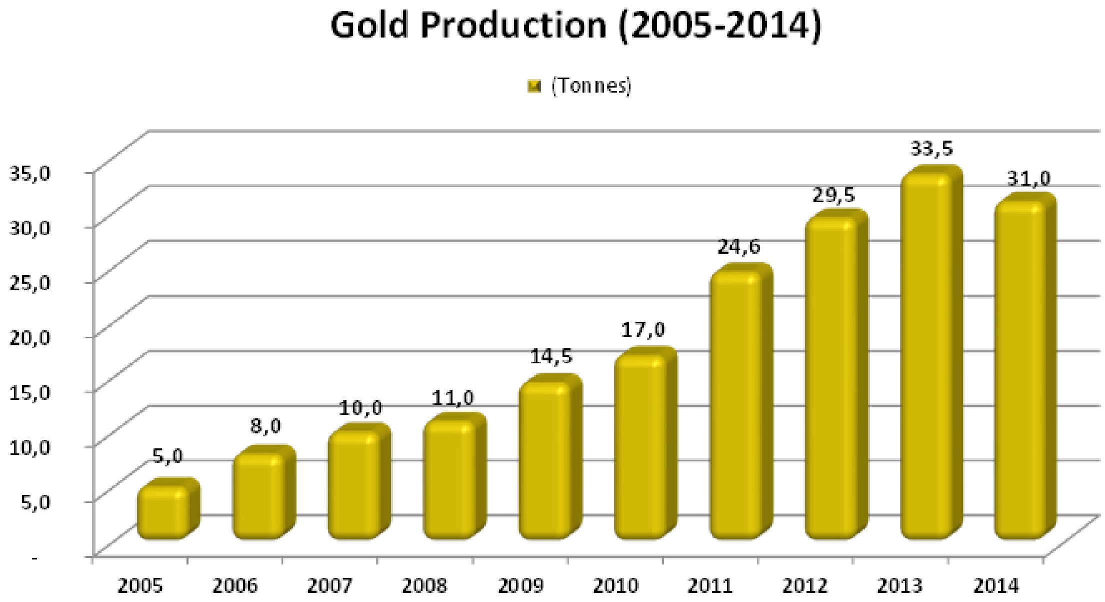

Figure 2.3. Gold Production in Turkey (2005-2014) (TGMA, 2014)

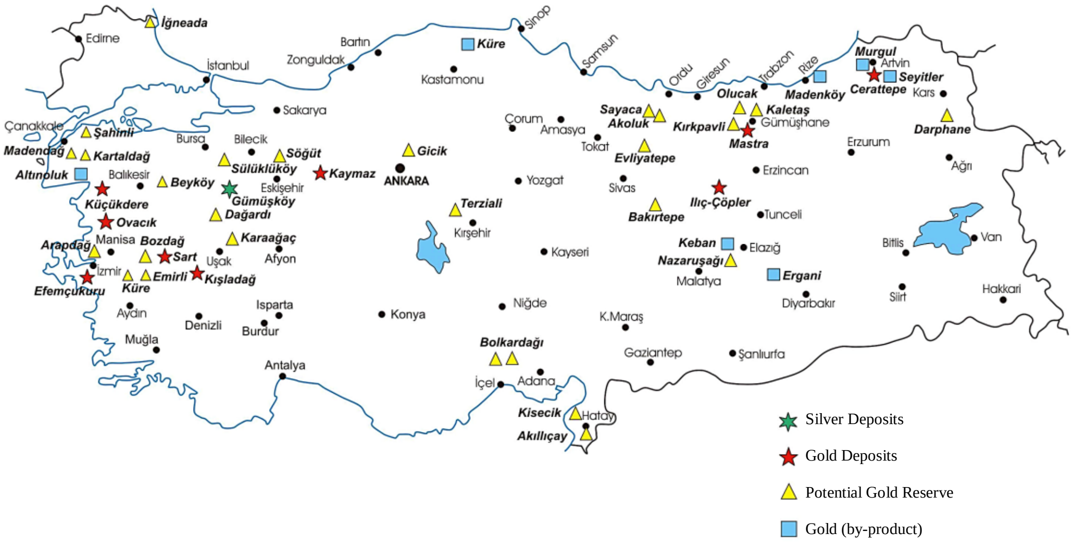

Figure 2.4. Turkish Gold and Silver Deposit Map (MTA)

2.4 Classification of Gold with respect to Cyanidation Behavior

In this respect it is possible to classify gold ores with respect to their response to cyanide leaching. First group is termed as “Free Milling Ores” and these deposits would yield Au recoveries greater than 90% by conventional cyanide leaching (Celep, et al., 2008). In other words, the size of gold particles in the first group are relatively coarse and are leachable by NaCN at all sizes. Free milling gold ores provide the maximum economical value (METS, 2009). Gravitational concentration methods can be applied. (Kongolo & Mwema, 1998).

“Complex Gold Ores” are the second group. Complex gold ores consist of reactive minerals that consume the leach reagents (mostly NaCN) which cause insufficient cyanide or oxygen in the pulp. They can be categorized by high cyanide consumption, high oxygen consumption and preg-robbing behavior (La Brooy et al., 1994). Cyanide consuming complex ores contain gold in electrum which causes increased cyanide consumption and slow leaching rate. Also, they are mostly copper containing sulphide ores and copper minerals consume cyanide. In order to increase the recovery of gold, pretreatment methods should be used before direct cyanidation. Oxygen consuming complex ores consist of reactive sulphides like pyrrhotite that need oxygen in order to oxidize sulphide. Using oxidative pretreatment methods may decrease oxygen consumption and increase the recovery of gold. Preg-robbing ores are mostly carbonates and clays that absorb dissolved gold. In order to treat such ores pretreatment methods (roasting, chlorination) should be used to decrease preg-robbing prior to direct cyanidation.

The third group are termed as ‘‘Refractory Ores’’ and are characterized typically with lower recoveries (50-80%) through conventional cyanide leaching. They consist of disseminated gold which is mostly fine to ultrafine in size and occur in locked/integrated in and/or associated with other minerals/rocks. In other words, refractory gold ores contain locked gold in the host mineral. Processing of these deposits may require complex procedures and higher capital and operating costs and are associated with higher technical risks (METS, 2009).

According to La Brooy (1994), gold can be classified in a more detailed manner as shown in Figure 2.5. Refractoriness may also have varying degrees as presented in Table 2.3. Classification of ore refractoriness (La Brooy et al., 1994).

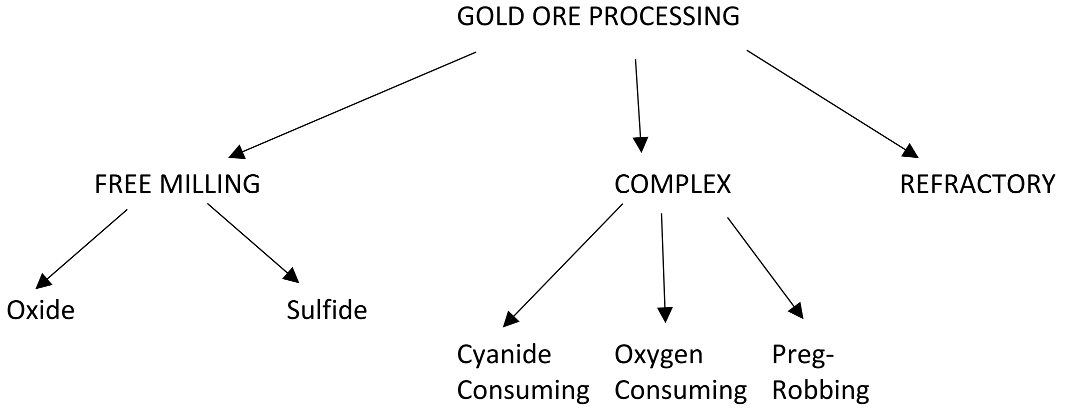

Figure 2.5. Classification of gold ores (La Brooy et al., 1994; Lunt & Weeks, 2016)

Table 2.3. Classification of ore refractoriness (La Brooy et al., 1994)

< 50% recovery	Highly refractory
50-80 % recovery	Moderately refractory
80-90 % recovery	Mildly refractory
90-100 % recovery	Non-refractory (Free Milling)

2.5 The refractoriness of Gold Ores

The refractoriness of gold ores arises due to the mineralogical occurrence of gold. Dissemination in fine sizes, being locked in other mineral matrix and/or presence of carbonaceous minerals are the main causes of refractoriness. Major reasons for refractoriness is as follows (Celep et al., 2008; McDonald, et al., 1990):

- Very fine and/or disseminated mineralization of gold in the form of entities finer than 10 μm,
- Presence of gold particles in pyrite, arsenopyrite matrix or within other arsenic sulfides,
- Presence of gold with tellurides and base metal sulfides of Pb, Cu and Zn,

- Presence of carbonaceous material which causes ‘‘Preg robbing”,
- Locked up ultrafine gold particles in the matrix of gangue minerals (mostly in quartz)
- Presence of gold within other minerals that react with cyanide, i.e. the ‘‘cyanicides’’ (Flatman et al., 2008)

A close link between ore mineralogy and treatment method obviously exists (Bayraktar & Yarar, 1981). Before selecting a suitable process, a detailed process mineralogy study is necessary.

2.6 Diagnostic Leaching

In order to select a suitable process for the gold ores diagnostic leaching can be used as a vital tool for establishing the distribution of gold within different mineral phases (Celep et al., 2009; Almeida, 2001). Diagnostic leaching is used to identify the unique attitude for a given gold ore (Alp et al., 2005; Lorenzen & Tumilty, 1992; Henley & Clarke, 2000). It consists of a series of leaching steps with a variety of chemical for selective destruction of mineral phases and each stage is followed by cyanide leaching to extract the encapsulated gold (Celep et al., 2008; Lorenzen, 1995; Lorenzen & Van Devender, 1993). In other words, it is conducted to determine invisible gold and mineral associations causing this encapsulation, through selective leaching steps.

Lorenzen (1995) indicated a set of guidelines for the design of a diagnostic leaching test and Figure 2.6 shows a typical setup for diagnostic leaching test. Also, diagnostic leaching procedures for destruction of different mineral phases are summarized in Table 2.4.

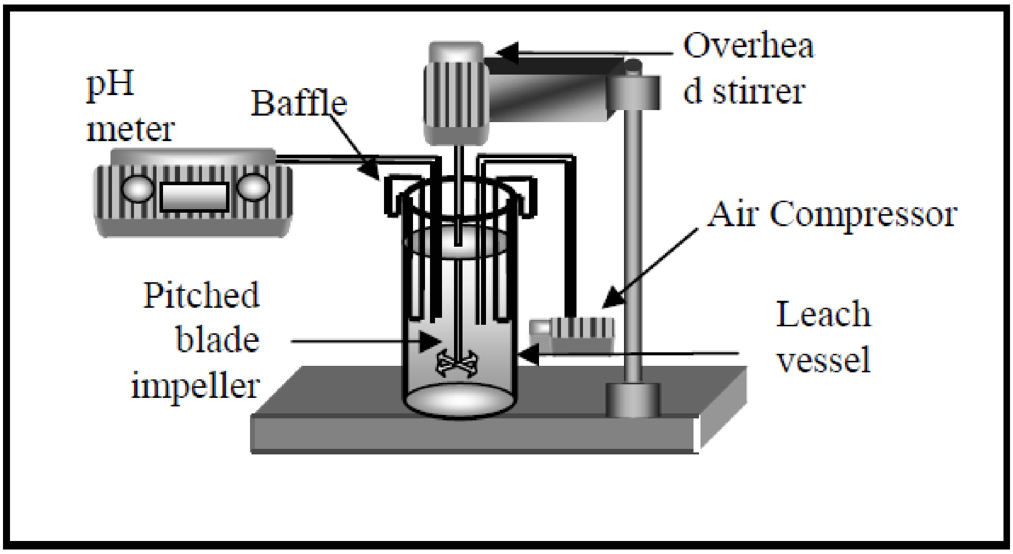

Figure 2.6. Diagnostic Leaching Equipment (Lorenzen & Tumilty, 1992; Celep et al., 2008)

Table 2.4. Diagnostic leaching procedure by selective destruction of gold ores (Lorenzen & Van Devender, 1993)

Treatment	**Minerals likely to be destroyed**
NaCN	Gold
Na_2CO_3	Gypsum and arsenates
HCl	Calcite, dolomite, galena, pyrhotite
HCl/$SnCl_2$	Hematite, calcine, ferrites
H_2SO_4	Cu-Zn sulphides, labile pyrite
$FeCl_3$	Sphalerite, labile sulphides, tetrahedrite
HNO_3	Pyrite, marcasite, arsenopyrite
Oxalic acid washes	Oxide coatings silicates
HF	Silicates
Acetonitrile elution	Gold adsorbed on carbon

2.7 Pretreatment Methods Applied in Gold Processing

To achieve higher recoveries from refractory ores, use of a pretreatment procedure is generally necessary prior to the main leaching phase. All these procedures aim at breaking down the mineral matrix to liberate gold values (Flatman et al., 2008).

Pretreatment methods can be classified as Thermal Methods, Pressure Oxidation, Biological Oxidation, Physical Treatment and Flotation (La Brooy et al., 1994; Çiftçi & Akçıl, 2009; Almeida, 2001). Selection of a proper method that ensures maximum liberation of gold is important as facilitating a pre-treatment procedure corresponds to additional process costs.

2.7.1 Thermal Pre-Treatment Methods

Thermal pre-treatment methods are applied to break down the sulphide matrix in the ore. Commonly three different processes are used, namely; Roasting, Pyrolysis and Microwave heating.

2.7.1.1 Roasting

Heating ores to elevated temperatures in an oxidizing environment is called roasting. Roasting is applied to break down the sulphide matrix in the ore and liberate the locked gold particles (Çiftçi & Akçıl, 2009). The degree of oxidation depends on mineralization of gold and the type of oxidation process used. Roasting is used to oxidize refractory sulfide, arsenical, and carbonaceous gold ores (Hammerschimidt et al., 2016).

According to Çiftçi and Akçıl (2009), main goals of roasting are listed below:

- Destruction of metal- sulphide matrix,
- Extraction of the gold inside the matrix,
- Hematite production with increased surface area,

- Decreasing the undesired cyanide-consumption by components other than gold
- Fixing arsenic and sulphur to an environmentally acceptable form (La Brooy et al., 1994).

Roasting of a sulphide mineral increases its porosity. This allows increased contact between the refractory gold and the leaching solution. Hence, by roasting calcines, refractoriness is reduced chemically and physically (Henly, 1975). Most of the sulfides oxidize quickly when heated to a high temperature. The process works best when the ore is crushed into smaller fragments before roasting. Additives, such as common rock salt, are often added to the mix before heating to enhance the effectiveness of the process. Once the ore is fully roasted, and the sulphur is driven off, the extraction of the gold or silver from the ore by cyanidation becomes relatively easy. Roasting is done generally between 450-820 °C and the effective roasting temperature depends on the mineralogy and complexity of the sulphide matrix (Çiftçi & Akçıl, 2009).

For arsenopyrite ores, a two-stage roasting is a conventional industrial practice. The mechanism of arsenic removal occurs as follows (Swash & Ellis, 1986):

$$4\ FeAsS(s) + 3\ O_2\ (g) \rightarrow 4\ FeS(s) + 2\ As_2O_3(g) \tag{1}$$

Arsenopyrite roasting is rather complicated as compared to other metallic sulphides with gold content, because several different intermediate compounds (e.g., As_2O_3, As_2O_5 and As_2S_3) form during the process. For instance, if the partial pressure of oxygen is too high, arsenic is converted into arsenate and becomes fixed as ferric arsenate in the calcine. In such a case, reagent consumption increases (Dunn & Chamberlain, 1997). For this reason, careful planning should be done before roasting the arsenopyrite ores and roasting should be closely controlled.

2.7.1.2 Pyrolysis

An alternative option to roasting is the pyrolysis of the refractory concentrate followed by cyanide leaching. It is a less common method than roasting. The concept here is to heat the concentrates in an inert atmosphere to decompose the sulfides according to the following (La Brooy et al., 1994).

$$FeS_2 + \text{heat} \rightarrow FeS + S \qquad (2)$$

$$FeAsS + \text{heat} \rightarrow FeS + As \text{ (Marsden \& House, 2006)} \qquad (3)$$

The pyrolysis reaction has a number of advantages:

- The decomposition of pyrite and arsenopyrite concentrates together leads to convertion into pyrrhotite.
- Fine invisible gold particles can be separated relatively easily from pyrrhotite.
- The porosity increases, increasing the access of leaching solutions.

The main advantage of the pyrolysis is the elimination or reduction of harmful gaseous effluents. Instead of SO_2, elemental sulphur is produced. Also, the arsenic component is converted to arsenic metal or solid arsenic oxide (Dunn & Chamberlain, 1997).

The main disadvantage of pyrolysis is the energy required to support an endothermic reaction. Also, pyrrhotites are one of the examples of the cyanicides which can rob oxygen and free cyanide ions from the solution. This may lead to decreases in the overall gold recoveries after cyanidation (Dunn et al., 1995).

2.7.1.3 Microwave Roasting

Microwaves could be used as an alternative source of energy for the treatment of ores in some of the unit operations such as drying, calcining, roasting and smelting. Carbon and metal sulphides are known to be good microwave absorbers and they can be rapidly and selectively heated (Amankwah & Pickles, 2009). Some of the advantages of microwave heating are:

- Rapid and selective heating;
- Heat transfer occurs effectively;
- Internal heating occurs;
- Environmentally friendly process;
- Continuous process is possible (Nathakumar et al., 2007).

Microwave roasting is done to decrease the mechanical strength and increase liberation by decreasing the energy required for size reduction (Amankwah & Ofori-Sarpong, 2011; Çiftçi & Akçıl, 2009). It is shown that by the removal of sulphur and arsenic as a result of microwave heating, cyanidation of refractory gold ores would be remarkably improved (Ma, et al., 2010).

2.7.2 Pressure Oxidation (POX)

Pressure oxidation became an important pre-treatment alternative in the last decades (Kongolo & Mwema, 1998). During this process, the sulphides are oxidized by oxygen at an elevated temperature and sulphides are broken down (Recovering Refractory Resources, 2012). It is important to know the thermodynamic properties of the chemical system and reaction kinetics while designing pressure oxidation pretreatment (Thomas & Pearson, 2016; Thomas, 2005)

Pressure oxidation requires high degree of operator skills, high pressure and temperature equipment (autoclave) and therefore it is a costly process (Flatman et al., 2008; Gao et al., 2009).

The ore consisting sulfide and arsenic minerals are liable to rapid oxidation. However, it is an unstable method for treatment of carbonaceous ores. Also, chlorination can be used in association with POX process (Marsden & House, 2006).

2.7.3 Biological Oxidation (BIOX)

Biological oxidation uses naturally occurring bacteria to accelerate sulfide oxidation. It is mostly used for arsenic-gold ores (Marsden & House, 2006). Bacteria is the catalyst in the series of chemical reactions that causes the destruction of gold involving phases (Marsden & House, 2006). Important parameters in biological oxidation are pH, temperature, O_2 and CO_2 transfer during reaction (Çiftçi & Akçıl, 2009). Broadly used bacteria for this pretreatment procedure is Thiobacillus Ferrooxidans (Yannopoloulos, 1991). When biological oxidation uses ultrafine ground feed, process kinetics and efficiency would increase (Miller & Brown, 2005).

However, rate of the reactions is relatively slow in bacteria assisted oxidation compared to other pretreatment methods. The other disadvantage is that bacteria species are very susceptible to changes in environmental conditions and require careful control (Linge & Welham, 1997). For instance bacteria become inactive at temperatures higher than 40°C (Yannopoloulos, 1991).

2.7.4 Ultrafine Grinding

When gold is locked in sulphides and silicates, it is necessary to reduce the particle size to a degree where gold is entirely or partially liberated. For refractory ores with possibility of physical gold liberation, this generally corresponds to grinding to less than 10 µm.

Ultra-fine grinding may be a solution applicable especially where gold is locked in siliceous matrix (Flatman et al., 2008). Advantage of ultrafine grinding is that the chemistry of the host and gangue rock does not change. However, ultrafine grinding is very energy-intensive and it also increases the surface area of other host minerals (Recovering Refractory Resources, 2012). This increases the possibility of higher cyanide consumption.

Mineral Engineering Technical Services compared four main pre-treatment methods, and highlights are presented in Table 2.5.

Table 2.5. Comparison of Pretreatment Methods (METS, 2009)

	ROASTING	**BACTERIAL LEACHING**	**PRESSURE OXIDATION**	**ULTRA FINE GRINDING**
Technical Risk	Low	High	High	Low
Environmental Risk	High-depending on gas clean up	Low	Lowest	Low
Capital Cost	Prohibitive	High	High	Low
Operating Cost	Low	High	High	Low
Simplicity	Not with gas cleaning	No complex plant	No materials of construct	Simple
Acid neutralization	Low	High	High	Low
Cyanide use	Medium	High	High	Modest
Power consumption	Low	Very High	High	Medium
Materials construct	Typical	Exotic	Exotic	Typical
Process Chemistry	Simple	Complex	Complex	Simple
Major Driver	Sulphur level	Power cost/Au:S ratio	Operating pressure & temperature	Power cost
History	Tried & true	Poor, failures	Difficult	Good, growing
Operability	Simple	Difficult, learning Curve applies	Fine, getting better all the time	Good, wear issues Power cost
Recovery	Very good	Subject to "Bacteria Health"	Excellent	Grind sensitive

2.8 Treatment of refractory gold ores

2.8.1 Cyanidation

The process of extracting gold from ore with cyanide is called cyanidation. The chemical reaction for the dissolution of gold by cyanide is defined by the well-known "Elsner Equation":

$$4\ Au + 8\ NaCN + O_2 + 2H_2O \rightarrow 4\ Na[Au(CN)_2] + 4\ NaOH \qquad (2)$$

Despite the fact that the amenability of cyanide for gold, cyanide will also form complexes with other metals from the ore, such as copper, iron and zinc. That complexes arrest cyanide so that it is not available to dissolve gold. For example, Copper cyanides are moderately stable. Their formation cause both operational and environmental problems, such as higher cyanide concentrations.

Also, the presence of free sulphur or sulfide minerals in the ore has negative effect on cyanidation. They cause high rate of oxygen consumption in order to oxidize. Therefore, in order to control pH and to avoid the volatilization of hydrogen cyanide (HCN), lime addition increases.

Cyanide refers to a group of compounds made of carbon and nitrogen. Cyanide solutions readily bond with gold, silver and some other metals, which is why the mining industry use them.

When cyanide is dry, it is stable and it usually in solid form. But, free cyanide is the most toxic form of cyanide in a gaseous or aqueous state that consists the cyanide anion and hydrogen cyanide (HCN). At pH between 9.3 - 9.5, CN^- and HCN are in equilibrium. At pH 11 the cyanide is found in solution as cyanide anion, and at pH 7 the cyanide remains in the form of HCN (Figure 2.7). Hydrogen cyanide has no color. It has a bitter almond odor and it is extremely dangerous.

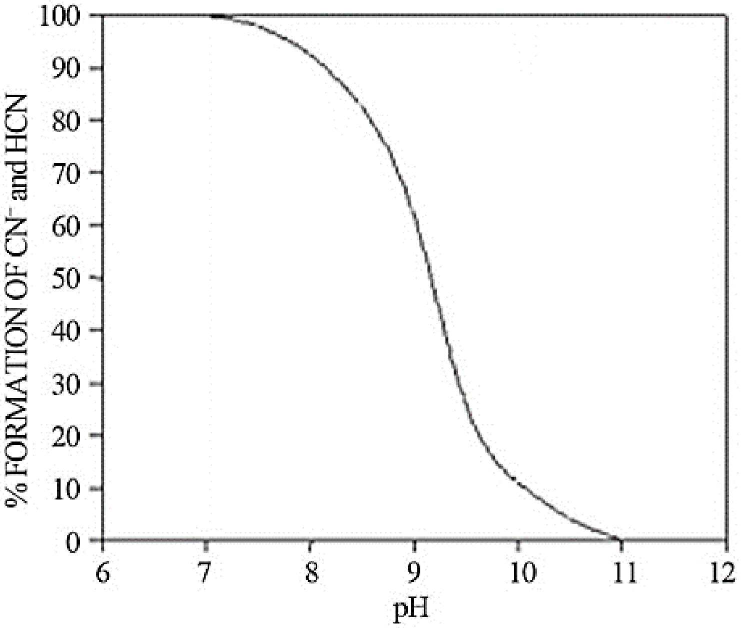

Figure 2.7. Relation Between pH and HCN

Cyanide-leaching of gold enabled exploitation of low gold ores once considered non-feasible. In the extraction of copper, nickel, cobalt and molybdenum, cyanide is used effectively during the milling and concentration processes (MPC, 2000).

2.8.2 Rapid Determination of Cyanidation Behavior of Gold Ores: Bottle Roll Test

The aim of this test is to determine the extent of gold recovery from gold ores and to gain an understanding of leaching kinetics of gold extraction and NaCN consumption.

It is a simple procedure that relies on rapid and forced leaching of a representative portion of gold ore in a bottle-type reactor over a rolling table. The test is conducted for a certain time period and the bottle is stopped at certain intervals to receive samples from pregnant solution. Analysis of this solution provides information about the extent of gold extraction, kinetics of leaching as well as NaCN consumption. In this respect, the test gives critical information about the liability of a given ore to cyanidation, the refractoriness of the ore, critical components such as preg-robbing material and/or cyanicides.

The test could be applied both at the very early stages, i.e. as a supplemental tool to help make decisions for a specific ore as well as for continuous monitoring of the gold extracted during a running operation. Determination of cyanide extractable gold by bottle roll tests is also a logical method for grade control in mines that employ CIL or CIP gold recovery circuits (ALS, 2009).

The procedure of bottle roll tests is described in further details in Chapter 3.

2.9 Challenges and opportunities

Major challenges associated with the refractory of gold ores are listed below:

- Capital and the operating costs are high
- Gold is disseminated
- Has higher technical risks
- Use of a pre-treatment method is generally necessary to attain acceptable Au recoveries
- Detailed mineralogical analysis is critical to ensure a successful processing route

Despite these challenges, processing of refractory gold ores has become a necessity. Due to the rapid depletion of free milling, easy-to-process gold reserves in the globe complex and refractory gold ore processing become important and has a peak potential. Also, demand for gold and gold prices increasing. There are several studies on that type of gold ores in order to decrease expenditures and technical risks. Moreover, closed gold ore mines can be reopened and tailings of early processed ore may be recoverable with cyanidation and pretreatment methods.

2.10 Previous Studies on Complex and Refractory Gold Ores

Linge, et al., (1997) used electrochemical in-situ chlorination technique to treat complex arsenopyritic gold ore. Gold recovery is improved from 7.3 to 94% by the treatment. According to the results electrochemical in-situ oxidation is much faster than bioxidation but it is an environment friendly process than carbon in leach (CIL) process.

Dunn et al., (1995) practiced pyrolysis of arsenopyrite in N_2, CO_2 and SO_2 atmospheres. In pyrolysis elemental sulphur was produced rather than SO_2 gas. In N_2 and CO_2 atmospheres similar results were achieved: Maximum mass loss occurred at 700°C and gold recoveries were 35 and 48 %, respectively. In SO_2 pyrolysis was faster with maximum mass loss occurring at 600°C and gold recovery was improved up to 61%.

Lehmann, et al., (2000), examined effects of three pre-treatment methods; fine grinding, pyrolysis-roast (Pyrox), and an oxidative pressure acid leach (PAL), on gold recoveries. The gold existed in an arsenopyrite matrix. Results showed that untreated ores did not yield high gold recoveries even after fine grinding. But, after oxidative treatment gold recoveries significantly increased. The recoveries after PAL process, Pyrox and fine grinding were 79%, 75%, and 55-60%, respectively Also, the dissolved oxygen consumption levels during cyanide leaching were significantly reduced by the two oxidative treatments, compared to the untreated ores.

Almeida (2001), made experiments on roasting a gold ore. Roasting was done at 450-700 °C for 1 h and it increased the recovery to 74% from 60%in a highly concentrated NaCN solution. Results showed that 600-700 °C temperature range for roasting was preferable to reduce cyanide and lime consumptions. Also, pre-washing the roasted product seemed not to reduce cyanide consumption.

For the roasting plant at Kwekwe, Zimbabwe (Gudyanga et al., 1999) complete oxidation of the sulphides was found necessary for entirely liberating gold. Operating conditions were temperature over 180°C, partial pressure of oxygen of over 15 bars and retention time of over 2 hours. Results showed that recoveries were highly dependent on the reaction temperature, retention time, percent solids, partial pressure of oxygen and particle size. Pressure oxidation pretreatment increased gold recoveries to more than 90%.

Robinson (1988) did an examination on the roasting characteristics of three flotation concentrates obtained from a gold ore from the Barberton area. It was found that the gold in the concentrates occurred mainly in two forms: As very fine gold particles distributed within the arsenopyrite, and in a slightly coarser form loosely associated with the pyrite. Direct cyanidation of the concentrates gave fairly low gold extractions from 16 to 30 %. After roasting the concentrates at 650 to 700°C for 20 minutes, extractions increased up to 86-90 %.

Dunn and Chamberlain (1997), examined a gold bearing arsenopyrite concentrate that consisted of 70% arsenopyrite and 25% pyrite with smaller extents of stibnite and silicates. With cyanide leaching on untreated ore, only 6% of gold could be extracted. When samples were first pyrolysed in N_2 or CO_2, and cyanide leached, recoveries increased to 40-50 %. In pyrolysis pyrite and arsenopyrite were decomposed to pyrrhotite. When samples were first pyrolysed at 700°C for 30 minutes, and then oxidized for two hours in O_2 at 550°C and cyanide leached, gold recovery increased to 97%. When the oxidation temperature was increased to 650°C, gold recovery decreased to 78%. In air, a maximum recovery of 86% was obtained after roasting at 550°C for 6 hours.

Ma, et al., (2010) studied microwave heating to remove sulphur and arsenic from refractory gold flotation concentrate. Above 400°C, removal of sulphur and arsenic occurred. When the pretreatment period was increased, removal efficiencies also increased. Results showed that removal of arsenic was easier than sulphur and, supplying oxygen during microwave heating enhanced sulphur removal.

Amankwah, et al., (2009) conducted a study on microwave treatment of the flotation concentrate of a refractory gold ore. Microwave roasting was done to oxidize both sulphides and carbonaceous matter. Studies showed that sample temperature increased with increased microwave power, processing time and sample mass. 600 W was found to be suitable for microwave roasting, as higher powers resulted in sintering and melting of the concentrate. After treatment gold recoveries increased to >96%. Total carbon removal rates and heating rates were comparatively higher and the specific energy consumptions were lower in microwave roasting as compared to conventional roasting.

In another work by Amankwah, et al., (2011) it was seen that microwave generated micro-cracks and reduced the crushing strength of a free-milling gold ore that consisted of magnetite, hematite silicates and quartz. These micro-cracks made diffusion faster, so leaching rates were enhanced. Microwave pretreatment improved grindability and increased the efficiency of leaching while improving leaching kinetics. After microwave treatment grindability of the ore and crushing strength were reduced by 31.2%. Results showed that gold extraction was higher than 95% within 12 hours after microwave treatment.

Nanthakumar, et al., (2007) conducted a study on microwave roasting of a refractory gold ore and compared the results with conventional roasting. Firstly, thermogravimetric analysis (TGA) was conducted and three major reactions in the roasting reactions were determined; oxidation of pyrite, oxidation of the carbonaceous matter and decomposition of carbonates. With direct microwave roasting, oxidation of organic carbon could not be achieved. In indirect microwave roasting, magnetite was used as a suscepter and preg-robbing was eliminated with 98% gold recovery after cyanide leaching. Conventional roasting gave similar results with indirect microwave roasting in terms of gold extraction. However, carbon removal was higher and similar results at lower specific energy consumption rates were achieved in indirect microwave roasting as compared to conventional roasting.

A study was conducted in Porcher Island Gold Mines (Vizsolyi & McElroy, 1986) on the effects of fine grinding and biological leaching on extraction of gold from a relatively complex gold ore. After fine grinding particle size of the ore was reduced to 90% -44 μm, and after cyanidation gold recovery increased to 80% at a sodium cyanide consumption of 5-6 kg/ton of ore. Biological pre-leaching of the coarse fraction of the crushed ore was not a viable option for treatment of this ore fraction. Gravity pre-concentration of +500 μm fraction of crushed ore was found as a solution to minimize the volume of material to be treated by direct cyanidation.

Sayın, et al., (2010) compared conventional leaching and cyanidation of gravity concentrated ore. Knelson concentrator was used for centrifugal gravity concentration as a means of pretreatment. A pre-concentrate with 93 g/t of gold was produced from a gold ore with 6.7 g/t Au, and by this application cyanide consumption was reduced significantly by 70%.

Celep, et al., (2006) also attempted to pre-concentrate Mastra gold ore, Türkiye by centrifugal gravity separation using a Knelson concentrator. It was seen that the ore consisted of coarse gold particles and this was the main motivation for use of Knelson. High grade concentrate (771.95g/t) obtained by Knelson concentrator with 47.2 % gold recovery. The concentrate can be recoverable with cyanide leaching with 98% gold extraction yield.

Celep, et al., (2009) used Akoluk (Ordu) ore to conduct a study on extraction of gold and silver by cyanide leaching. Firstly, diagnostic leaching tests were performed, then leaching tests were applied. Direct leaching after 24 hours resulted low extraction of gold and silver (47% and 19.2%, respectively). Even grinding to -38 μm did not improve the results. Using diagnostic leaching, the reason of refractoriness was identified as fine dissemination of gold and silver mainly in sulphide minerals.

Örgül and Atalay, (2002) applied thiourea for leaching of gold as an alternative approach to conventional cyanidation. Gold ore from Kaymaz, Türkiye was used in this study and gold was finely disseminated in the host rock. Thiourea consumption for different leaching parameters was investigated in details. Results showed that, by thiourea leaching of a feed with 100% -53 μm particle size for 6 hours, recoveries as high as 85.8% could be achieved.

Vukcevic (1996) studied different alkali and acid techniques on low grade copper-gold ores/tailings in Australia. He used ammonia-cyanide and sulphuric acid in the tests. The ore used for ammonia-cyanide leaching consisted of 1.5% Cu. It was found that, at optimized conditions, with only 0.55 kg/t NaCN and 3 kg/t NH_3 consumption, 89% of gold could be extracted with only 200 ppm of copper remaining in the solution.

Kasaini, et al., (2008) used tailing samples assaying 1.35 g/t gold and 155 g/t silver for further extraction of these values. Samples were exposed to a two-step processing. First step was alkaline pre-treatment to decompose jarosite minerals. The second step was cyanide leaching. The parameters changed during experiments were NaCN dosages (2.5–10 kg/ton of ore) and particle size (96–200 μm). Results showed that combination of alkaline pre-treatment (with $Ca(OH)_2$) and cyanide leaching increased gold and silver extractions from 41% and 25% to 55% and 81%, respectively.

Önal, et al., (2007) made an investigation on the Bolkardağ ore, Türkiye. The ore assayed 14.5 gr/t gold, 415 gr/t silver and 7.03 % lead. The ore was chemically and mineralogically characterized and was subjected to gravity concentration using shaking table. The obtained pre-concentrate was cyanide leached. Through this method, recoveries of 91.2 % of gold, 69.6% silver and 36.7% of lead were possible.

Shin, et al., (2013) carried out a study on bioleaching of a gold ore. A cyanide producing bacteria, ‘‘Chromobacterium Violaceum’’, was used in this work. The optimal conditions for bacterial activity were identified as pH 9 and addition of 5 g/L glycine. The highest gold bioleaching results were achieved at finer particle sizes (-180 μm) at 20% pulp density. This study showed that Chromobacterium Violaceum could be used as an alternative to conventional cyanidation.

Murthy, et al., (2007) applied thiourea leaching on a low grade gold ore (1.4 g/t). Prior to leaching a sulphide concentrate with 6.1 g/t of gold was obtained by flotation. Without flotation pre-concentration of this ore gold extraction by thiourea was limited to 16.4%. The ore was then subjected to flotation coupled with pressure oxidation and gold recovery dramatically improved up to 74%. It was concluded that gold recoveries could be effectively improved by effective combination of physical beneficiation, oxidation and chemical procedures.

Ayata and Yıldıran, (2001) studied silver extraction with thiosulphate complexing reagent from Ag_2S. pH, the reaction temperature, the complex equilibrium of Ag^+ ion with thiosulphate and the solubility of Ag_2S were examined. After electrolysis, purity of silver was very high (99%).

Özgen, (2014) investigated flotation behavior of sulphide gold ores. Flotation was used for pre-concentration of Kaymaz ore, Türkiye. Also, an attempt was made to find a link between gold and sulphur recoveries: A direct and linear relationship between recoveries of gold and sulphur was identified. Results showed that pre-concentration by flotation increased overall gold recoveries.

Şen & Çilingir, (2009) conducted a study on the effect of reagents on gold flotation to produce a pre-concentrate for subsequent leaching Ore from Ovacık operation Bergama, Türkiye was used for the study. The most effective reagent combination was found as potassium amyl xhanthate, dialkyl dithiophosphate and dialkyl dithiophosphinate. It was also seen that increasing the Na_2S addition up to 85 g/t increased rate of the flotation. On the other hand, Na_2S dosages higher than 85 g/t, caused decreased the flotation rate and gold recoveries.

CHAPTER 3

MATERIALS AND METHOD

In this thesis possibility of gold extraction from a given arsenic-rich ore through cyanide leaching was determined. For determination of the leaching behavior, ‘‘Bottle Roll Tests’’ were conducted. Three important aspects of the cyanidation behavior of the ore were identified: Extent of Au extraction, Au extraction profile with respect to time and cyanide consumption of the ore, and cyanide extraction of the ore. The influence of roasting as a pre-treatment procedure on enhancing gold extraction from the ore and on reducing cyanide consumption was also investigated. The exact location and source of the gold ore cannot be declared in this work owing to commercial restrictions set by the ore-provider company.

3.1 Sample Preparation

The ore was delivered in the form of drill-core fragments in separate bags. Each separate bag contained samples from different drilling intervals retained within a certain drill well. Details and weights of the drill-core fragments and corresponding logs/intervals are presented in Table 3.1. The drill core fragments weigh approximately 72 kg, (Figure 3.1). Before sample preparation, coarse fragment pieces in each drill log bag were inspected and characteristic sample pieces that reflect specific mineralogical features of the ore were separated for preparation of thin sections and microscopic mineralogical investigations. Next, drill core fragments were made ready for the study after several stages of comminution and representative sampling. A flow chart that briefly presents the procedures followed for sample preparation for different phases of the study is shown in Figure 3.1.

Table 3.1. Details of Drilling Cores

Drill Well	**Log No**	**Drilling Interval**		**Length (m)**	**Weight**		
		from (m)	**to (m)**		**½ (g)**	**¼ + ¼ (g)**	
KT-01	236	95.20	96.00	0.80	946.21	987.16	
	237	114.00	115.00	1.00	1222.07	1261.77	
	238	115.00	116.00	1.00	1284.76	1283.95	
	239	116.00	117.00	1.00	1309.54	1307.40	
	240	117.00	118.00	1.00	1230.24	1253.56	
	241	118.00	119.00	1.00	1703.11	1695.92	
	242	120.00	121.00	1.00	1359.01	1390.82	
	243	121.00	122.00	1.00	1440.02	1442.89	
	244	123.00	123.80	0.80	726.88	736.21	
	245	123.00	124.40	1.40	1000.19	1027.82	
	246	124.40	125.00	0.60	702.26	704.61	
	247	125.00	126.00	1.00	1117.59	1120.56	
	248	126.00	126.50	0.50	508.92	553.36	
	249	126.50	127.50	1.00	488.58	505.97	
	250	127.50	128.30	0.80	736.00	761.94	
	251	128.30	129.40	1.10	1018.13	1017.95	
	252	129.40	130.00	0.60	705.94	743.13	
	253	130.00	131.00	1.00	1096.61	1144.21	
	254	131.00	131.80	0.80	1028.06	1033.45	
	255	131.80	133.00	1.20	1183.58	1180.75	
KT-02	256	102.70	104.10	1.40	1598.37	1609.42	
KT-03	257	41.00	41.80	0.80	1640.51	1633.59	
	258	41.80	42.80	1.00	1445.94	1448.87	
	259	42.80	43.70	0.90	1544.28	1537.75	
KT-04	260	74.30	75.00	0.70	993.89	973.44	
	261	75.00	76.00	1.00	1350.79	1339.16	
	262	76.00	77.00	1.00	1300.37	1271.84	
	263	77.00	78.00	1.00	1076.94	1063.30	
	264	78.00	79.00	1.00	1037.48	1043.73	
	265	79.00	80.00	1.00	1085.37	1033.47	
	266	80.00	81.00	1.00	1116.47	1109.55	
	267	81.00	82.00	1.00	976.38	972.26	**TOTAL(g)**
					35974.49	**36189.81**	**72164.3**

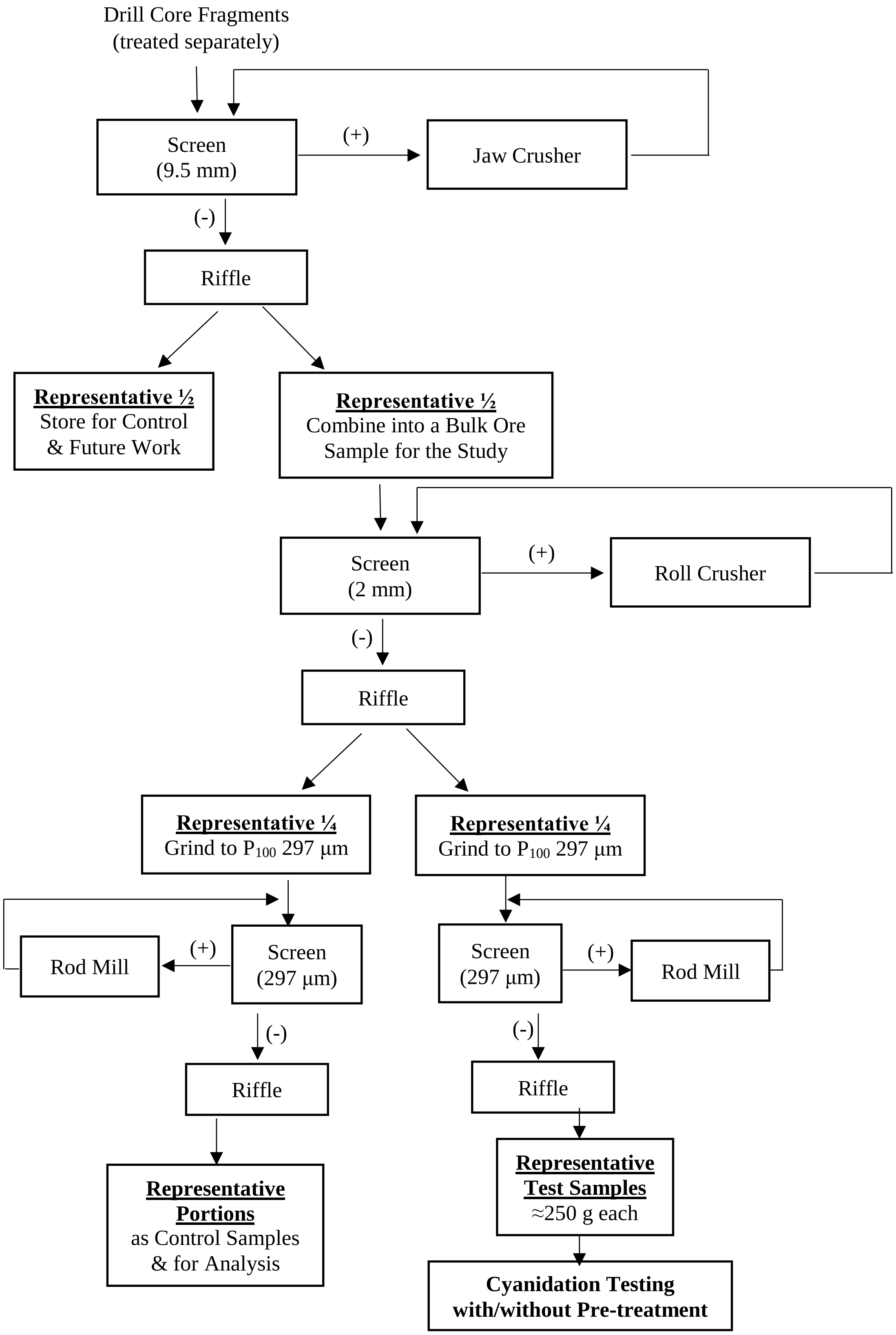

Figure 3.1. Sample preparation for various stages of the study

The fragments for each drill log & interval were separately reduced to 100% -9.5 mm size using a laboratory jaw crusher. -9.5 mm material for each drill log was then representatively divided into two portions by riffling. One of the ½ portions were packed and preserved for possible future work and/or control tests/analysis. The other ½ portion of each drill log were combined and blended into a bulk. This bulk was used as the ore sample to be used for further stages of the leaching work. The ore sample was further crushed down to 100% -2 mm size using a laboratory roll crusher. Particle size distribution of 100% passing -2mm is presented in Table 3.2 and Figure 3.2. The analysis showed that P_{80} is 1.5 mm and P_{50} is 811 μm.

Table 3.2. Particle size distribution of the -2 mm ore

Particle Size (μm)	**Weight (g)**	**Weight (%)**	**Size (μm)**	**Cumulative Undersize (%)**
-2000+1000	454.79	40.88	2000	100.00
-1000+500	268.22	24.11	1000	59.12
-500+250	132.51	11.91	500	35.01
-250+150	64.66	5.81	250	23.10
-150+106	40.57	3.65	150	17.29
-106+75	31.69	2.85	106	13.64
-75+53	29.99	2.70	75	10.79
-53+38	35.57	3.20	53	8.10
-38	54.53	4.90	38	4.90
	1112.53	**100.00**		**100.00**
P_{80}	1510.75 μm			
P_{50}	810.8 μm			

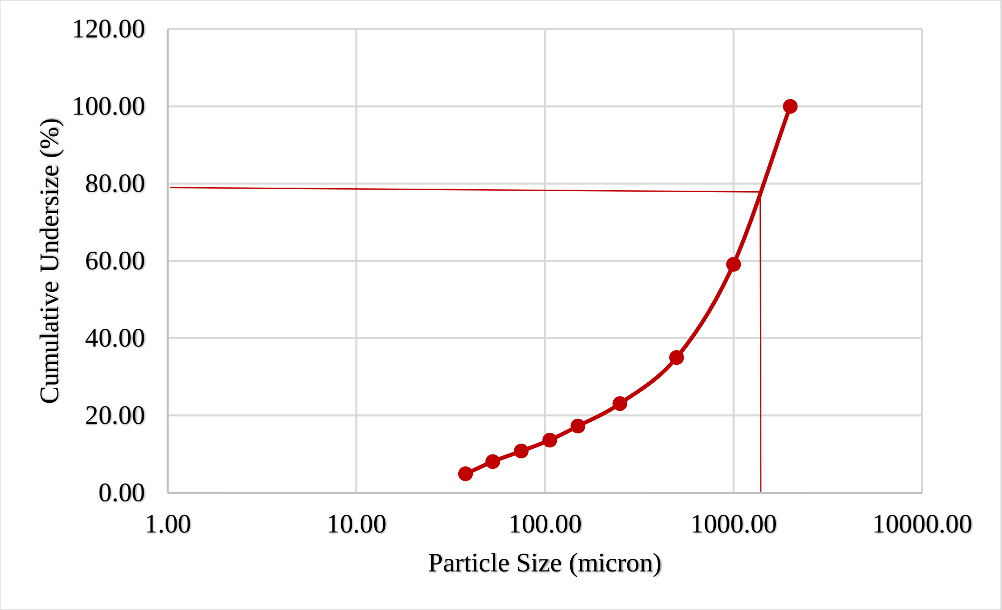

Figure 3.2. Particle size distribution of the -2mm ore

Following this stage, the ore was riffled into two ¼ portions and each portions was separately ground to -297 µm (-48 mesh) using a rod mill. One of the portions were reserved as a check-sample as well as analytical determinations (head assaying, XRD, XRF, ICP, TG, etc.). The other portion was divided into representative samples of approximately 250 g using different sized riffles, to be used solely for cyanidation work with and without pretreatment, i.e. bottle roll tests with and without roasting.

3.2 Analytical Tools Used, Mineralogical & Chemical Analysis

For characterization of the ore sample, assessment of the cyanidation behavior and evaluation of the influence of pretreatment on gold extraction, several analytical tools, mineralogical and chemical procedures were facilitated.

Mineralogical and compositional characterization of the ore was conducted by optical microscopy and X-Ray Diffraction (XRD), qualitatively. Mineralogical analysis by optical microscopy were performed on polished thin sections taken from characteristic and specific sections of the drill cores. XRD was performed on representatively prepared ore samples using a Rigaku-SmartLab diffractometer. The sample was ground to 100% -75 μm size for the analysis and dried at 50°C in a laboratory furnace. XRD patterns were obtained on samples in free powdered form in the 0° -100° range (°2θ). XRD was also used to compare the effect of roasting at varying conditions on the compositional characteristics of the ore.

The scanning electron microscopy (SEM) investigations in this study were conducted using a JEOL SEM 7700F instrument. Energy-dispersive X-ray spectroscopy (EDS) and elemental mapping were also performed where necessary, using an analyzer system that works in conjunction with the JEOL SEM 7700F. SEM images were taken on various sections of the run-of-mine (ROM) ore as well as on ore samples roasted for 60 minutes at three different temperature levels of 550 °C, 650 °C, 750 °C. In the thesis, the SEM images that provide useful information for interpretation of the obtained results are presented and discussed.

For quantitative analysis of the major and minor chemical components in the ore Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) and X-Ray-Fluorescence were used (where applicable). These analyses were outsourced, but samples for analysis were ground and prepared at Mineral Processing Laboratories of METU Mining Engineering.

For determining gold assay in the head sample, analysis of gold dissolved in pregnant solutions from bottle roll tests and gold assay in the tailing of bottle roll tests, Atomic Adsorption Spectroscopy (AAS) was used. For these analysis a Shimadzu AA-7000 spectrophotometer was used. Gold in pregnant solution samples were directly fed to the AAS and detected. For determining the gold assays in the head and remaining ore after bottle roll tests, the gold was first dissolved by chemical procedures using aqua regia solution. In this procedure a solution that includes a mixture of strong acids (HCl and HNO_3) at certain quantities (proportions of 1 to 3) is used to dissolve gold in the solid ore and removal of undesired metal constituents sequentially. Eventually, solutions that involve dissolved gold were obtained to be used for Au detection by

AAS analysis. The steps of dissolving the ore in aqua regia and subsequent chemical procedures are given in the Appendix.

To gain an understanding of the thermal behavior of the ore, regarding the roasting pretreatment, the ore was subjected to thermal analysis using Thermogravimetric Analysis (TGA). Thermogravimetric determinations were conducted on 10 mg representative ore samples, in oxidizing environment by supplying O_2 to the test chamber using ore samples with P_{100} sizes of 37, 53, 74, 106 and 150 µm. Thermal behavior of the ore in a temperature range of 0-1000 °C was detected in the form of weight loss profiles with increasing temperature. The TG profiles indicated the major temperature dependent degradations/reactions regions within the applied temperature zone. TG analysis were performed with a Perkin Elmer TG/DTG instrument at a heating rate of 10 °C/min.

3.3 Ore Characteristics

The major components in the ore include quartz (SiO_2), calcite ($CaCO_3$).gold (Au), orpiment (As_2S_3), realgar (As_4S_4), pyrite (FeS_2). Minor components in the ore were seen as silicates other than quartz, gypsum ($CaSO_4 \cdot 2H_2O$), arsenopyrite (FeAsS), and hematite (Fe_2O_3). The gold is present in disseminated form. In order to recover the gold, bottle roll tests were used.

Microscopic investigations showed that significant components in the ore were quartz and calcite. Presence of pyrite, realgar, orpiment and dolomite ($CaMg(CO_3)_2$) were also remarkable, but in relatively lower quantities as compared to quartz and calcite. In the microscopic analysis, gold was very hardly identified in the ore and when detected it was seen as associated with and/or locked in arsenic sulfides, i.e. realgar and orpiment, and rarely as very fine entities locked in pyrite. Presence of arsenic sulfides and strong association/encapsulation of gold with these minerals point to challenges in gold extraction from this ore and possible refractory character of the reserve. Orpiment is more abundant than realgar. Both arsenic sulphides were found as fillings in voids, veines, fractures and microcracks within the ore. Pyrite is seen along with orpiment and realgar as fillings in voids, veins and fractures. Traces of elemental sulphur in this ore should also be mentioned, particularly in microcracks.

The minerals detected are labeled above or next to the major/meaningful peaks recorded in the X-ray Diffraction pattern. XRD of the ROM ore (Figure 3.3) confirms the mineralogical analysis. The pattern shows several peaks for quartz and calcite, some of which occurred with outstanding intensities. These correspond to the abundance of quartz and calcite, as observed in the mineralogical analysis. Remarkable presence of orpiment, realgar and pyrite were also identified. Relatively higher presence of orpiment seen in the microscopic investigations was justified by XRD, with higher number of peaks compared to realgar and pyrite (Figure 3.3).

Analysis on the gold content of the showed that the ore assays approximately 2.5 ppm Au. Determination of the compositional characteristics verified the high arsenic content and presence of relatively high amount of sulphur in the ore (Table 3.3).

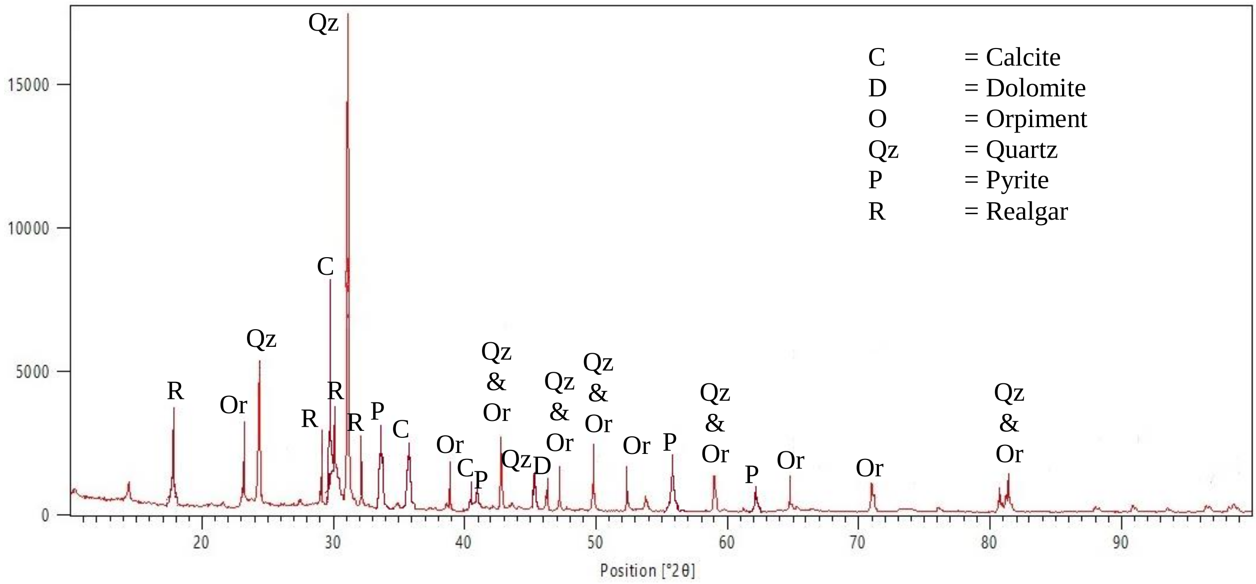

Figure 3.3. XRD Pattern of Run-of-Mine (ROM) Ore

Table 3.3. Compositional characteristics of the ore

Compound	%	Element	
SiO_2	56.12	Au	2.50 ppm
CaO	19.12	Ag	2.00 ppm
Al_2O_3	3.20	Cu	750 ppm
Fe_2O_3	1.12	Pb	362 ppm
K_2O	0.16	Zn	250 ppm
MnO	0.20	Mo	6.5 ppm
MgO	0.20	As	5.52%
		S	7.52%

3.4 Bottle Roll Testing

For determining the liability of the given ore to cyanide leaching, Bottle Roll Tests were conducted. Bottle Roll Testing is recognized worldwide and commonly used for studies that aim at identification of the leaching behavior of gold ores. Bottle roll tests were performed with the roll-setup at the Mineral Processing Laboratories of Mining Engineering Department of the Middle East Technical University. The setup can hold up to six bottles at a time and the rotational speed is adjustable. The setup is shown in Figure 3.4.

Bottle roll testing is typically based on treatment of a gold ore sample in a certain amount, with NaCN solution, under specific conditions. Solid ore sample and the NaCN solution are put into bottles and rotated at a constant speed (30 rev/min) for a certain period. Forced leaching of the ore by the NaCN is aimed as the NaCN solution is intended to contact with the ore in an effective manner. With prolonged and imposed contact, the theory is to leach (chemically dissolve) the gold in the ore as much as possible. The liquid solution containing dissolved gold as a result of leaching is called "pregnant solution".

The liability of the given ore to gold extraction is determined by sampling of the pregnant solution after certain leaching periods. The solution samples are then analyzed for their gold content and NaCN consumption. A plot of the extraction of gold and NaCN consumption vs. time provides the NaCN leaching profile of the ore, identifying the extent gold extraction, rate of leaching and possible NaCN consumption per ton of ore during the leaching process.

In this study, the ore was subjected to the 72 hours leaching in the bottle roll tests. The pregnant solution was sampled periodically. Dissolved gold in the periodical pregnant solutions was determined by Atomic Adsorption Spectrometry (AAS). The detailed experimental conditions of the bottle tests are described in Chapter 4. The steps followed in the application of the bottle roll test are provided in Appendix.

As stated above, in determining the liability of a given ore to cyanidation, in addition to the extent of gold extraction, consumed NaCN per ton of ore during leaching is of crucial importance. NaCN consumption should be as low as possible owing to economic and environmental constraints. NaCN consumption of the ore was found analyzing the periodical pregnant solution samples. The well-known and proven Silver Nitrate ($AgNO_3$)-Rhodanine indicator titration method was used for determining the NaCN consumption amount after certain leaching periods. The method is described in details elsewhere (Ryan & Culshaw, 1944; Breuer et al., 2011; Van Loon & Barefoot, 1989). In this way, the relation between NaCN consumption and gold extraction at different conditions were revealed with respect to leaching time.

Figure 3.4. Bottle Roll Test Setup

3.5 Pretreatment of the ore Prior to Cyanidation: Roasting

As the characterization work revealed, the ore sample comes from an arsenic rich, low-grade Au reserve. Mineralogical work showed presence of some of the most problematic minerals, such as realgar, orpiment and pyrite that are likely to adversely impact gold extraction. The adverse impact of such minerals may be two-fold: The gold may be embedded and/or locked in within these mineral phases and this imposes a refractory character to the gold ore. It is well established that refractoriness causes relatively lower extents of gold extraction. Besides low Au extraction rates, presence of these minerals could cause higher cyanide consumptions, since these minerals, known as ‘‘cyanicides’’ are liable to chemically react with NaCN.

In this respect, it was anticipated that for enhancing gold extraction performance pretreatment would be necessary for this ore. On accounts of the presence of remarkable quantities of arsenic sulphides and due to the locked form of Au in As-sulphides and pyrite, roasting was selected as an effective, rapid pretreatment procedure. The goal in this phase was, identifying the influence of roasting on gold extraction and NaCN consumption, determining the most effective roasting conditions and comparison of the cyanide leaching behavior of the ore with and without pretreatment.

Roasting temperature and time were varied as the major pretreatment parameters. Within this context, ore samples with 100% -150 μm size were subjected to roasting at 550, 650 and 750°C and for periods of 30, 60 and 90 minutes for each temperature level. Constant flow of air was provided to the fixed-bed reactor and oxidizing thermal conditions, essential for effective roasting, was ensured. The samples in the reactor were rabbled every 15 minutes in order to ensure a homogenous pretreatment process. After roasting, the calcines were subjected to the NaCN leaching by the before mentioned Bottle Roll Testing procedure to obtain Au extraction vs. time and NaCN consumption vs. time profiles for each pretreatment condition.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Determination of the Amenability of the Ore to Consume NaCN

The purpose of the first tests conducted was to determine the amount of sodium cyanide (NaCN) that needs to be used in the leaching solutions for the bottle roll tests.

At this stage, the representative ore samples ground to P_{100} 150 μm size were subjected to the bottle roll tests and the ore was treated with solutions containing 500, 1000, 1500 and 2000 ppm NaCN. The bottle roll device was stopped every 30 minutes, a liquid sample was taken from the pregnant solution in order to determine the gold extraction yield and NaCN consumption, and afterwards the tests were continued. When NaCN added was totally consumed the test was ended. In addition, pure O_2 were given as the oxidizing agent to all bottles through tubes in the system.

Table 4.1 presents the detailed conditions for the bottle roll leaching tests performed at this stage.

Table 4.1. Experimental Conditions to Identify NaCN Consumption

Particle Size	100% -150 µm
pH	10.5±0.3
Chemicals	For pH regulation: NaOH As leaching agent: NaCN
NaCN amount (ppm)	500, 1000, 1500, 2000
Maximum leach time	180 min.
Rotational speed	30 rev/min
% Solids by weight	40
O_2 addition	25±5 liters/hour

Results of gold extraction yield and NaCN consumption with respect to time are seen in Table 4.2 - Table 4.5.

A major part of 500 ppm NaCN at the end of 30 minutes was consumed by the ore. Yet, gold extraction yield remained very limited (7%). This situation indicates that addition of 500 ppm NaCN is not sufficient for gold extraction from the ore, and furthermore, 30 minutes' duration is insufficient for completing leaching (Table 4.2).

Table 4.2. Leaching Results with 500 ppm NaCN addition

NaCN Amount = 500 ppm (≈764 g/ton) *Feed Au Grade = 2.78 g/ton*		
Leaching time (Minute)	**Gold Yield (%)**	**NaCN Consumption (g/ton)**
0	0.0	0
30	7.0	600
60	8.0	750

Leaching results obtained after addition of 1000 ppm NaCN are seen in Table 4.3. 1000 ppm NaCN was entirely consumed by the ore at the end of 120 minutes of leaching, and as a result, gold extraction yield hardly reached 12.5%. In other words, according to the test which involved addition of 500 ppm, the leaching process continued until 120 min. and gold extraction yield increased by 4.5%. This situation shows that if the solution contains a sufficient amount of NaCN, leaching process and dissolution of the gold in the ore is likely to go on.

Table 4.3. Leaching Results with 1000 ppm NaCN addition

NaCN Amount = 1000 ppm (≈1500 g/ton) *Feed Au Grade = 2.72 g/ton*		
Leaching time (Minute)	**Gold Yield (%)**	**NaCN Consumption (g/ton)**
0	0.0	0
30	7.4	650
60	10.7	1100
90	12.1	1400
120	12.5	1500

Gold extraction yield increased to 16.6% after the addition of 1500 ppm NaCN (Table 4.4). In addition, the cyanide was almost entirely consumed by the ore. At this when the gold extraction of 16.60% was obtained and the reaction time was 120 minutes. Furthermore, it was seen that leaching decelerates after 90 minutes; in other words, when 30-minute periods are examined, despite NaCN consumption continues, the rate of gold extraction decreases between 90-120 minutes. Obtaining a higher degree of gold extraction yield in all periods as compared to the test with 1000 ppm is another supportive fact that NaCN concentration of 1000 ppm was insufficient for proper leaching of the ore.

Table 4.4. Leaching Results with 1500 ppm NaCN addition

NaCN Amount = 1500 ppm (≈2250 g/ton) *Feed Au Grade = 2.62 g/ton*		
Leaching time (Minute)	**Gold Yield (%)**	**NaCN Consumption (g/ton)**
0	0.0	0
30	8.8	1100
60	13.2	1600
90	15.6	2100
120	16.6	2200

Table 4.5. Leaching Results with 2000 ppm NaCN addition

NaCN Amount = 2000 ppm (≈3000 g/ton) *Feed Au Grade = 2.72 g/ton*		
Leaching time (Minute)	**Gold Yield (%)**	**NaCN Consumption (g/ton)**
0	0.0	0
30	9.6	1300
60	14.4	1900
90	16.3	2200
120	16.9	2400
150	16.9	2500
180	16.9	2500

With regard to the fact that NaCN was almost entirely consumed when the NaCN dosage was 1500 ppm and the gold continued to dissolve; another test was conducted by increasing the amount of NaCN addition to 2000 ppm. The results are seen in Table 4.5.

Increasing the NaCN addition from 1500 ppm to 2000 ppm did not lead to a significant increase in the gold extraction yield. When the results are examined, it is seen that leaching was almost complete within 120 minutes. Gold extraction obtained at this point was 16.9%. Similarly, NaCN consumption became almost constant after 120 minutes: Extending the leaching duration to 180 minutes did not increase gold extraction or NaCN consumption (Table 4.5).

When the results are collectively considered, it is seen that the ore consumes significant amounts of NaCN in leaching. This could be attributed to the high refractory characteristic of the ore as well as the presence of cyanicides such as orpiment, realgar and pyrite. In addition, despite high NaCN consumption, gold extraction remained limited.

Another possibility regarding low gold extraction would be insufficient liberation of gold bearing matrix at -150 µm grind size. It was seen that gold entities in the ore are finely distributed, and an effective NaCN-Au interaction might not be achieved.

With regard to the leaching process carried out at -150 µm size with different NaCN concentrations, gold extraction and NaCN consumption amounts with time are comparatively presented in Figure 4.1 and Figure 4.2, respectively. As a result of these first stage tests, it was seen that leaching cannot be completely finished with 500 ppm and 1000 ppm NaCN. When 2000 ppm NaCN was added, NaCN in the solution cannot be consumed by the ore within the given period. Furthermore, metallurgical results obtained with the addition of 2000 ppm NaCN does not show a significant increase when compared to those obtained with 1500 ppm cyanide (Figure 4.3 & Figure 4.4). In the light of these findings, it was deemed appropriate to proceed to the second stage tests, with the addition of 1500 ppm NaCN.

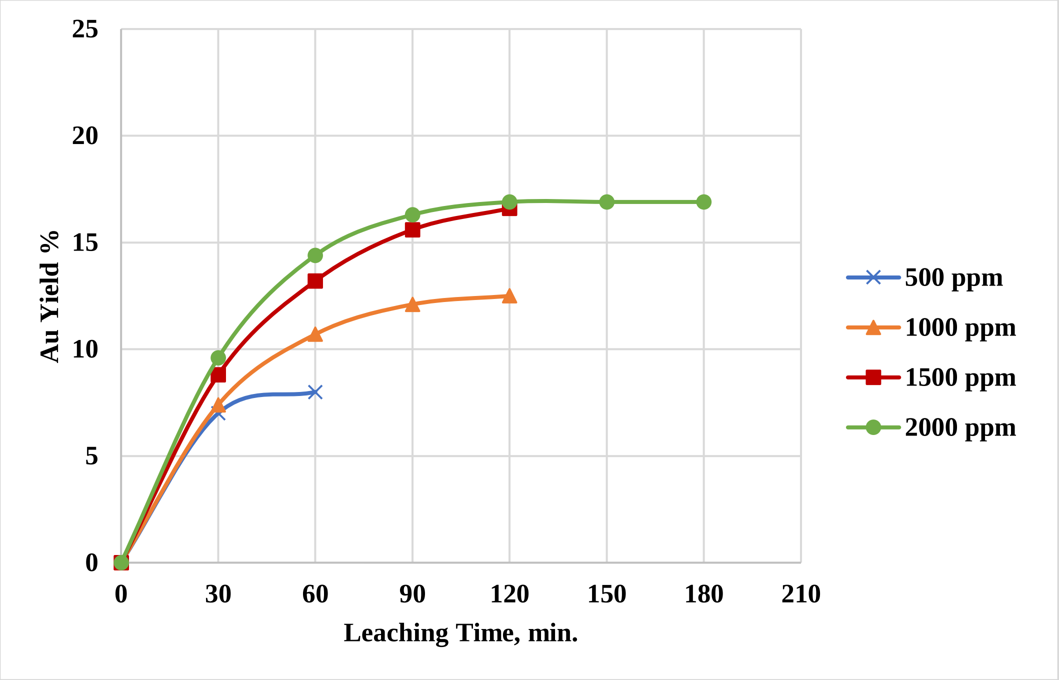

Figure 4.1. Gold recovery at -150 µm particle size with the addition of NaCN

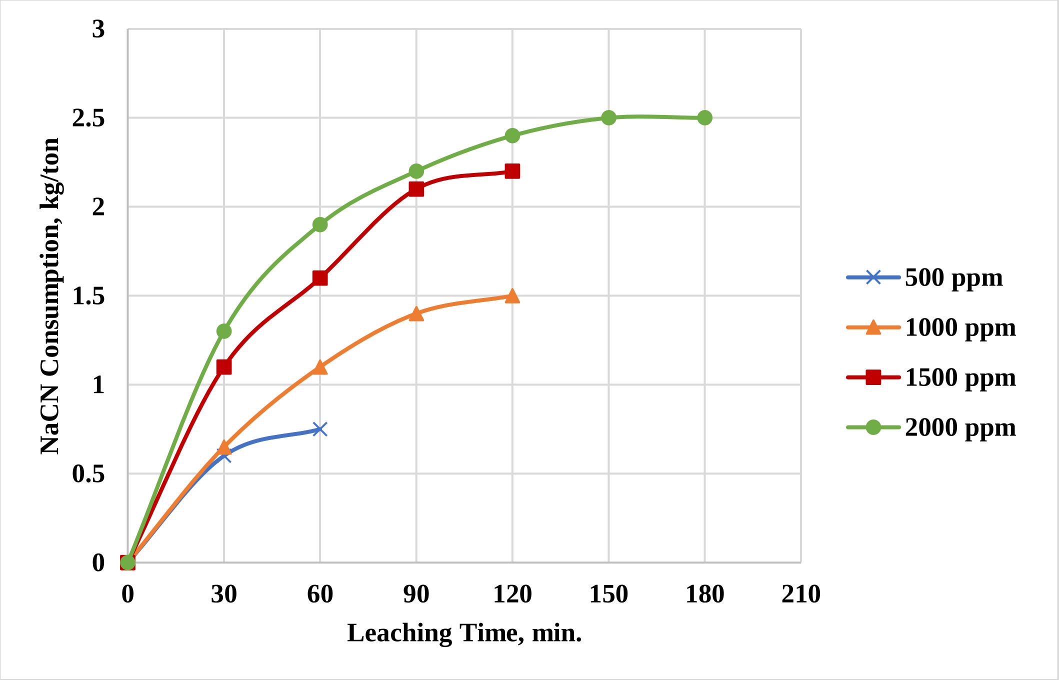

Figure 4.2. The consumption of NaCN at -150 µm particle size with respect to leaching time

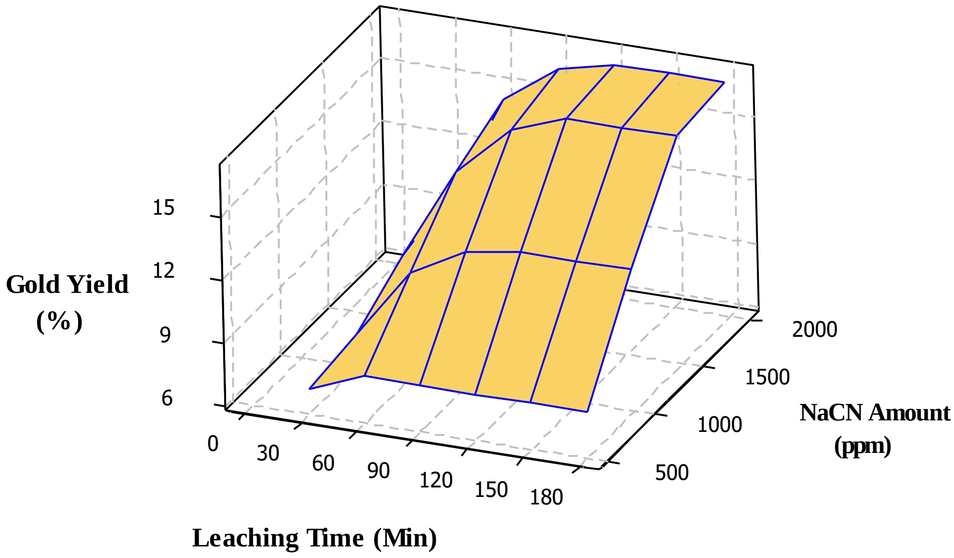

Figure 4.3. 3D Surface Response of Gold Yield (%) vs NaCN Amount (ppm) with respect to Leaching Time (min)

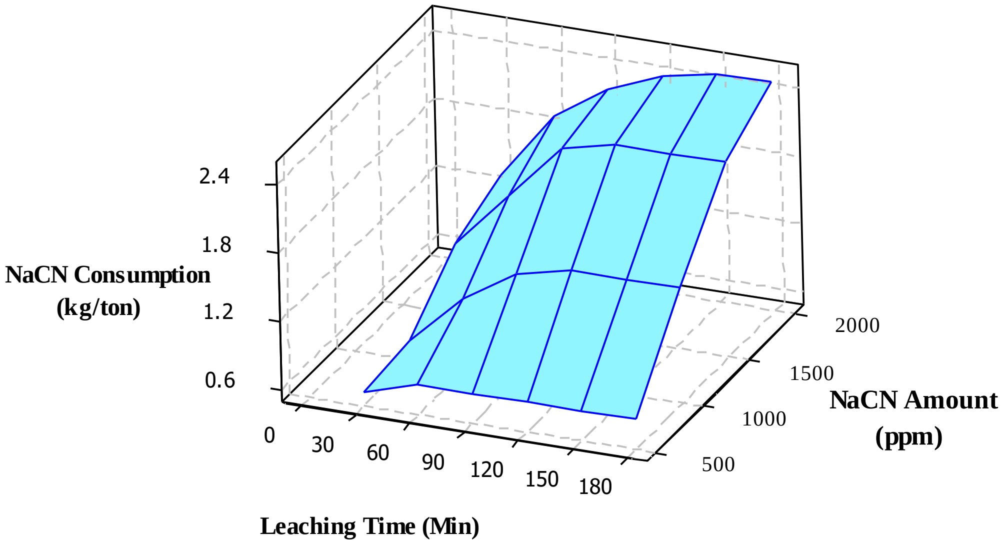

Figure 4.4. 3D Surface Response of NaCN Consumption (kg/ton) vs NaCN Amount (ppm) with respect to Leaching Time (min)

4.2 Determination of Impact of Particle Size on Gold Extraction Yield

At the first stage of the study, in addition to determination of NaCN concentration to be used in further testing, it was also seen that the size of -150 µm did not allow complete liberation of the gold in the ore in leaching tests. So, the second phase of the study was conducted to determine the relation between gold recovery and particle size of the ore.

At the second stage of the study, in order to increase liberation and thus to reveal the possibility to obtain higher gold extraction, bottle roll tests were performed at finer sizes, starting with -150 µm. Table 4.6 presents the detailed conditions of the bottle roll leaching tests performed at this stage. The main purpose was produce shorter distances in the particles for diffusion or better exposure of gold on particle surfaces.

Table 4.6. Experimental Conditions for determining the effect of particle size on Au extraction

Particle Size	100% -150, -106, -74, -53, -37 µm
pH	10.5±0.3
Chemicals	For pH regulation: NaOH As leaching agent: NaCN
NaCN amount (ppm)	1500 ppm
Maximum leach time	72 hours
Pregnant Solution Sampling Times	2, 6, 12, 24, 48 and 72 h.
Rotational speed	30 rev/min
% Solids by weight	40
O_2 addition/ amount	25±5 l/hour

As seen in Table 4.6, leaching tests were conducted with an initial NaCN concentration of 1500 ppm; on samples with P_{100} sizes of 150, 106, 73, 57 and 37 µm. Leaching was continued for 72 hours for all particle sizes. Within this period, the device was stopped at the end of the 2nd, 6th, 12th, 24th, 48th and 72nd hours and samples were taken from the pregnant solution to determine the gold dissolved in the solution and NaCN consumption. At the end of the monitoring periods the consumed amount of NaCN was re-added to the bottles to ensure that 1500 ppm NaCN dosage remained constant throughout the test.

This also applies to the pH of the solution; the initial pH level was set to pH 10.5±0.3 at the start of the tests. At each monitoring period it was ensured that this pH level was preserved and adjustments were made adding NaOH, when necessary. Since the ore has a refractory character; pure O_2 was supplied at a rate of 25 l/h during the tests to ensure proper chemical interaction between Au-NaCN. The results obtained at this stage on gold extraction, and cyanide consumption with respect to time are seen in Table 4.7- Table 4.11 and Figure 4.5 - Figure 4.9. As seen in Table 4.7, the gold extraction of the ore with -150 μm size was 37.5% after 72 hours. This extraction was with NaCN consumption of 5.6 kg per ton of ore. It was seen that leaching significantly decelerated and NaCN consumption decreased after the 24-hour period. In fact, when the results obtained at the end of the 48th and 72nd hours are considered, it can be said that dissolution of gold was almost completed at the end of the 48th hour (Table 4.7 and Figure 4.5). At this point, 37.3% gold extraction was obtained with a NaCN consumption of 5.5 kg per ton of ore. Here it should be emphasized that gold extraction remains low in spite of high level of NaCN consumption.

The results of the bottle roll test conducted on the ore with -106 μm size are seen in Table 4.8 and Figure 4.6. At this size gold extraction increased to 44.2% at 6.5 kg/ton NaCN consumption after 72 hours of leaching. Decreasing the particle size to -106 μm had a positive impact on gold extraction and an increase by approximately 7% was achieved as compared to the results with -150 μm ore. However, using a finer particle size increased the total consumption of NaCN. As with the size of -150 μm, it is seen that the leaching process was completed at the end of 48 hours. After 48 hours, 44.2% gold was extracted at 6.3 kg/ton NaCN consumption. Despite the achieved increase, gold extraction yield with this size is also a low level.

Table 4.7. Leaching results of -150 µm sample

P_{100} = 150 µm NaCN amount = 1500 ppm Feed Au Grade = 2.44 g/ton			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.0	0.0	0.0
2	16.4	2.4	2.4
6	23.5	1.1	3.5
12	29.4	0.8	4.3
24	34.0	0.9	5.2
48	37.3	0.3	5.5
72	37.5	0.1	5.6

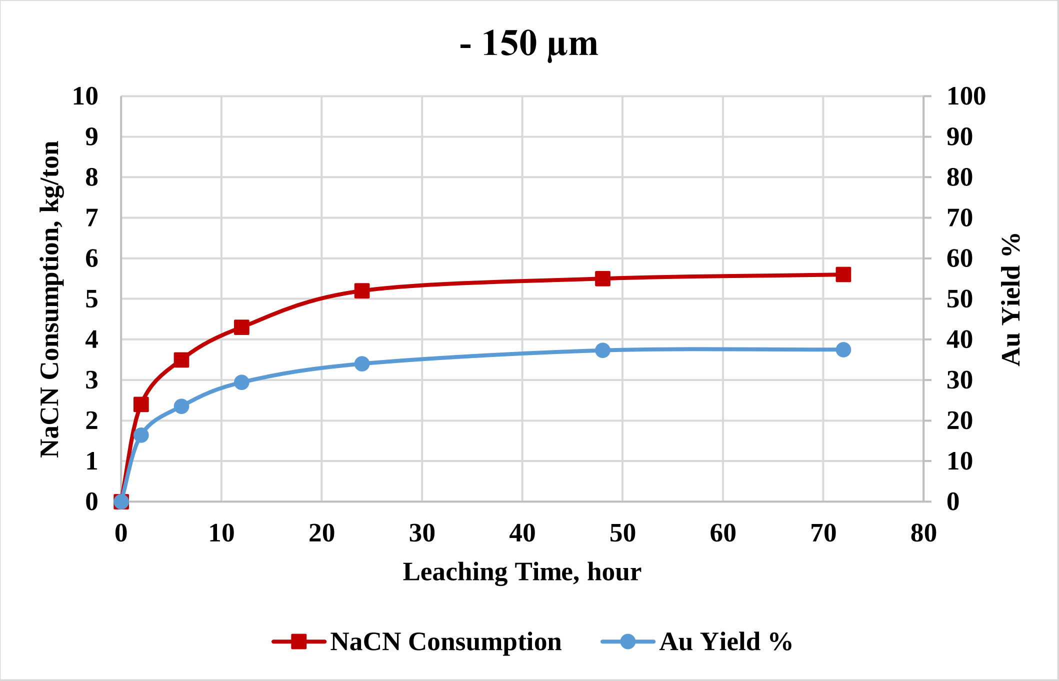

Figure 4.5. NaCN Consumption and Gold Recovery at -150 µm

Table 4.8. Leaching results of -106 μm sample

P_{100} = 106 μm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.21 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.0	0.0	0.0
2	19.0	2.8	2.8
6	27.6	1.2	4.0
12	35.1	0.9	4.9
24	40.1	0.9	5.8
48	44.2	0.5	6.3
72	44.2	0.2	6.5

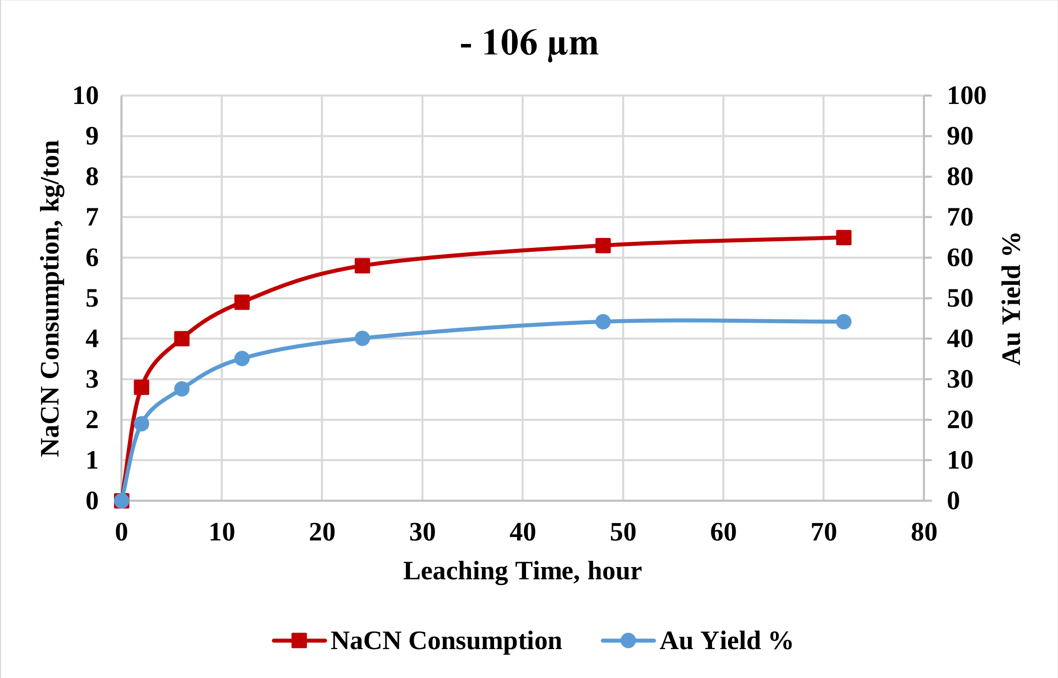

Figure 4.6. NaCN Consumption and Gold Recovery at -106 μm

The results of the bottle roll test conducted with the -74 µm ore are seen in Table 4.9 and Figure 4.7. After 72 hours of leaching gold extraction of 54.5% was obtained. At the end of this period, NaCN consumption was 7.7 kg per ton of ore. With this size, an increase by more than 10% was achieved in gold extraction compared to the results obtained with -106 µm ore. In terms of NaCN consumption, an increase of 1.2 kg per ton was recorded. In spite of the achieved increase in gold extraction, metallurgical efficiency is still low. However, it is remarkable that with reduction of the particle size, gold extraction higher than 50% was achieved in the tests for the first time. Increase in the gold extraction with increasing fineness indicates that as the particle size of the gold in gold ore decreases, the liberation level increases. With higher increased liberation, an increase in the amount of the gold contacting with the NaCN solution is also enhanced and this results in an increase in gold extraction.

The results of the bottle roll test conducted with the -53 µm ore are seen in Table 4.10 and Figure 4.8. It was seen that the highest gold extraction obtained with this size is 67%. This value was obtained with 8.1 kg/ton NaCN consumption after 48 hours. During the period between 48 and 72 hours, no increase was seen in the gold extraction. The gold extraction at this size corresponds to an increase of 12.5% compared to the extraction the -74 µm ore. However, NaCN consumption also increased, by 0.6 kg per ton of ore, and reached to 8.1 kg/ton. The increase achieved in the gold extraction yield with this size supports the fact that leaching efficiency is enhanced due to increase of the liberation as the ore was made finer.

Table 4.9. Leaching results of -74 μm sample

P_{100} = 74 μm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.42 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.0	0.0	0.0
2	23.5	3.6	3.6
6	34.6	1.4	5.0
12	43.5	1.1	6.1
24	48.9	1.0	7.1
48	53.3	0.4	7.5
72	54.5	0.2	7.7

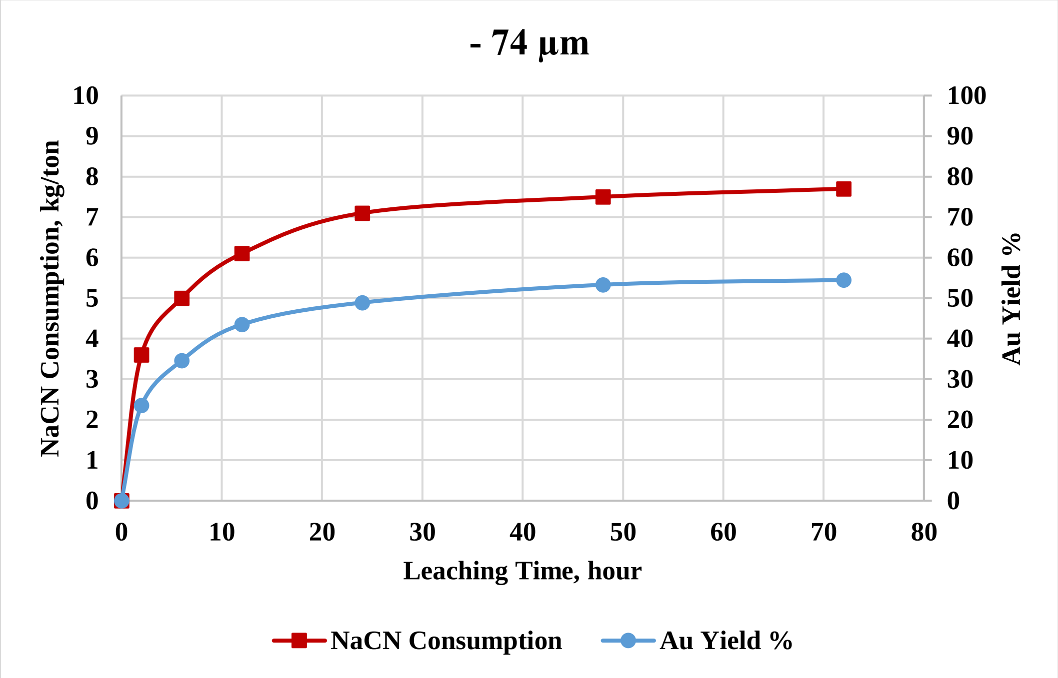

Figure 4.7. NaCN Consumption and Gold Recovery at -74 µm

Table 4.10. Leaching results of -53 µm sample

P_{100} = 53 µm

NaCN amount = 1500 ppm

Feed Au Grade = 2.61 g/ton

Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.0	0.0	0.0
2	29.9	4.0	4.0
6	45.0	1.6	5.6
12	55.5	1.2	6.8
24	61.8	0.8	7.6
48	67.0	0.5	8.1
72	67.0	0.1	8.2

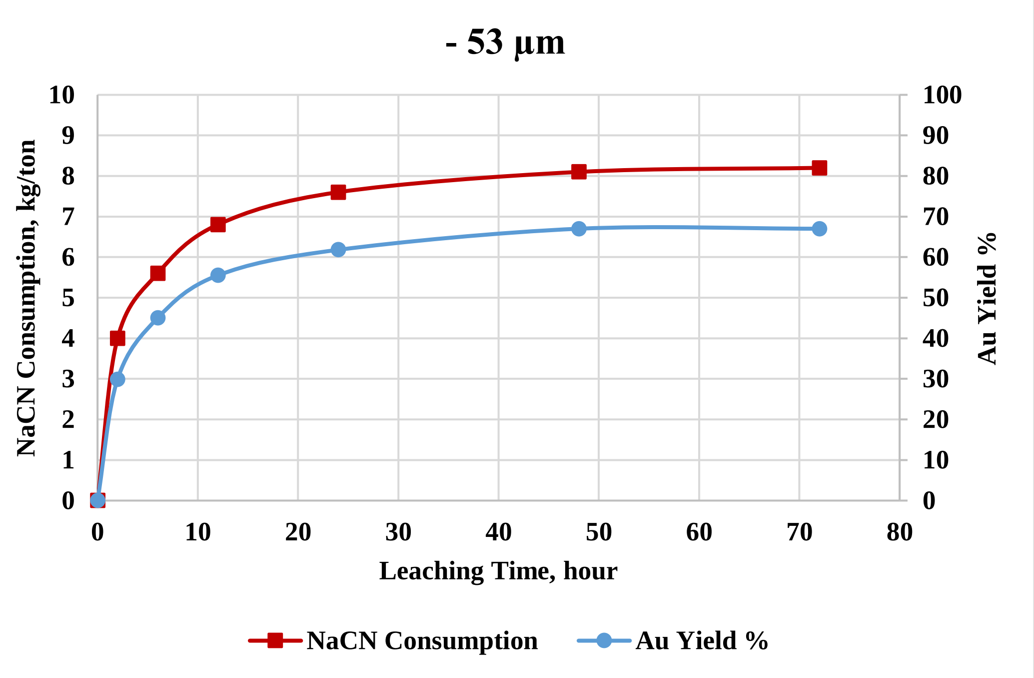

Figure 4.8. NaCN Consumption and Gold Recovery at -53 µm

The results on the bottle roll testing of the -37 µm ore are seen in Table 4.11 and Figure 4.9. With this size, leaching was completed after 48 hours. At the end of this period, 71.1% of gold extraction was obtained, which the highest metallurgical yield is obtained in the study up to this point. For leaching 8.8 kg/ton NaCN was consumed. The extracted gold is 4.1% higher. This is in fact a limited improvement as compared to the increase in gold extraction when the particle size was reduced to -53 µm from -75 µm (12.5%). In addition, it is seen that, in spite of the increase in the gold extraction, NaCN consumption was 0.7 kg/ton higher at this size compared to the NaCN consumption of -53 µm ore (Table 4.10 and Table 4.11). Consistent increase in gold extraction with decreasing particle size verifies that gold in this ore could be exposed only after fine grinding. Besides, although NaCN consumption per ton was very high, gold extraction yield was barely 70%.

In addition, as mentioned previously, high consumption of NaCN points to the presence of undesired constituents in the ore that behave as cyanicides. The rise in NaCN consumption with increasing fineness implies increased liberation of these cyanide consuming components, too. In this respect, the ore is a challenging type with apparent refractory character and inclusion of remarkable extent of cyanicides. Figure 4.10 and Figure 4.11 show 3D surface response of gold yield and NaCN consumption vs particle size; leaching time.

Table 4.11. Leaching results of -37 µm sample

P_{100} = 37 µm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.73 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.0	0.0	0.0
2	33.3	4.3	4.3
6	50.2	1.9	6.2
12	63.0	1.4	7.6
24	70.3	1.0	8.6
48	71.1	0.2	8.8
72	71.1	0.0	8.8

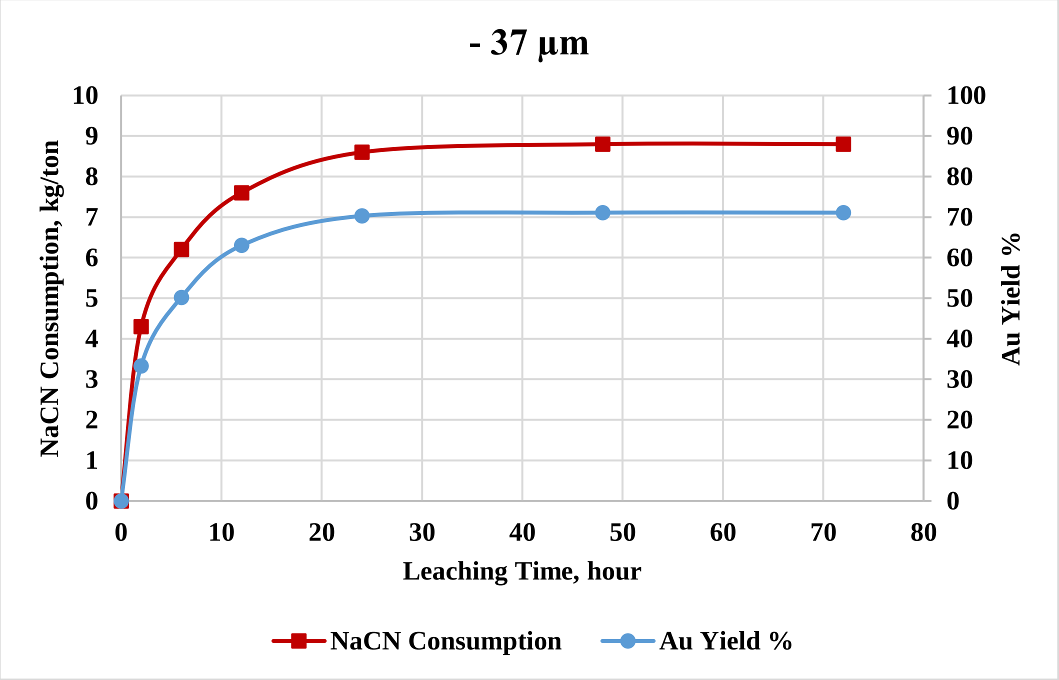

Figure 4.9. NaCN Consumption and Gold Recovery at -37 µm

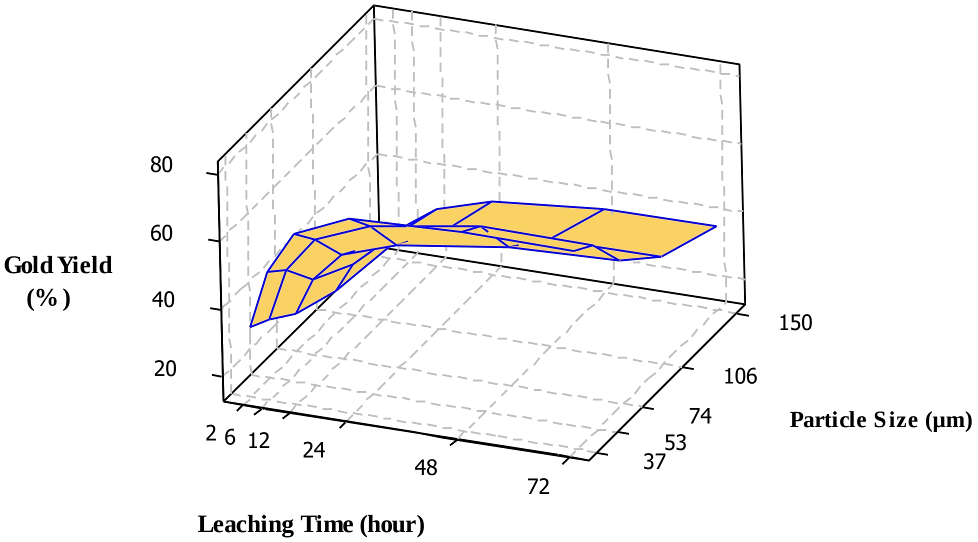

Figure 4.10. 3D Response of Gold Yield (%) vs Particle Size (µm) with respect to Leaching Time (hour)

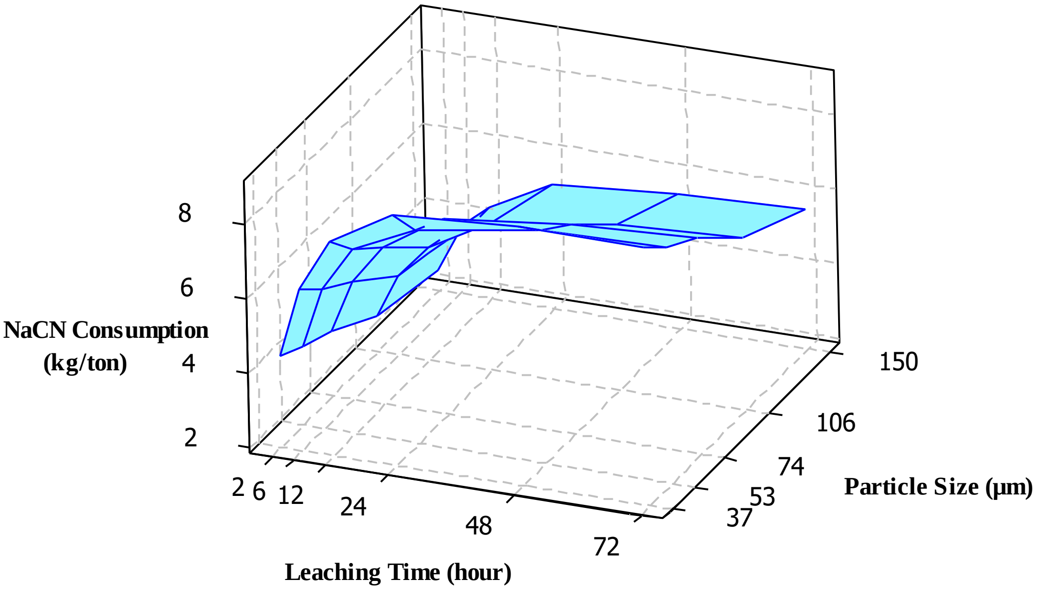

Figure 4.11. 3D Response of NaCN Consumption (kg/ton) vs Particle Size (µm) with respect to Leaching Time (hour)

4.3 Determination of Thermal Behavior of the Ore

Roasting is a thermal treatment procedure and in order to investigate the behavior of the given ore in oxidizing-thermal conditions, thermogravimetric analysis (TGA) were performed. The aim was identifying particular weight loss zones as an indication to possible structural changes in the ore with increasing temperature. The TGA profiles are presented in Figure 4.12 -Figure 4.16.

When the thermal behavior and weight loss profile of the ore in oxidizing environment was assessed, it was seen that major weight losses occurred in approximately 30-120 °C, 230-260 °C, 280-330 °C, 370-520 °C and 520-630 °C. The highest value of the weight loss of the sample at the end of 1000 °C is approximately 12% and this value changes slightly according to the particle size (Figure 4.12 -Figure 4.16). Around 1.5% weight loss occurred within the range of 30-120 °C indicated the moisture loss from the sample. Weight losses starting from 230 °C indicates presence of heat-dependent reactions, and oxidative degradation due to such reactions.

Weight losses occurred within the ranges of 370-520 °C and 520-630 °C are very significant; therefore, it can be said that significant changes and degradations occurred in the structure of the ore within these ranges. These could be attributed to the destruction of arsenic sulfides and pyrite. When it is considered that 400 °C and above temperatures are typically used for roasting of gold ores, the obtained results are significant. Around 6% total weight loss (3.5% in the first range (370-520 °C) and 2.2% in the second range (520-630 °C)) was determined. Degradation reactions leading such weight losses imply degradation of the structural components in the ore as a result of oxidation and increased temperature, i.e. roasting. This is expected to be confirmed upon leaching of the roasted ore. On the basis of the thermal profiles roasting pretreatment of the ore was performed at temperatures higher than 400 °C for different durations.

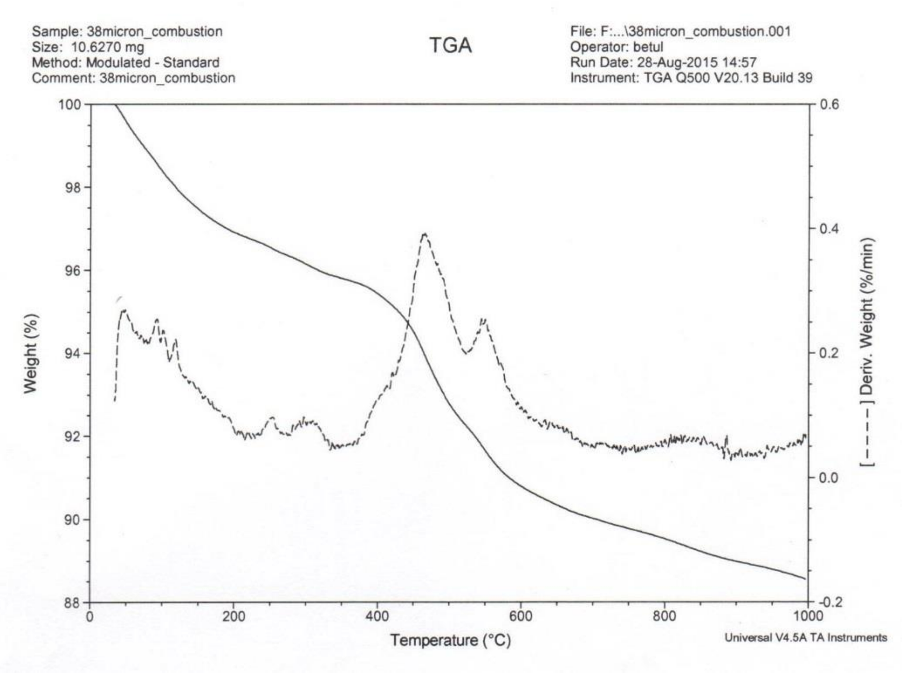

Figure 4.12. Thermal Behavior of -37 μm ore in oxidizing conditions

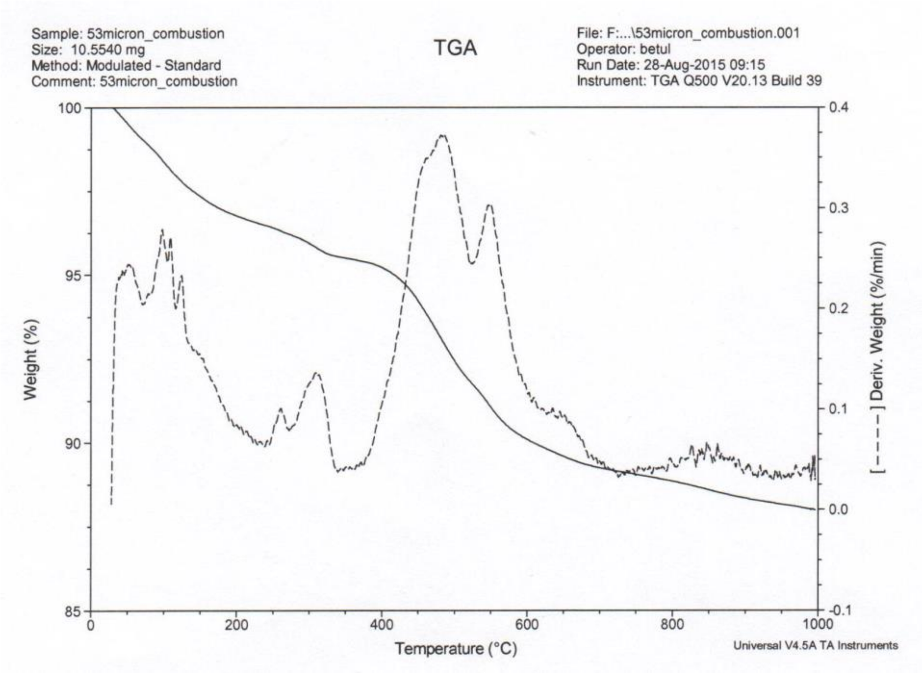

Figure 4.13. Thermal Behavior of -53 μm ore in oxidizing conditions

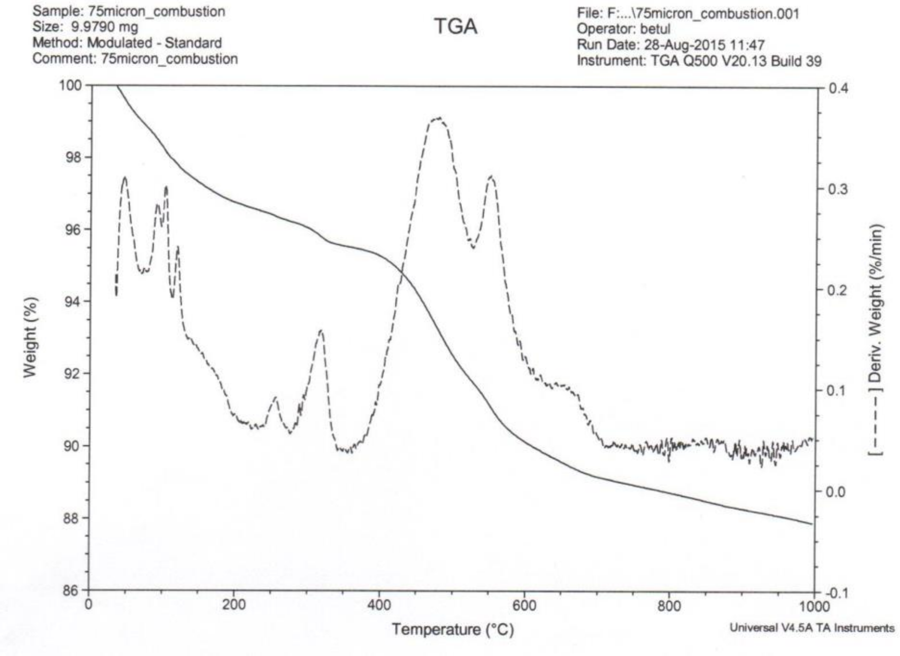

Figure 4.14. Thermal Behavior of -74 μm ore in oxidizing conditions

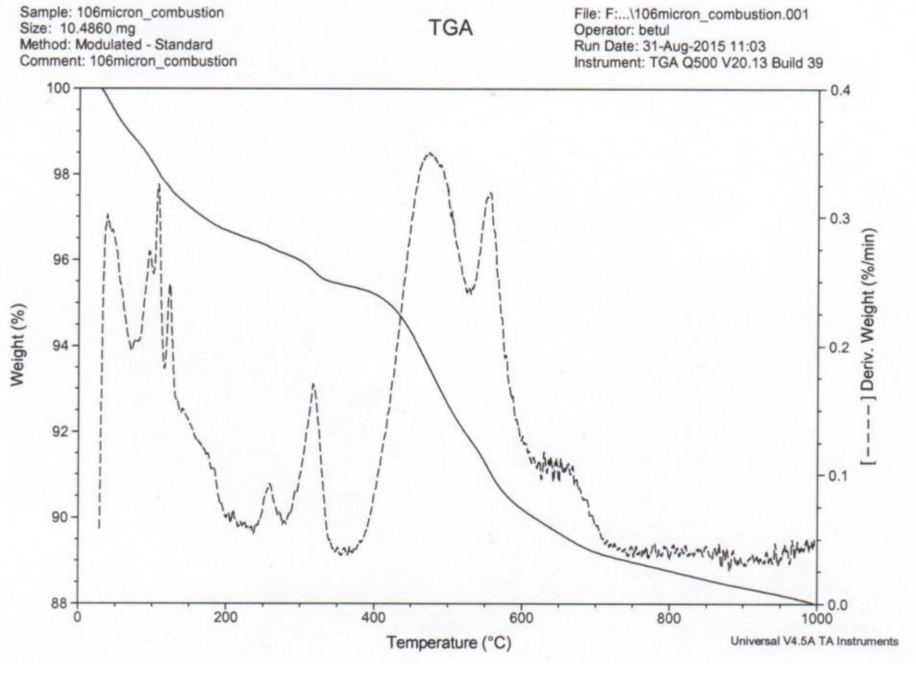

Figure 4.15. Thermal Behavior of -106 μm ore in oxidizing conditions

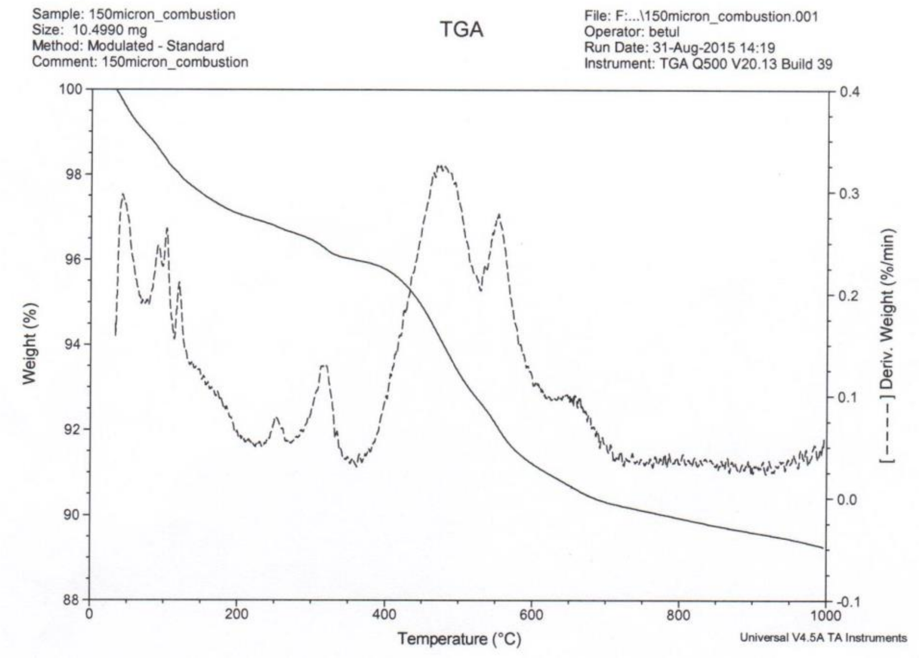

Figure 4.16. Thermal Behavior of -150 μm ore in oxidizing conditions

4.4 Impact of Roasting on Gold Extraction and NaCN Consumption

For roasting pretreatment representative ore samples were first ground to P_{100} 150 μm size, roasted at varying temperature levels and pretreatment periods and subjected to Bottle Roll Testing. Conditions of the Bottle Roll Tests applied on roasted ores are shown in Table 4.12.

Initial concentration of NaCN was set as 1500 ppm in the leaching performed after roasting, and the process was continued for 72 hours. The device was stopped at the end of the 2nd, 6th, 12th, 24th, 48th and 72nd hours and samples were taken from the pregnant solution in order to determine gold extraction yield and NaCN consumption. In addition, at the end of each period, NaCN in the amount of the consumed NaCN was added to the solution so that NaCN concentration of the solution was ensured to remain at 1500 ppm level during leaching. pH of the solution was set to pH 10.5±0.2 level at the beginning of leaching; and it was ensured to remain at the same level during the testing by measuring at the end of each period in which solution samples were taken and adding CaO when necessary. Furthermore, as it was in the previous stages, the tests were conducted by providing pure O_2 (20-25 l/hour) to the bottles during leaching.

Table 4.12. Experiment Conditions to identify relation between gold recovery and roasting

Particle Size	100% -150 µm
pH	10.5±0.3
Chemicals	For pH regulation: CaO As leaching agent: NaCN
NaCN amount (ppm)	1500 ppm
Maximum leach time	72 hr.
Pregnant Solution Control Times	2, 6, 12, 24, 48 and 72 hour
Rotational speed	30 rev/min
% Solids by weight	40
O_2 addition/amount	25±5 l/h

The results obtained at this stage on gold extraction and cyanide consumptions with time are seen in Table 4.13 -Table 4.27 and Figure 4.17 -Figure 4.25.

4.4.1 Roasting at 550 °C

Gold extraction obtained after 72 hours of leaching after 30 minutes roasting at 550 °C is 45.04% and the total NaCN consumption per ton of ore is 5.1 kg. When Table 4.13 is examined, it was seen that leaching efficiently continued for 48 hours, and then decelerated in the 48-72 hours period. After 24 hours period, a deceleration was also seen in NaCN consumption (Figure 4.17).

Table 4.13. The results of leaching after roasting at 550 °C for 30 minutes

P_{100} =150 µm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.44 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.0	0	0
2	16.80	1.9	1.9
6	24.43	1.3	3.2
12	30.53	0.8	4
24	35.11	0.6	4.6
48	43.51	0.3	4.9
72	45.04	0.2	5.1

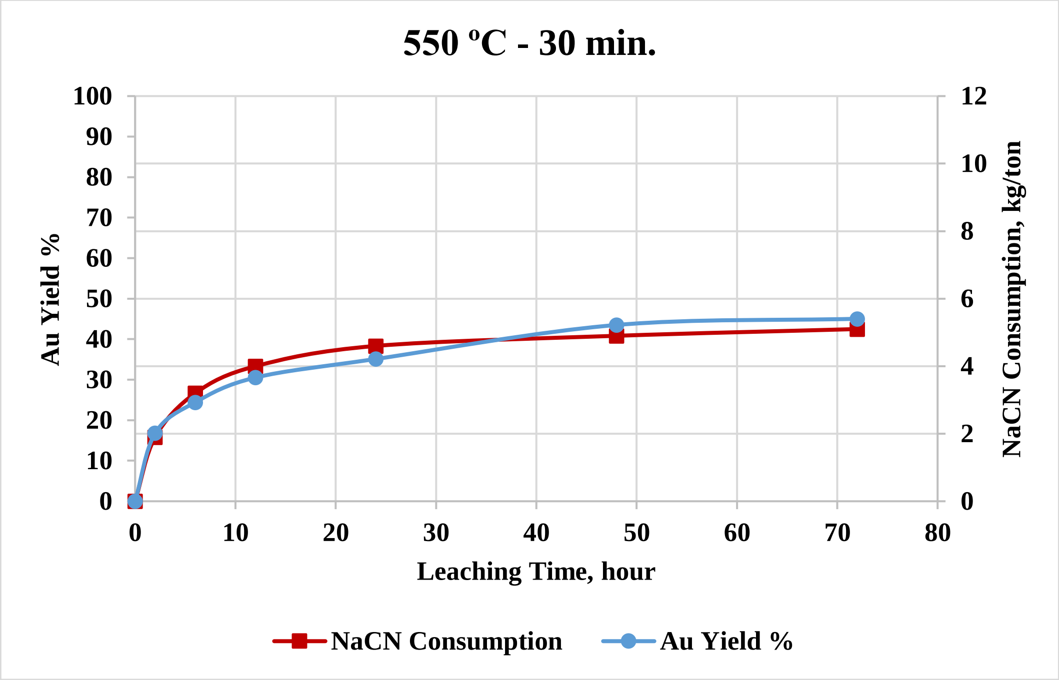

Figure 4.17. Gold Yield and NaCN consumption after roasting at 550 °C for 30 minutes

The results of leaching on 60-minute roasted ore at 550 °C are seen in Table 4.14. The time-dependent leaching profile of the sample is seen in Figure 4.18. Leaching decelerated after 24 hours and NaCN consumption increased slightly. Gold extraction increased to 50% and a total of 4.6 kg/ton NaCN was consumed.

When the results are examined, it is seen that the gold extraction value is approximately 5% higher compared to the roasting at 550 °C for 30 minutes and this increase was achieved with a decrease of 0.5 kg in total NaCN consumption per ton of ore. Therefore, extending the pretreatment duration from 30 to 60 minutes increased efficiency of the roasting process. This situation corresponds to a decrease in refractoriness of the ore and an increase in its liability to leaching.

Table 4.14. The results of leaching after roasting at 550 °C for 60 minutes

P_{100} =150 *µm* *NaCN amount = 1500 ppm* *Feed Au Grade = 2.48 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.00	0	0
2	17.34	1.6	1.6
6	28.23	1.2	2.8
12	36.29	0.8	3.6
24	43.55	0.5	4.1
48	47.98	0.3	4.4
72	50.00	0.2	4.6

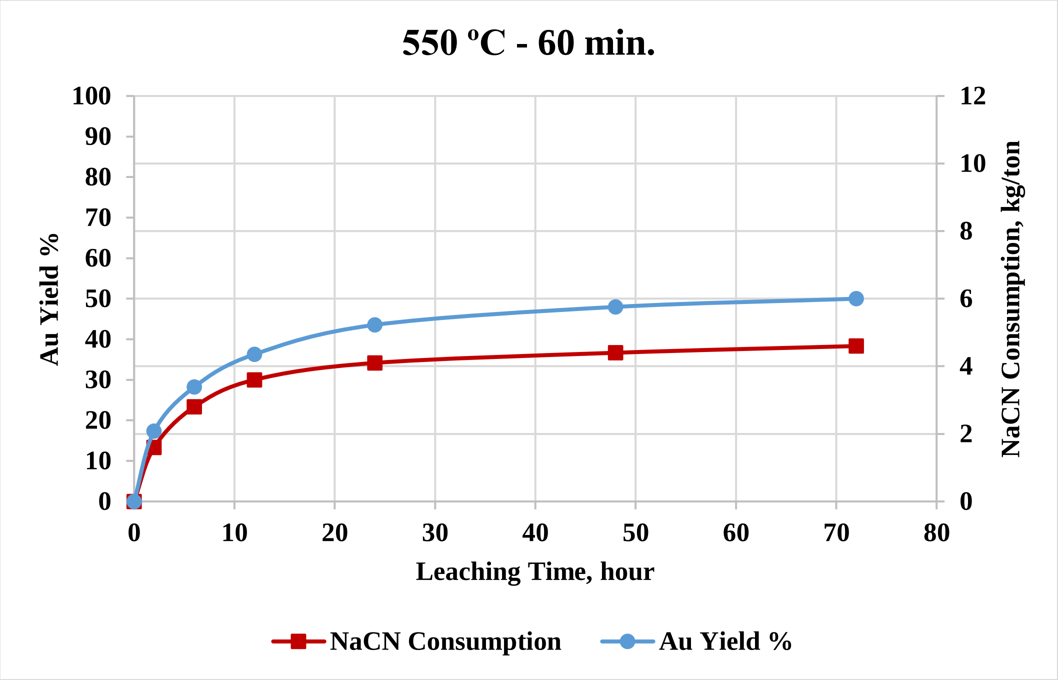

Figure 4.18. Gold Yield and NaCN consumption after roasting at 550 °C for 60 minutes

The results of leaching 90 minutes roasted ore at 550 °C is seen in Table 4.15. The time-dependent leaching profile of the sample is seen in Figure 4.19. After 90 minutes of roasting, contrary to pretreatment at 30 and 60 minutes, leaching completely ended at the end of 48 hours and this situation is clearly seen from gold extraction and NaCN consumption profiles (Table 4.15, Figure 4.19). While gold extraction increased to 53% after 48 hours, a total of 4.4 kg NaCN was consumed per ton of ore.

When 90 minute and 60 minutes of roasting were compared, an increase by 3% in gold extraction and a decrease of 0.2 kg/ton in total NaCN consumption was achieved (Figure 4.18 and Figure 4.19). In other words, the improvement achieved by increasing the pretreatment time from 60 to 90 minutes is relatively limited compared to the increase in leaching performance when the roasting period was extended from 30 to 60 minutes.

Table 4.15. The results of leaching after roasting at 550 °C for 90 minutes

<table>
<tr><td colspan="4">P_{100} =150 <i>μm</i>
<i>NaCN amount = 1500 ppm</i>
<i>Feed Au Grade = 2.52 g/ton</i></td></tr>
<tr><th>Leaching time (Hour)</th><th>Gold Yield (%)</th><th>NaCN Consumption (kg/ton)</th><th>Cumulative NaCN Consumption (kg/ton)</th></tr>
<tr><td>0</td><td>0.00</td><td>0</td><td>0</td></tr>
<tr><td>2</td><td>17.86</td><td>1.6</td><td>1.6</td></tr>
<tr><td>6</td><td>29.76</td><td>1.1</td><td>2.7</td></tr>
<tr><td>12</td><td>36.51</td><td>0.8</td><td>3.5</td></tr>
<tr><td>24</td><td>48.41</td><td>0.6</td><td>4.1</td></tr>
<tr><td>48</td><td>53.02</td><td>0.3</td><td>4.4</td></tr>
<tr><td>72</td><td>52.98</td><td>0.1</td><td>4.5</td></tr>
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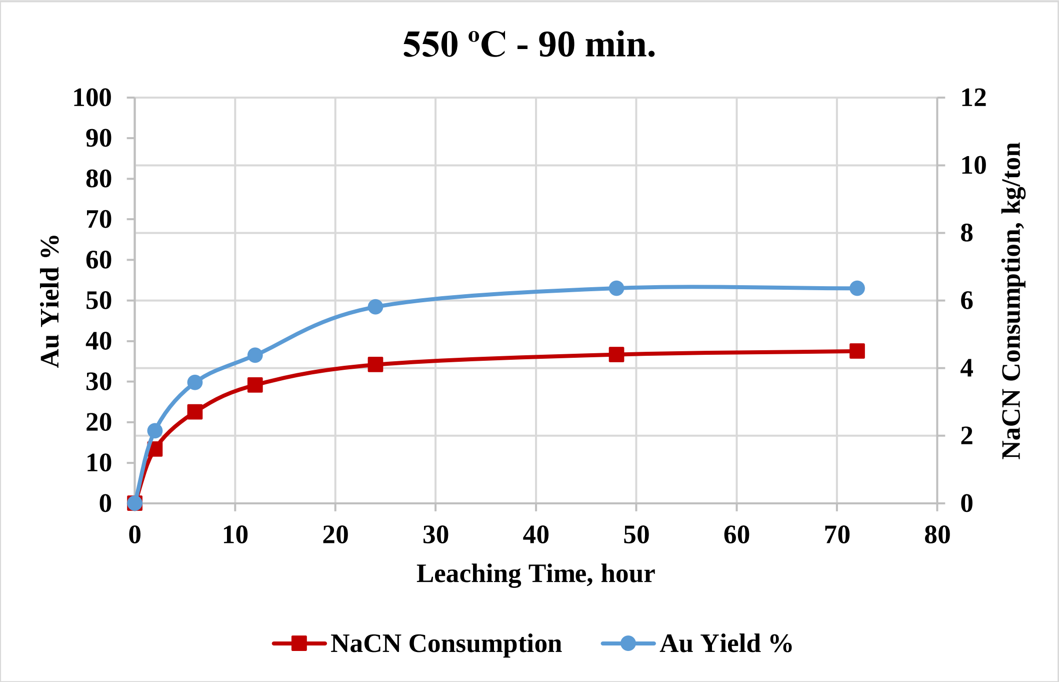

Figure 4.19. Gold Yield and NaCN consumption after roasting at 550 °C for 90 minutes

The results of leaching on roasted ore at 550 °C confirms high refractory character of the ore. An overall evaluation of the results (Table 4.16), shows that gold extraction significantly increased (15.5%) in comparison with the gold extraction achieved with the leaching of the ROM ore. NaCN amount consumed per ton of ore also decreased by 1.2 kg (Table 4.17). On the basis of these results it could be deduced that structures of the phases containing gold in the ore degrade/change, and after these changes, the liability of the ore to gold extraction increases. Similarly, the decrease in cyanide consumption points to degradation/removal of the cyanicides through oxidative pretreatment. However, gold extraction of 53% after 90 minutes of roasting at 550 °C is still lower than the 71.1% Au extraction obtained with the ROM ore with P_{100} size of 37 µm. Also, despite notable decrease, NaCN consumption of 4.4 kg per ton of ore is still a high level.

Table 4.16. Comparison of Gold Extraction after roasting at 550 °C and without roasting

	Leaching without Roasting		**Leaching after Roasting at 550 ˚C**		
			Roasting Time		
Leaching Time	**P_{100} = 150 µm**	**P_{100} = 37 µm**	**30 min**	**60 min**	**90 min**
			P_{100} = 150 µm		
(Hour)	**Gold Yield, %**		**Gold Yield, %**		
0	0	0	0	0	0
2	16.4	33.3	16.8	17.34	17.86
6	23.5	50.2	24.43	28.23	29.76
12	29.4	63	30.53	36.29	36.51
24	34	70.3	35.11	43.55	48.41
48	37.3	**71.1**	43.51	47.98	**53.02**
72	**37.5**	71.1	**45.04**	**50**	52.98

Table 4.17. Comparison of NaCN consumption after roasting at 550 °C and without roasting

	Leaching without Roasting		**Leaching after Roasting at 550 °C**		
			Roasting Time		
Leaching Time	**P_{100} = 150 µm**	**P_{100} = 37 µm**	**30 min**	**60 min**	**90 min**
			P_{100} = 150 µm		
(Hour)	**Cumulative NaCN Consumption**		**Cumulative NaCN Consumption**		
0	0	0	0	0	0
2	2.4	4.3	1.9	1.6	1.6
6	3.5	6.2	3.2	2.8	2.7
12	4.3	7.6	4	3.6	3.5
24	5.2	8.6	4.6	4.1	4.1
48	5.5	**8.8**	4.9	4.4	**4.4**
72	**5.6**	8.8	**5.1**	**4.6**	4.5

4.4.2 Roasting at 650 °C

The results of leaching after 30 minutes roasting at 650 °C are seen in Figure 4.20 and Table 4.18. As a result of the test, gold extraction after 72 hours is 65.16% and NaCN consumption per ton of ore is 4.3 kg. When the results and the leaching profile of the ore (Figure 4.20) are examined, it is seen that leaching almost ended after 48-hours. At this point, gold extraction was 64.75% and total NaCN consumption was 4.2 kg/ton ore.

When compared to leaching without roasting under the same conditions, Au extraction after 30 minutes roasting at 650 °C (Table 4.7, Figure 4.5) increased from 37.5% to 64.75% and NaCN consumption per ton of ore decreased from 5.6 to 4.2 kg. When the roasting temperature levels were assessed, roasting at 650 °C provides a decrease in NaCN consumption and an increase by approximately 20% in gold extraction, compared to roasting at 550 °C, (Table 4.13 and Table 4.18).

Table 4.18. The results of leaching after roasting at 650 °C for 30 minutes

P_{100} =150 µm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.44 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.00	0	0
2	18.85	1.4	1.4
6	31.56	1.1	2.5
12	38.00	0.8	3.3
24	54.92	0.6	3.9
48	64.75	0.3	4.2
72	65.16	0.1	4.3

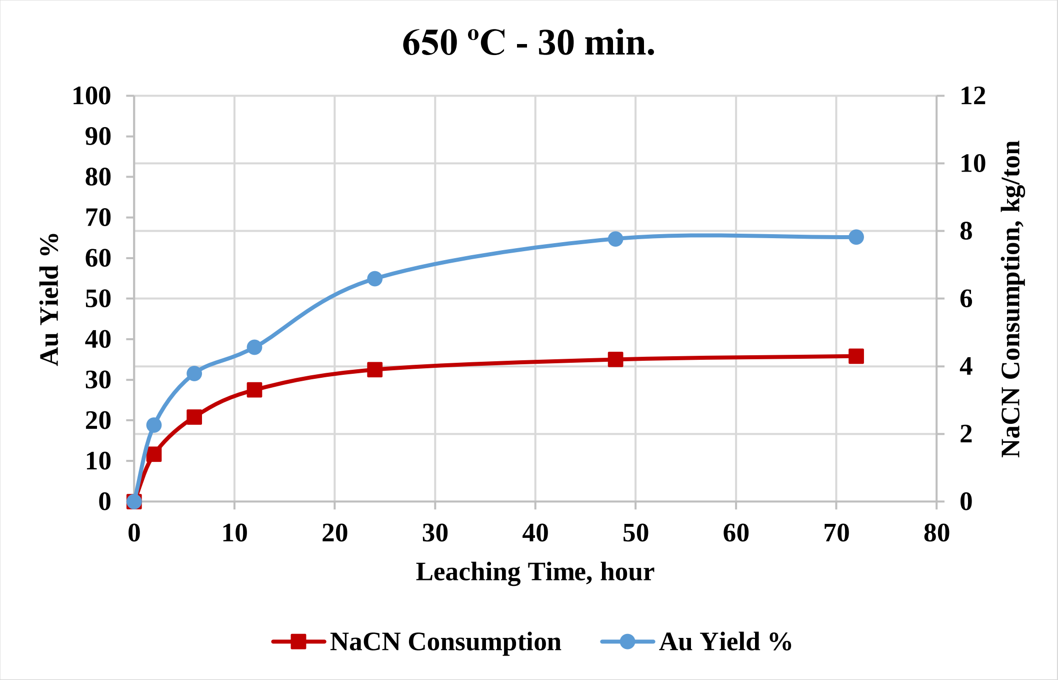

Figure 4.20. Gold Yield and NaCN consumption after roasting at 650 °C for 30 minutes

The results of leaching of 60 minutes roasted ore at 650 °C are seen in Table 4.19 and Figure 4.21. Leaching decelerates after 24 hours ends after 48 hours. Gold extraction after 48 hours reached to 68.42% at 3.9 kg NaCN consumption per ton of ore (Table 4.19, Figure 4.21). In other words, for the first time in the study, NaCN consumption could be reduced below 4 kg/ton ore along with a relatively acceptable Au extraction.

When the results of roasting for 30 and 60 minutes at 650 °C were compared, it was seen that gold extraction increased by >3% after 60 minutes of roasting. Total NaCN consumption per ton of ore showed a limited decrease to 3.9 kg from 4.2 kg (Table 4.18 and Table 4.19).

Increasing the roasting duration to 60 minutes from 30 minutes improves the effectiveness of roasting. In addition, when the roasting for 60 minutes at 550 °C and 650 °C are considered (Table 4.14 and Table 4.19), the positive impact of increasing the roasting temperature is clearly seen: Gold extraction increases to 68.42% from 50% and NaCN consumption decreases to 3.9 kg from 4.6 kg per ton of ore.

Table 4.19. The results of leaching after roasting at 650 °C for 60 minutes

P_{100} =150 μm
NaCN amount = 1500 ppm
Feed Au Grade = 2.40 g/ton

Leaching time (Hour)	Gold Yield (%)	NaCN Consumption (kg/ton)	Cumulative NaCN Consumption (kg/ton)
0	0.00	0	0
2	22.50	1.3	1.3
6	32.92	1.1	2.4
12	50.83	0.7	3.1
24	64.58	0.5	3.6
48	68.42	0.3	3.9
72	68.00	0.1	4

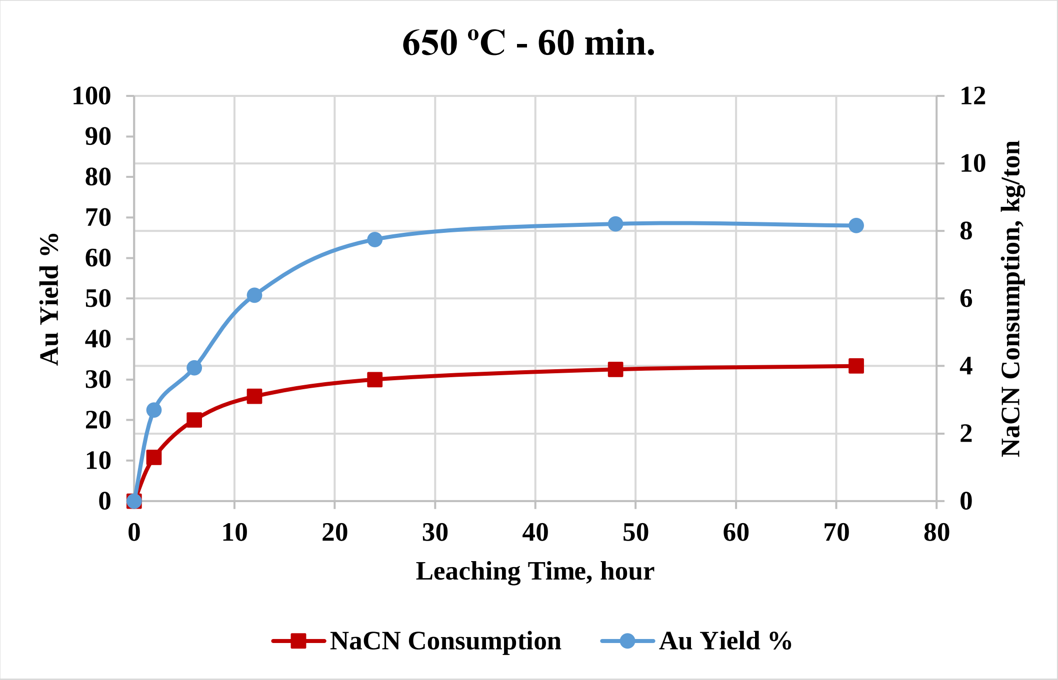

Figure 4.21. Gold Yield and NaCN consumption after roasting at 650 °C for 60 minutes

The results of leaching 90 minutes roasted ore at 650 °C are seen in Table 4.20 and the time-dependent leaching profile of the ore is presented in Figure 4.22. After 90 minutes of roasting, leaching effectively continues for 24 hours, decelerates after 24 hours and completely ends at the end of 48 hours, similar to the leaching behavior after roasting at 550 °C for 90 minutes (Figure 4.22). Gold extraction after 48 hours was 68.40% with 3.8 kg NaCN consumption per ton of ore (Table 4.20).

When roasting for 60 and 90 minutes were compared, it was seen that gold extraction remained at the same level (68.40%) and NaCN consumption per ton of ore was almost equivalent (3.8 kg). In other words, increasing the duration of the pretreatment from 60 to 90 minutes at 650 °C does not provide any remarkable improvement in leaching performance. This indicates that 60 minutes of roasting was sufficient for achieving the highest possible leaching after roasting at 650 °C.

Table 4.20. The results of leaching after roasting at 650 °C for 90 minutes

P_{100} =150 µm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.50 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.00	0	0
2	21.60	1.2	1.2
6	36.00	1	2.2
12	46.00	0.8	3
24	66.00	0.5	3.5
48	68.40	0.3	3.8
72	68.08	0.1	3.9

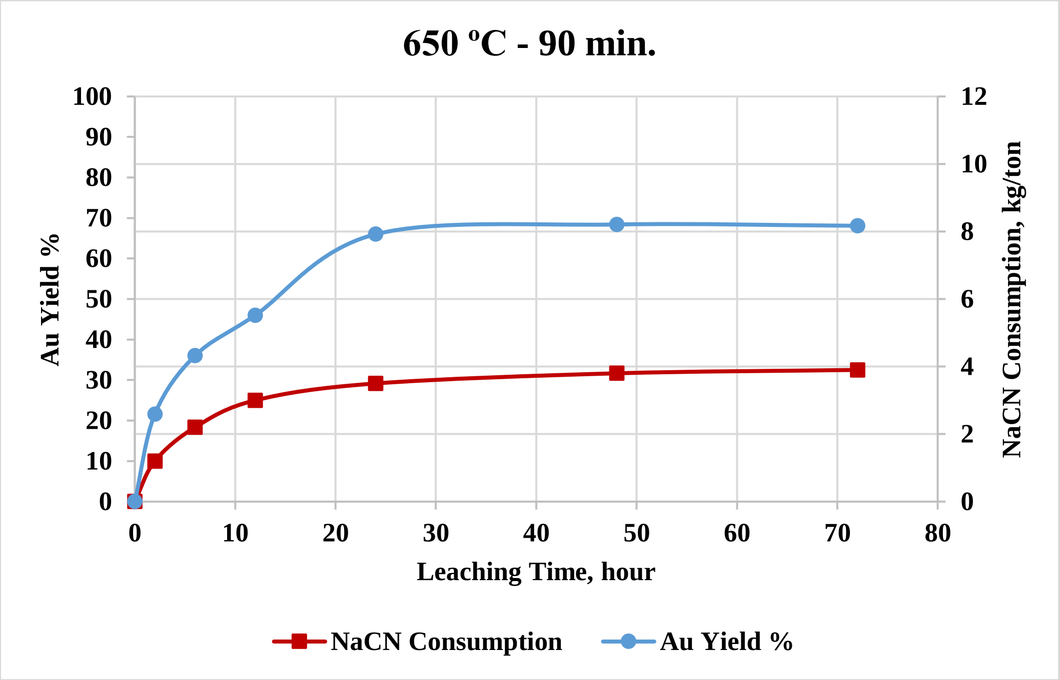

Figure 4.22. Gold Yield and NaCN consumption after roasting at 650 °C for 90 minutes

The results obtained from leaching applied after roasting at 650 °C are collectively summarized in Table 4.21. Roasting at 650 °C provides a significant improvement in terms of both gold extraction and NaCN consumption compared to roasting at 550 °C. This situation indicates that 550 °C temperature level was not high enough to reduce refractoriness of the ore. In addition, 60 minutes of roasting increases gold extraction yield and causes a decrease in NaCN consumption, compared to 30 minutes. The leaching tests performed after 60 and 90 minutes of roasting provide quite similar results.

On the basis of these findings, it could be stated that 30 minutes of roasting at 650 °C was not sufficient and 90 minutes of roasting does not provide a significant advantage. Hence, 60 minutes of roasting comes to the forefront as the ideal roasting duration.

After 60 minutes of roasting at 650 °C, an increase by 30.92% in gold extraction (Table 4.21) and a decrease of 2.7 kg in total NaCN consumption per ton of ore (Table 4.22) could be achieved, compared to the leaching of the ROM ore with P_{100} size of 150 µm. Also, after roasting at 650 °C, gold extraction proximate to the highest extraction level obtained without roasting (P_{100} 37 µm, 71.1% Au extraction) was achieved at a quite coarser size (150 µm) with remarkably lower NaCN consumption (Table 4.21).

Table 4.21. Comparison of Gold Extraction after roasting at 650 °C and without roasting

	Leaching without Roasting		**Leaching after Roasting at 650 ˚C**		
			Roasting Time		
Leaching Time	**P_{100} = 150 µm**	**P_{100} = 37 µm**	**30 min**	**60 min**	**90 min**
			P_{100} = 150 µm		
(Hour)	**Gold Yield, %**		**Gold Yield, %**		
0	0	0	0	0	0
2	16.4	33.3	18.85	22.5	21.6
6	23.5	50.2	31.56	32.92	36
12	29.4	63	38	50.83	46
24	34	70.3	54.92	64.58	66
48	37.3	**71.1**	64.75	68.42	**68.4**
72	**37.5**	71.1	**65.16**	**68**	68.08

Table 4.22. Comparison of NaCN consumption after roasting at 650 °C and without roasting

Leaching Time	Leaching without Roasting		Leaching after Roasting at 650 °C		
			Roasting Time		
	P_{100} = 150 µm	P_{100} = 37 µm	30 min	60 min	90 min
			P_{100} = 150 µm		
(Hour)	Cumulative NaCN Consumption		Cumulative NaCN Consumption		
0	0	0	0	0	0
2	2.4	4.3	1.4	1.3	1.2
6	3.5	6.2	2.5	2.4	2.2
12	4.3	7.6	3.3	3.1	3
24	5.2	8.6	3.9	3.6	3.5
48	5.5	**8.8**	4.2	3.9	**3.8**
72	**5.6**	8.8	**4.3**	**4**	3.9

4.4.3 Roasting at 750 °C

The results of leaching of 30 minutes roasted ore at 750 °C are given in Table 4.23 and Figure 4.23. Leaching effectively continues for 48 hours and 65.8% gold extraction was obtained at 4.1 kg/ton of ore NaCN consumption after 48 hours. Roasting at 750 °C also led to a decrease in NaCN consumption.

Although the gold extraction obtained for 30 minutes of roasting at 750 °C (65.48%) is much higher than the gold extraction after roasting at 550 °C (45.04%, Table 4.13), it corresponds to a limited improvement as compared to the results of roasting at 650 °C (64.75%, Table 4.18). Similar situation is applicable for NaCN consumption per ton of ore. Although NaCN consumption is almost identical after roasting at 650 °C and 750 °C (4.2 and 4.1 kg/ton ore, respectively), it is approximately 1 kg lower per ton of ore compared to NaCN consumption after roasting at 550 °C.

Table 4.23. The results of leaching after roasting at 750 °C for 30 minutes

P_{100} =150 µm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.52 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.00	0.0	0.0
2	20.24	1.4	1.4
6	30.16	1.0	2.4
12	46.03	0.8	3.2
24	55.56	0.6	3.8
48	65.48	0.3	4.1
72	64.68	0.0	4.1

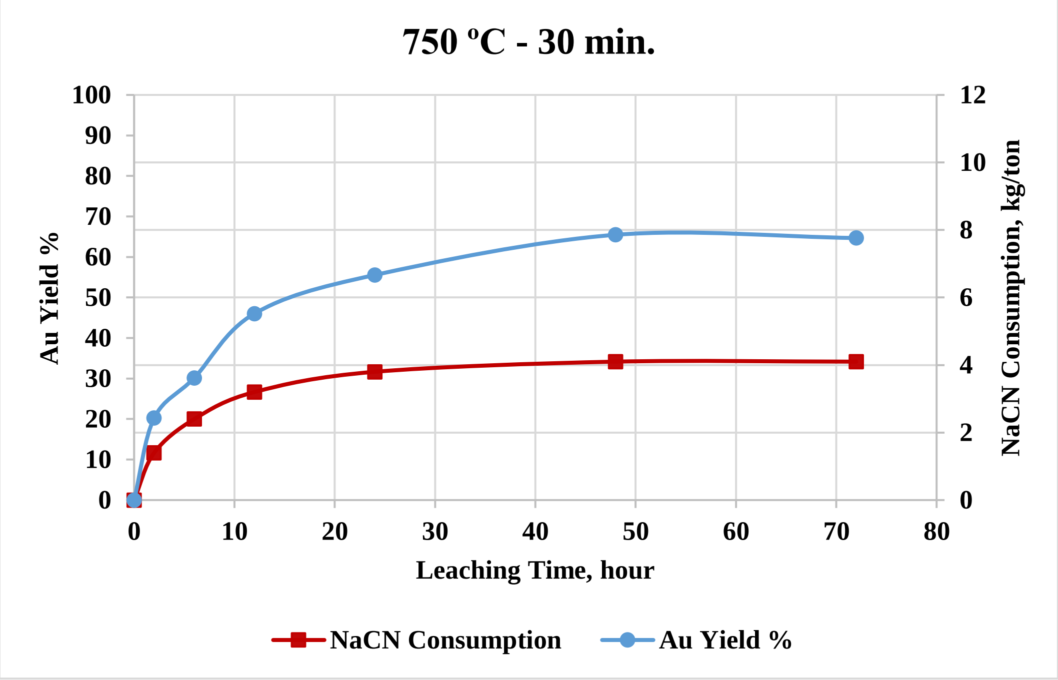

Figure 4.23. Gold Yield and NaCN consumption after roasting at 750 °C for 30 minutes

The results of leaching of 60 minutes roasted ore at 750 °C are seen in Table 4.24 and Figure 4.24. Leaching significantly decelerates after 24 hours and completely ends at the end of 48 hours. Gold extraction at the end of 48 hours of leaching reaches to 69.11% with 3.7 kg NaCN consumption per ton of ore. This value is very close to the 70% level, which is the highest gold extraction obtained without pretreatment of the ore, and NaCN consumption is quite lower, <4 kg/ton ore.

Similar to the results obtained after 30 minutes of roasting; although gold extraction after 60 minutes of roasting at 750 °C is significantly higher compared to roasting at 550 °C (>19% increase, Table 4.14 and Table 4.24), this increase is very limited compared to the results after roasting at 650 °C (<1%, Table 4.19 and Table 4.24). A similar amenability is valid for NaCN consumption levels.

Table 4.24. The results of leaching after roasting at 750 °C for 60 minutes

P_{100} =150 μm *NaCN amount = 1500 ppm* *Feed Au Grade = 2.46 g/ton*			
Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.00	0.0	0.0
2	21.95	1.2	1.2
6	35.37	1.1	2.3
12	49.59	0.7	3.0
24	65.45	0.4	3.4
48	69.11	0.3	3.7
72	69.23	0.1	3.8

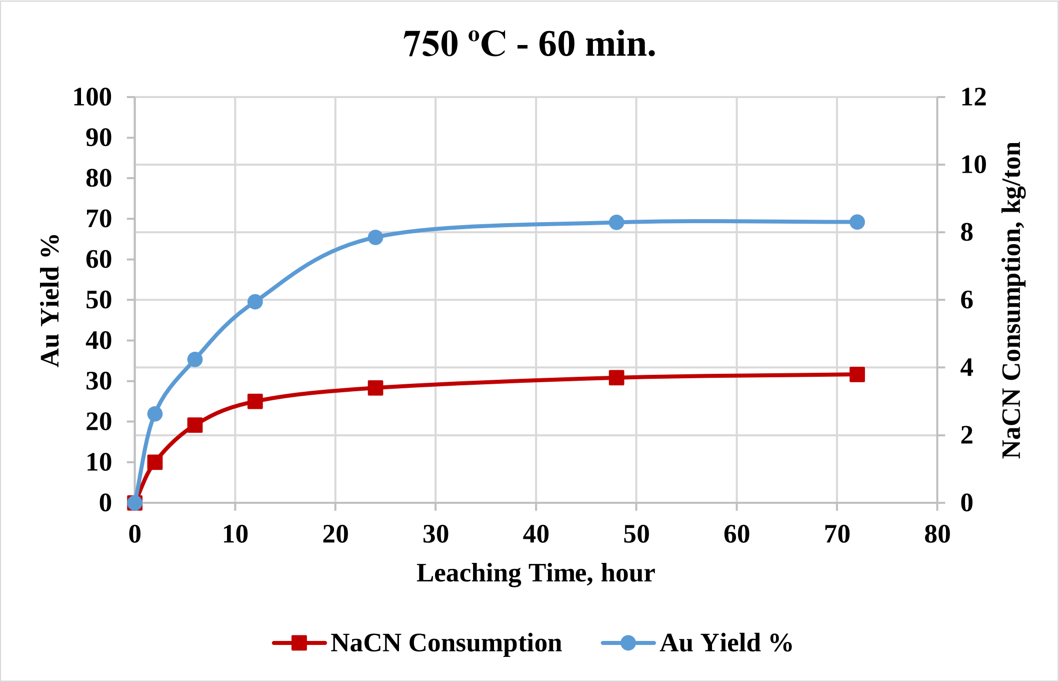

Figure 4.24. Gold Yield and NaCN consumption after roasting at 750 °C for 60 minutes

The results of leaching of 90 minutes roasted ore at 750 °C are seen in Table 4.25 and the time-dependent leaching profile of the sample is seen in Figure 4.25. Leaching effectively continues for 24 hours and significantly decelerates after 24 hours. At the end of 48 hours, leaching is completed and gold extraction of 69.84% is obtained with 3.7 kg NaCN consumption per ton of ore (Table 4.25).

When 60 and 90 minutes roasting are compared, it was seen that gold extraction increased very slightly (<1%) and NaCN consumption per ton of ore was the same (3.7 kg) (Table 4.24 and Table 4.25). In other words, increasing the roasting duration from 60 to 90 minutes at 750 °C could not yield an increase in the effectiveness of the pretreatment.

Table 4.25. The results of leaching after roasting at 750 °C for 90 minutes

P_{100} =150 μm

NaCN amount = 1500 ppm

Feed Au Grade = 2.52 gr/ton

Leaching time (Hour)	**Gold Yield (%)**	**NaCN Consumption (kg/ton)**	**Cumulative NaCN Consumption (kg/ton)**
0	0.00	0.0	0.0
2	22.22	1.1	1.1
6	36.11	1.0	2.1
12	50.79	0.7	2.8
24	66.27	0.6	3.4
48	69.84	0.3	3.7
72	69.44	0.0	3.7

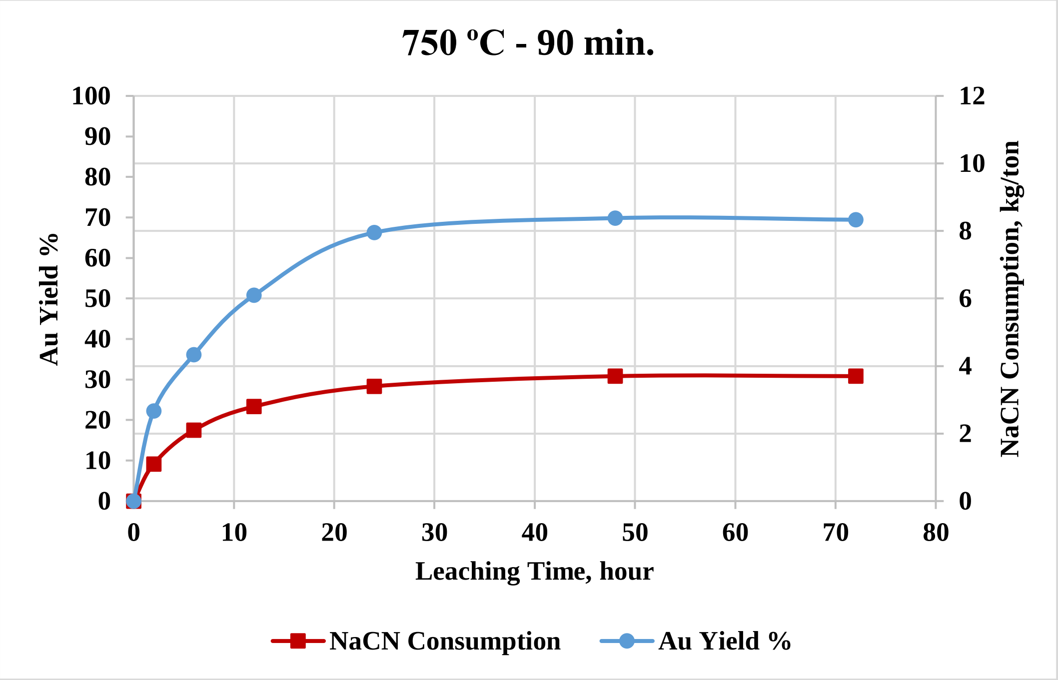

Figure 4.25. Gold Yield and NaCN consumption after roasting at 750 °C for 90 minutes

Gold extraction after roasting at 750 °C for different durations are summarized in Table 4.26 and total NaCN consumption values are presented in Table 4.27. For roasting at 750 °C, 60 minutes of application increases gold extraction and provides some decrease in NaCN consumption compared to 30 minutes of roasting. Gold extraction and NaCN consumptions after 60 and 90 minutes of roasting were almost identical, and it was understood that 30 minutes of roasting was not sufficient but 60 minutes of roasting is adequate.

Table 4.26. Comparison of of Gold Extraction after roasting at 750 °C and without roasting

	Leaching without Roasting		**Leaching after Roasting at 750 °C**		
			Roasting Time		
Leaching Time	**P_{100} = 150 µm**	**P_{100} = 37 µm**	**30 min**	**60 min**	**90 min**
			P_{100} = 150 µm		
(Hour)	**Gold Yield, %**		**Gold Yield, %**		
0	0	0	0	0	0
2	16.4	33.3	20.24	21.95	22.22
6	23.5	50.2	30.16	35.37	36.11
12	29.4	63	46.03	49.59	50.79
24	34	70.3	55.56	65.45	66.27
48	37.3	**71.1**	65.48	69.11	**69.84**
72	**37.5**	71.1	**64.68**	**69.23**	69.44

Table 4.27. Comparison of NaCN consumption after roasting at 750 °C and without roasting

	Leaching without Roasting		Leaching after Roasting at 750		
			Roasting Time		
Leaching Time	$P_{100} = 150$ µm	$P_{100} = 37$ µm	30 min	60 min	90 min
			$P_{100} = 150$ µm		
(Hour)	Cumulative NaCN Consumption		Cumulative NaCN Consumption		
0	0	0	0	0	0
2	2.4	4.3	1.4	1.2	1.1
6	3.5	6.2	2.4	2.3	2.1
12	4.3	7.6	3.2	3	2.8
24	5.2	8.6	3.8	3.4	3.4
48	5.5	**8.8**	4.1	3.7	**3.7**
72	**5.6**	8.8	**4.1**	**3.8**	3.7

When roasting temperature levels were comparatively assessed, it was seen that as the roasting temperature was increased from 550 °C to 650 °C and then to 750 °C, gold extraction increased (Figure 4.26) and NaCN consumptions decreased for all roasting periods (Figure 4.27). This situation indicates that refractory character of the ore decreased along with reductions in the adverse influence of cyanicides on NaCN consumption over the leaching process. However, here attention should be paid to the amount of the changes incurred in gold extraction and NaCN consumptions with roasting temperatures: When roasting temperature was increased from 550 °C to 650 °C, gold extraction increased by ≈15-20% and a decrease of 0.6-0.9 kg per ton of ore NaCN consumption was achieved. On the contrary; improvement in gold extraction was quite limited (≈0.7-1.4%) when the roasting temperature was further increased from 650 °C to 750 °C, and no significant reduction in NaCN consumption could be obtained (Figure 4.26 and Figure 4.27). In other words, the highest possible metallurgical improvement was almost achieved after roasting at 650 °C. When a similar assessment is made in terms of roasting durations, changes in gold extraction and NaCN consumption were very limited when roasting periods were extended from 60 to 90 minutes particularly for roasting at 650 and 750 °C. Therefore, 60 minutes of roasting at 650 °C corresponds to the optimum pretreatment conditions for this ore (Figure 4.28 and Figure 4.29).

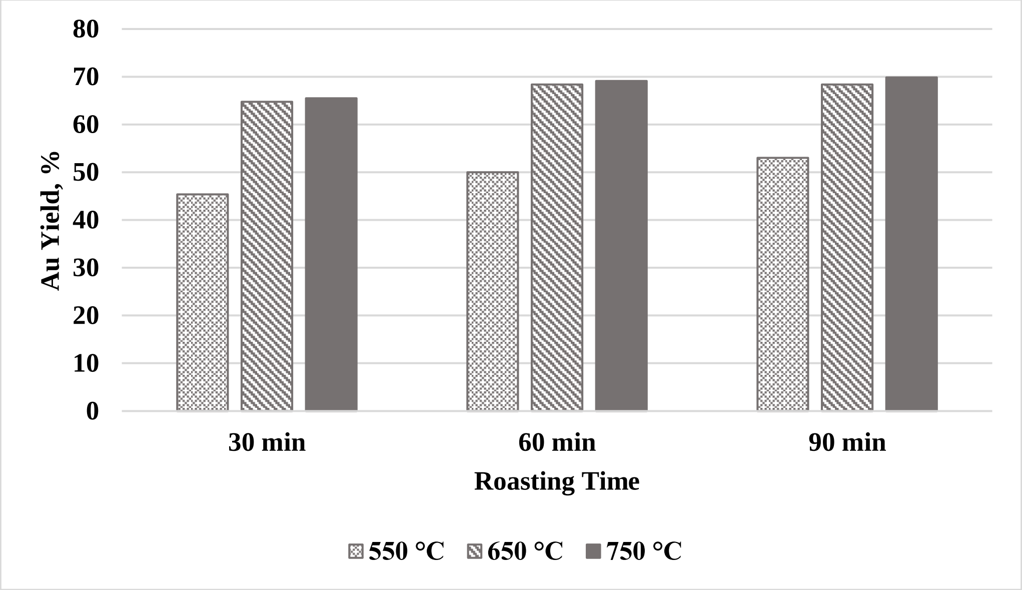

Figure 4.26. Variation of Gold Extraction with respect to Roasting time at Different Roasting Temperatures

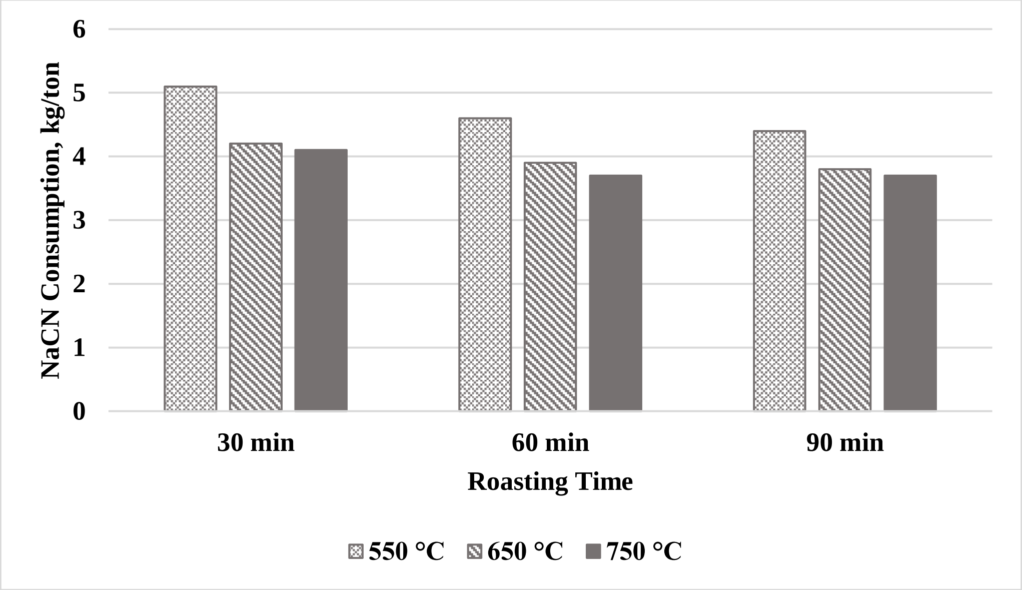

Figure 4.27. Variation of NaCN Consumption with respect to Roasting Time at Different Roasting Temperatures

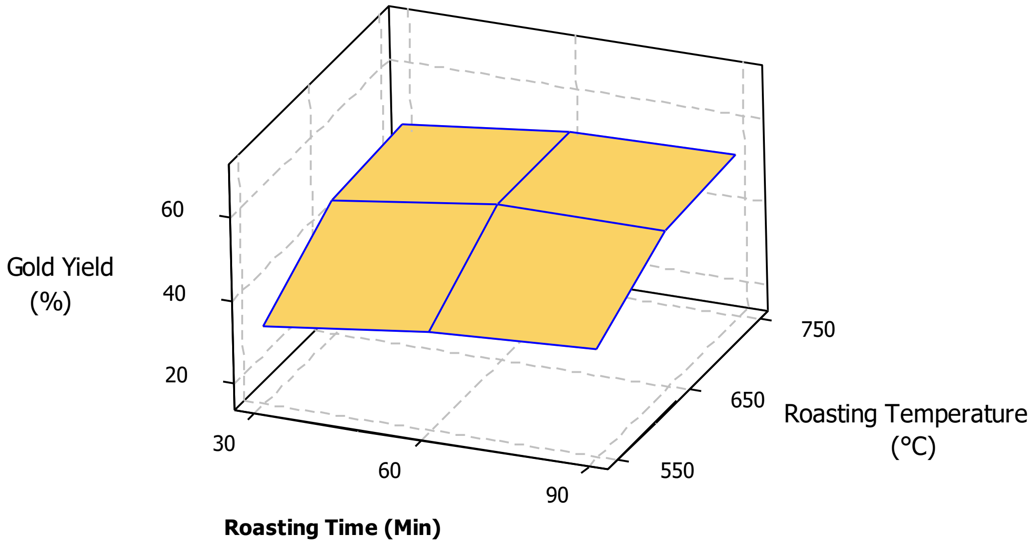

Figure 4.28. 3D Surface Response of Gold Yield (%) vs Roasting Temperature (°C) with respect to Roasting Time (min)

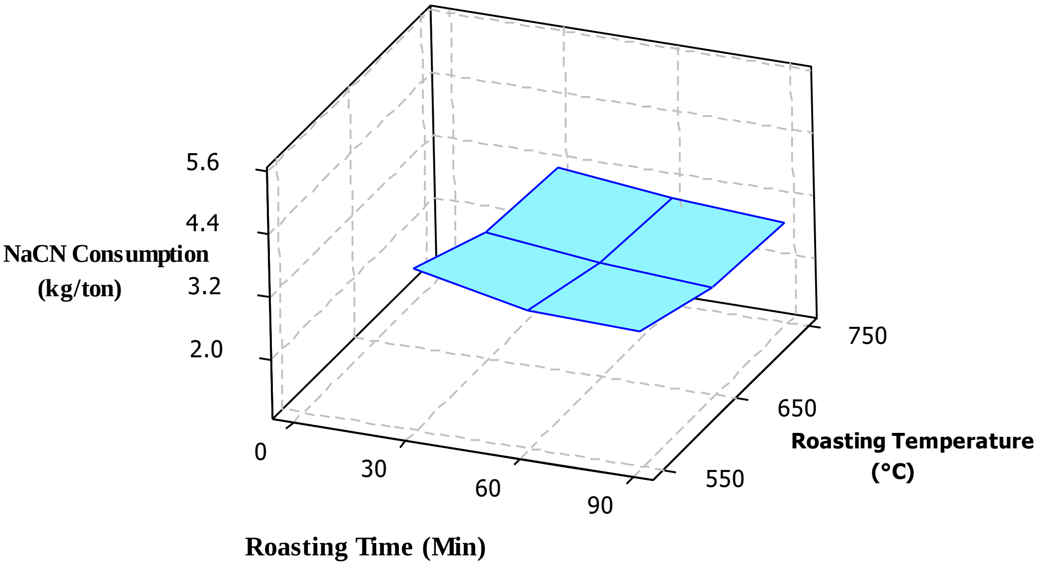

Figure 4.29. 3D Surface Response of NaCN Consumption (kg/ton) vs Roasting Temperature (°C) with respect to Roasting Time (min)

4.5 Assessment of the Changes in the Compositional Characteristics of the Ore after Roasting

Figure 4.30 compares the XRD patterns of pretreated ore by roasting at 550, 650, 750 °C for 60 minutes. Comparative XRD spectra clearly revealed the changes incurred in the ore due to the roasting application at different temperatures. The XRD pattern of the ore roasted at 550 °C showed similarity to the XRD pattern of the ROM ore: The minerals seen at the XRD pattern of the ROM ore were also seen after roasting at 550 °C. Peaks showing the presence of arsenic sulphides, pyrite, quartz and calcite were clearly seen. Despite such similarities in terms of the minerals detected, the intensities of the peaks of arsenic sulphides and pyrite were different: Characteristic orpiment, realgar and pyrite peaks occurred with lower intensities. Another remarkable difference after roasting at 550 °C is the occurrence of hematite peaks at approximately 58° and 74° which were not recorded in the XRD pattern of the ROM ore. These peaks show that structural changes started in the ore due to pyro-oxidative treatment. It could be anticipated that hematite formation is due to the thermal oxidizing transformation of pyrite into iron oxides. It could be implied that change of pyrite into porous hematite increases the liability of locked gold entities to contact with NaCN solution. Slight hematite formation is verified by the slight increase in the gold extraction of roasted ore at 550 °C.

Comparison of the XRD pattern of the roasted ore at 650 °C with roasting at 550 °C and ROM ore corresponds to more remarkable differences (Figure 4.30). The intensities of arsenic sulphides and pyrite significantly decreased and some of the characteristic peaks of these minerals were totally lost. Also, higher number of characteristic hematite peaks were recorded, some of which occurred with higher intensities as compared to roasting at 550 °C. These differences imply successful destruction of the arsenic sulphides with pyro-oxidative treatment. Also, significant reductions in pyrite peaks and occurrence of apparent hematite peaks correspond to more effective oxidation of pyrite into hematite, i.e. increase in porous structures in the ore. All these are anticipated to contribute to reduction of the refractoriness. This is reflected by the apparent increase in the gold extraction performance after roasting at 650 °C. Also, arsenic sulphides and pyrite are well-known cyanicides. The destruction of these minerals or the change in their structures to other forms would decrease cyanide consumption potential of the ore. This claim is confirmed by reduced amount of NaCN consumption after roasting the ore at 650 °C.

XRD pattern of the roasted ore at 750 °C showed notable differences in comparison with the XRD pattern of the ROM ore. Also, the peaks of the arsenic sulphides were lower and hematite peaks were stronger as compared to roasting at 550 °C and 650 °C (Figure 4.30). It was seen that arsenic sulphides and pyrite were almost entirely destructed after roasting at 750 °C. The most characteristic feature in the XRD pattern after roasting at 750 °C was the occurrence of magnetite peaks (Figure 4.30). These peaks showed that, increasing roasting temperature from 650 to 750 °C, caused reduction of hematite into magnetite. This is, in fact, not a favorable situation in roasting of gold ores: With transformation into magnetite, the porous structure of hematite is lost. This phenomenon is termed as over-roasting. In case of over-roasting despite the increase in the extent of the destruction of arsenic and/or iron sulphides, gold yield may not change and in some cases it may even decrease. This decrease is due to the re-encapsulation of gold entities with the change of the porous hematite structure to the ‘‘firm’’ magnetite form. In compliance with this, no significant change in gold extraction or NaCN consumption could be achieved when roasting temperature was increased to 750 °C in this work.

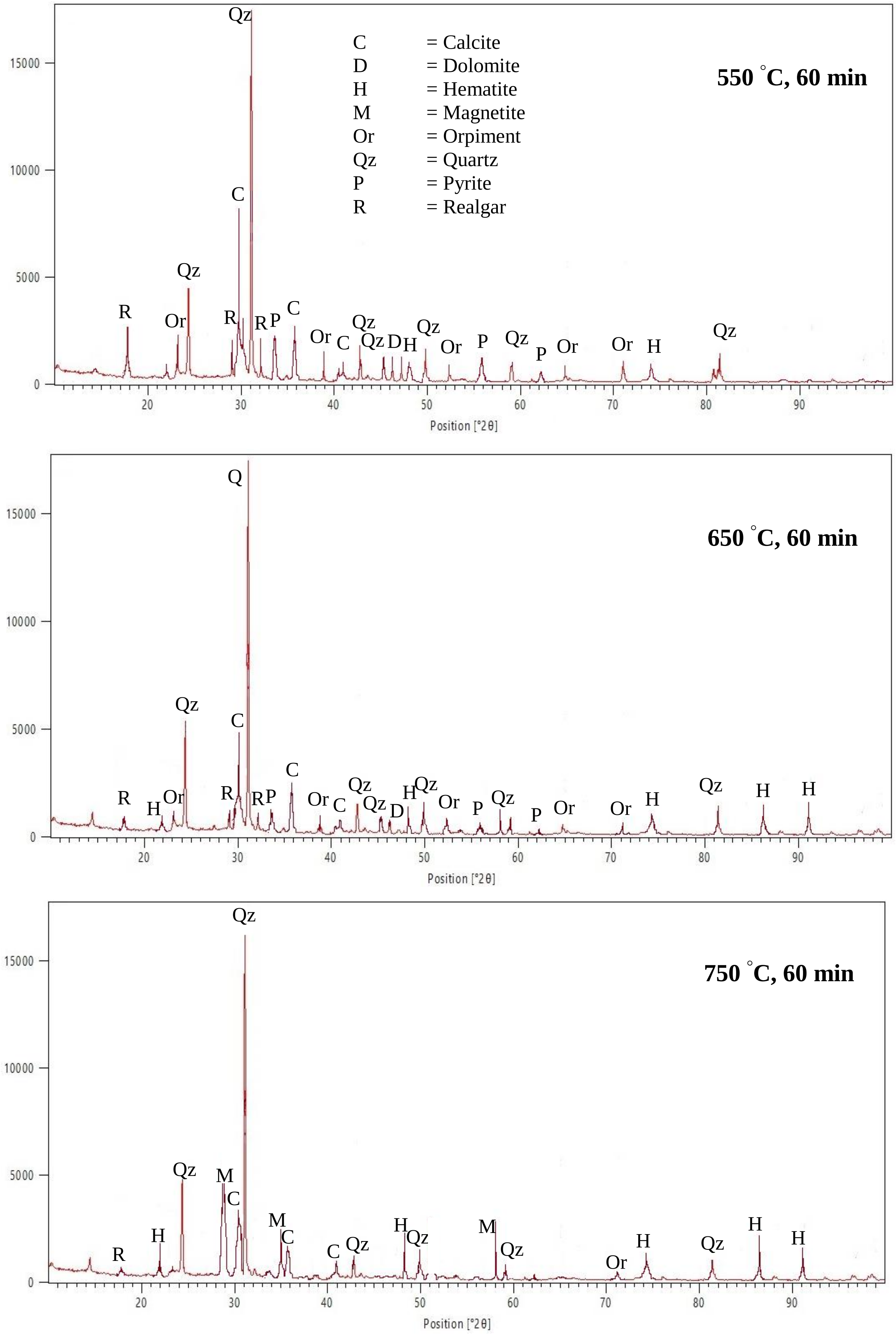

Figure 4.30. Comparison of XRD patterns of pretreated ores roasted at different temperatures

4.6 Assessment of the Run-of-Mine (ROM) Ore and Roasted Ores by Scanning Electron Microscopy

Figure 4.31 demonstrates the scanning electron microscopy (SEM) image of the run-of-mine gold ore and energy-dispersive X-ray spectroscopy (EDS) analysis of the SEM image. Apparent elements identified by EDS are labeled above relevant peaks. EDS analysis reveals abundance of silicon, iron and oxygen in the ore represented by high-intensity peaks. Presence of aluminum, sulphur and arsenic are also apparent with peaks of moderate intensities. Gold was clearly identified in the sample with two specific peaks (Figure 4.31). SEM and EDS analysis are in compliance with the aforementioned mineralogical characteristics of the ore: Silicon and oxygen peaks are attributed to quartz, iron peaks are due to the presence of pyrite and aluminum peak corresponds to the clay minerals in the ore. Apparent arsenic peaks along with characteristic sulphur peaks confirm remarkable arsenic sulphides content of the ore. Elemental sulphur might also contribute to the sulphur peaks. Clarification of iron, sulphur and arsenic in the EDS analysis imply that the area covered by the SEM image shows a representatively characteristic section of the given ore.

The SEM image was also analyzed through elemental mapping. The results are presented in Figure 4.32. Oxygen and silicon in the sample were detected almost at equivalent extents and at the same sections of the image, implying that quartz is widely distributed in the ore. Iron is another element that is highly distributed in the ore (Figure 4.32). Presence of sulphur, arsenic and gold is relatively lower as compared to oxygen and silicon, as revealed by the elemental map of the image. Rather than a homogenous distribution, these three elements tend to concentrate specifically at certain sections of the ore. Focusing distinctively on the distribution of S, As and Au show that, at sections with notable gold presence, sulphur and arsenic were also observed in concentrated forms. In other words there are certain sections in which As, S and Au specifically occur together – these sections are shown in Figure 4.33. Based on this observation, it is confirmed that gold association with arsenic sulphides, (realgar and orpiment in this ore), is one of the distinctive characteristics of this ore - a challenging mineralogical feature against gold extraction from this ore.

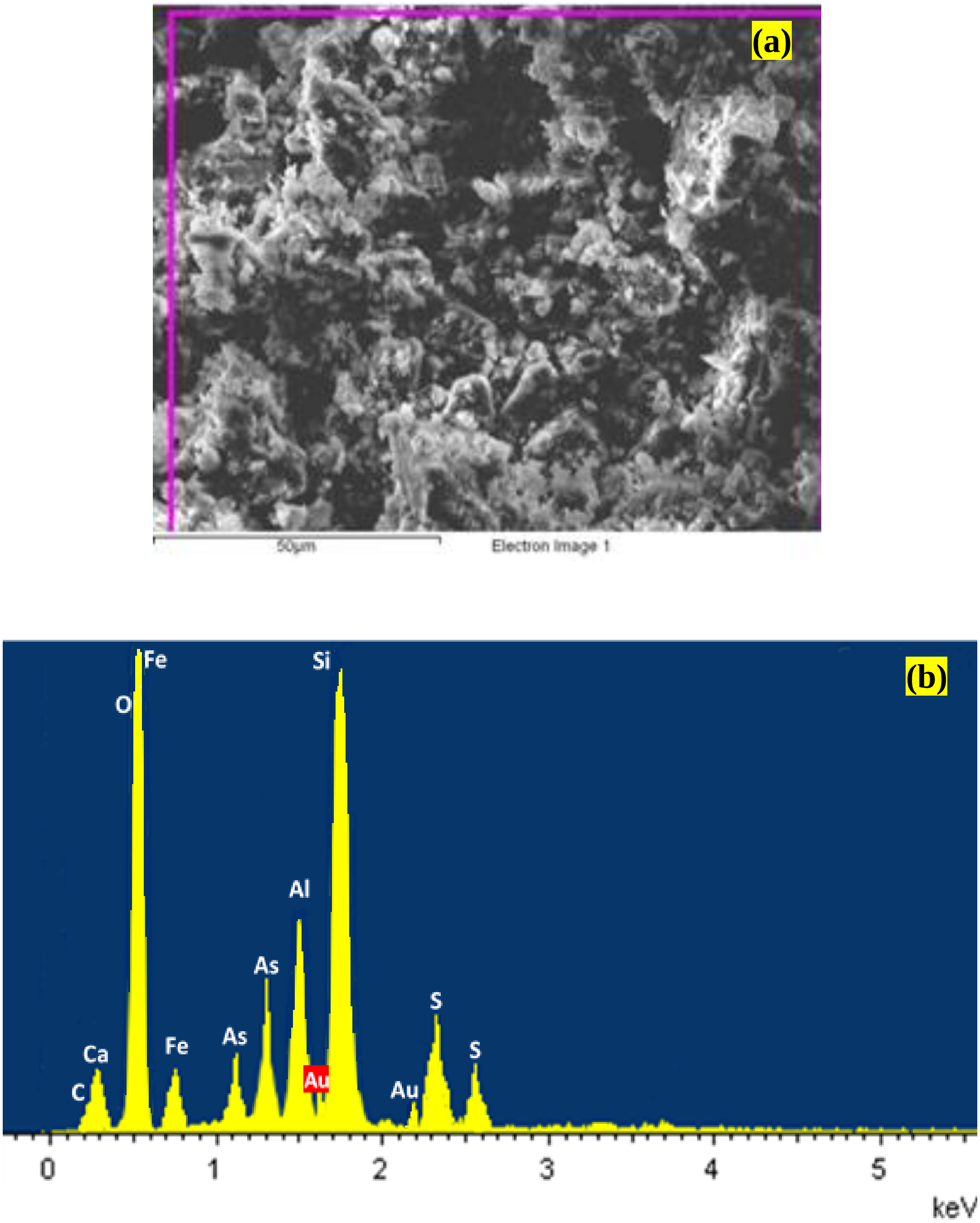

Figure 4.31. (a) SEM Image and (b) EDX Analysis of the of ROM Ore

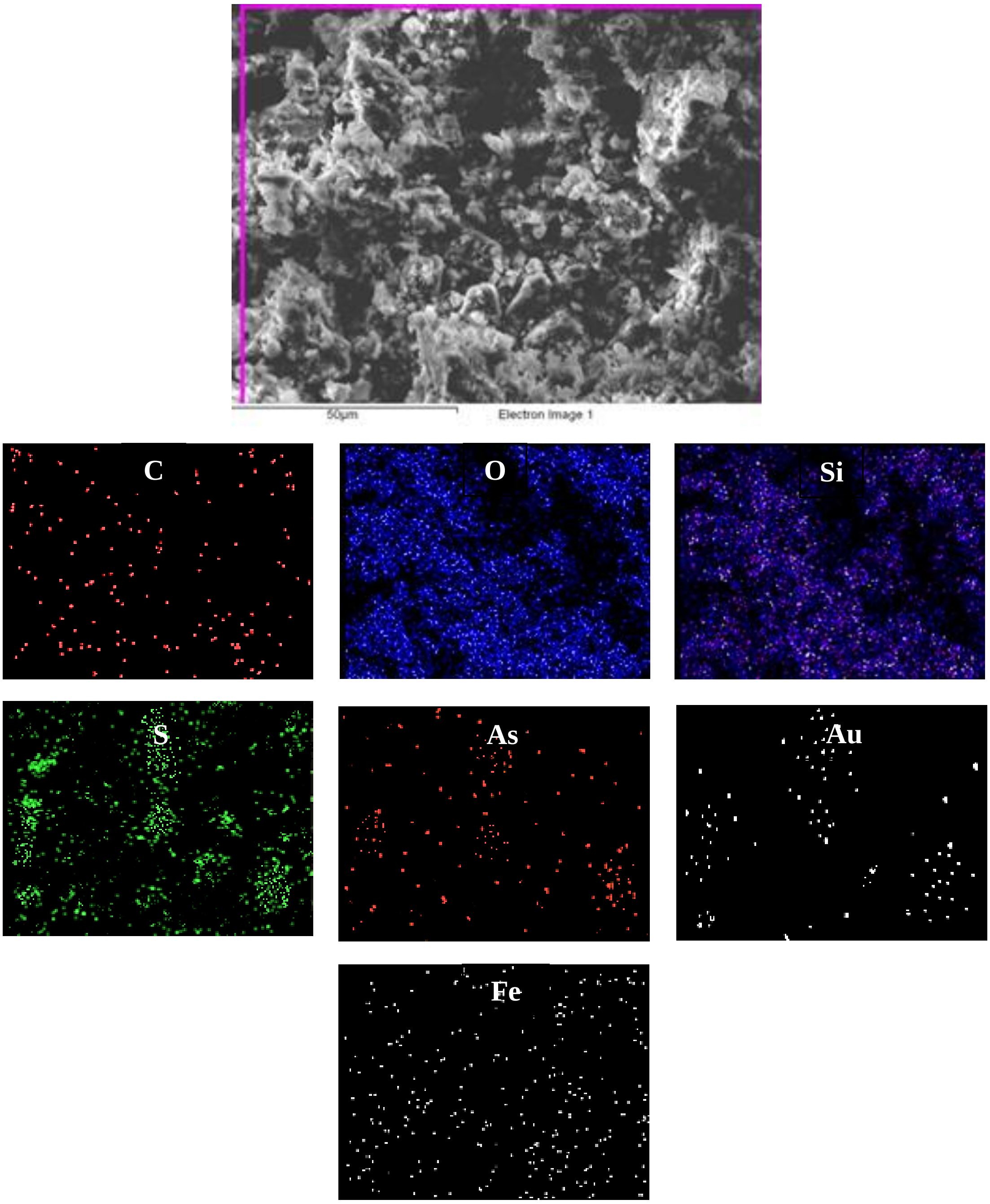

Figure 4.32. SEM Image (at the top) and Elemental Mapping of the of Ore:
C= Carbon, O= Oxygen, Si= Silicon, S= Sulphur, As= Arsenic, Au = Gold, Fe = Iron

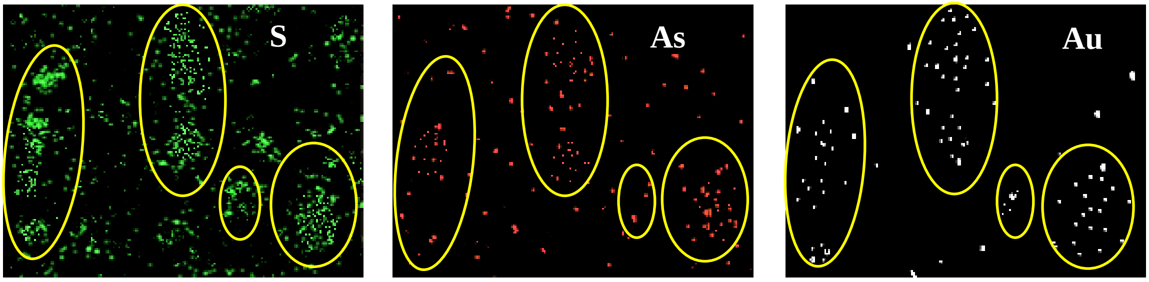

Figure 4.33. Elemental Mapping of Sulphur (S), Arsenic (As) and Carbon (C) in the ROM ore and Concentration of These Elements at Certain Sections (shown in yellow circles)

Ores roasted for 60 minutes at 550 °C, 650 °C and 750 °C were also subjected to SEM investigation. Characteristic SEM images of the roasted ores are given in Figures 4.34-4.36 along with the EDS analysis of the given SEM images. Comparative assessment of the EDS results show that roasting influences the compositional characteristics of the ore, particularly with regard to sulphur (S) and arsenic (As): After roasting, changes were recorded in the intensities of the specific peaks of arsenic (As) and sulphur (S). The EDS analysis shows two peaks for sulphur and two peaks for arsenic in the ROM ore (Figure 4.31). The intensities of the two sulphur peaks and the two arsenic peaks decrease after roasting at 550 °C (Figures 4.31 & 4.34). When the roasting temperature was increased to 650 °C only one arsenic peak and one sulphur peak could be seen (Figure 4.35). Further, the intensities of these remaining single arsenic and sulphur peaks are apparently lower as compared to those seen after roasting at 550 °C (Figures 4.34 & 4.35). With the increase of the roasting temperature to 750 °C no arsenic and sulphur peaks could be detected (Figure 4.36). These observations prove that pre-treatment of the ore by roasting directly affected the realgar and orpiment content of the ore. SEM-EDS analysis justify that oxidative pre-treatment of this arsenic-rich gold ore at elevated temperatures caused chemical decomposition of orpiment and realgar, and most probably their eventual loss in the form gaseous compounds.

It should also be emphasized that gold is clearly identified by the EDS analysis in all samples, regardless of the roasting temperature (Figures 4.34-4.36). Besides the visibility of gold by EDS is relatively higher in the roasted ores as compared to the ROM ore (Figures 4.31 & 4.34-4.36).

Presence of iron were seen both in the ROM ore and roasted ores (Figures 4.31 & 4.34-4.36) – This is expected because XRD patterns showed that ROM ore consisted of pyrite (FeS_2) and roasted ores had hematite (Fe_2O_3) and limited extent of magnetite (Fe_3O_4), depending on the applied roasting temperature. Hence, the Fe peaks observed in the EDS results of the ROM ore and roasted ores could be attributed to these iron-bearing compounds.

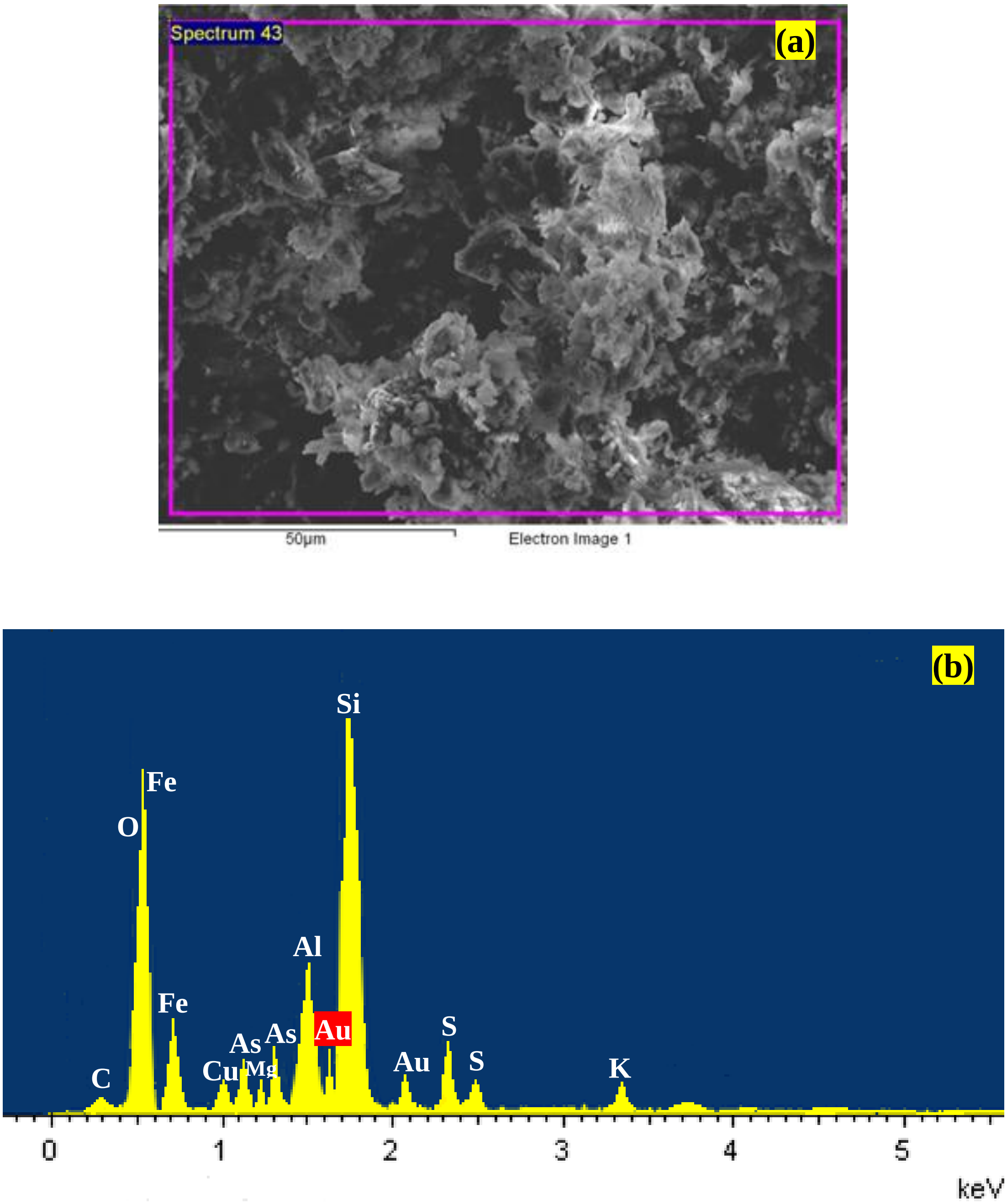

Figure 4.34. (a) SEM Image and (b) EDX Analysis of the Ore Roasted at 550 °C for 60 minutes.

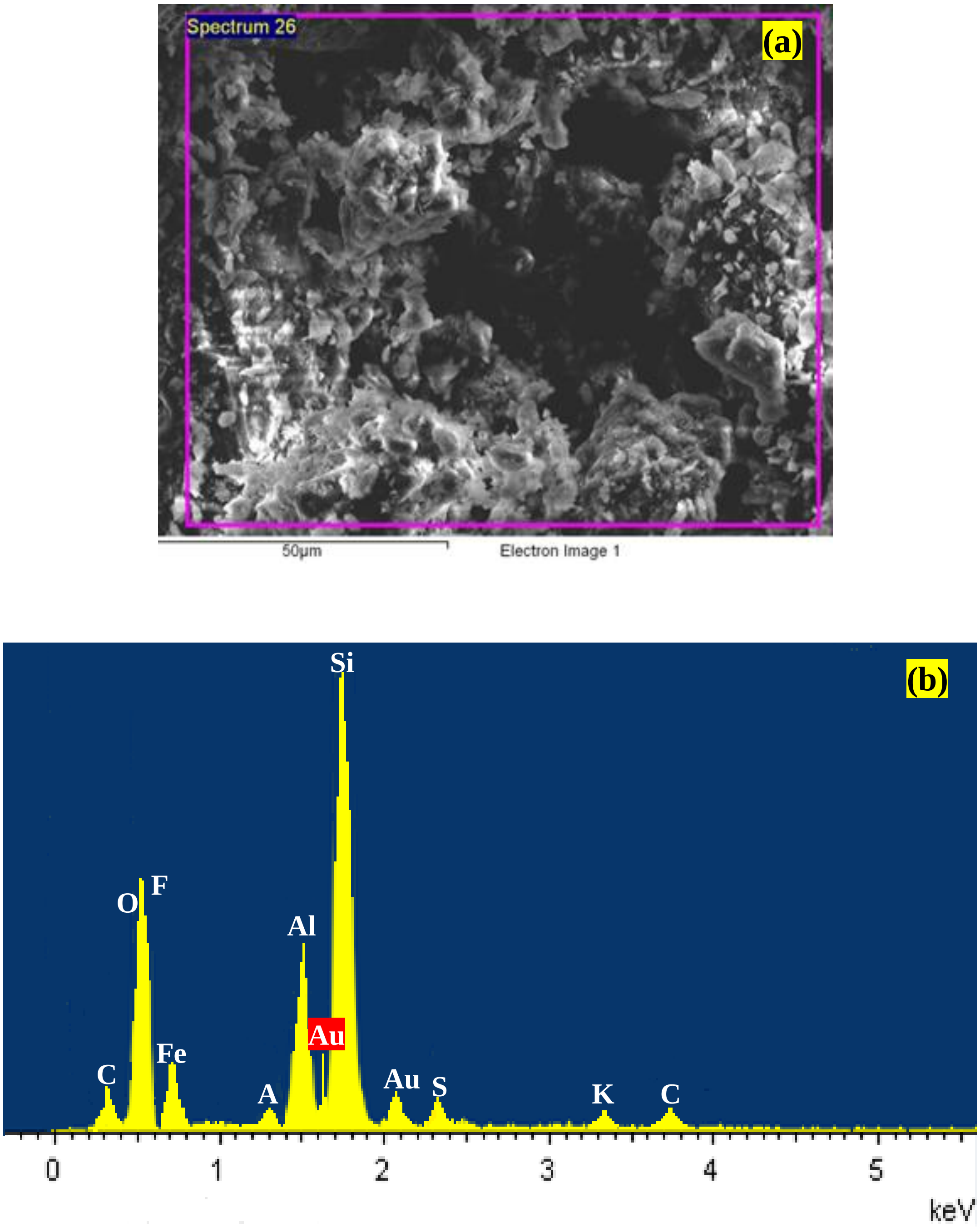

Figure 4.35. (a) SEM Image and (b) EDX Analysis of the Ore Roasted at 650 °C for 60 minutes.

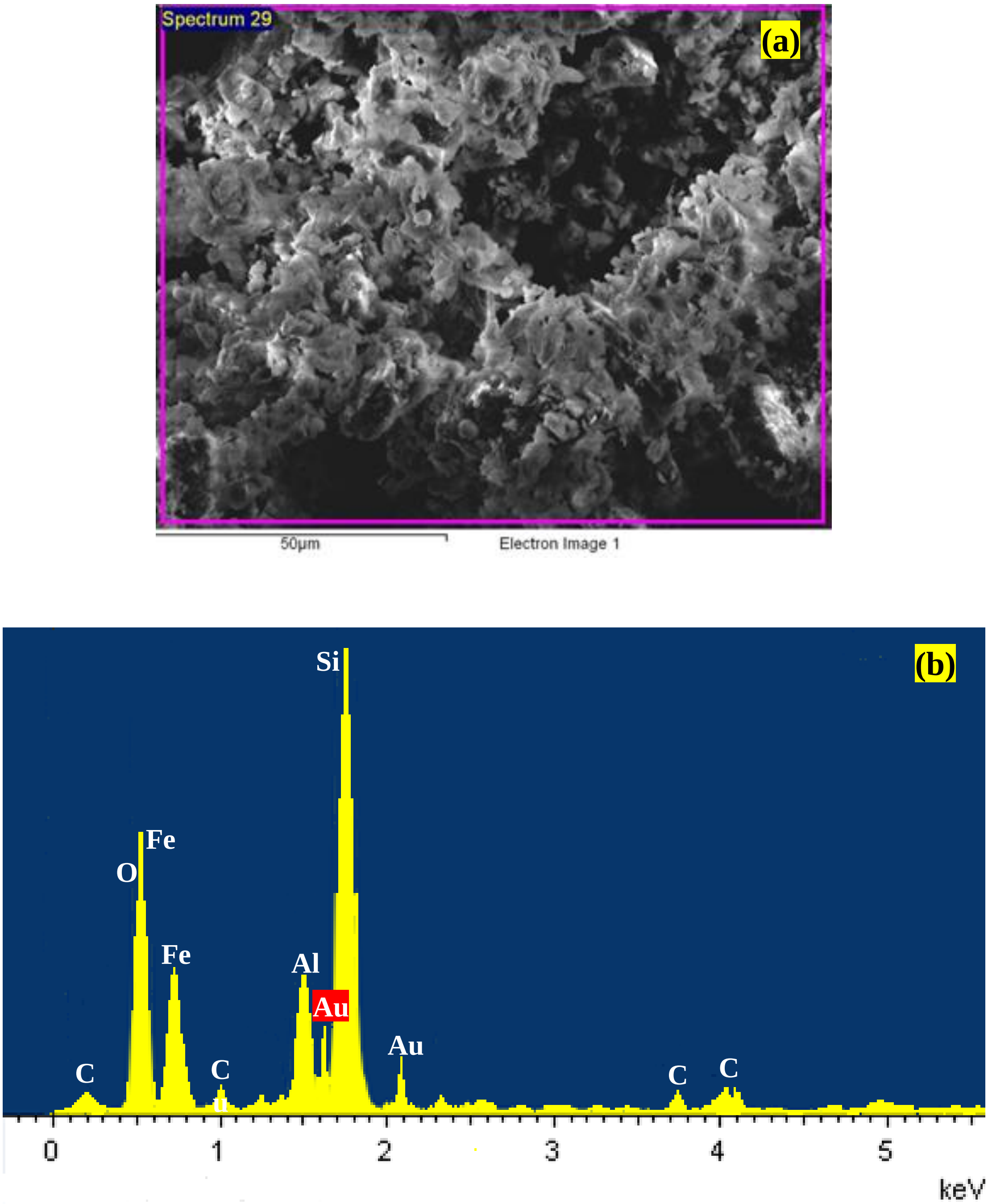

Figure 4.36. (a) SEM Image and (b) EDX Analysis of the Ore Roasted at 750 °C for 60 minutes.

Previous investigations and analysis revealed that the refractoriness of this gold ore and hence the difficulty of Au extraction depended on several factors; including the presence of Au bearing arsenic sulphides (AsS & As_2S_3 in this ore) and iron sulphides (mainly FeS_2 in this ore). It was suggested that Au is in locked form in these sulphide bearing mineral grains and this is anticipated to be limiting/obstructing the contact of Au entities with the leaching lixiviant. As confirmed by the aforementioned SEM images, roasting pre-treatment proved useful in increasing the possible exposure of Au entities locked in As-sulphide grains, with NaCN solution. In addition, roasting in an oxidizing environment is well known to change the structure of pyrite, i.e. thermal oxidation causes the change of pyrite into hematite. This, in fact, not only a chemical transformation, but structural change of Fe-bearing grains into a more porous form occur. With regard to the pyrite content in this ore, this results was anticipated in this work. Figure 4.37 shows SEM images of roasted Fe-bearing grains at 650 °C and 750 °C for 60 minutes. It is clearly seen that roasting on pyrite particles provided a significant change at both temperatures into the anticipated porous structure. The grain shown in Figure 4.37.a is distinguished with its sponge-like form, which consists of numerous pores in varying dimensions. Gold entities could be identified with a brighter tone in the SEM images and those observed in this grain are shown in white circles. It is seen that Au entities that were probably in locked/embedded form in the original FeS_2 grains became remarkably exposed and liable to contact with NaCN solution with the formation of the pores after roasting. This justifies the increase in the Au extraction from this ore after the roasting pretreatment. It should also be noted that Au entities encapsulated n Fe-bearing grains are extremely fine, usually in 1-2 μm size.

The SEM image in Figure 4.37.b is more interesting, since it captures a view of a Fe-bearing grain which possessed porous as well as non-porous sections after roasting. It should be noted here again that Figure 4.37.b is an image taken from the ore roasted at 750 °C. The grain has smaller and wider pores, but it also has a non-porous section in the inner part and randomly located smaller non-porous sections all across the grain. Also, there are specific sections that incurred re-closure after gaining a porous form. It is known that excessive roasting may/would cause further transformation of hematite into magnetite. This is a non-favorable condition in the roasting of gold ores, since this transformation results in the loss of the porous structure and thus, reduces the exposure of Au entities in the pores. In this study, the XRD pattern obtained at 750 °C showed presence of magnetite, a major compositional difference compared to the XRD patterns of the ROM ore and the ore roasted at 650 °C. In this respect, the SEM image taken from the ore roasted at 750 °C is quite meaningful: It reveals exposed Au entities randomly distributed in the porous sections as well as Au entities that are in locked/embedded form in sections that were likely to be porous, but incurred re-closure due to excessive roasting, i.e. due to hematite-magnetite transformation (Figure 4.37.b). In this respect, the SEM investigations are in compliance with the XRD results and interpretations made on the Au extraction results prior to- and after roasting pretreatment.

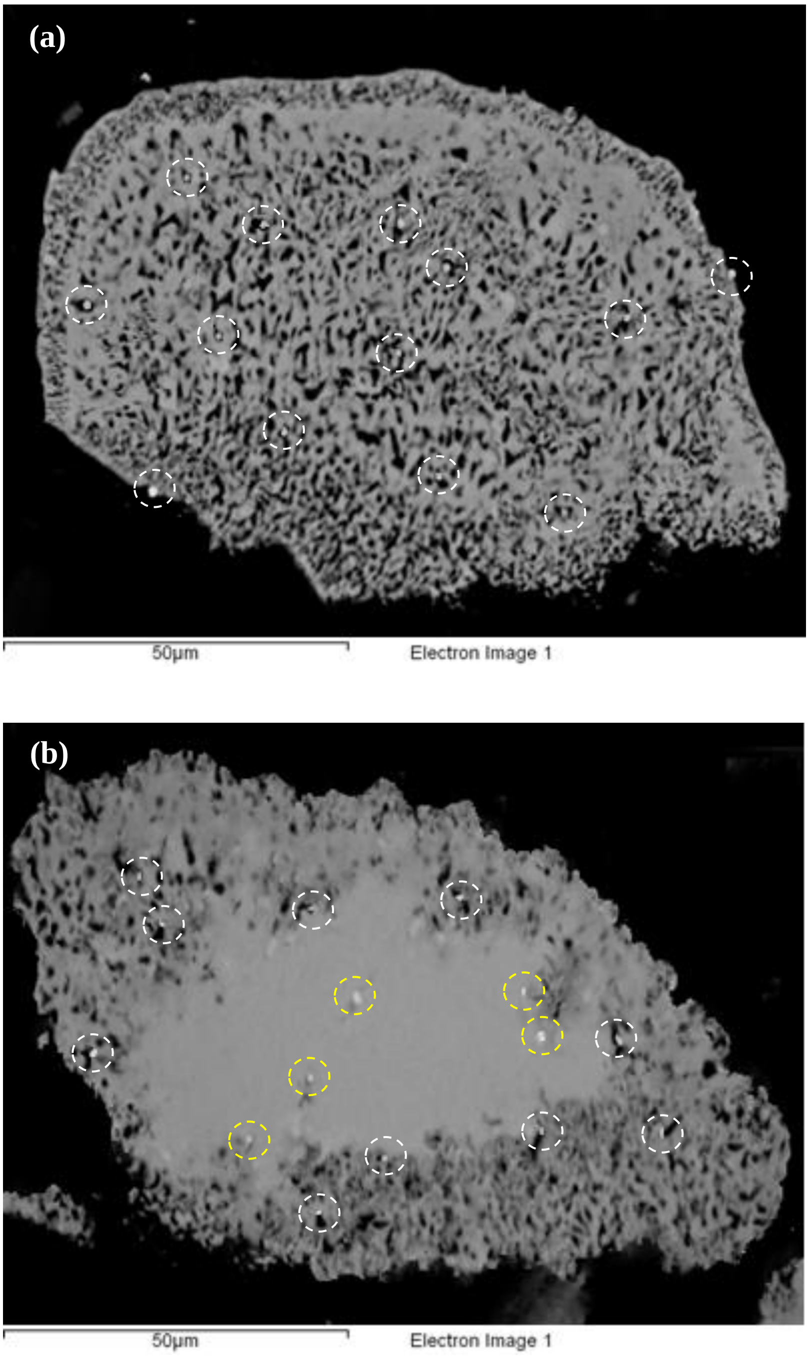

Figure 4.37. SEM Images of; a) Roasted Fe-bearing Grain at 650 °C b) Partially Roasted Fe-bearing Grain at 750 °C

(white circles: Au entities with exposure; yellow circles: Au entities locked in the grain)

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

Turkey has high potential of refractory gold ore reserves. Also, free milling ores are being depleted throughout the world and low grade ores should be recovered. So, refractory gold ore processing has become important more than ever. However, the operating and capital costs for these ores are high and their treatment is challenging.

The results of this study are expected to provide a development in this topic and understanding the leaching behavior of these ores, particularly those with an abundance of arsenic sulphides, realgar and orpiment, which could form the basis for further work and research.

In the tests conducted to determine NaCN consumption tendency of the gold ore and to investigate the impact of particle size on leaching it was seen that the ore has a highly refractory character and contains high amounts of cyanicides. In other words, this ore is classified as "difficult" in terms of Au extraction through NaCN leaching. In the leaching tests conducted on ore samples with different particle sizes, it was seen that Au extraction increased with increasing fineness. This situation indicates that exposure of the gold in the ore increases with decreasing particle size. Within this context, when the particle size was reduced down to P_{100} 37 μm from P_{100} 150 μm, gold extraction became almost twice higher increasing from 37.5% to around 70%.

However, reduction of the particle size had a negative impact on NaCN consumption. Very high levels of NaCN consumed by the ore indicates that other elements/compounds, which consume significant cyanide, i.e. reacts with NaCN, are present in the ore other than gold. When mineralogical structure of the ore is inspected, such constituents are likely to be sulphides. When the particle size was reduced to P_{100} 37 µm, a remarkable increase in NaCN consumption was observed. This indicates that the constituents referred to as cyanicides became more exposed and more influential with increasing particle size.

In view of these findings, it was seen that leaching this As-rich gold ore is not viable to cyanide leaching-at least- without pre-treatment.

The second stage of the work therefore focused on pre-treatment of the ore in order to enhance its liability to NaCN leaching. Within this scope and based on previous literature, the ore was subjected to roasting, which is a thermal-oxidizing pre-treatment. Leaching behaviors of the ore roasted at various temperatures and roasting periods were determined. Two major goals of roasting this ore are:

- To increase extent of Au leaching by reducing its refractory character,
- To reduce/eliminate the influence of cyanicides.

As a result of the leaching of the roasted ore samples, significant improvements in leaching performance and reductions in NaCN consumption were achieved. Gold extraction as high as 70% was achieved after roasting the ore with P_{100} size of 150 µm at 750 °C for 90 minutes, with 3.7 kg NaCN consumption per ton of ore. This situation indicates that similar extent of Au extraction to the level achieved only at very fine size ROM ore (P_{100} 37 µm) with the ROM ore could be achieved at a relatively coarser particle size after pretreatment with a much lower NaCN consumption. In other words, the pretreatment procedure applied provides the anticipated improvement, particularly, in reducing the refractoriness of the ore and rejection of cyanide consuming components other than gold. Post-roasting metallurgical performance of 70% Au extraction at 3.7 kg/ton of ore NaCN consumption indicates metallurgically acceptable levels of gold processing, i.e. rendering an As-rich refractory gold ore workable by a pre-treatment is possible., When constraints such as process economy, time, energy consumption, etc. are considered, roasting at 650 °C and 60 minutes was found optimum for this ore.

Technically roasting is optimum for this type of gold ore, yet, in practice due to the additional energy consumption it may not be economically feasible.

For further studies, it is recommended that application of other pre-treatment methods, particularly, "Pressure Oxidation - POX", is strongly suggested. POX is a method being successfully applied for refractory gold ores at the industrial scale because it provides a significant solution to the harmful-toxic emissions and wastes that would emerge due to roasting. Also, "Biological Oxidation – BIOX + Leaching" may be considered.

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APPENDIX

7.1 Chemical Dissolution of Gold in Solid Samples for Analysis with AAS

The steps of the chemical analysis are presented below.

1. Put 10 grams of dried sample into a 400 ml beaker.
2. Add 25 ml 37% HCl into the beaker and start heating the breaker.
3. Wait for 15-25 minutes to dissociate the sample and to assure its contact with HCl. Stir the solution occasionally using a glass baguette.
4. Add 15 ml 65% HNO_3 and wait for another 15-20 minutes while beaker is heated. Stir the solution occasionally.
5. Add 25 ml distilled water to the beaker and wait until boiled.
6. After boiling, wait until no gas fumes from the beaker is observed, then remove the beaker from the heater.
7. Wait for the solution to cool down.
8. Put an analytical grade filter paper into a glass cone and place a 250 ml flask under the cone. Pour the solution to the filter paper gently and carefully. Filter the entire solution.
9. When filtration is done, complete the solution to 250 ml with distilled water.
10. Fill the separation funnel with the 250 ml solution in the flask.
11. For metal analysis (analysis of metals other than gold) draw 50 ml solution and pour into a flask. Analyze this solution using an Atomic Absorption Spectrometer (AAS).
12. Add 15 ml analytical grade isobutyl methyl ketone to the remaining solution and gently shake the separation funnel. Wait until separated phases could be observed (a transparent phase and a yellow phase – separation takes approximately 5 minutes).
13. Draw the yellow phase (bottom phase) carefully and pour it off. This phase aims at the removal of dissolved iron from the solution.

14. Add 40 ml 10% HCl to the remaining solution in the flask and shake the separation funnel. Wait until separated phases could be observed again (a transparent phase and a yellow phase - takes approximately 5 minutes). This step aims at further removal of dissolved iron from the solution.
15. Repeat previous step (step 14) until no yellow phases could be observed, i.e. all the dissolved iron is removed from the solution – this requires repeating of step 14 at least two times.
16. Take the remaining solution to a 25 ml flask and complete it to 25 ml with isobutyl methyl ketone.
17. Shake the flask well and analyze with AAS.
18. The result given by AAS should be further processed by the following equation:

$$\frac{\text{Value from AAS x Dilution Factor x Volume of flask}}{\text{Sample Amount}} = \%\ \text{Element}$$

7.2 General Procedure of Bottle Roll Testing

The bottle test steps are briefly as follows:

1. Grind the ore to the proposed size.
2. Adjust ore and water amounts to form a slurry of 40% solids by weight.
3. Measure the initial pH value of the slurry and adjust it to 10.5 using $Ca(OH)_2$ (if necessary).
4. Add proposed amount of NaCN.
5. Place bottles on the bottle-roller, adjust rotational speed at ≈30 rev/min, start the roller.
6. Start O_2 supply (optional) at the determined flow rate. Check the flow rate on the flow meters.
7. Stop the bottle-roller after pre-determined periods. Allow some time for solids in the slurry to settle down (not more than 10 minutes). Draw 30 ml of solution from the relatively clarified surface layer. Centrifuge this solution if necessary.
8. Measure the pulp pH; if necessary re-adjust to 10.5 using $Ca(OH)_2$.
9. For determining NaCN consumption in leaching, use 10 ml of drawn-centrifuged solution for silver nitrate ($AgNO_3$) titration. Add consumed amount of NaCN to the solution.
10. Repeat steps 5-10 after each pre-determined period.
11. After terminating the tests separate filter the slurry. Perform analysis for gold (and/or other metals of interest) separately on the filtrate and the remaining solid, and analyze the filtrate for NaCN consumption over the last testing period.