KARADENİZ TECHNICAL UNIVERSITY
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

DEPARTMENT OF MINING ENGINEERING

RECOVERY OF GOLD FROM COPPER-RICH ORES USING ION EXCHANGE RESINS

MASTER THESIS

Deus Albert MSUMANGE

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TRABZON

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Department of Mining Engineering
Deus Albert MSUMANGE

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Approved By

Chairman : Prof. Dr. Ata Utku AKÇİL ....

Member : Prof. Dr. Hacı DEVECİ ....H.Deveci....

Member : Assoc. Prof. Dr. Ersin Yener YAZICI ....

Prof. Dr. Asim KADIOĞLU
Director of Graduate School

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Deus Albert Msumange
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DECLARATION

I Deus Albert Msumange, hereby certify that this thesis titled ‘Recovery of Gold from Copper-rich Ores using Ion Exchange Resins’ is the result of my own handwork under the full supervision of Prof. Dr. Hacı DEVECİ and that no any previous submission for a degree has been done here or elsewhere. Also, all the parts in this thesis which are not my own work have been identified. I hereby also declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. 26.06.2019

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Karadeniz Technical University
The Graduate School of Natural and Applied Sciences
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In this study, the treatment of a refractory copper-rich gold ore was investigated. Initial studies focused on the characterization of the refractory behaviour of the ore, which yielded only 18.4% extraction in direct cyanide leaching. Diagnostic leaching tests have shown that 70% of gold which were recovered at HCl +NaCN and HNO_3 +NaCN stages were linked with different mineral phases such as sulfide minerals. Ultrafine grinding did not offer extractions to the desired extent, even when cyanide concentration was increased up to 8 g/L as it only enhanced gold recoveries by 36% (from 18% to 54%). Chemical pretreatment of the ore using acid leaching and roasting proved to be the best treatment route as it offered 96% of gold recovery. In this study, adsorption tests on pregnant leach solutions obtained from cyanide leaching of copper-rich gold ore were also investigated. Activated Carbon (GAC 1240 M-1448), strong base resin (Purogold A194) and medium base resin (Purogold S992) were used as adsorbents for gold recovery from cyanide leaching solutions of the ore. In these tests effects of adsorbent type, dosage, cyanide concentration, process type (CIP/CIL/RIL/RIP) were also investigated. The experimental results showed that adsorbent dosage and cyanide concentration are important parameters affecting the extent of gold adsorption and its selectivity. Increasing cyanide concentration remarkably improved the selectivity allowing high recoveries even at low activated carbon/resin dosages. The adsorption tests have also demonstrated that the activated carbon has higher gold loading capacity than Purogold A194 and Purogold S992. Each adsorbent offered good selectivity for gold over copper. Purogold S992 appeared to be the most selective adsorbent that barely loaded copper. This distinctive trait makes the resin preferable for gold recovery from cyanide liquors containing a significant amount of copper values.

Keywords: Gold, Copper, Activated Carbon, Adsorption, Ion exchange resins, Recovery, Selectivity, Cyanide.

Yüksek Lisans Tezi

ÖZET

İYON DEĞİŞTİRİCİ REÇİNELER KULLANILARAK YÜKSEK BAKIR İÇEREN CEVHERLERDEN ALTIN KAZANIMI

Deus Albert Msumange
Karadeniz Teknik Üniversitesi
Fen Bilimleri Enstitüsü
Maden Mühendisliği Anabilim Dalı
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Bu çalışmada, yüksek bakır içeren bir refrakter altın cevherinin değerlendirilmesi araştırılmıştır. İlk olarak, doğrudan siyanür liçinde %18,4 gibi düşük altın kazanımına yol açan, cevherin refrakter davranışının karakterizasyonuna odaklanılmıştır. Tanımlayıcı liç testlerinde, HCl+NaCN ve HNO_3+NaCN aşamalarında %70'i kazanılmıştır. Bu veriler altının , sülfit mineralleri gibi farklı mineral fazları ile bağlantılı olduğunu göstermiştir. İnce öğütme işlemi, altın kazanımında istenilen düzeyde bir iyileşme sağlamamış; siyanür konsantrasyonu 8 g/l'ye yükseltildiğinde dahi, yalnızca %36 oranında (%18'den %54'e kadar) bir artış sağlamıştır. Asit liçi (1M H_2SO_4) ve kavurma gibi kimyasal ön hazırlık işlemleri sonucunda, cevherden altın kazanımı %96'ya ulaşmıştır. Bu ön hazırlık işlemlerinin cevherden yüksek altın kazanımı açısından en uygun işlemler olduğu görülmüştür. Bu testlerde, adsorban olarak aktif karbon (Karbon GAC 1240 M-1448), kuvvetli bazik reçine (Purogold A194) ve orta bazik reçine (Purogold S992) kullanılmıştır. Adsorban tipi, dozajı, siyanür konsantrasyonu ve proses tipinin (CIP/CIL/RIL/RIP) etkileri incelenmiştir. Deneysel sonuçlar, adsorban dozajı ve siyanür konsantrasyonunun, altın adsorpsiyonu ve seçimliliğini etkileyen önemli parametreler olduğunu göstermiştir. Siyanür konsantrasyonunun arttırılması, seçimliliği arttırmış ve daha düşük aktif karbon/reçine dozajlarında bile yüksek geri kazanımlarına olanak sağlamıştır. Adsorpsiyon testleri, aktif karbonun altın adsorpsiyon kapasitesinin Purogold A194 ve Purogold S992 reçinelerinin kapasitelerinden daha yüksek olduğunu göstermiştir. Bütün adsorbanlar, altın için bakıra kıyasla daha yüksek bir seçimlilik göstermiştir. Purogold S992 en yüksek seçimliliğe sahip adsorban olarak belirlenmiştir. Bu ayırt edici özellik, Purogold S992 reçinesinin, önemli miktarlarda bakır konsantrasyonu içeren siyanür çözeltilerinden altın kazanımı için tercih edilmesini sağlayacaktır.

Anahtar Kelimeler: Altın, Bakır, Aktif karbon, Adsorpsiyon, İyon değiştirici reçineler, Kazanım, Seçimlilik, Siyanür.

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1. INTRODUCTION

1.1. Overview

Following the rapid diminution of free milling gold ores, production of gold from refractory ores is gradually receiving attention across the globe. Copper-rich gold ores are among the types of refractory ores. The process of recovering gold from gold-copper ore presents various technical drawbacks ascribed to the ready dissolution of most minerals of copper in cyanide solutions, leading to high cyanide consumption and exorbitant cost. Certain copper minerals (e.g., chalcopyrite) can also exhibit preg-borrowing traits which are reversible when subjected to cyanide-deficient solutions (Marsden and House, 2006).

Traditionally the process of adsorption by activated carbon is employed for the recovery of Au from cyanide leachates. However, significant technical difficulties are encountered in the use of this method depending particularly on ore properties. At low concentrations of cyanide, copper adversely affects the gold recovery from the solution as adsorbed to activated carbon (Sceresini, 2005; Marsden and House, 2016). For gold adsorption from leach liquors with appreciable copper content, high CN/Cu ratios (>2) should be maintained to improve selectivity of activated carbon for the gold adsorption. The justification for this is the relatively low affinity of activated carbon for highly charged copper-cyanide complexes (e.g. $Cu(CN)_4^{3-}$, $Cu(CN)_3^{2-}$), which forms at high CN/Cu ratios. However, even if adsorption is undertaken at a high concentration of cyanide, a certain amount of copper is still adsorbed onto activated carbon (Dai et al., 2012; Van Deventer et al., 2014; Dai et al., 2012).

Activated carbon adsorption is not a suitable method in the gold recovery from cyanide leach liquors if the ore is associated with carbonaceous materials (preg-robbing). Because, carbonaceous materials also act as activated carbon adsorbing the dissolved gold leading to gold losses of 20-90% (Marsden and House, 2006; Miller et al., 2005; Celep., 2015). The Carbon-in-Leach (CIL) process or a chemical pretreatment can be employed to reduce gold losses in cyanide leaching of carbonaceous ores (Dunne et al., 2012; Miller et al., 2005). However, since the direct CIL method is not effective for ores with strong preg-robbing traits, it is necessary to remove reactive carbons before cyanide leaching (CIL) by suitable pretreatment techniques such as roasting (SGS, 2009). However, such pre-treatment processes increase operating costs.

There is a need to develop new and more effective alternative methods due to the low performance of the activated carbon adsorption method especially in the gold recovery from leach solutions having high carbonaceous material and/or high levels of copper (Kotze et al., 2016; Sceresini, 2005). IX resins have appeared to be the promising alternative to activated carbon (Marsden and House, 2016; Kotze et al., 2016; van Deventer et al., 2014). Very recently, a considerable attention has been paid to the invention of special resins for the selective recovery of gold from selective cyanide solutions (Kotze et al., 2016).

There are six chapters in this thesis. Chapter one introduces the work; its focus, aims and problem statement. Chapter two gives the literature review to provide a background information focusing particularly on the current practise for gold recovery from copper rich ores. In the third chapter, the research methodology used in the experimental studies is outlined. This includes characterisation of the as-received ore, ore preparation, experimental and analytical procedures for leaching and adsorption tests.

Chapter four gives a detailed report on the experimental results and their discussion. Chapter five covers the conclusions while recommendations on the future work are covered in chapter six.

1.2. Problem Statement

Gold ores may contain copper minerals. The extraction of gold by cyanide leaching is a process which is naturally unselective with ready dissolution of most minerals including most Cu minerals available in the ore. The availability of appreciable levels of copper in leach solutions interferes with downstream recovery of Au by activated carbon. In this regard, a proper choice of selective gold resin(s) can be considered as potential alternatives to activated carbon for the maximum recovery of Au from cyanide leach liquors.

1.3. Aims and Objectives

The aim of this research work is to investigate an effective utilization of ion exchange (IX) method for the recovery of gold from cyanide leach liquors of copper-rich gold ores. In this research, treatment and characterization of refractory copper-rich gold ore from Mastra (Turkey) were investigated. Within the scope of this study, pretreatment, cyanide leaching, activated carbon adsorption (CIL/CIP) and resin in leach/pulp (RIL/RIP) tests were carried out. The findings of this project are expected to contribute to the potential exploitation of IX resins for the recovery of Au from refractory gold ores especially the ones containing considerable amount of copper.

The main objectives of this study are:

I. To characterize the refractory behaviour of the ore in cyanide leaching through physical, analytical, mineralogical and diagnostic leaching tests

II. To test and develop suitable pre-treatment processes for elimination of refractoriness of the ore

III. To overcome copper interference with gold cyanidation

IV. To demonstrate how ion exchange resins are more selective than activated carbon for the recovery of gold from cyanide liquors containing high levels of copper.

V. To make scientific / technical contribution to the current knowledge for the treatment of copper-rich gold ores.

2. LITERATURE REVIEW

2.1. Introduction

The distinctive role that gold plays in society is considerably connected to the fact that it is the most noble of all metals. The vast use of gold in various industries is linked to its longevity (Nicol et al., 1984). In 2013, the total gold reserves exploitable in the world were around 56,000 tons (Celep et al., 2009; Celep, 2011; MTA, 2016). Figure 1 illustrates the distribution of world gold reserves according to countries and Figure 2 demonstrates the prices of gold from 2000 to 2018. Turkey appears to have around 2% of the world reserves (Figure 1) with 15 operating gold mines (Table 1).

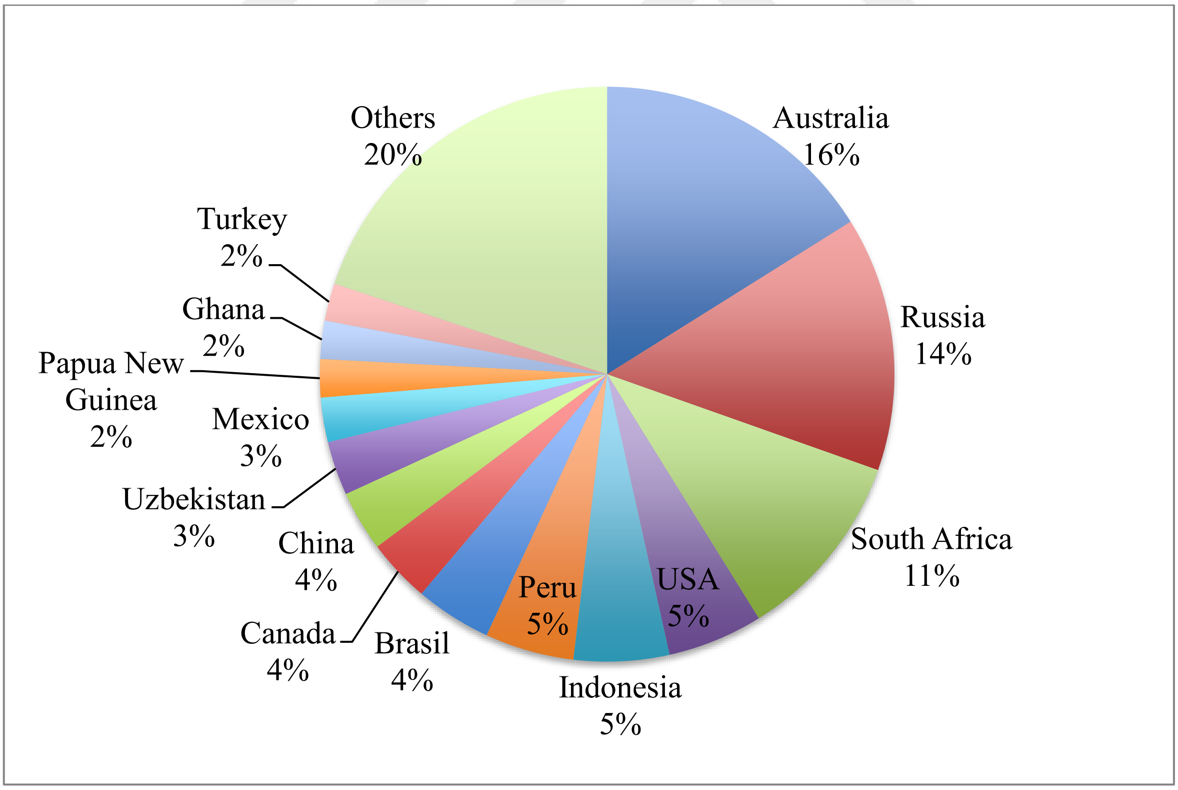


Figure 1. Distribution of world gold reserves (MTA, 2016)

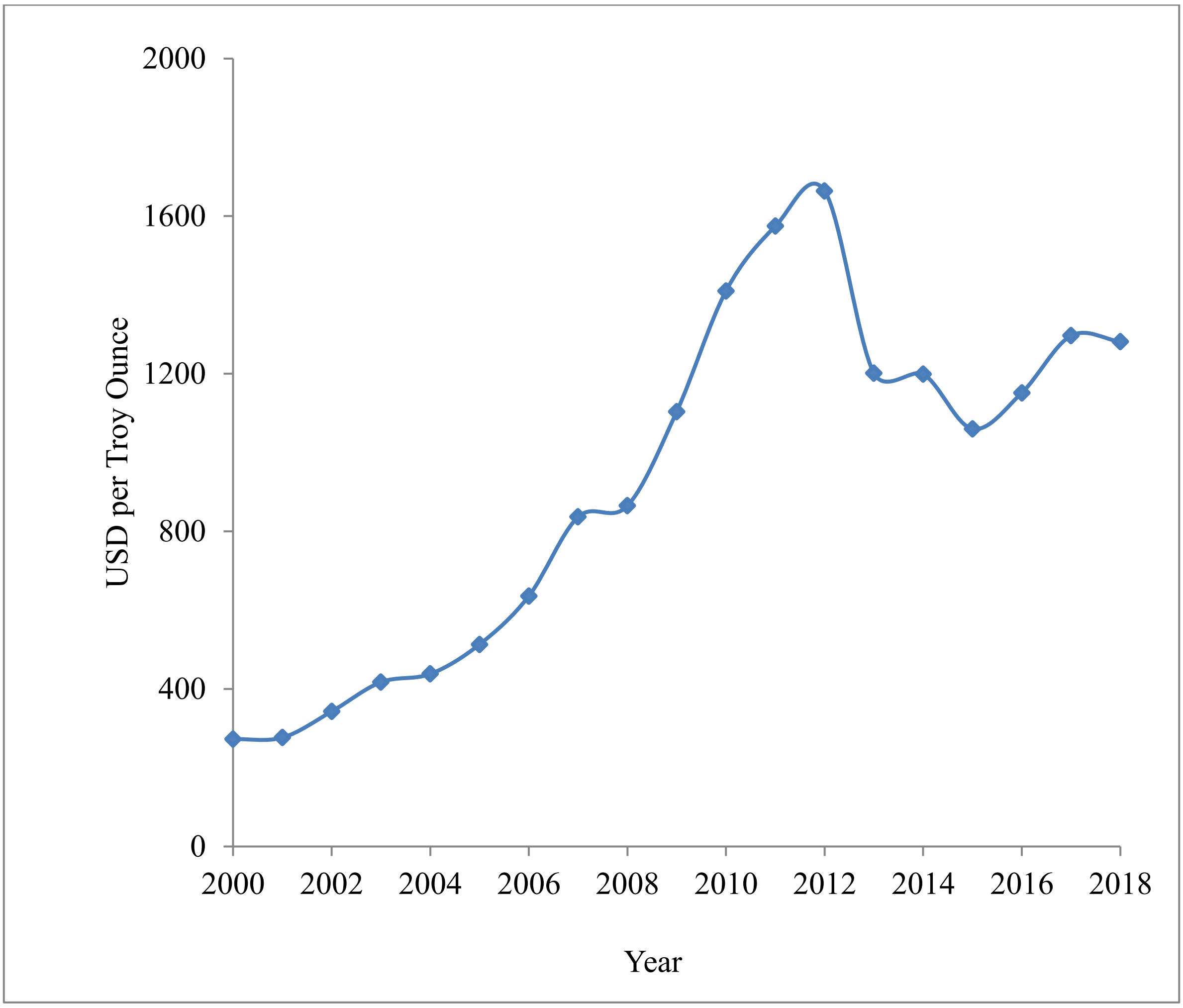


Figure 2. Evolution of gold price from 2000 to 2018 (URL-2, 2019)

Various factors such as the depth of the ore deposit, social consideration, location, safety, and environmental concerns determine the choice of the mining and extraction methods of gold. Open pit and underground mining are the common methods applied worldwide, the latter one being more costly. Leaching by cyanide is currently the principal technique employed for gold extraction from run-of-mine ores (ROM) (Marsden and House, 2006).

Table 1. Gold mining companies in Turkey (Madencilik Türkiye, 2018)

No	Location	Company	Start of Production
1	Ovacık	Koza	2001
2	Sart	Pomza	2002
3	Kışladağ	Tüprag	2006
4	Mastra	Koza	2009
5	Çöpler	Anagold	2010
6	Efemçukuru	Tüprag	2011
7	Kaymaz	Koza	2011
8	Bolkardağ/Tepeköy	Gümüştaş	2012
9	Midi	Yıldızbakır	2012
10	Himmetdede	Koza	2014
11	Altıntepe	Bahar	2015
12	Bakırtepe	Demirexport	2015
13	Kızıltepe	Zenit	2017
14	Konya İnlice	Esan	2017
15	Lapseki	Tümad	2018

2.2. Gold Ores

From a metallurgical frame of reference, gold ores can be widely grouped into refractory and free-milling ores based on their response to traditional cyanide leaching. The latter type of ores are fairly easy to process yielding acceptable Au recoveries (>90% with $d_{80} < 75\mu m$) in cyanide leaching. Whereas the refractory ores are problematic to process and offer poor Au recoveries of often much lower than 80%, sometimes even <50% (Celep et al., 2009; Alp et al., 2005; Marsden and House, 2006). There appear to be many reasons for refractoriness of gold ores. The most common and best understood is gold locked in sulfides such as pyrite and arsenopyrite in particular. Carbonaceous matter (i.e. organic and/or inorganic carbon) present in the ore is also a frequent cause of poor recovery because it readily adsorbs gold in solution and leads to preg-robbing phenomenon (Habashi, 1999; Marsden and House, 2006). Another reason for refractory behaviour is the presence of excessive cyanide and oxygen consuming phases i.e. reactive copper minerals, pyrrhotite, etc. Furthermore, gold can also occur as minerals (e.g. $AuTe_2$, $AuSb_2$ etc.), which are refractory to dissolution in cyanide leaching (Chryssoulis and McMullen, 2016) which affects recoveries even at low concentrations.

Basically, ore pretreatment is indispensable prior to leaching by cyanidation, this enhance gold recoveries from refractory ores. Figure 3 shows the pre-treatment techniques. The conditions such as ore, gold content, mineralogy, gold/sulfur ratio, amount of other minerals (cyanicides) that lead to high cyanide consumption, can determine the pre-treatment method to be selected (Alymore and Jaffer, 2012). Moreover, economic and environmental outcomes play a distinctive role in deciding these methods (Marsden and House, 2006). Table 2 shows some of the free milling and refractory gold/silver ores in Turkey.

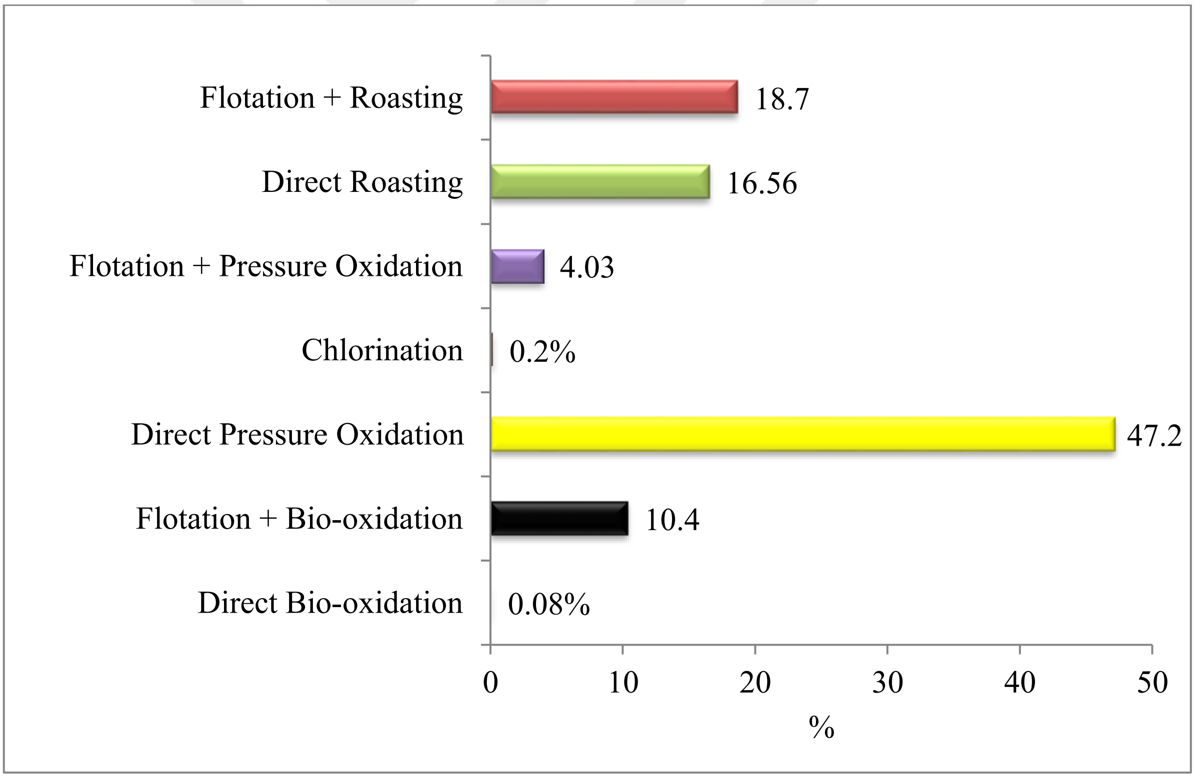


Figure 3. Pre-treatment and leaching methods applied to refractory type ores (Marsden and House, 2006)

Table 2. Free milling and refractory gold/silver ores in Turkey

Mine	**Au grade**	**Ag grade**	**Cu Content**	**Leaching Method**	**Class**
Bergama,Ovacık(İzmir)	9 gt^{-1} Au [a]	11 gt^{-1} Ag [a]	-	A.T.L.	FM
Küçükdere (Balıkesir)	5 gt^{-1} Au [a]	11.8 gt^{-1} Ag [a]	-	A.T.L.	FM
Kaymaz (Eskişehir)	7.9 gt^{-1} Au [a]	6.4 gt^{-1} Ag [a]	-	A.T.L.	FM
Kışladağ (Uşak)	1.2 gt^{-1} Au [a]	0.9 gt^{-1} Ag [a]	-	H.L.	FM
Mastra (Gümüşhane)	12.13 gt^{-1} Au [b,c]	5.5 gt^{-1} Ag [b,c]	<1.1% [b]	A.T.L.	FM&R
Çöpler (Erzincan)	1.4 gt^{-1} Au [d]	-	0.03%Cu (Some zones ≤ 3.9 Cu) [d]	H.L.	FM&R
Efemçukuru (İzmir)	13.3 gt^{-1} Au [e] 62 gt^{-1} Au [f]	110 gt^{-1} Ag [e]	0.35%Cu [f]	F. + H.L.	R
Gümüşhane köyü (Artvin)	0.3 gt^{-1} Au [e,g]	-	0.3% [e,g]	Under development	R
Cerattepe (Artvin)	4.2 gt^{-1} Au [e]	151 gt^{-1} Ag [e]	5.2% [e]	-	R
Hot Maden (Artvin)	10 gt^{-1} Au [h]	-	2.2% [h]	-	R
Gümüşköy (Kütahya)	-	127 gt^{-1} Ag [a]	-	A.T.L.	R

[a] Celep (2011); [b] Bas et al., (2012); [c] Koza Altin (2013); [d] Bascombe et al., (2013); [e] Yiğit (2006); [f] Ahlatcı et al., (2016); [g] Akçay ve Gündüz (2004); [h] Mariana Resources Ltd. (2016).

A.T.L.:Agitation tank leaching; H.L.:Heap leaching; F.:Flotation FM.: Free milling R.:Refractory

2.3. Diagnostic Leaching for Characterization of Gold Ores

An effective pre-treatment approach is often necessary to deal with refractory ores. As far as the economics of the recovery of gold is concerned, a suitable pre-treatment route must be employed (Lorenzen, 1995). In general aspects, diagnostic leaching offers a comprehension of the comportment of an ore. Usually it includes selective break-down of different phases of mineral and recover gold at the cyanidation stage following its liberation from the decomposed ore (Lorenzen and Van Deventer, 1992; Lorenzen,1995; Celep et al., 2008). Table 3 illustrates the procedures employed in diagnostic leaching.

The chief information concerning the characteristic of refractory ores can be studied by the use of this technique (Tumilty, 1992; Lorenzen and Van Deventer, 1992; Torres and Costa, 1995). This method gives a clear picture of how the decomposed phase of minerals affected the gold recoveries when standard direct cyanidation is performed (Henly et al., 1995). The main advantage of this technique is that an ore is evaluated quantitatively (Chryssoulis and Cabri,1990). The amount of gold which is encapsulated in other mineral phases present in the ore is vulnerable to be destroyed by a sequence of leaching by acids such as HCl, HNO_3, and HF (Lorenzen, 1995; Celep et al., 2013). Following acid treatment of the ore, direct cyanidation is done to recover free gold.

Van Deventer and Lorenzen (1993) applied diagnostic leaching to provide an insight into the refractory behaviour of gold ores. They found that less than 2% of the Au present was amenable to direct cyanidation and breakdown of Au bearing phases with HNO_3 & H_2SO_4 was required to render the remaining gold (above 95%) available for cyanide leaching. Celep et al. (2008) investigated the refractory character of Kaletaş ore via diagnostic approach, they found that 72% of gold was recoverable from non treated ore by direct cyanidation, however the latter recovery changed up to 100% when the same ore was treated with a series of acids (HCl, H_2SO_4, HNO_3, HF) .

An ore containing 0.9% pyrite, 5.9% pyrrhotite and 1.3% arsenopyrite with bulk concentrates originated from the experiments of flotation was investigated by Teague et al. (1998) with the aid of diagnostic leaching. The findings revealed that appreciable fraction of gold was locked inside the refractory pyrite (around 20%) and pyrrhotite (approximately 10%). That means the percentage of gold which was free in the concentrate was around 70%.

Table 3. Selective destruction of gold ores by diagnostic leaching (Lorenzen and Van Deventer, 1993)

Treatment	**Possible minerals to be destroyed**
NaCN	Gold
Na_2CO_3	Arsenates and Gypsum
HCl	Dolomite, Calcite, Galena, Geothite, Pyrhotite
$HCl/SnCl_2$	Ferrites, Hematite, Calcine
H_2SO_4	Labile pyrite, Cu-Zn sulphides
$FeCl_3$	Tetrahedrite, Labile Sulphides, Sphalerite
HNO_3	Arsenopyrite, Marcasite, Pyrite
Oxalic Acid Washes	Oxide Coatings Silicates
HF	Silicates
Acetonitrile elution	Gold adsorbed on carbon

2.4. Treatment of Refractory Gold Ores

2.4.1. Ultra-fine Grinding (UFG)

Various industries such as mining, metallurgy, ceramics, cement etc. employ the approach of ultrafine grinding for reducing particle size of different materials to their size of interest. Due to the rapid fall of easy bodies, complex ore bodies are encountered in most cases. Locked gold from refractory ores can be treated by this technique (Celep et al., 2015). In the time of ultrafine grinding, the breakage of particles occurs either by attrition or impact. The choice of equipment must consider the nature of reduction, as wet or dry. However, the most employed equipments are stirred media mills (Celep and Yazici, 2013).

Several researches have shown that physically locked gold are amenable to lixiviants after ultrafine grinding of gold ores less than 11 μm (Ellis, 2003). Though the operation is expensive (Ellis, 2003) it was shown to be still more economical than some oxidation processes (Bowell,1999; Saunders,1990; Frondel, 1938). Not all physically locked refractory gold ores are amenable to fine milling (Ellis, 2003). The selection of the most suitable method should be based on location, economic evaluation, and legislative issues (Ellis, 2003).

Celep and Yazici (2013) investigated the tailings of the silver plant from a refractory ore by the utilization of the UFG technique, the charge ratio of ball used was between fifty and eighty percent. The original tailing (as received) had a size of 100 µm (d_{80}), and when was leached by cyanidation only 36% was achieved. Notwithstanding, the recovery increased up to 84% when the same tailing was ground to 1.2 µm (d_{80}) prior to bringing into contact with cyanide solution. This difference of 48% suggested that UFG may be employed as a tool to treat ores which demonstrate refractory comportments. In addition their research results revealed that factors like size of the media, stirring speed (rpm), and ball charge ratio are very important in the determination of the product's fineness.

Celep et al. (2016) examined the effect of ultra fine grinding on a copper bearing pyritic gold concentrate, they pointed out that direct cyanidation consumed reagent exorbitantly with poor gold recoveries, however when the same concentrate was initially ground to 4.2 µm (d_{80}) and then leached by 5 g/L NaCN, a remarkable gold recovery of 98% was attained.

2.4.2. Bio-oxidation

It has been well established that gold extractions are low, when the route of its extraction from sulphide minerals is achieved by conventional cyanidation process (Bas et al., 2012; Celep et al., 2015). The refractory deportment of ores can be ascribed mainly to finely distribution and encapsulation of the gold as microcrystalline particles within sulphides such as pyrite and arsenopyrite in particular (Marsden and House, 2006). Pretreatment of pyritic/arsenopyritic gold ores via oxidation is vital to enhance the accessibility of gold to the lixiviant action of cyanide in subsequent cyanidation process (Amankwah, 2005). Gold locked inside the matrix of sulfides is exposed out when gold bearing arsenopyrites, pyrite, and arsenian-pyrite phases are oxidized by microorganisms (Brierley, 1999; Marais, 2008). Following the oxidation process, a conventional cyanidation process is then performed.

Mesophilic bacteria such as *Leptospirillum ferrooxidans, Acidithiobacillus ferrooxidans* and *Acidithiobacillus thiooxidans* which are these chemolithotropic bacterias are dependent on sulfur and/or iron as their source of energy are most commonly used microorganisms in the commercial biooxidation processes (Deveci, 2001; Deveci, 2002; Deveci, 2004; Akcil et al., 2007; Akcil and Deveci, 2010; Akcil et al., 2013; Ndlovu, 2009). These bacterias oxidize the matrices of sulfide ore, setting free the entrapped gold and removing interfering ore constituents. The microorganisms can be directly attached to mineral surface and oxidize the phases of sulfide releasing sulfuric acid or oxidize ferrous iron into ferric iron, which moderates oxidation of sulphides. Figure 4 illustrates the mechanism for sulfide oxidation by bacteria (Marsden and House, 2006). The bacteria are also competent of leaching or solubilizing metals, such as manganese, nickel, zinc, and copper, from their sulfides (Deveci, 2001; Deveci et al., 2003 and 2008; Bailey, 1993). The bacteria play essentially a secondary role in leaching of sulfide mineral by (re)generating ferric iron as oxidant in required quantities for effective oxidation of sulphide minerals as well as by removing the encrusted sulfur on mineral surface converting it into sulfate/acid (Deveci et al., 2003). The general oxidation reactions for iron sulfide mineral (FeS_x) in acidic media are given as follows (Marsden and House, 2006).

$$FeS_x+xO_2+2xH_2O \rightarrow FeSO_4+4XH^{+}+(X-1)SO_4^{2-}+(2X+2)e \tag{1}$$

$$4FeSO_4+O_2+2H_2SO_4 \rightarrow Fe_2(SO_4)_3+2H_2O \tag{2}$$

$$FeS_x+Fe_2(SO_4)_3 \rightarrow 3FeSO_4+xS \tag{3}$$

$$2S+3O_2+2H_2O \rightarrow 2H_2SO_4 \tag{4}$$

The reaction chemistry for arsenopyrite is as follows

$$FeAsS+13O_2+6H_2O \rightarrow 4H_3AsO_4+4FeSO_4 \tag{5}$$

$$2FeAsS+7O_2+H_2SO_4+2H_2O \rightarrow 2H_3AsO_4+Fe_2(SO_4)_3 \tag{6}$$

$$2FeAsS+Fe_2(SO_4)_3+6O_2+4H_2O \rightarrow 2H_3AsO_4+H_2SO_4+4FeSO_4 \tag{7}$$

The reaction chemistry for chalcopyrite is as follows

$$CuFeS_2+O_2+2H_2SO_4 \rightarrow CuSO_4+Fe_2SO_4+2S+2H_2O \tag{8}$$

$$4CuFeS_2+17O_2+2H_2SO_4 \rightarrow 4CuSO_4+2Fe_2(SO_4)_3+2H_2O \tag{9}$$

$$CuFeS_2+2Fe_2(SO_4)_3 \rightarrow CuSO_4+5FeSO_4+2S \tag{10}$$

The potential challenges that bacterial oxidation poses are as follows (LaBrooy et al., 1994)

- The oxidation rate is slow e.g. days required for achieving the desired level of sulfide oxidation
- Meticulous control of temperature is required
- Corrosion due to low pH operating conditions
- Potential adverse effect of dissolved organic residue from microorganisms on carbon fouling
- Foaming problems may occur.

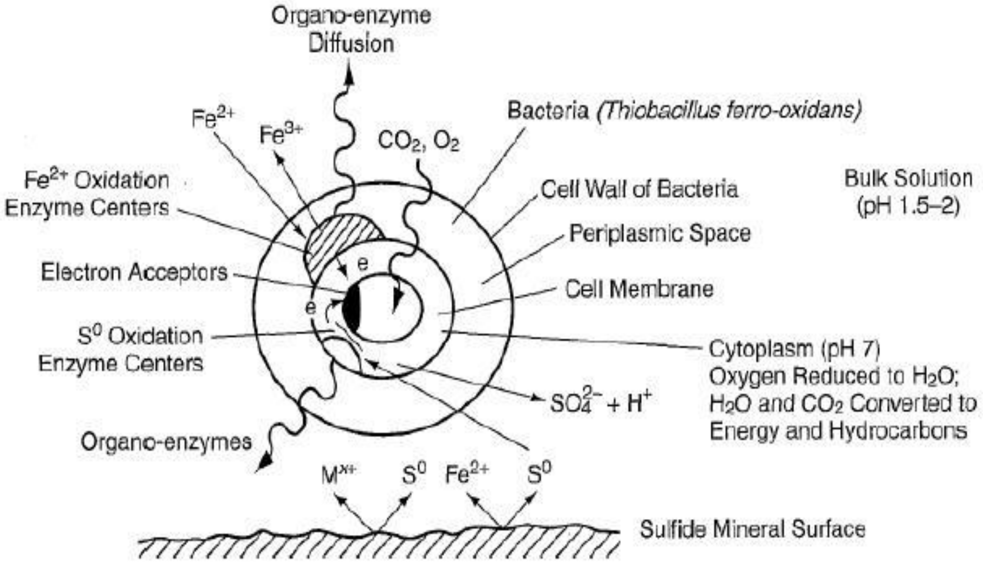


Figure 4. The simplified mechanism for bacteria-catalyzed sulfide oxidation (Marsden and House , 2006)

2.4.3. Pressure Oxidation

Even after fine grinding of the ore (i.e. -75 µm) gold recoveries less than 80% in cyanide leaching indicate refractory ore although a clear boarder between refractory and non-refractory ore in term of gold recovery is not defined (Fleming, 1992). The pressure oxidation destructs gold bearing phases with concomitant exposure of the occluded gold to subsequent cyanidation of refractory ores. Pressure oxidation (POX) process involves oxidation of sulfides to sulfates with oxygen at high pressures and temperatures (180-230°C) (Thomas and Pearson, 2016). Figure 5 demonstrates process options for high-pressure oxidation.

High-temperature POX was initially carried out at the Homestake McLaughlin plant in California in 1985 (Thomas, 2005). At this plant 3 autoclaves (4.2 x 16.2 m) were used to oxidize 2700 tons of low pyritic sulfur containing ore per day at 180°C. This was the first industrial application of pressure oxidation on ore. The first application of pressure oxidation of flotation concentrate started at the end of 1986 at Sao Bento Mineracao plant in Brazil (Fraser et al., 1991). Sherritt-Gordon Company offers the technical know-how of this technique.

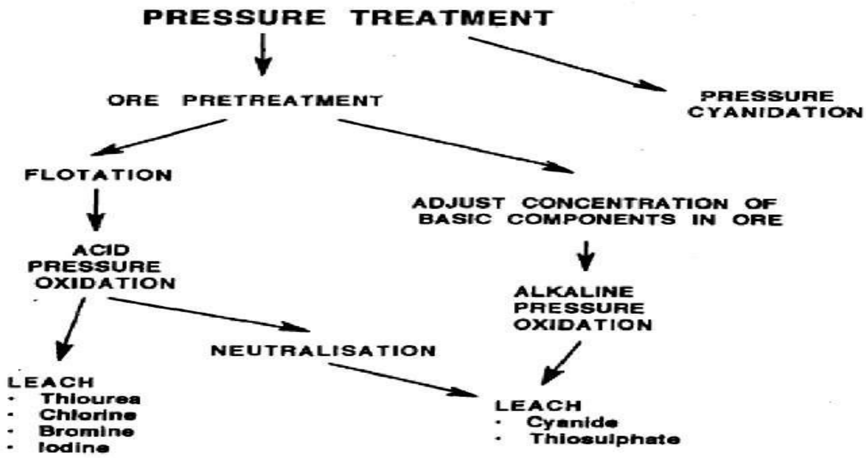


Figure 5. Process options for pressure pre-treatment leaching of refractory materials (La Brooy et al ., 1994)

POX is a commercially proven, mature technology and well suited to ores/concentrates that need extensive sulfide oxidation (La Brooy et al., 1994). However, the industrial application of this technology is often impeded by its high operating and capital costs (Iglesias and Carranza, 1994). Autoclaves are used for pressure oxidation, which is often carried out using sulphuric acid and oxygen at a temperature which ranges from 180°C to 230 °C, (pressure 1800-2200 kPa) with the leaching period between 1- 3 hours (Komnitsas and Pooley, 1989; Thomas and Pearson, 2016). The main reactions that take place for oxidation of arsenopyrite and pyrite are as follows:

$$2FeS_2 + 7O_2 + 2H_2O \rightarrow 2FeSO_4 + 2H_2SO_4 \tag{11}$$

$$4FeAsS + 11O_2 + 2H_2O \rightarrow 4HAsO_2 + 4FeSO_4 \tag{12}$$

$4FeSO_4 + 2H_2SO_4 + O_2 \rightarrow 2Fe_2(SO_4)_3 + 2H_2O$ (13)

$4HAsO_2 + Fe_2(SO_4)_3 + O_2 + 2H_2O \rightarrow 2FeAsO_4 + 3H_2SO_4$ (14)

Temperature and acidity have a major influence on the whole mechanism of leaching by pressure oxidation (Komnitsas and Pooley, 1989; La Brooy et al., 1994; Marsden and House, 2006). High acidity promotes sulfide oxidation and controls the formation of jarosite and basic iron sulphates (Komnitsas and Pooley, 1989; Fleming, 2010). Low temperatures promote the formation of elemental sulfur. Formation of jarosite, basic iron sulphates and elemental sulphur adversely affect gold and silver extraction in cyanidation (Fleming, 2010; Thomas and Pearson, 2016).

2.4.4. Roasting

Roasting is applied under supply of air to remove volatile matter (Maycock et al., 2007). Roasting is the most commonly employed technique for pretreatment of double refractory gold ores (Paktunc et al., 2006). Not mechanical loss by draught (URL-9, 2012) Table 4. below shows comparison of roasting with other pretreatment methods for refractory gold ores (Charitos et al., 2013). Through roasting the sulfide matrix (host sulfide minerals) is decomposed to liberate the encapsulated submicroscopic gold (Taylor, 2013; Marsden and House, 2006; Fraser et al., 1991; Paktunc et al., 2006; Charitos et al., 2013; Runkel and Sturm, 2009). In roasting, gold bearing iron sulphides are transformed into a porous iron oxide, which allows the penetration of cyanide to react with gold grains (Komnitsas and Pooley, 1989). Sulphides oxidation involve the release of energy, the contained sulphides serve as fuel source for roasting (Charitos et al., 2013). Autogeneous roasting may occur when sulphide content of the feed is above 15% (Thomas and Cole, 2016). In addition, roasting effectively oxidizes any organic/inorganic carbon present in carbonaceous (pregrobbing) refractory ores (Marsden and House, 2006; Afenya, 1991; Goode, 1993; Brittan, 1995). This makes roasting an efficacious pretreatment process for double refractory gold ores. The efficiency of roasting is strongly dependent on various factors including temperature, partial pressure of oxygen and particle size (Taylor, 2013; Marsden and House, 2006; Amankwah et al., 2005; Paper and Aylmore, 2013; Afenya, 1991; Fraser et al., 1991; Paktunc, 2006; Neuvonen, 2013). For the recovery of gold to be optimum, a fairly inert and porous calcine is recommendable (Alymore and de Klerk, 2013). Once a calcine product is inert, the cyanide lixiviant can easily access the gold for recovery (Robinson, 1988; Marsden and House, 2006; Aylmore and de Klerk, 2013). This calcine can be achieved by typical roasting conditions such as pressure of oxidizing gas, particle size, roasting temperature and sample composition (Aylmore and de Klerk, 2013). Depending upon the comportment of the ore, roasting is carried out by the use of a configuration of either two stage or single roaster. The roasting temperature of 650°C to 700°C is proposed (Robinson, 1988). The rate of oxidation of sulphides and volatilisation of arsenic minerals tends to improve with the increase of partial pressure of oxygen and temperature in the gas phase (Paper and Aylmore, 2013). The amenability of the calcine product to cyanide leaching is often regarded as a measure of the roasting efficiency (Marsden and House, 2006; Paper and Aylmore, 2013; Paktunc, 2006; Brooy et al., 1994). Marsden and House (2006) reported that during the roasting of gold-bearing sulfides, the gold tends to drift towards pores or grain boundaries where it coalesces in the liquid phase. Further coalescence may also takes place on the mineral surface due to the volatilization of arsenic and sulfur forming gold particles up to 1 µm in diameter (Marsden and House, 2006).

During roasting of refractory gold ores, loss of gold may be experienced through volatilization given that the feed contains chloride and fluoride.The loss may also be attributed by the deposition of gold inside the roaster itself (Marsden and House, 2006). It commonly ranges from 2% to 5%. In 1856, Plattner pointed out that during roasting of standard auriferous pyrites, gold loss may occur given that the process is done so swiftly that via the draught, the contained fine particles are mechanically carried off. This inference, with regard to arsenides and sulphides has been endorsed by Kustel. A 20% of gold loss was recorded by Kustel when roasting of some tellurides of gold and silver was carried out. He pointed out as the loss was due to volatilization and not mechanical loss by draught (URL-9, 2012).

Table 4 . Attributes of different refractory ore-handling technologies (Charitos et al., 2013)

	Roasting	**POX**	**BIOX**	**UFG**
Energy recovery	Excess heat of reaction can be recovered	Oxygen generation is energy intensive.	Low energy consumption, no heat recovery.	No heat recovery, energy consumption depending on the extent of milling
Sulfur	S converted into SO_2 gas or partial sulfidation	S converted into H_2SO_4	S partially converted into sulfate	Virtually no oxidisation
Carbon (Lignite, Anthracite)	Oxidation >700°C to CO_2	No reaction	No reaction	No impact
Arsenic	Oxidation and separation as arsenic trioxide	Oxidation in slurry	Oxidation in slurry	No impact
Mercury	Almost complete volatilization, Gas cleaning for Hg with the Boliden Norzink Process, Almost Hg-free calcine	Majority remains in solids	Remains essentially in solids phase	No impact
Residence time	0.5 h	1h	4-5 days	Not applicable
CN Consumpti-on	1-2 kg/ton	1-2 kg/ton	10-20 kg/ton	No comparison available

2.5. Gold-Copper Ores

In gold ores, copper can be present at high levels (Adams, 2016). As of 2009, above 20% of the total world's Au production was from Cu-Au ores (Fleming, 2011). Porphyry copper deposits are essentially one of the basic types of mineral body containing gold. The typical gold and copper grade in these deposits are 0.2-2 g/ton and 0.2-1% respectively (Fleming, 2011; Kesler et al., 2002; Sinclair, 2007). Porphyry deposits alone was reported to account for approximately 10% of total gold production (Sinclair, 2007). There are also many copper-rich gold ore deposits exploited in Turkey (Yiğit, 2006). Table 5 presents metal grades and applied leaching methods of some Au-Cu ores in Turkey.

Table 5. Metal grades and applied leaching methods of some Au-Cu ores in Turkey

Mine/Location	Grade	Extraction Method	Source
Çöpler (Çukurdere)/Erzincan	1,4 g/ton Au [1] 0.03% Cu (Some zones up to 3.9% Cu)	Heap Leaching	Bascombe et al., (2013)
Efemçukuru/İzmir	13.3 $gton^{-1}$ Au [2] 61.5 $gton^{-1}$ Au [3] 110 g/ton Ag [3] 0.35% Cu [3]	Flotation + Leaching	Yiğit (2006)
Mastra/Gümüşhane	12 g/ton Au [2] 5.5 g/ton Ag [2] <1.1% Cu [3]	Tank Leaching	Bas et al., (2012) Koza Altın (2013)
Cerattepe/Artvin	4.2 g/ton Au [2] 151 g/ton Ag [2] 5.2% Cu [2]	-	Yiğit (2006)
Gümüşhane/Artvin	0.3 g/ton Au [1] 0.3% Cu [1]	In the project phase	Akçay & Gündüz (2004) Yiğit (2006)

1 Source [2] Reserve [3] Grades of flotation concentrates (Celep et al., 2013)

2.5.1. Technical Difficulties In The Extraction of Gold From Cu-Rich Ores

High reagent consumption is the major impediment to the treatment of gold ores, which contain significant levels of cyanide-soluble copper. This excessive consumption of cyanide (Eqs. (15)−(19)) is attributed by the formation of stable copper cyanide complexes. Au extraction from copper bearing ores is often unacceptably low when unsufficient level of cyanide is used (Marsden and House, 2006; Koç et al., 2014; Yazıcı et al., 2015). Approximately thirty kilograms of NaCN is consumed per 1 ton of an ore for each 1% cyanide soluble copper, which negatively influences the process economics (Muir et al., 1991). The cyanide consumption can increase up to 51.5 kg/ton as a result of thiocyanate formation (Eqs. (17) and (19)) and cyanate (Eq. (18)) as well when copper is present as sulphide such as covellite (Sceresini, 2005). Cyanide leaching of gold ores containing >0.5% Cu are often uneconomic (Muir et al., 1991).

$$Cu^{2+}+2CN^{-}\rightarrow Cu(CN)_2 \tag{15}$$

$$Cu_2O+6CN^{-}+H2O\rightarrow 2Cu(CN)_3^{2-}+2OH^{-} \tag{16}$$

$$Cu_2S+7CN^{-}+1/2O_2+H2O\rightarrow 2Cu(CN)_3^{2-}+2OH^{-}+CNS^{-} \tag{17}$$

$$2CuO+7CN^{-}+H_2O\rightarrow 2Cu(CN)_3^{2-}+2OH^{-}+CNO^{-} \tag{18}$$

$$2CuS+8CN^{-}+1/2O2+H_2O\rightarrow 2Cu(CN)_3^{2-}+2OH^{-}+2CNS^{-} \tag{19}$$

As far as the process economy of the treatment of a copper-rich gold ore is concerned, an invention of viable alternate method to cyanidation is highly needed (Bulatovic, 1998). In connection with the point previously mentioned, various options and strategies including flotation (Bulatovic, 1998; Forrest et al., 2001), different lixiviant systems like ammoniacal cyanide or thiosulphate, copper leaching before cyanidation (Bas et al., 2012), cyanide recovery from effluents (e.g. the process of SART) (Dai et al., 2012), the introduction of air before the cyanidation , and addition of $Pb(NO_3)_2$ (Deschenes et al., 2012) have been suggested/tested for the treatment of copper-rich gold ores. Lead nitrate addition or pre-aeration could alleviate complications affiliated with copper sulphides during gold cyanidation (Deschenes et al., 2012). Also, reactive copper can be removed by leaching with ammonia and sulphuric acid before the cyanide leaching of gold (Bas et al., 2012; 2015). In comparison with acid leaching, high selectivity over gang minerals, application in alkaline condition and low corrosivity are the advantages possesed by ammonia leaching

Table 6 . Solubility of copper minerals in 0.1% NaCN solutions (Marsden and House, 2006)

Mineral	Formula	%Cu dissolved	%Cu dissolved
		23 °C	25 °C
Azurite	$Cu_3(CO_3)_2(OH)_2$	94.5	100.0
Malachite	$Cu_2CO_3(OH)_2$	90.2	100.0
Chalcocite	Cu_2S	90.2	100.0
Covellite	CuS	-	-
Native Copper	Cu	90	100.0
Cuprite	Cu_2O	85.5	100.0
Bornite	Cu_5FeS_4	70	100.0
Chalcopyrite	$CuFeS_2$	5.6	8.2
Tetrahedrite	$(Cu,Fe,Ag,Zn)_{12}Sb_4S_{13}$	21.9	43.7

Most copper minerals are eminently soluble in cyanide solutions (Table 6) (Marsden and House, 2006). Dissolution of minerals of copper present interferes with cyanide leaching of gold leading to low gold extractions under conventional cyanide leaching conditions (0.5-1.5 g/L NaCN) (Sceresini, 2005; Bas et al., 2015). Head et al. (2015) reported that gold extraction could be substantially improved from <10% in standard leaching conditions (1.5 g/L NaCN) to ~99% by raising the concentration of cyanide to 7.5 g/L NaCN. Copper cyanide complexes formation; $Cu(CN)_2^-$, $Cu(CN)_3^{2-}$ and $Cu(CN)_4^{3-}$ in the liquid phase causes the depletion of free CN present for leaching of gold (Figure 6)

(Marsden and House, 2006). Therefore, high gold extractions can be possible only at sufficiently high levels of cyanide.

The Pourbaix diagram for the "Cu-CN-H_2O" system depicted in Figure 7 shows that under conventional conditions for Au cyanidation (pH 10-11, Eh -0.2 − 0.2 V, excess of free cyanide), copper is available predominantly as the charged copper cyanide complexes of $Cu(CN)_3^{2-}$ and $Cu(CN)_4^{3-}$. Nonetheless, speciation of copper in cyanide solutions depends essentially on pH and cyanide to copper ratio (Bas et al., 2015; Breuer et al ., 2007) .

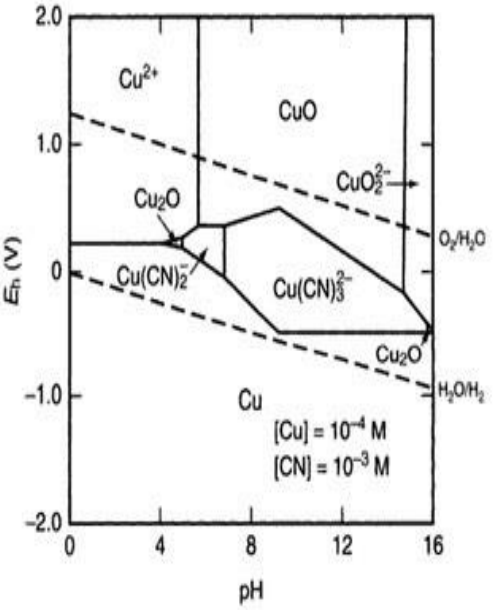


Figure 6 . Eh-pH diagram for the CU-CN-H_2O system at 25°C (Marsden and House, 2006)

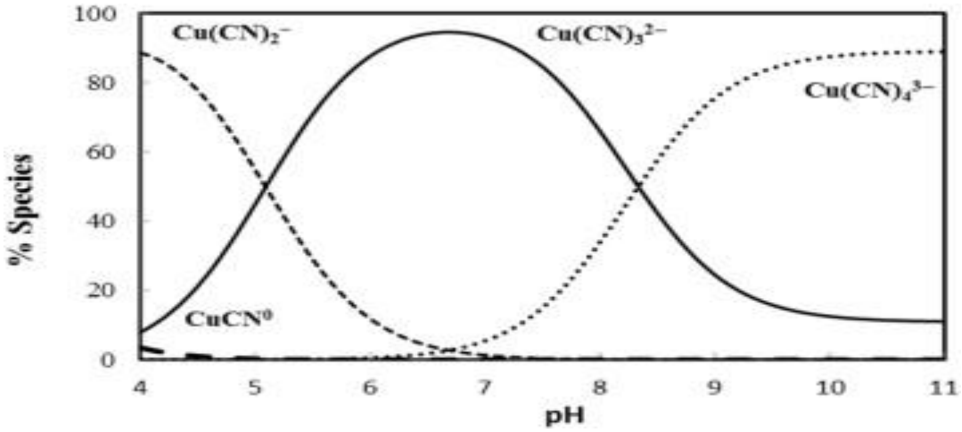


Figure 7. Species distribution for the Cu(I)-CN$^-$ system as a function of pH in 1M Cu$^+$ at 25°C (Breuer et al., 2007)

2.6. Cyanide Leaching

Since the beginning of 20th century, the extraction of Au or Ag from ores is done mostly by the use of cyanide. This process is done in an alkaline media where by the primary condition for the gold dissolution is the oxidation of gold by oxygen. The oxidized Au is dissolved via the formation of the stable complex Au $(CN)_2^-$. In addition to $Au(CN)_2^-$, there is also Au(III)-cyanide complex ($Au(CN)_4^-$), but, it is not stable under cyanide leaching conditions (Marsden and House, 2006). Figure 10 illustrates Eh-pH diagram for the Au-CN-H_2O system and stability region of Au $(CN)_2^-$ at 25°C. H_2O_2 which is formed as an intermediate product through the reduction of oxygen in the first phase (Eq. 20), is employed as an oxidizing agent in the second phase (Eq. 21) (Marsden and House, 1992; Kondos et al, 1995; Ling et al., 1996; de Andrade Lima and Hodouin, 2005; Senanayake, 2004). The following reactions represent the mechanism of gold dissolution.

$$2Au + 4CN^- + O_2 + 2H_2O \rightarrow 2Au(CN)_2^- + H_2O_2 + 2OH^- \qquad (20)$$

$$2Au + 4CN^- + H_2O_2 \rightarrow 2Au(CN)_2^- + 2OH^- \qquad (21)$$

The overall reaction as proposed by Elsner is as follows:

$$4Au + 8CN^- + O_2 + 2H_2O \rightarrow 4Au(CN)_2^- + 4OH^- \qquad (22)$$

Lime is employed to control the desired level of pH (10.5-11). In cyanide leaching pH is maintained above 10.5 to ensure 95% of the cyanide ions present in the medium to be in the free cyanide (CN^-) form. However, in highly alkaline conditions (>pH 11), the dissolution of Au and Ag in the cyanide solution is adversely affected (Figure 8). The free CN concentration is an aggregate of the CN^- and HCN concentrations; the latter has no lixiviant action on gold and silver. Cyanide is principally present as HCN at pH values below 7 where by following its high vapor pressure, it is readily volatilized. The equilibrium point is shifted in favor of cyanide ion formation at pH values above 7 (Figure 9).

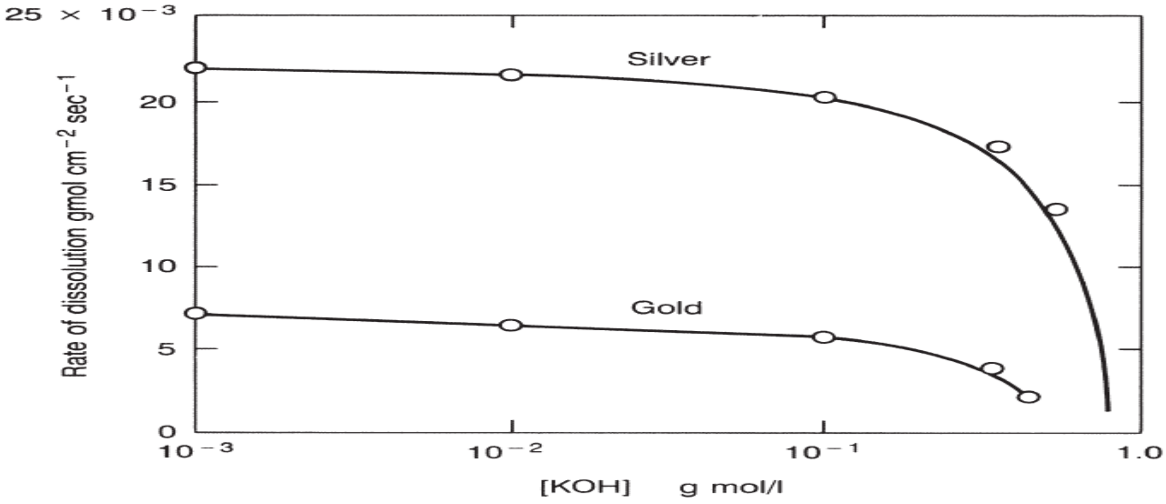


Figure 8. Effect of pH on the rate of dissolution of gold and silver in KOH (Kakovskii and Kholmanskikh, 1960

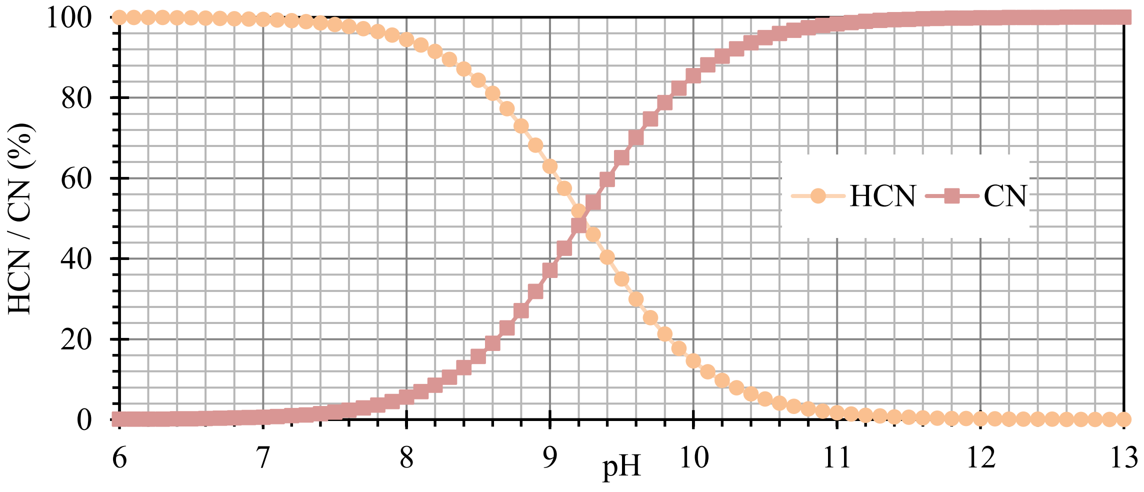


Figure 9. Equilibrium distribution for cyanide as a function of pH

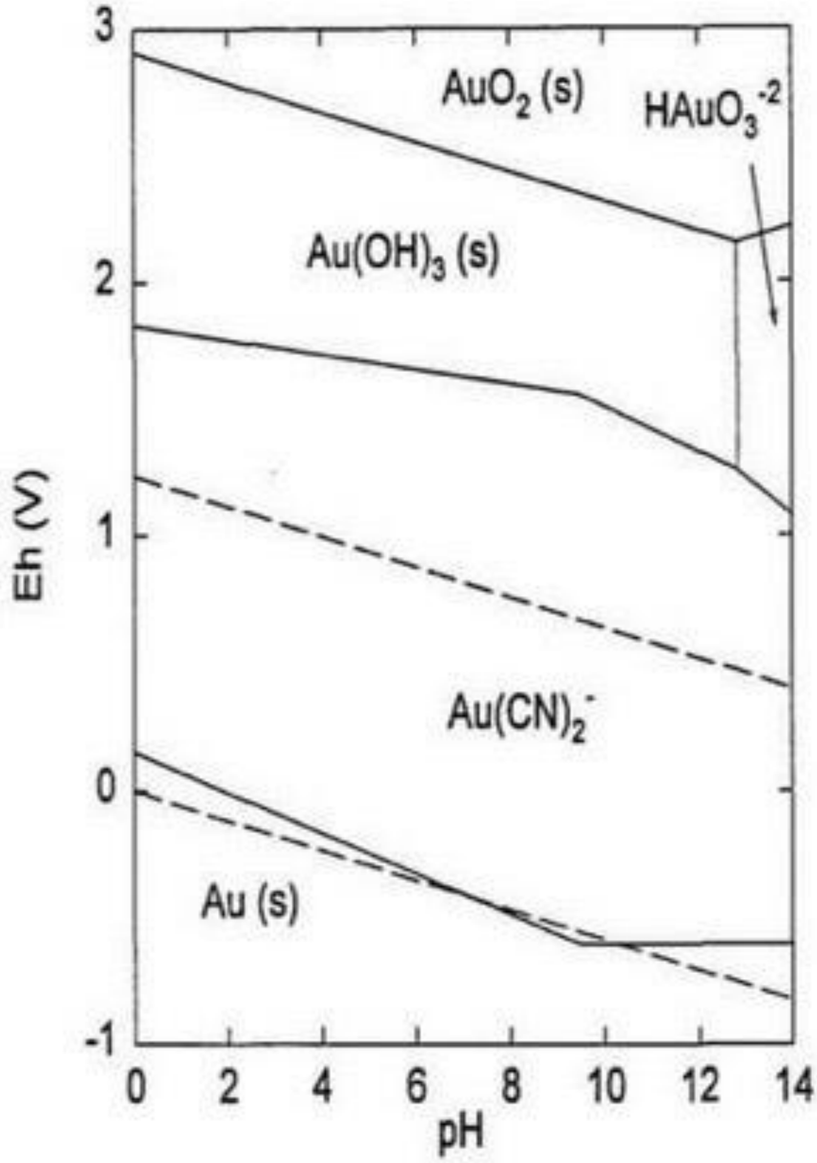


Figure 10. Eh-pH diagram for the Au-CN-H_2O system at 25°C, assuming an activity of 10^{-5} for all dissolved gold species and a cyanide activity of 4×10^{-2} (Zhang, 2005 ; Marsden and House, 2006)

2.7. Gold Recovery from Pregnant Leach Solutions (PLS)

The main gold recovery techniques from pregnant leach solutions (PLS) employed around the globe are ion exchange resins, activated carbon adsorption, and Merrill Crowe process.

2.7.1. Activated Carbon Processes

Eversince 1847 the technique of gold adsorption from aqueous liquors onto activated carbon has evidently been within the scope of knowledge (Fleming, 1992). From the beginning of 1970s the CIL and CIP have been broadly employed as the continuous processes for gold adsorption (Lima, 2002). These circuits (CIP & CIL) save operating and capital expenses of solid-liquid separation (Marsden and House, 1992). Figure 11 illustrates that CIP and CIL processes are the most important methods for the recovery of gold in use globally, making an aggregate of 44% of total global production (Marsden and House, 1992). Other techniques used are around 30% for Zn cementation and liquid-solid separation (CCL & Zn) and 19% for gravity concentration and flotation (MISC).

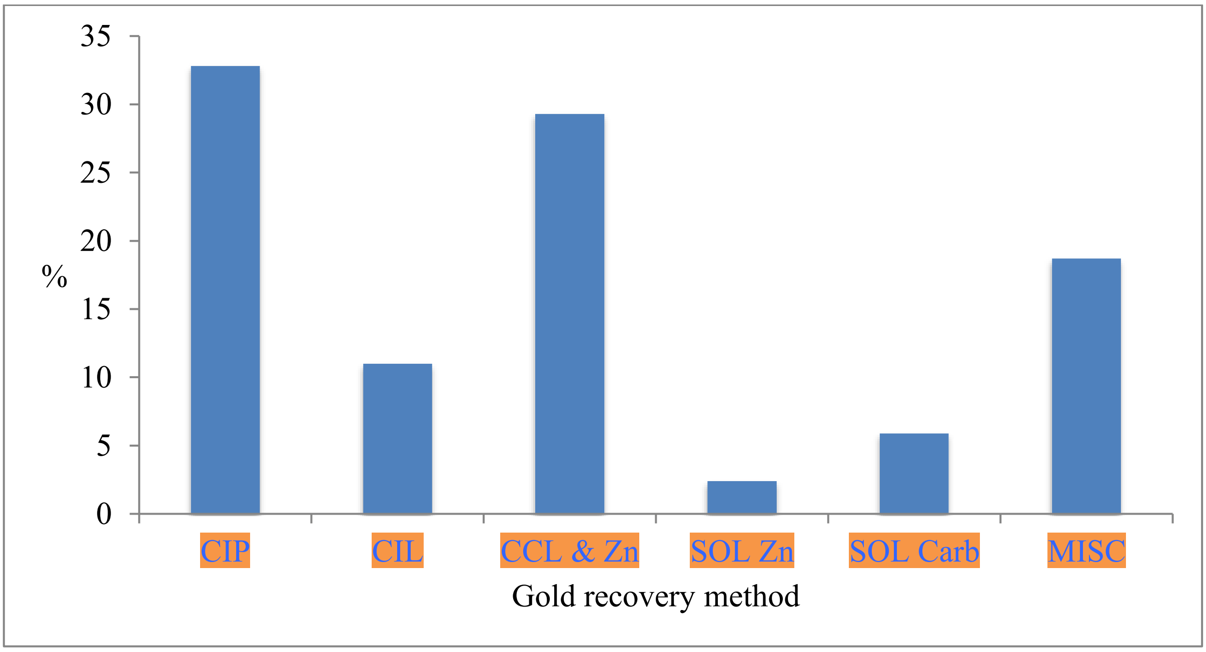


Figure 11. Gold production globally by recovery method (Marsden and House, 2006)

2.7.1.1. Carbon-in-pulp (CIP) Process

Recently, CIP is regarded as the most considerable advance in the technology of gold recovery. Countries like Australia, Canada and South Africa have paid an incredible attention to the simulation of this process (de Andrade Lima, 2007). It involves the recovery or concentration of metal through adsorption onto activated carbon (Marsden and House, 2006). In this process, within the agitated tanks the adsorption and leaching phases dont take place concurrently (Fleming, 2004). Prior to the commencement of the process, carbon (around -8 to +20 mesh) and lixiviant cyanide are introduced into the tanks. Carbon is conveyed from one tank to another in an opposite direction to ore pulp. When carbon is loaded by aurocyanide ion, it is sent out into the circuit of adsorption prior to being sent to the column of elution. CIP is used for recovery of gold from ores exhibiting various comportment (de Andrade Lima, 2007). A typical modern CIP plant is depicted in Figure 12. Earlier studies have pointed out the main parameters influencing the extent of adsorption of gold onto carbon. These include carbon type and its size, temperature, pH, mixing efficiency, the slurry density, cyanide concentration, gold concentration in solution, carbon fouling, dissolved oxygen, concentration of other metals and ionic strength (Fleming, 2004; Marsden and House, 2006).

Claus and Weiss (1977) studied the gold cyanide adsorption on different activated carbons, they came with a conclusion that the surface of carbon does not play a role during the adsorption of gold, because some of the carbon treatment processed chemically (e.g. with a mixture of HCl and HNO_3 at 80°C) hamper the process of gold adsorption. According to the literature of previous studies, the rate of adsorption of gold can be reduced when the pulp contains metals such as Ag, Ni, and Cu (Sayiner and Acarkan, 2014). When the silver to gold concentration ratio reaches 2:1, silver also impeded the adsorption of gold on activated carbon (Adams, 1992).

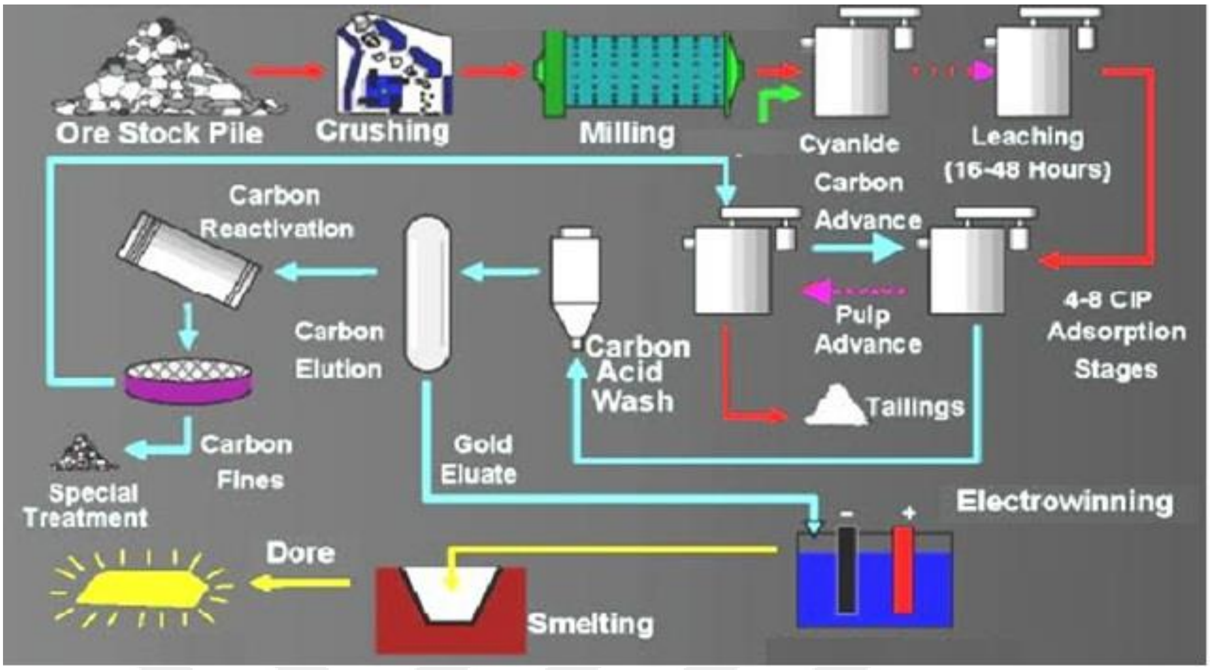


Figure 12 . Flow sheet of a modern CIP plant (Fleming , 2004)

2.7.1.2. Carbon-in-Leach (CIL) Process

The CIL process mainly started in Australia and South Africa in 1980s and then spread rapidly to all zones with gold mines across the globe (Fleming et al., 2011). Standard CIL process operates as the same as the CIP process except that in this process the adsorption and leaching take place concurrently (Marsden and House, 2006; Van Deventer et al., 1999; Ibragimova et al., 2007). The addition of carbon in to leach tanks is to allow the gold adsorption onto carbon almost as shortly as the cyanide lixiviant solution comes into contact with it (Marsden and House, 2006). This method is largely employed for the treatment of ores containing preg-robbing species and organic matter that are capable of adsorbing complexes of aurocyanide anion (Rees and Van Deventer, 2000; Marsden and House, 2006). In this approach, the amount of Au recovered is always lower than the CIP process. The main reason is that the carbonaceous matters reduce the gold yield through adsorbing gold meant for the carbon (Marsden and House, 2006). Figure 13 demonstrates the carbon in leach (CIL) process. Carbon attrition is a well-known source of gold loss in this process. Also carbon inventory is always larger than that in CIP.

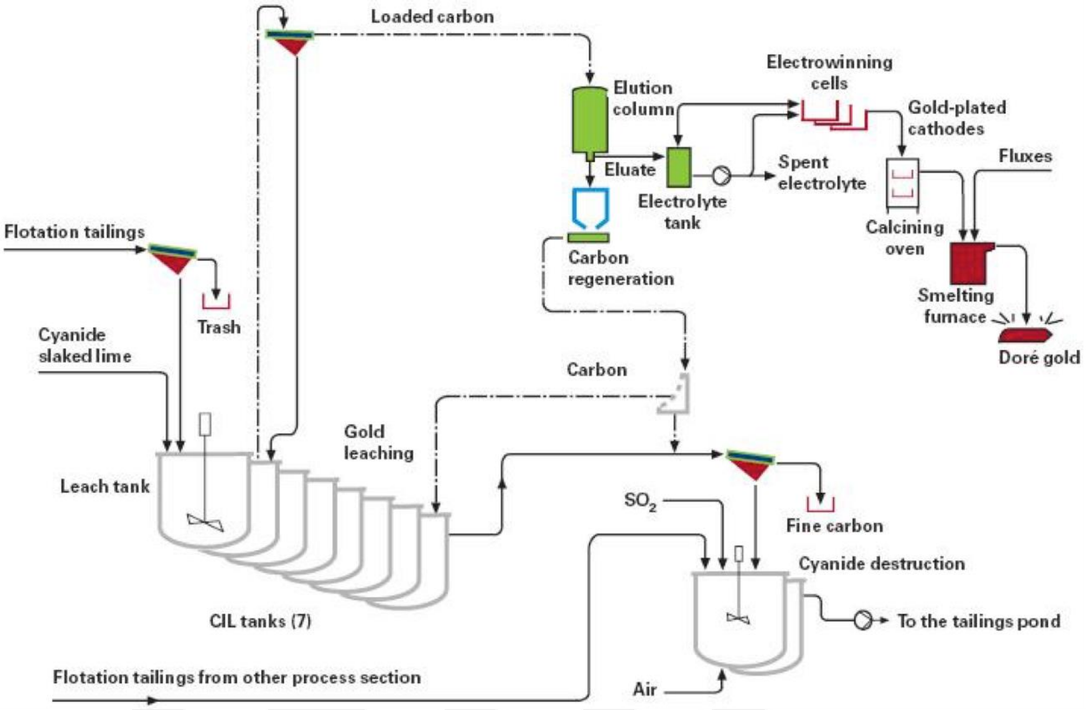


Figure 13 . The carbon in leach process (Outokumpu, 2004)

Table 7 . Advantages and disadvantages of carbon adsorption processes (Marsden and House, 2006)

Advantages	Disadvantages
Pregnant liquor pretreatment is not required	When the pregnant liquor contains high levels of silver, high carbon movement may be experienced.
It can handle carbonaceous and muddy ores	Carbon is vulnerable to fouling by magnesium and calcium salts
Very promising recoveries, regardless of the concentration of precious metal coming in	The stripping and regeneration of carbon is labor intensive

2.7.2. Merrill Crowe Process (Zinc Cementation)

The precipitation of silver and gold metals from pregnant leach solutions by the employment of zinc cementation approach has been well known eversince 1888 (Marsden and House, 2006). Zn cementation is the most frequently used cementation process for the gold recovery. Oxidation and reduction reactions are involved in this process. Gold recovered from cyanide leachates by Zn cementation is always high ranging from 98% to 99% (Fleming, 1992; Marsden and House, 2006). The chemistry of zinc precipitation depends mostly on the fact that silver and gold metals are more noble than zinc (Nicol et al., 1979). This means that they are reduced to their native states (i.e., Au^0 and Ag^0) from gold/silver-cyanide complex. The cementation reaction may be represented as shown in the equation below (Zadra et a1., 1952) .

$$Zn + 2Au(CN)_2^- \rightarrow Zn(CN)_4^{2-} + 2Au^0 \qquad (23)$$

When the pregnant leach solution (PLS) contains an ample amount of silver compared to gold (i.e. [Ag]/[Au] >3) the process becomes more effective (Marsden and House, 2006). Various metallic ions have an adverse effects on Zn cementation. The most problematic ones are those of arsenic and antimony (Nicol et al., 1979). This approach is also conducive for the pregnant solutions containing an appreciable amount of mercury content, high organic content and high flotation reagents. Nonetheless, just after grinding operations, the solid to liquid separation stage in this process is expensive and here its most important disadvantage compared to adsorption process via activated carbon (Marsden and House, 2006).

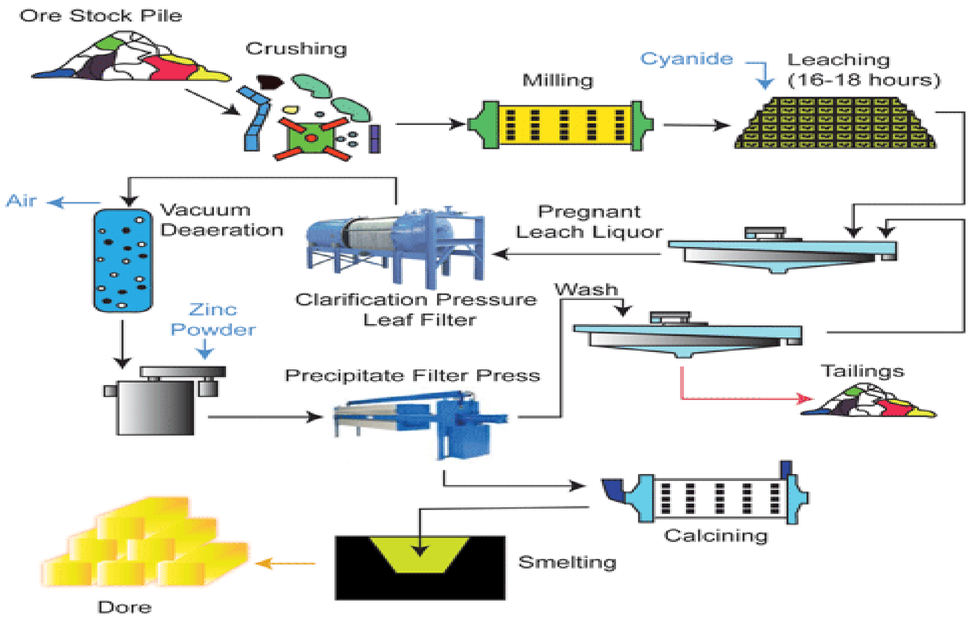


Figure 14 . Merrill Crowe Process (URL-9, 2012)

Table 8 . Advantages and disadvantages of Merrill Crowe process (Marsden and House, 2006)

Advantages	Disadvantages
Inexpensive labour costs for maintenance and operation	Pretreatment must be done to a pregnant solution in advance of precipitation
Capital costs are low	Process is reactive to intrusive ions
Can manage substantial silver to gold ratios present in the pregnant liquor	Low concentration of precious metal in solution escalate the quantity of zinc indispensable to precipitate approximately 28.3495 grams of metal

2.7.3. Ion Exchange Resins

In a tradition way, ion exchange (IX) resins have been utilized for water purification and metal contaminants removal from dilute stream of wastes originated from domestic and industrial zones. Notwithstanding, in the very recent days of this century there has been a substantial increase of its applications in getting rid of various trace impurities relating to metals from hydrometallurgical process streams (Leão and Ciminelli, 2000).

In recent years, IX resins have received an incredible attention from different gold mines across the globe (Sole et al., 2018). As readily exploitable ore deposits are becoming exhausted, the centre of curiosity is shifting to the more difficult ore-bodies that were formerly deemed economically unappealing (Habashi, 1999). They are often preg-robbing, refractory or contain high concentrations of contaminants such as copper. The use of IX resins could allow for the economic exploitation of these difficult ore bodies (Sole et al., 2018). The vital advantages of ion exchange resins encompass their low energy demand and superior selectivity for gold (Leão and Ciminelli, 2000; Van Deventer et al., 2012).

The history of IX applications in hydrometallurgy goes back to 1940s. During World War II within the project of Manhattan (Seaborg, 1963), uranium was recovered from leach solutions by the employment of IX resins. The same resins were utilized for the separation of the chemical elements with atomic numbers greater than 92. Uranium industry has stimulated the development of resins for metals recovery in hydrometallurgical circuits. Presently different gold mines are endeavouring to use RIL and RIP processes for extraction purpose (Sole et al., 2018). Generally, IX resins show technical advantages when compared with solvent extraction (SX), precipitation, activated carbon and membrane processes (Sole et al., 2016).

2.7.3.1. Properties of IX Resins

IX resins can be defined as polymers holding unattached ions which are capable of exchanging some ions inside the polymer with other ions in a solution which come in contact with them. The exchange happens without any alteration to the IX material (Purolite, 2017). Ion exchange resins are insoluble bases or acids containing insoluble salts. This allows them to interchange either negatively charged ions or positively charged ones. The following are the properties of ion exchange resins:

- Swelling
- Particle Size
- Stability
- Capacity

Swelling

Ion exchange resins are absorptive (Purolite, 2017). Their moisture retention is between 40% and 60%. The nature of functional group determines the moisture content hydrated by a resin. A considerable amount of water resulting in swelling is contained in the gel resins accompanied by functional groups of quaternary ammonium or H_2O_3S . The life span of resin may be reduced by persistent shrinkage and swelling (Marsden and House, 2016).

Particle Size

Common IX resins have a particle size range of 500-800 microns (Dow, 2016). They are manufactured in form of beads, which are spherical. During the exchange of ions, smaller particles may enhance the dynamics of the reaction, but, they provoke an increment in the difference of total pressure of water (URL-12, 2016).

Stability

The prevalence of shrinking and swelling succession and the firmness of the structure of the polymer are the predominant factors employed to determine the physical stability of resins. Precipitates of compounds like iron hydroxide may foul the resin and cause its chemical breakdown (Purolite, 2016).

Capacity

Capacity is defined as the maximal metal load grasped by one gram of resin. Two types may be used to categorize the capacity of the resin. These are operating and total capacity. The SI unit of capacity is eq/kg (Habashi, 1999).

2.7.3.2. Application of IX resins in Hydrometallurgy

For the recovery of metal(s) of interest or increasing the freshness and value of the end product by the removal of specific contaminants, then ion exchange technology is employed (Purolite, 2016). Table 9 shows miscellaneous applications of IX resins in hydrometallurgy. So far there are a bunch of metals which have been recovered by the use of ion exchange technical knowledge worldwide, they include radioactive elements, platinum group metals (PGM), precious and rare earth metals. Common resins employed in hydrometallurgical processes are illustrated in Table 10.

Table 9 . Applications of ion exchange in hydrometallurgy (Purolite, 2016)

GENESIS OF STREAM TO BE TREATED	ION EXCHANGE APPLICATION
Ore, after agitated or heap leach	Primary extraction of metal(s) of interest, e.g. uranium, gold
Vaporous compounds grasped in gases emitted from roasters and smelters	Extraction of rhenium from gases discharged following the smelting of Cu concentrates and roasting of molybdenite
Calcines and slags from smelting and roasting operations	Extraction of miscellaneous metal(s) of interest after leaching
Electrolyte	Removal of Zn and Cu impurities from Co and Ni advance electrolytes to ensure higher metal purity
Tailings treatment	Recovery of Cu, Co, Au etc. from historical mine tailings, as well as current arisings
Acid mine drainage	Treatment of process water to enable recycling or safe disposal

Table 10 . Common resins employed in hydrometallurgy with their properties

Resins	Type	I/ F	pH range	Moisture (%)	Size (mm)	Capacity (meq/ml)	A/G
Weak Basic Anion							
Amberlite IRA-93	M	FB	0-9	57	0.4-0.5	1.2	TA
DiAnion CR20	M	OH^-	4-6	50-60	0.4-0.6	0.8	PA
Lewatit MP62	M	OH^-	0-8	50-55	0.3-1.3	1.7	TA
Strong Base Anion							
Amberlite IRA-400	G	Cl^-	0-14	44-48	0.6-0.8	1.4	QA
Amberlite IRA-410	G	Cl^-	0-14	42	0.48	1.4	QA
Amberlite IRA-910	M	Cl^-	0-14	52	0.46	1.0	QA
Dowex G51	G	Cl^-	0-14	43-48	0.3-0.9	1.4	QA
Dowex G55	G	Cl^-	0-14	43-48	0.6	1.0	QA
Dowex 21K	G	Cl^-	0-14	43-48	0.6-1.2	1.2	QA
Purolite A500/2788	M	Cl^-	0-14	53-58	0.8-1.3	1.2	QA
Purolite A600u	G	Cl^-	0-14	43-48	0.3-1.2	1.4	QA

Note: A/G = Active group ; G= Gel; M= microporous ; FB= Free base ; TA= Tertiary amine; PA= Polyamine; QA= Quarternary amine; meq= milliequivalent (Zhang and Dreisinger, 2002)

2.7.3.3. Gold Selective Resins

Gold selective resins that appear to be commercially viable are strong and medium base anion exchange resins (Kotze et al., 2016). Generally during adsorption, medium-base resins are more selective for gold over base metals and their elution is simply done by aqueous NaOH (Van Deventer, 2012). They are also very sensitive to pH changes, the optimum pH range lies between 10 to 11 (Voiloshnikova et al., 2014). Strong resins have high metal loadings compared to medium- base ones, their loading is not pH dependent. The selectivity of strong base resins is reduced due to the tendency for loading base metals during the process of adsorption. Elution of these resins is undertaken by the use of a mixture of thiourea or sulphuric acid (Marsden and House, 2006; Van Deventer, 2012; Kotze et al., 2016).

2.7.3.3.1. Strong-Base Resins

These resins act similar to strong bases. Generally their functional groups are made of quatenary ammonium and they contain positive charges which are fixed. Any metal cyanide anion is able to take the place of the counter ion affiliated with the resin site, which is positively charged and begin to be connected through the generation of a pair of ion. These anions bond robustly to the resin because they are eminently polarizable (Kotze et al., 2016). Their reactions are not affected by pH change in the cyanide leachate (Van Deventer, 2012). The mechanism of adsorption is illustrated in Equation 24 below. Minix ^{TR} containing a functional group of tributylamine is a good example of strong-base resin (Kotze et al., 1993). Others are Purogold A194, Purolite A660/4759, Purolite A500U/2788 and Purolite A560/4790 (URL-10, 2015).

$$\text{P-NR}_3^+\ \text{Cl}^-\text{+Au (CN)}_2^- \rightarrow \text{P-NR}_3^+\ \text{Au (CN)}_2^- + \text{Cl}^- \qquad (24)$$

Where

P= Polymer Matrix,   R= Alkyl Chain

2.7.3.3.2. Medium-Base Resins

These resins have a combination of primary, tertiary, quaternary, and secondary amine groups (Van Deventer, 2012; Kotze et al., 2013; Kotze et al., 2016). Protonation of their functional group is a prerequisite, so that the extraction process takes place (Kotze et al., 2016). pKa values play a vital role in the process of protonation (Van Deventer, 2012; Kotze et al., 2016). An example of medium base resin is Purogold S992 (Van Deventer, 2012; URL-10, 2015). The adsorption and protonation reactions are presented in the equation below. Figure 15 shows how the basicity of the functional group affect the rate of elution and loading. The features of the common used gold selective resins are presented in Table 11.

$$\text{Protonation: } P\text{-}NR_2 + H^+ \rightarrow P\text{-}NR_2H^+ \qquad (25)$$

$$\text{Adsorption: } P\text{-}NR_2 + H^+Cl^- + Au(CN)_2^- \rightarrow P\text{-}NR_2H^+\,Au(CN)_2^- + Cl^- \qquad (26)$$

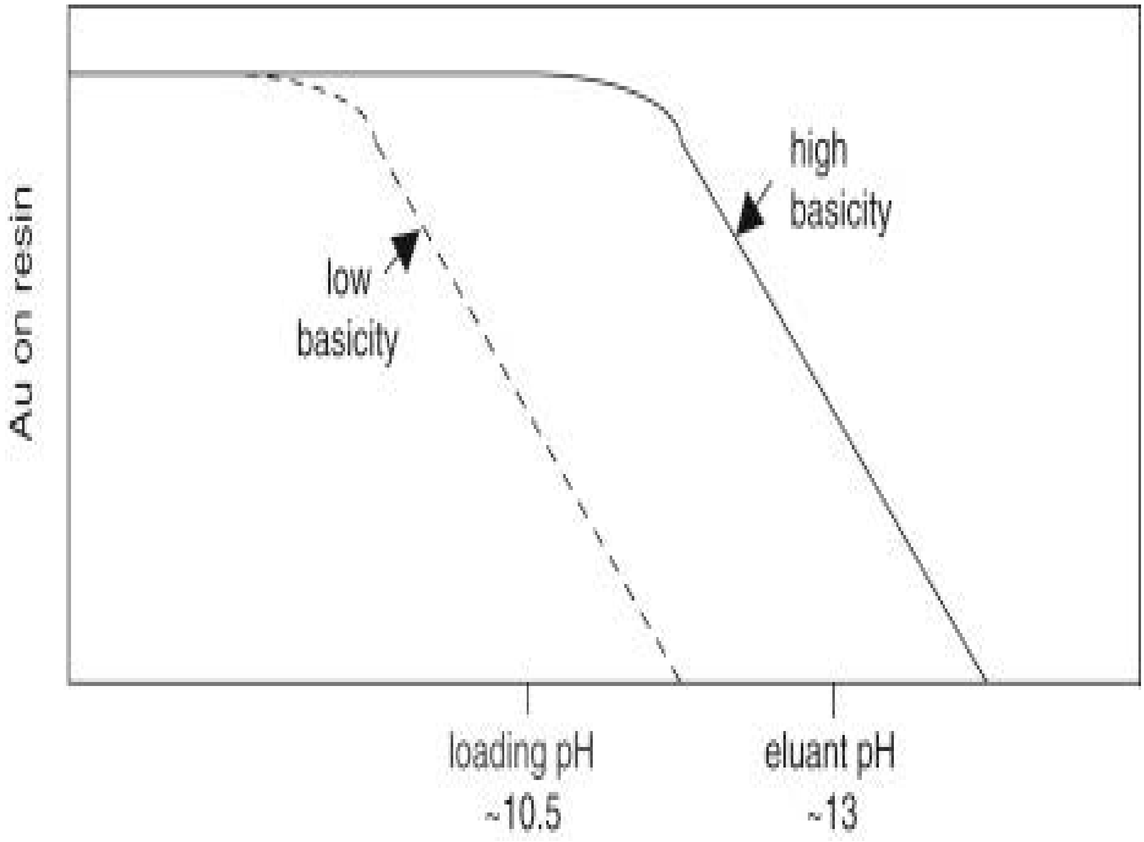


Figure 15. Effect of functional group basicity on degree of protonation and gold loading (Kotze et al., 2016)

Table 11 . Characteristics of the common used gold selective resins in cyanide liquors (Purolite, 2016)

Name of the resin	**Type**	**Functional Group**	**Ionic Form**	**Capacity**	**Moisture retention (%)**
Purogold™ A193	Macroporous Mixed Base Anion	Mixed Tertiary & Quaternary Amines	Cl^-	3.8 eq/kg	46 – 56
Purogold S992	Macroporous Medium Base / Chelating	Mixed Amines	FB	4.4 eq/kg	47 – 55
Purogold A194	Macroporous Mixed Base Anion	Quaternary Amines	Cl^-	3 eq/kg	44-52

There seem many operations using IX processes for extraction of gold (Table 11). IX offers a lot of advantages over activated carbon. They include their property of not being fouled by organic matter contents, their none thermal regeneration and their better selectivity for gold over other base metals such as Ni, Zn, Cu etc. (Kotze et al., 1993, and 2016; Van Deventer, 2012; Sole et al., 2018).

Table 12 . Gold processing resin operations (Estay et al., 2012; URL-11, 2016).

Company	**Mine**	**Location**	**Start up**	**Throughput (Mtpa)**
Barrick Gold	Goldstrike	USA	2014	4.0
Petropavlovsk	Albyn	Russia	2011	4.5
Petropavlovsk	Pioneer	Russia	2008	7.0
Alhambra Resources	Uzboy	Kazakhstan	2006	1.0
Petropavlovsk	Pokovskiy	Russia	2004	2.0
Petropavlovsk	Malomir	Russia	2003	4.5
J Resources	Penjom	Malaysia	1999	0.5
Golden Jubilee Mining	Golden Jubilee	South Africa	1988	Closed
NGMK	Muruntau	Uzbekistan	1971	Approximately 23
Anglo Asian Mining	Gedabek	Azerbaijan	2009	1

Where Mtpa: Million Tons Per Annum

2.7.3.4. Resin-in-leach (RIL) Process

This process works as same as the CIL process, that’s to say, resins are introduced at the beginning of the process of leaching, hence the adsorption of metals onto resins take place as the leaching process continues (Marsden and House, 2006; Sole et al., 2018). Among the 50% of gold production in the Soviet Union back in 1970s and 1980s was obtained by RIL (Sole et al., 2018). In Malaysia specifically at Penjom gold mine, the RIL technology has received an appealing attention. At this mine where the head feed grade is between 6 g/ton and 10 g/ton, poor gold recoveries were yielded via CIL (Lewis, 2000; Kotze et al., 2016). These low recoveries were due to the presence of preg-robbing ores. However, recoveries have improved when the circuit was shifted to RIL with the utilization of Minix ^{TR} which is characterized by its high selectivity for Au over base metals (Kotze et al., 2016). There was an increment of approximately 20% of recoveries by RIL compared to the ones of CIL. RIL method proved to be a best option to treat such an ore. It should also be remembered that for the gold recovery, RIL is an effective technology for treating ores with preg-robbing traits, because they are less vulnerable to fouling by organic matters (Kotze et al., 2016; Lewis, 2000).

Mubarok and Irianto (2016) investigated the enhancement of the gold recovery from a preg-robbing ore. The resin used was MP-800 in a resin in leach (RIL) process with the aid of an agent for blinding (BA). The recovery obtained after leaching without pretreating the ore was 65.5%. However when the ore was pretreated and then leached by RIL, the recovery improved to 99.5%. They also noted a very rapid kinetics of the adsorption of gold and high loading capacity.

2.7.3.5. Resin- in- Pulp (RIP) Process

RIP process was innovated for the sake of recovering metals of value from huge volumes of pulps with low grades. In this process, resins are introduced after the leaching process has done. The uranium industry in USA used resin in pulp technology between 1960’s and 1970’s (Fleming, 1992).

CIL process was shifted to RIP at the Golden Jubilee Mine in South Africa (Fleming, 1992 and 1993). Following the installation of RIP, the overall recovery of gold increased from 65% to 85%, this resulted in a production of gold of more than 200%. Gold loadings with RIP were between 4000-6000 g/ton, while for activated carbon was less than 1500 g/ton. The overall operating costs in RIP process were lower than in CIL tanks. Losses, which occurred due to breakage, were 8-10 g/ton of treated ore and 50-100 g/ton for resin and activated carbon respectively (Fleming, 1992).

Other known RIP plants include Gedabek, Muruntau, Barbrook, and Penjom gold mine located in Azerbaijan, Uzbekistan, South Africa and Malaysia respectively. The mechanism of a RIP process is depicted in Figure 16.

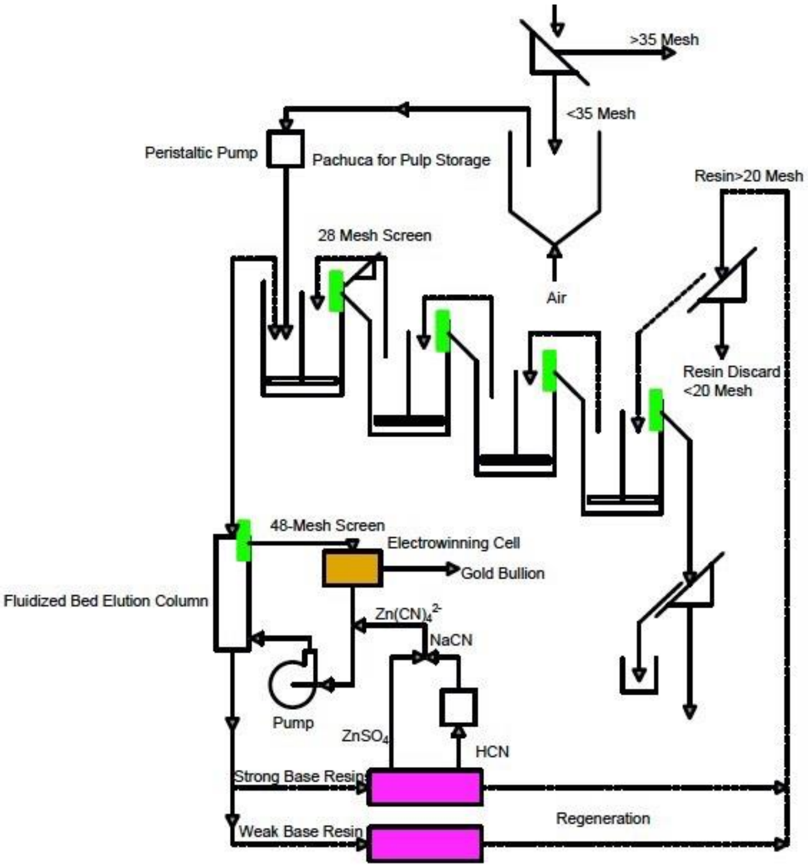


Figure 16 . Flowsheet for RIP gold extraction system (Fleming, 1984)

3. MATERIALS AND METHODS

3.1. Introduction

Within this study, the feasibility of recovering gold from a copper-rich gold ore by the utilization of ion exchange resins was investigated. To compare the rate of recovery of gold from the refractory ore, miscellaneous adsorption experiments were conducted. Before the leaching experiments characterization of the ore encompassing a detailed chemical, mineralogical, and analysis of the particle size were carried out. The refractoriness of the ore was identified by the use of diagnostic leaching. Various strategies or pretreatment options (i.e. acid leaching, roasting) were tested to eliminate refractoriness of the ore. In all experiments, representative sub-samples were employed.

The ore sample utilized in this research work was taken from copper-rich zones of Mastra-Gümüşhane deposit. Figure 17 shows the location map of the Mastra gold deposit. In this thesis, the ore sample was named as MLG. In all experimental studies, stock solutions were prepared with deionized water. The reagents with their properties used in different experiments and analysis are shown in Table 13. All equipment and glassware used were cleaned, rinsed with tap water and then acid washed with 4% HCl. Following acid washing, all glasswares were thoroughly rinsed with deionized and distilled water.

Figure 17 . A map showing a location of Mastra gold deposits (Pilot Gold, 2014)

Table 13 . Different reagents used during the scope of this project

Chemical Name	Formula	Mol.w(g/mol)	Purity	Purpose	Mark
Sulphuric Acid	H_2SO_4	1L= 1,84 kg	95-98 %	L/ T	MERCK
Hydrochloric Acid	Hcl	1L= 1,19 kg	36.5-37%	L/ T	MERCK
Nitric Acid	HNO_3	1L= 1,39 kg	65%	L/ T	MERCK
Sodium Hydroxide	NaOH	40	99.5	L/ T	MERCK
Sodium Cyanide	NaCN	49	$\geq$ 95%	L/T	MERCK
Copper Sulfate	$CuSO_4$	159.61		F	
MIBC	$C_6H_{14}O$	102.17		F	
Lime	CaO	56.07		F + L/T	
Aerophine	$C_8H_{18}NaPS_2$	232.32		F	
SIPX	$(CH_3)_2CHOCSSNa$	158.2		F	
Sodium Sulphate	$Na_2SO_4 .10H_2O$	322.19		A	

L/T : Leaching Test F: Flotation SIPX : Sodium Isopropyl Xanthate
MIBC: Methyl Isobutyl Carbinol A: Adsorption F: Flotation

Mastra Gold Mine is situated at 22km of northwest of Gumushane and northeast of Mastra (Demirkaynak) village. Ore is processed at Mastra plant with tank leach method to produce gold. Ore is grinded in ball and rod mills and fed into leach tanks. The copper contained in the ore is recovered by SART system and fed into the plant. The SART system is presented in Figure 18.

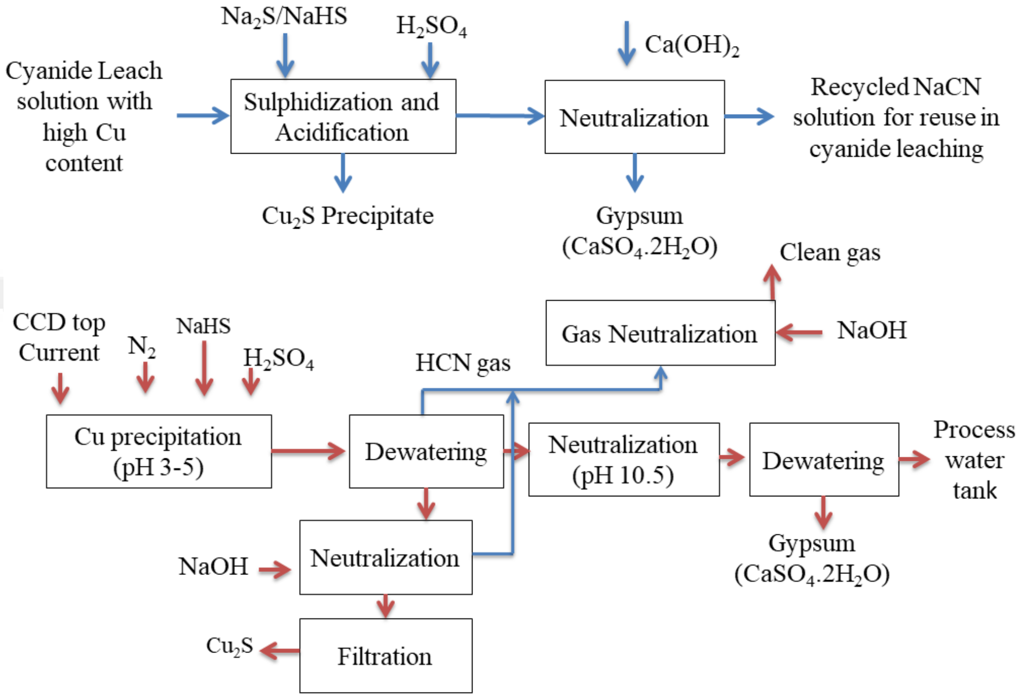


Figure 18. Flowsheet of the SART process at Mastra Gold Mine

3.2. Sample Preparation

The ore sample was received from Mastra gold deposit situated in Gümüşhane-Turkey. About 14 kilograms of crushed ore were ground to -73 µm (P_{80}) by using a laboratory rod mill (length: 31 cm, diameter: 20.5 cm). The time used for grinding was 30 minutes. The ground ore was riffled and then divided by rotary sample divider (Fritsch 27) to obtain representative sub-samples for the tests. Figure 19 depicts the device used for the auto-division of the sample. The milling process was carried out in the form of a dry mill illustrated in Figure 20 and the milling conditions for the ore samples are given in Table 14.

Figure 19 . Rotary sample divider (Fritsch 27)

Figure 20. Rod mill used during the grinding process

Table 14. Grinding conditions used for MLG sample

Sample's Name	Mass (g)	Charge rate (%)	Rotational speed (rev/min)	Grinding time (min)	Product Particle Size-d_{80} (µm)
MLG	1000	21.55	82	30	73

3.2.1. Wet Sieve Analysis

A known amount (242 g) of ground MLG was sieved via a sequence of sieves ranging from +150 µm to -38 µm. Any material corresponding to certain size was retained in these sieves. Figure 21 represents the size distribution of the sample.

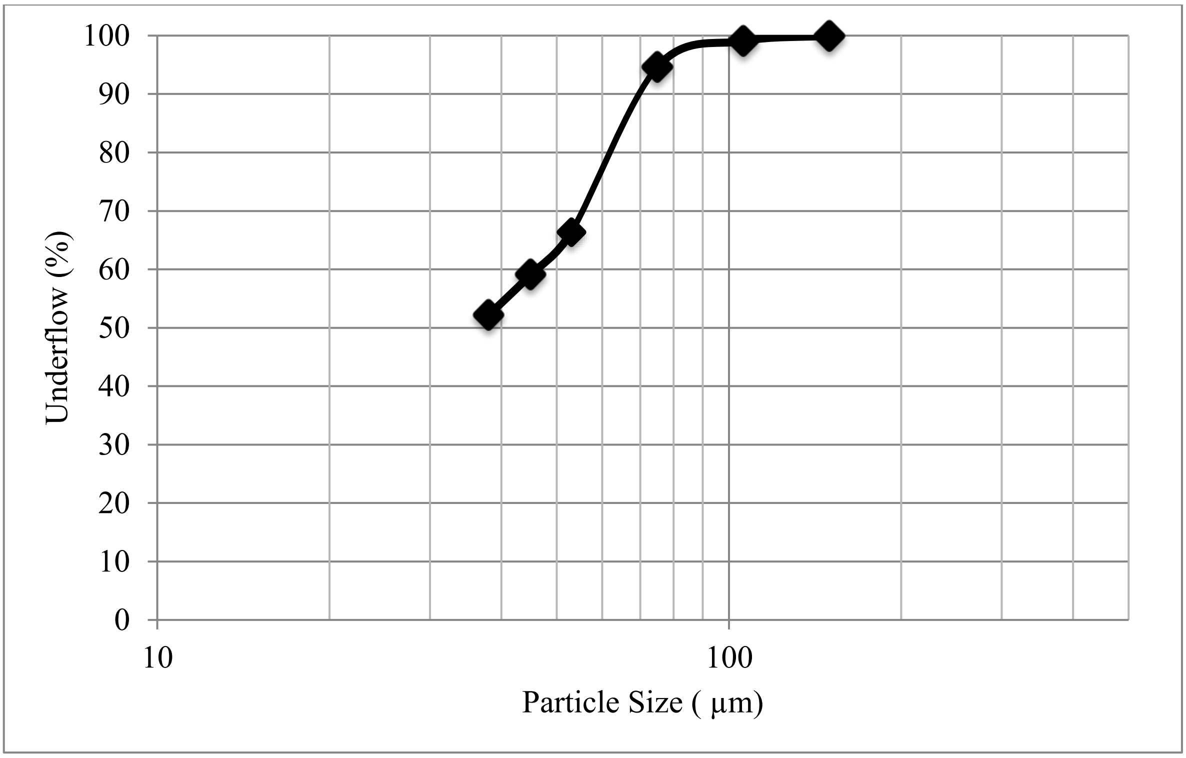


Figure 21. Particle size distribution of the sample (ground for 30 min) used in the tests

3.3. Mineralogical Analysis

Prior to the main series of experiments, 120 grams of MLG was sent to ARGETEST, an accredited analytical laboratory in Ankara-Turkey for chemical analysis using an in house method.

Table 15 illustrates the head assay of the ore sample. The analysis revealed that the ore sample contained high levels of copper (1.6%) (Table 15). According to the data collected from XRD system, and ICP-MS for trace elements, the main phases of the ore were identified as quartz (SiO_2) and pyrite (FeS_2). Other phases were reported to be chalcopyrite ($CuFeS_2$) and sphalerite (ZnS) (Bas et al., 2012).

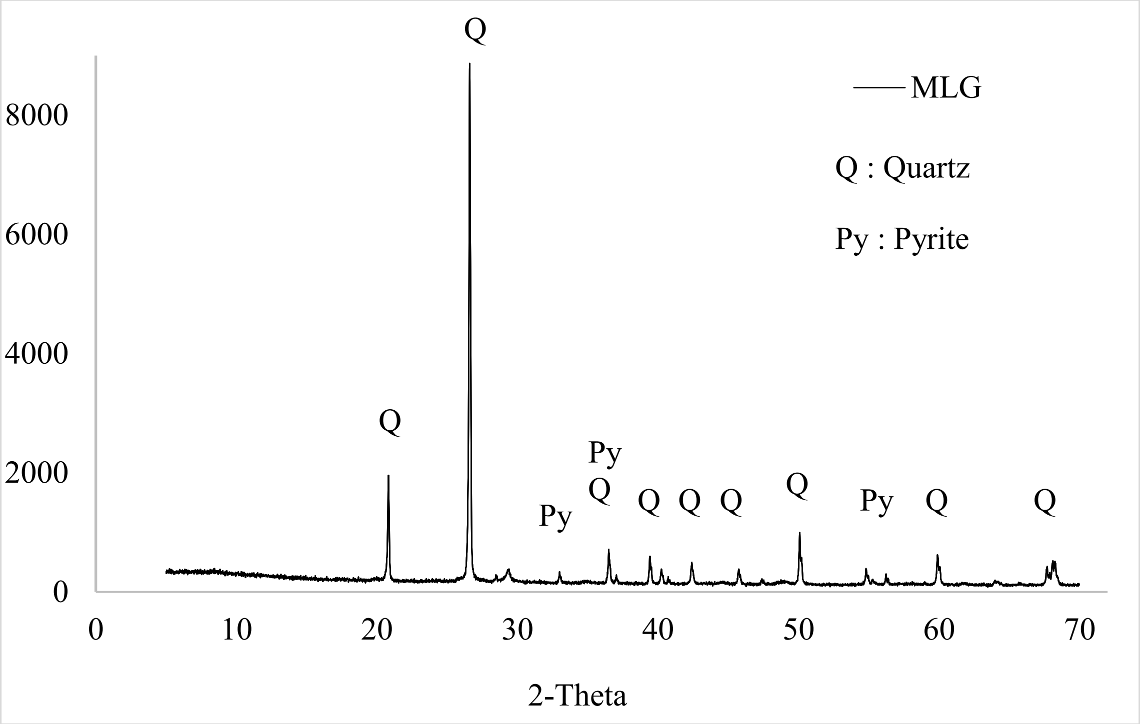


Figure 22. The XRD profile of the MLG sample

Table 15. Chemical composition of the ore

Element	Grade	Unit	Element	Grade	Unit	Compound	Content (%)
Au	107.7	ppm	Ni	604.5	ppm	SiO_2	67.3
Be	<1	ppm	Cu	15960	ppm	Al_2O_3	11.51
Na	0.027	%	Zn	926.1	ppm	Fe_2O_3	6.06
Mg	0.071	%	Ga	<5	ppm	MgO	0.45
Al	0.39	%	As	225	ppm	CaO	0.46
P	<0.001	%	Ag	9.3	ppm	Na_2O	0.06
S	3.46	%	Ba	1211	ppm	K_2O	2.58
K	0.21	%	Bi	<5	ppm	TiO_2	0.02
Ca	0.22	%	Cd	10.7	ppm	P_2O_5	0.19
Sc	1.94	ppm	Hg	<5	ppm	MnO	0.18
Ti	0.0023	%	La	1.64	ppm	Cr_2O_3	0.015
V	16	ppm	Mo	24.64	ppm	LOI	6.03
Mn	174	ppm	Pb	838.1	ppm	Total C	0.15
Fe	4.02	%	Sb	93.21	ppm	Total S	4.56
Co	15.57	ppm				Sum	95.31

3.4. Physical Pre-treatment of MLG ore: Ultra-fine Grinding

A laboratory scale pin type vertical stirred media mill was used for having products of ultra-finely ground samples. Figure 23 illustrates the device used in these tests. This was done using 2 mm ceramic balls (alumina-based zirconia toughened, DMM AZ 2000®) at 29% w/w (S/L) ratio and 600 rpm stirring rate. The beads had Al_2O_3 content of 80%, specific gravity (SG) of 3.75-3.80 and Vickers hardness (HV) of 1314 m. The grinding time was 15, 30 and 45 minutes. The particle sizes of 23 µm, 15 µm, and 8 µm were produced in 15, 30, and 45 minutes respectively. Cyanide leaching tests (8 g/L NaCN) were carried out for the as-received ore and the ground ones for a period of 24 hrs. During cyanidation, the stirring speed and solid to liquid ratio were 600 rpm and 25% w/w respectively. Some of samples (6 ml) were taken at prearranged intervals (30minutes, 1hr, 2hrs, 4hrs, 8hrs, and 24hrs) to monitor leaching of metals (Au, Ag, Cu, Fe, Zn) and cyanide consumption using silver nitrate titration. NaCN was added to keep the concentration of titratable NaCN at the initial level over the period of leaching. Following the termination of leaching experiments, filtration of residues was done and then dried prior to chemical analysis. The metal values of solutions and residues were determined by AAS (Perkin Elmer AAnalyst 400) after hot digestion of residues in an acid mixture (HCl, HNO_3). Metal content of residues determined the calculation for extractions.

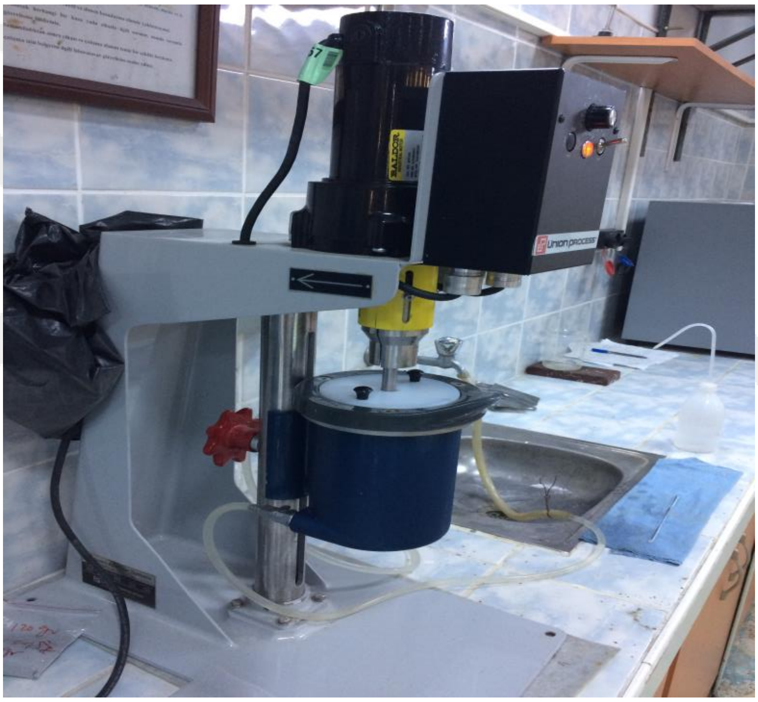


Figure 23. The stirred mill used in the ultra-fine grinding process

3.5. Diagnostic Leaching

A sequence of acid leaching steps aiming at breaking down targeted mineral phases and to set free gold associated was undertaken. At each stage of treatment the residue was leached by 1.5 g/L NaCN so as to recover gold liberated after the treatment. The series and prerequisites of acid leaching steps in this approach are presented in Table 16. With the aid of AAS, liberated gold at each stage was carefully analyzed. For a proper establishment of a mass balance, leach residues of all the steps involved were also analyzed for Au, so the recovery of gold is calculated. The experimental set-up is depicted in Figure 24.

Table 16. Chronology and parameters used in diagnostic leaching of the MLG ore

Stage of Treatment	Leach Condition(s)	Possible Minerals to be destroyed
NaCN, 1.5 g/L	pH 10.5 (with lime), 24 h	Au
HCl (12%)	S/L =1:2, 60°C, 8 h	$CaCO_3$, $CaMg(CO_3)_2$, PbS, $Fe_{(1-x)}S$ (x=0 to 0.2), Fe_2O_3
HNO_3 (33%)	S/L=1:2, 60°C, 6 hours	FeS_2, FeAsS, Marcasite

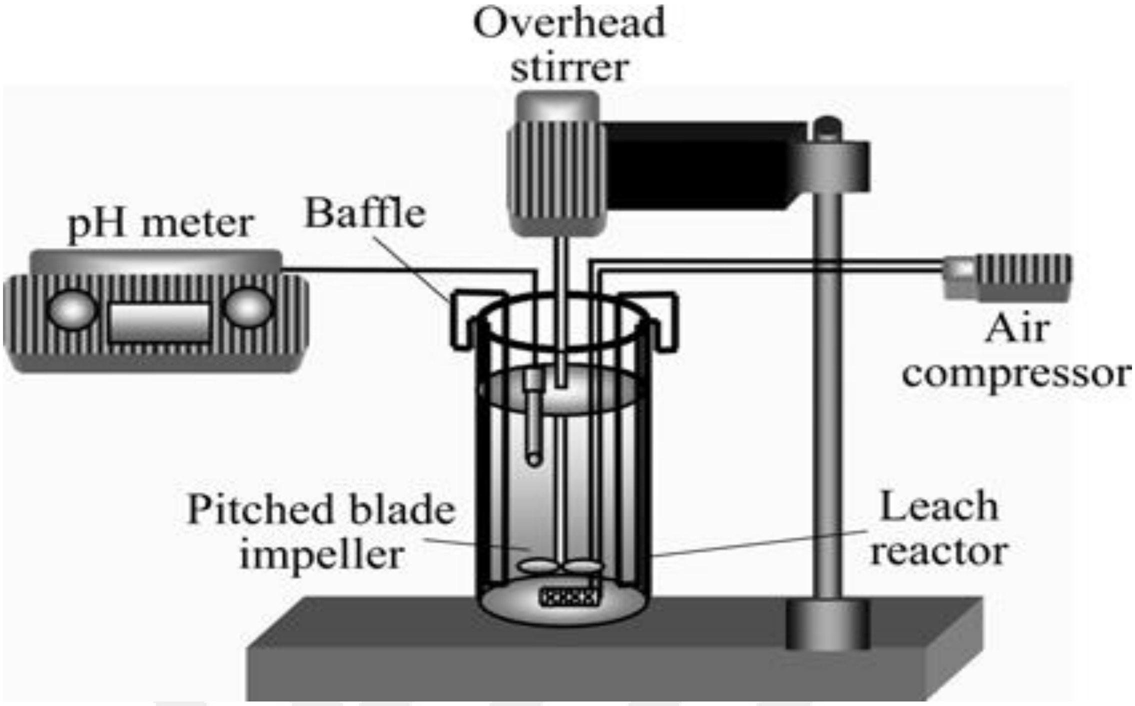


Figure 24. Set up for the diagnostic leaching experiment (Celep et al., 2008)

3.6. Roasting Pretreatment Tests

A Protherm Furnace was used to roast about 32 packets (≈ 125 g each) of MLG ore. The target operating temperature for roasting was 650°C.

I. The 125 g sample to be roasted was placed in a bowl made of SiO_2, its dimensions were 145 mm long, 83 mm wide and 18 mm deep. This left 3 to 5 mm of freeboard above the sample.

II. At room temperature, the ore was introduced in the furnace, then the thermostat was set at 200°C and turned on. The door of the furnace remained about 20- 25 mm open throughout the period of roasting, so as to ensure an unrestricted supply of oxygen.

III. The sample was rabbled every 30 minutes by the use of a rabbler consisting of 2-toothed steel plates fastened to a steel rod for maximizing the efficiency of mixing.

IV. Every one hour, the temperature was elevated by about 100°C, this was done up to the point when the target temperature (650°C) reached. This was done in four steps, about 25°C every 15 minutes. It took about 6 hours to reach 650°C.

V. The process of rabbling after every 30 minutes was done for further 120 minutes, when the target temperature reached. This made an aggregate of 8 hours.

Figure 25 illustrates the device (Protherm furnace) used during roasting and the colour of the roasted and unroasted sample respectively.

Figure 25. The Protherm furnace used in the roasting of samples as well as roasted and unroasted samples

3.7. Sulphuric Acid Pretreatment Tests

Two samples (roasted and unroasted) were treated with 1M H_2SO_4, in order to dissolve oxidized or the acid soluble copper minerals. The pH was controlled below 2 through out the experiment, the solid-liquid ratio was 25%, leaching time 2 hrs , and agitation speed 600 rpm. The colour of the solution changed swiftly into blue which implies the dissolution of copper minerals just after the introduction of H_2SO_4. Figure 26 shows the set up of this leaching test. The pH range for the roasted sample ranged between

0.74 and 0.80 whilst the one for unroasted ranged between 0.69 and 0.80 (at the commencement and the termination of experiment respectively).

Figure 26. Experimental set up for sulphuric acid leaching tests

3.8. Cyanide Leaching Experiments

Leaching tests by cyanide were conducted in 1000-mL Pyrex beakers over a period of 24 h. Mechanical mixers equipped with four-blade, PTFE coated, with 45^0-pitched blade impellers made of stainless steel were used; the diameter of these blade impellers was 6.4 cm. Figure 27 shows the experimental set up used in cyanide leaching. Stirring speed employed in reactors was 650 rpm. NaCN (≥95%, Merck) was used to prepare leach solutions with a concentration of 1.5 g/L NaCN. A pulp ratio of 25 % w/w (120 g ore sample + 360 ml cyanide solution) was maintained in cyanide leaching tests. The flow rate of air transferred by an air pump into the medium during the leaching tests was 1.5 L/min. Samples were taken at certain intervals (30min, 1hr, 2hrs, 4hrs, 8hrs and 24hrs) and passes to a centrifuge to get clear solutions for analysis of copper, gold, silver, iron and zinc by AAS. The free cyanide concentration was analyzed by silver nitrate (0.02M $AgNO_3$) titration using p-dimethyl amino-benzyl-rhodanine as the indicator to determine cyanide consumption as well as to maintain the NaCN concentration at 1.5 g/L. pH was adjusted to 10.5-11 by the use of lime. It is pertinent to note that silver nitrate titration method leads to overestimation of free cyanide level since silver nitrate extracts a part of cyanide from copper cyanides present in the leach solution. Following a leaching period of 24 h, filtration was done for separation of leach residues. They were then passed to an oven at 105 °C for drying purposes, afterwards they were digested in a hot aqua regia for the metal analysis. With the aid of the results of analysis of residues, metal extractions in the leaching tests were determined.

Figure 27. Experimental set-up used in cyanide leaching test

3.9. Preliminary Tests For Selection of Resins

Before adsorption tests (CIP, CIL, RIP and RIL), preliminary adsorption tests were conducted to compare the effectiveness of various adsorbents for gold recovery from cyanide leachates having significant levels of copper. With the use of an activated carbon (GAC 1240 M-1448), a strong base (Purogold A194), Dowex 21K XLT, Purolite A193, and medium-base (Purogold S992) gold selective resins, gold selectivity over copper and the loading capacity were evaluated. A real cyanide solution containing 28.2 mg/L Au and 2804 mg/L Cu was prepared in 50-ml flasks, which were then placed onto an orbital shaker (Wiggen Hauser). pH was adjusted to 10.5-11 by the use of 1M NaOH. The resins and activated carbon were then introduced into flasks. The sampling was carried out at 15, 30, 60, 120, 240, 480 and 1440 minutes by removing 1 ml solution from each flask during sampling. The concentration of resins and carbon used was 5 g/L. At the end of the tests, solutions were diluted with 1.5g/L NaCN and metal concentrations (Au, Cu, Fe, Ag, Zn) were analyzed by the use of atomic absorption spectroscopy (AAS). Following these tests a proper selection of resins was achieved. Table 17 illustrates the parameters employed in these tests.

Table 17. Parameters employed in preliminary tests for the selection of resins

	Dowex 21K XLT	Activated Carbon	Purolite A193	Purogold S992	Purogold A194
Concentration (g/L)	5				
Temperature (°C)	25				
Stirring Speed (rpm)	170				
Leaching Time (hours)	24				

Another series of experiments were undertaken where by the employed medium base resin was firstly transformed to the form of sulphate before the adsorption tests. Protonation of this type of resin is a prerequisite for the extaction process to occur. They were contacted with two bed volumes (BV's) of a 0.5M $Na_2SO_4.10H_2O$ solution in a column. The rate of flow was 2-bed volume per hour (volume of resin used is defined as a BV). 4 BV's of water was utilized to cleanse the excess reagent from the resin.

The selectivity (μ) and adsorbent loading (A) were calculated using the following formulas.

$$\mu = \frac{D\,(Au)}{D\,(Cu)}$$

Or

$$\mu = \frac{[Au]_{\mathbf{adsorbent}} \times [Cu]_{\mathbf{Solution}}}{[Au]_{\mathbf{Solution}} \times [Cu]_{\mathbf{adsorbent}}}$$

$$A = (C_o - C_f) \times V \times m^{-1}$$

Where, C_o: Initial concentration of adsorptive in solution (mg/L), C_f: Equilibrium concentration of adsorptive in solution (mg/L) , A: Adsorbent loading (mg/g) , V: Volume of solution(L) , m: Adsorbent mass (gr)

3.9.1. Carbon-In-Leach (CIL) and Carbon-In-Pulp (CIP) Experiments

These experiments were carried out by using activated carbon (Carbon-GAC 1240 M-1448). In CIL tests, the required amount of activated carbon (AC) was introduced into the leach system to produce concentrations of 2 g/L, 5 g/L and 10 g/L AC. The pH adjustment to the desired level was performed by the dropwise addition of 1 M NaOH. The leach tests were done in 1000-mL Pyrex beakers for 24 hrs. Each beaker was placed in the experimental set-up and agitated at 650 rpm. MLG sample (120 grams) was added to each beaker to keep constant a solid to liquid ratio of 25%. The leach solution (360 ml) was adjusted to a pH of 10.5 before the addition of activated carbons and cyanide. The cyanide concentration used was kept at 1.5 g/L with activated carbons of 0 g/L, 2 g/L, 5 g/L, and 10 g/L. A high concentration of 5 g/L NaCN was also tested at 5 g/L activated carbon. Air was supplied into these beakers at 1.5 L/min and the tests were undertaken at an ambient temperature. Sampling was done in the course of the leach period at 0.5, 1, 2, 4, 8, and 24 hours respectively. A known volume (6 ml) of sample was removed at each sampling interval. The volume change caused by the sample removal was accounted for in the data processing procedure. The sample solutions were assayed for the concentration of Au, Ag, Cu, and the final concentration of Zn and Fe. With the aid of a 250-µm sieve, activated carbons were separated from the pregnant leach solution following the termination of each experiment. The slurry was then filtered by the use of Whatman^{TM} filter paper (125 mm Cat No 1001 125) for the separation of residue. The gold, silver, copper, zinc and iron in the residue were determined to establish a metallurgical balance to determine metal extraction. Table 18 illustrates the parameters used in these tests. The only variation between CIP and CIL tests is that in CIP, activated carbons were introduced after leaching the ore for a period of 24 hours. Adsorption tests lasted for 24 hours, making an aggregate of 48 hours, whilst in CIL activated carbons were introduced at the beginning of the experiment.

Table 18. Parameters for CIL tests

	Carbon Conc	NaCN Conc	pH	S/L ratio	Time (hours)	Stirring speed (rpm)
Test 1	0	1.5	10.5-11	25%	24	650
Test 2	2	1.5				
Test 3	5	1.5				
Test 4	5	5				
Test 5	10	1.5				

3.9.2 Resin-In-Leach (RIL) and Resin-In-Pulp Experiments

These tests involved the use of two different resins namely Purogold S992 and Purogold A194 respectively. In RIL and RIP tests, these resins were added into the leach solution. The same set up for experiments and procedure as those in activated carbon tests were used. The pH was set to 10.5 since resins especially medium base are highly sensitive to pH. The resins dosage was the same as the one used in activated carbon for comparison. Other conditions were the same with the exception of using resin in the place of activated carbon.

4. RESULTS AND DISCUSSION

4.1. Direct Cyanide Leaching Experiments

Direct cyanide leaching of the ore was initially tested at 1.5 g/L NaCN. The results indicated a very limited gold extraction of 18.4 % at 24 h (Figure 28).

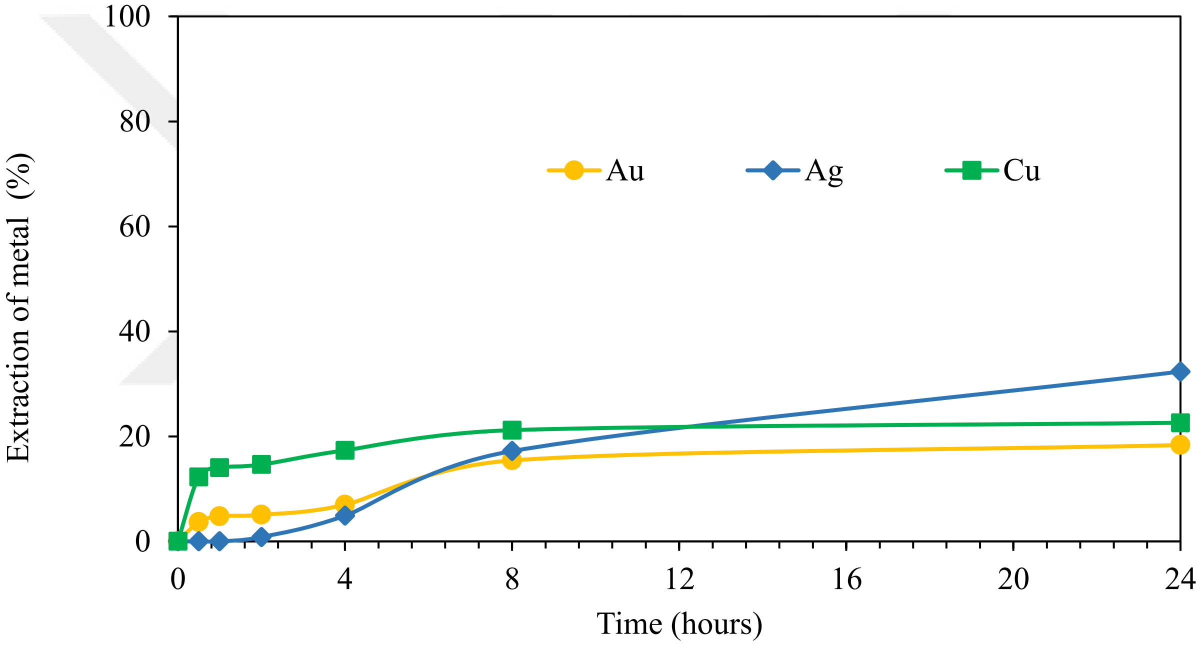


Figure 28. Direct cyanide leaching at 1.5g/L NaCN

These data suggested that the ore is refractory in character. The total cyanide consumed in a direct leaching test was around 6.75 kg/ton, this showed that cyanide consuming elements like copper and other metal ions could have a deleterious effect on the extraction of Au. The cyanide consumption was reduced to 1.2 kg/ton when the same ore was pretreated by sulphuric acid whereby about 95% of copper dissolved. This difference of around 5.55 kg/ton implies the deleterious effect of copper in the whole process of leaching. Bas et al. (2015) indicated that the process of gold leaching by cyanide is intervened by the copper containing sulphides when this process is carried out at concentrations below 5 g/L NaCN. Pyrite and arsenopyrite are the common sulphide minerals which have a tendency of locking fine gold particles. A typical extraction of less than 20% by direct cyanidation is offered by the ores containing these minerals. The researchers (Dawson et al, 1997; Aylmore and Muir, 2001; Bas et al., 2012 and 2014) pointed out that dissolution of copper minerals may adversely affect cyanidation leading to elevated consumption of cyanide and very limited recovery of gold. Marsden and House (2006) also claimed that sulphide ion (S^{2-}) that is generated from sulphide minerals such as copper sulphides which are available in the ore can also provoke the gold surface passivation.

Bas et al. (2014) claimed that leaching by cyanide is an unselective process. As far as the process economy is concerned, 30 kg/ton NaCN, could be consumed for every one percent of reactive copper available in the ore (Muir, 2011). Copper sulphides like covellite can increase the cyanide consumption up to 51.5 kg/ton for every percent of copper included, this is linked with the formation of thiocyanate and cyanate complexes.

In an attempt to improve extraction, a series of other leaching tests were conducted using an elevated levels of cyanide concentration up to 8 g/L NaCN. Increasing cyanide concentration did not produce the desired effect (Figure 29). Only 47.2% of gold was extracted at a NaCN concentration of 8 g/L. This gave a clear picture of how reagent consumption could be wasted in treating such kind of ores, yet the extractions remain poor. These findings suggested that the low gold extraction (i.e. refractoriness deportment of the ore) is not linked only with the presence of copper. Therefore, further pretreatment tests and diagnostic leaching were carried out to characterize and eliminate the refractoriness of the ore.

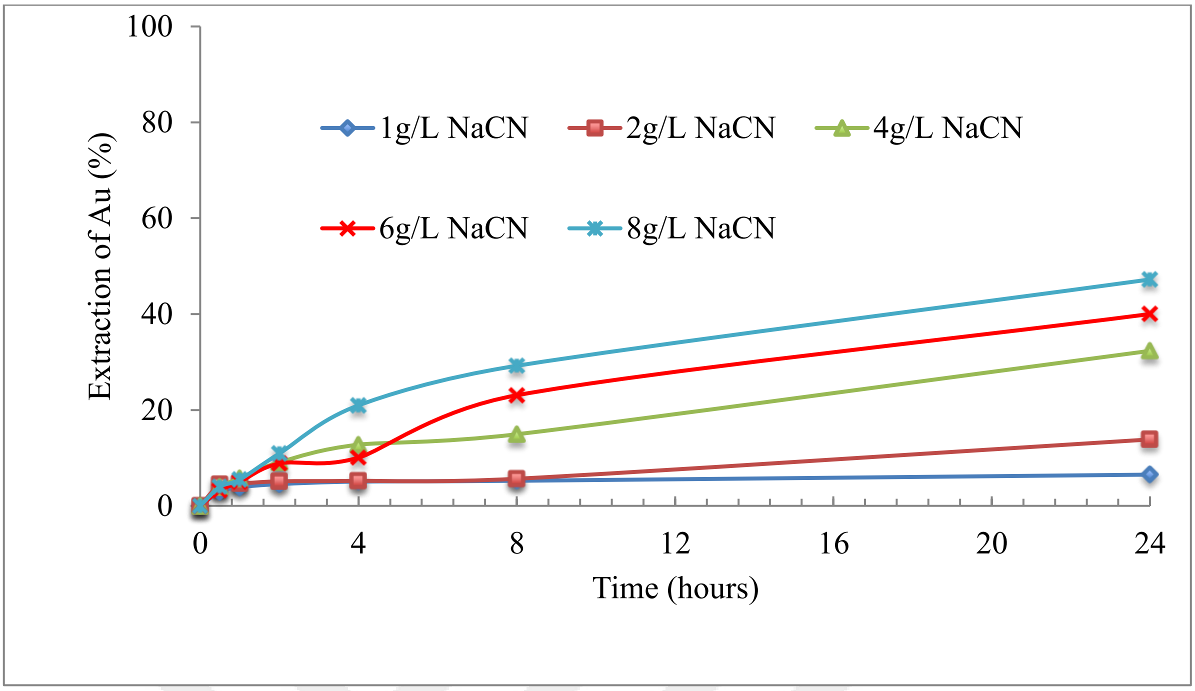
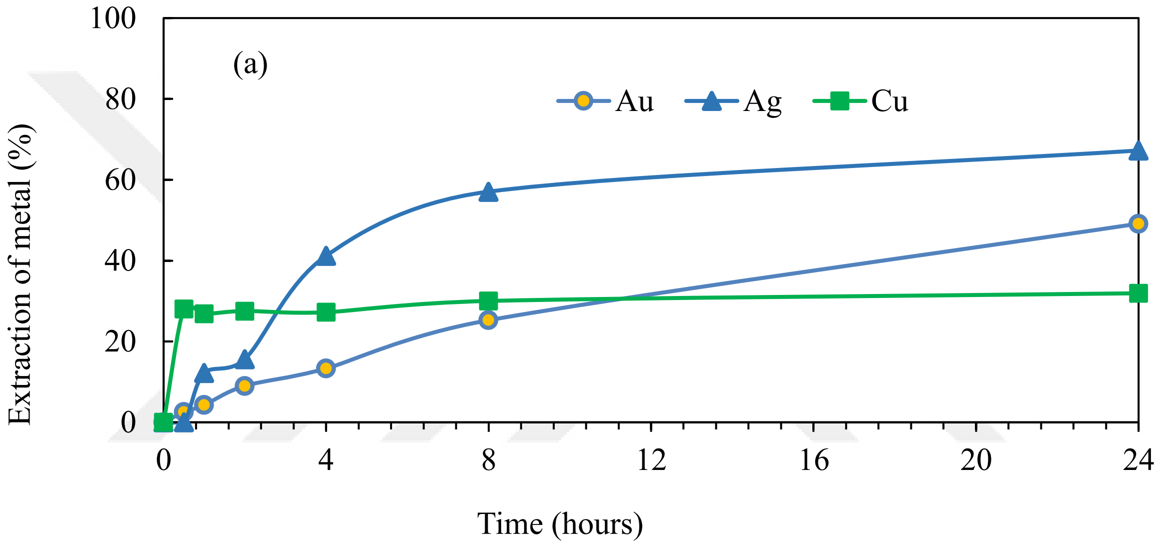


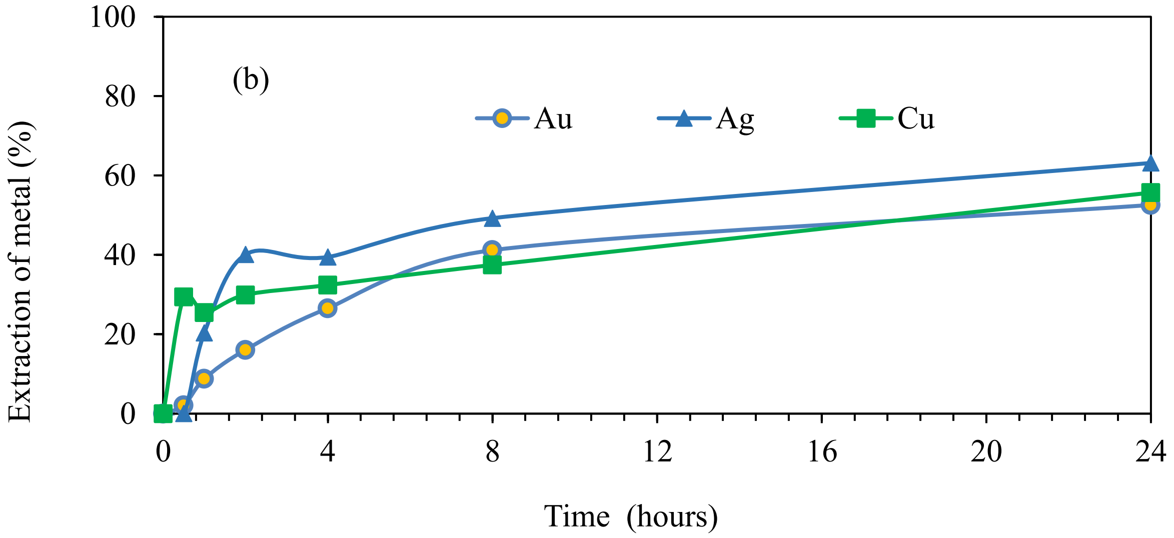
Figure 29. Direct effect of cyanide at high concentrations

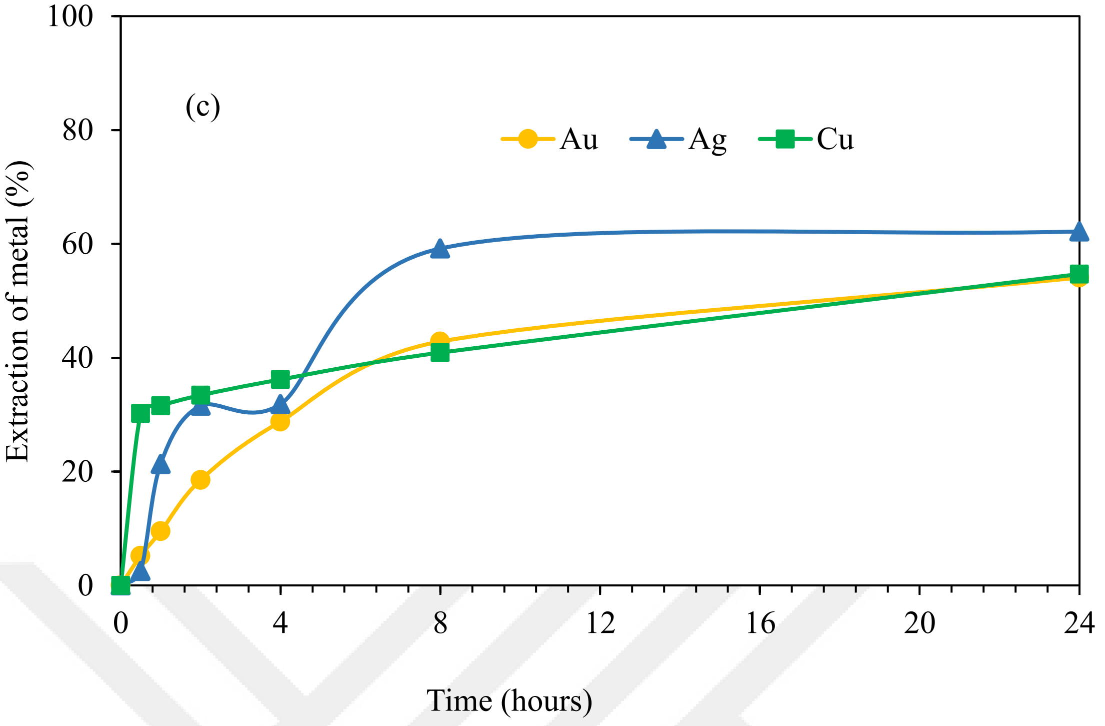
4.1.1. Effect of Ultra-fine Grinding

This approach was employed as a physical pretreatment to enhance the extraction of gold. This technique is applied to set free the grains of gold finely distributed in gangue minerals. The various grinding times (15, 30 and 45 minutes) resulted in the production of samples with sizes (d_{80}) of 23 µm, 15 µm, 8 µm respectively. Figure 30 illustrates the results of cyanide leaching of the ground samples for 24 hours. There was no a remarkable effect of the ultra-fine grinding on the extraction of gold, despite the fact that sodium cyanide concentration used in this experiment was increased to 8 g/L (Figure 30). The highest extraction obtained was 54 % when the sample was ground for 30 minutes. This may be due to physical encapsulation of invisible gold, which apparently impedes the contact between gold and cyanide. Bas et al. (2012), investigated the direct cyanidation of a copper-rich gold ore, they found poor recoveries (<12%) of gold leached from the ore under cyanide concentration of less than or equal to 1.5 g/L. Celep et al. (2016), noted an increment of about 40% (from 45% to 85%) in the gold extraction from a pyritic concentrate just after applying ultra-fine grinding as a pretreatment method before the cyanidation. Their findings suggest that concomitantly grinding the concentrate and increasing cyanide concentration (to 5 g/L) are required to achieve acceptably high gold extractions. Ellis and Gao (2003) noted an appealing extraction of gold at Kalgoorlie Consolidated Gold Mines (KCGM) after using this method as a pretreatment.

It is well established that direct leaching of copper-rich gold ores/concentrates by cyanide results often in low gold extractions (<80%) with high consumption of cyanide due to the ready dissolution of some minerals of copper in cyanide solutions. This phenomenon was vindicated by some investigators (Celep et al., 2015; Celep et al., 2016; Ellis and Gao, 2003; Malange and Olubambi, 2012).







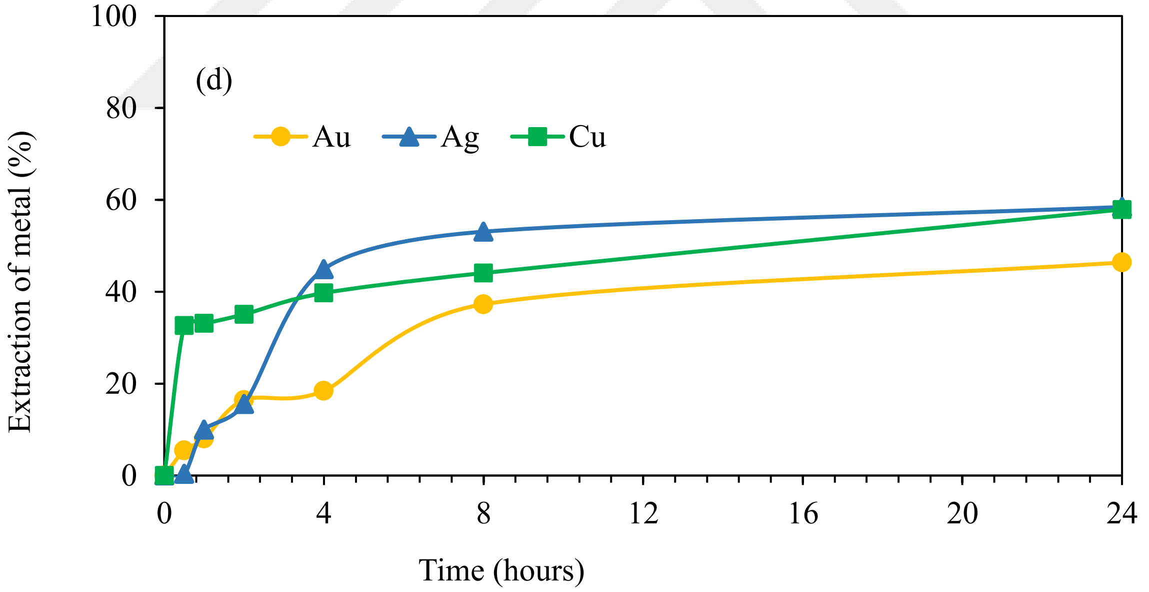


Figure 30. Effect of ultra-fine grinding on the extraction of gold (a) As received (73µm, 8 g/L NaCN, S/L: 25%) (b) Ground sample (15 min, 23µm, 8 g/L NaCN, S/L: 25%) (c) Ground sample (30 min, 15µm, 8 g/L NaCN, S/L: 25%) (d) Ground sample (45 min, 8µ, 8 g/L NaCN, S/L: 25%)

4.1.2. Diagnostic Leaching Tests

It is well established that a variety of mineral phases would be destroyed by the use of HCl and HNO_3 in a sequential acid leaching as a pretreatment route. It is very hard to determine the phases decomposed and thus make a robust inference from the data. This is attributed by the non-selectivity nature of treating refractory ores by acid leaching. However, in the initial phases of acid leaching by hydrochloric acid, the carbonates and oxides would be utterly broken down to set free the gold associated (Lorenzen and Van Deventer, 1993). For better comprehension of the behaviour of MLG ore, diagnostic leaching was applied and the results are presented in Figure 31.

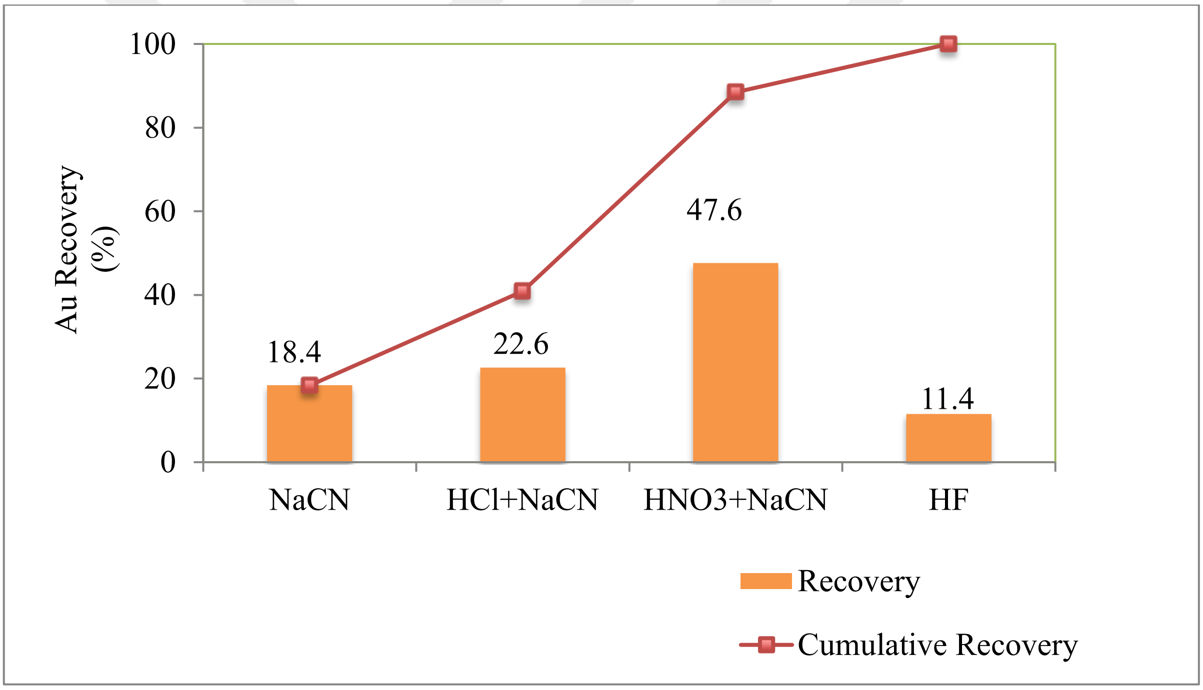


Figure 31. Extraction of gold following each stage of acid treatment (NaCN leaching:1.5 g/L NaCN, 24 h, pH= 10.5-11; HCl leaching: HCl (12%), L/S=2:1, 8 h, 60 °C; HNO_3 leaching: HNO_3 (33%), L/S=2:1, 6 h, 60 °C)

As depicted in Figure 31, the extraction of gold following each step of acid treatment increased stepwisely. It can be inferred that the primary reason for the low gold extractions appeared to be attributed by the obstruction of fine particles of gold within sulfide, oxide and carbonate mineral phases. Moreover 47.6% of gold which were extracted at HNO_3 + CN stage shows that approximately half of the refractory gold were associated with sulfide minerals. After the treatment of the ore with a series of HCl, and HNO_3, about 88.6% of gold was extracted. The remaining 11.4 percent may show the amount of gold encapsulated in silicates which may be treated by hydrofluoric acid (HF).

4.1.3. Effect of Roasting

Roasting of refractory ores is performed to oxidize the sulfides and remove the organic matter prior to leaching for gold extraction (Marsden and House, 2006). The roasting is principally to get a calcined product less or more porous, thus increasing the availability of the occluded gold in the subsequent cyanide leaching. Therefore in the current research roasting was employed as a pretreatment step before the leaching procedure by cyanide. Figure 32 demonstrates a substantial improvement in the extraction of gold after roasting.

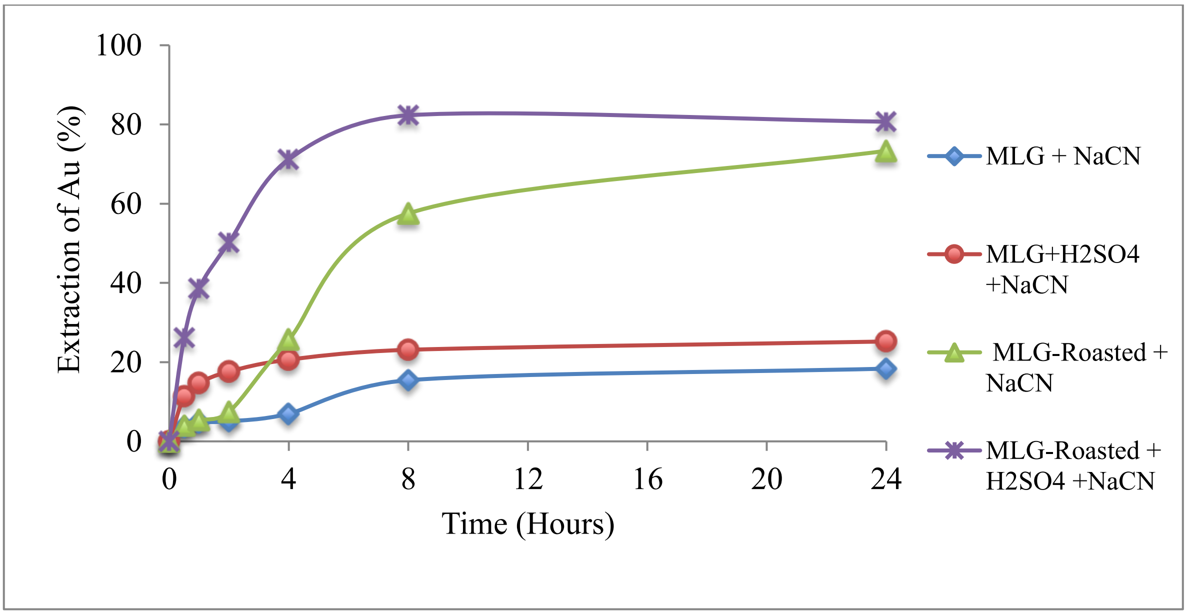


Figure 32. Effect of roasting and sulphuric acid as a pre-treatment on the extraction of gold (Roasting: 650°C, Acid Leaching: 1M H_2SO_4, CN leaching: 1.5 g/L , 3 g/L and 5 g/L NaCN, S/L = 25%)

It can be deduced from Figure 32 that when the MLG sample (as received) was leached by direct cyanidation, the recovery was just 18.4%. However, when the same sample was pre treated by sulphuric acid prior to cyanidation, there was an increment of about 6.9% in the amount of gold extracted in the cyanide leaching. A remarkable increase in gold extraction was observed when MLG was roasted at 650°C and then leached by cyanide leaching. About 73.4% of gold was extracted, this indicated the importance of roasting the sample prior to cyanidation. Similarly, the dissolution rate of gold increased when the roasted sample was firstly pre treated with sulphuric acid to eliminate the interference of cyanide soluble copper.

It can also be seen from Figure 33 within the first 8 hours the dissolution of gold was about 96.2%. Thereafter extractions were very slow. When the same roasted sample was leached at 3 g/L and 5g/L NaCN, about 86.5 and 96% of gold respectively were extracted (Figures 33 and 34). These improvements with increasing cyanide concentration can be linked with the formation of copper cyanide complexes of high cordination numbers which have a less significant effect to the process.

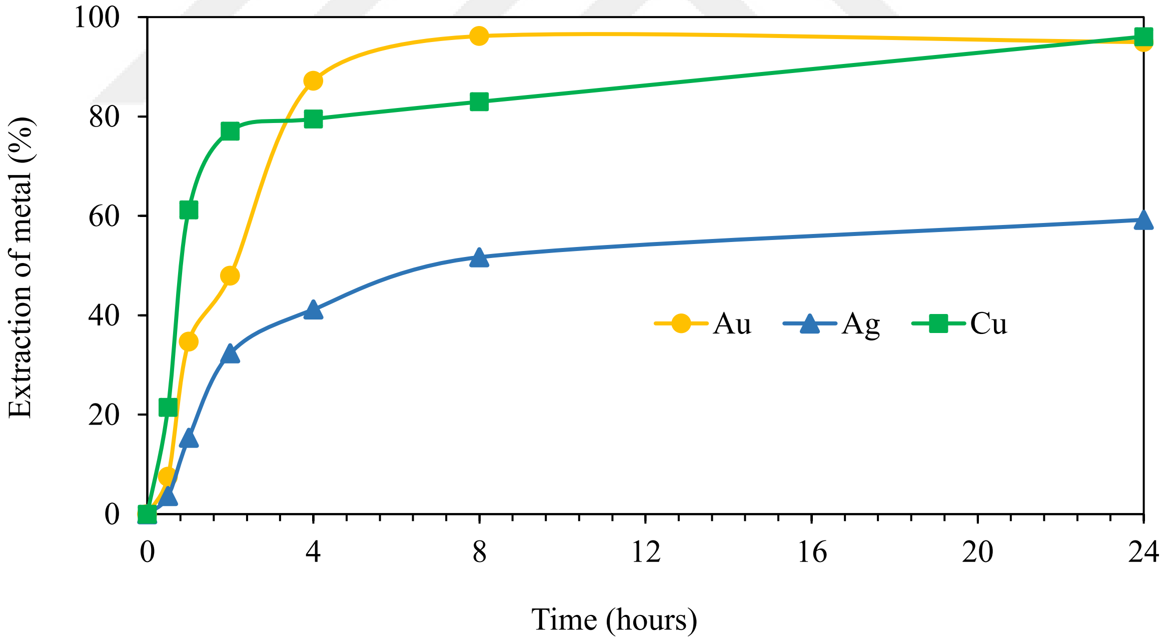


Figure 33. Cyanide leaching of roasted MLG ore at 5 g/L NaCN, S/L: 25%

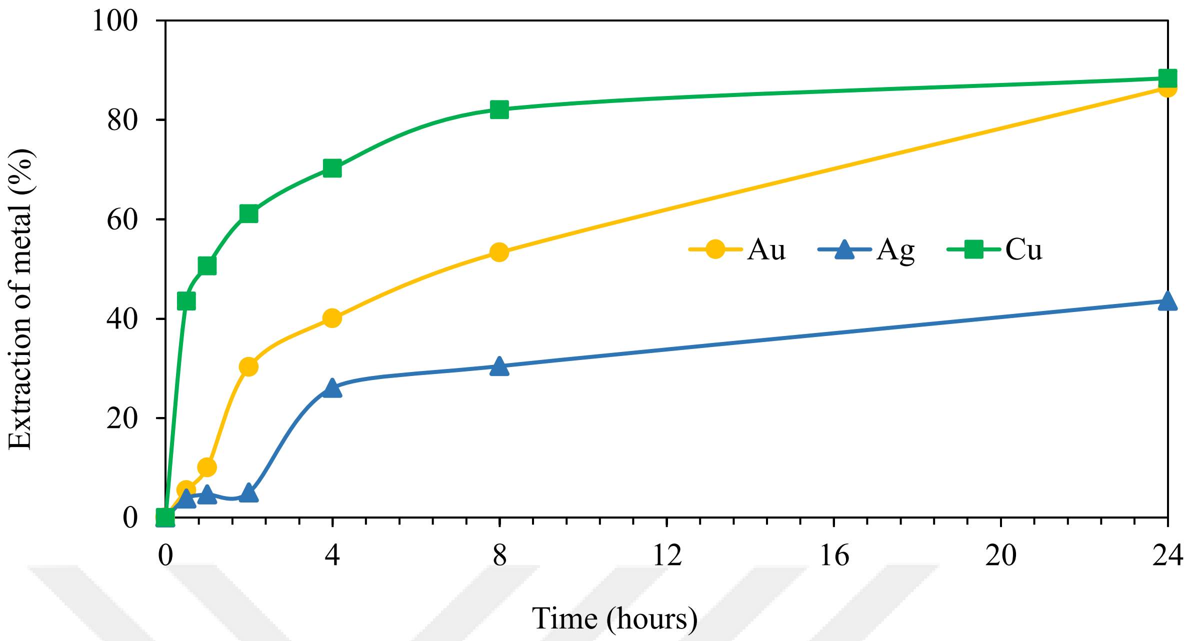


Figure 34. Cyanide leaching of roasted MLG ore at 3 g/L NaCN, S/L: 25%

4.2. Preliminary Tests used for Resins Selection

Shake flask experiments were carried out as a way to select most selective adsorbents for gold over copper. Cyanide leach liquors containing different levels of copper were tested under the conditions presented in Table 19. The first cyanide liquor tested contained 28.2 mg/L of gold and 2804 mg/L of copper, which was obtained from a sample leached at 1.5 g/L NaCN. In the adsorption tests, distribution ratios of gold and copper, selectivity coefficient and amount of metal loaded on adsorbent (g/ton) were calculated. Amongst the adsorbents Dowex XLT showed a non-selective trait as it was loading both Au&Cu. This strong base resin operates in a pH range of 0-14 with excellent kinetics. Other strong base resins used (Purolite A193 and Purogold A194) showed a high loading capacity and fast loading rates, but, poor selectivity due to adsorption of metals such as Cu, Zn, Fe, &Ag. Activated carbon was found to have a high loading capacity compared with resins despite the fact that some copper metal was also loaded onto it. This showed its low selectivity (gold over copper). The only adsorbent which hardly adsorbed copper was the medium base resin (Purogold S992). This indicates its high selectivity compared with other resins and activated carbon tested. Using the latter resin will not need extra inventory for the loading of copper hence the productivity of the cell-house will be higher.

Table 19. Parameters used during preliminary tests

Adsorbent	Concentration of Adsorbent (g/L)	Solution Volume (ml)	pH	Stirring Speed (rpm)	Temperature (°C)
Dowex XLT	5	50	10.5	170	25
Activated Carbon					
Purolite A193					
Purogold S992					
PurogoldA194					

Table 20 illustrates the results of the adsorption tests. From the results a higher distribution ratio for gold was achieved by activated carbon followed by Purogold A194. However, Dowex XLT attained the higher distribution ratio for copper, followed by Purolite A194. The selectivity of an adsorbent is defined as its gold loading relative to copper loading (it was formulated as the ratio of distribution of gold to that of copper.

Table 20. Comparison of activated carbon with IX resins for adsorption of Au and Cu from a real cyanide leach solution

Adsorbent	D (Au)	D (Cu)	µ (Selectivity)	Au on Adsorbent (g/ton)	Cu on Adsorbent (g/ton)
Dowex XLT	82	145	0.56	1814	220676
Activated Carbon	619.6	68.4	9.06	4406	137256
Purolite A193	138.8	82.2	1.69	2510	156004
Purogold S992	257.9	12	21.5	3373	31648
Purogold A194	404.3	115.4	3.5	3946	193672

It is noticeable that the amounts of gold loaded onto activated carbon and Purogold A194 are higher than that loaded onto Purogold S992. However, the latter has the superior selectivity for gold with its characteristic of having the lowest copper loading. Earlier works (Van Deventer et al., 2012) demonstrated that no copper was loaded onto Purogold S992 from conventional gold leach liquor. Figure 35 and Figure 36 demonstrate the amount of metals loaded onto adsorbents in terms of percentage. Activated carbon was found to have the highest gold recovery from the pregnant solution under the same conditions for adsorption (Figure 35). These findings also indicate that Purogold A194 has also high capacity for adsorption of gold compared with other resins tested. On the other hand, Dowex 21K XLT appeared to show the highest recovery for copper. A similar level of copper recovery was also observed for Purogold A194. Consistent with its high selectivity, Purogold S992 recovered the lowest level of copper amongst the adsorbents tested. It should be noted that Purogold S992 is very sensitive to pH, and performs better at pH lying between 10 and 11. Van Deventer et al. (2012) showed that optimum Au loading and minimum Cu co-loading of Purogold S992 was between 10.4 and 10.5. Voiloshnikova et al. (2014) showed that the optimal pH value is 10.1-10.3. In this study the pH was maintained between 10-10.5 in the tests where Purogold S992 was used.

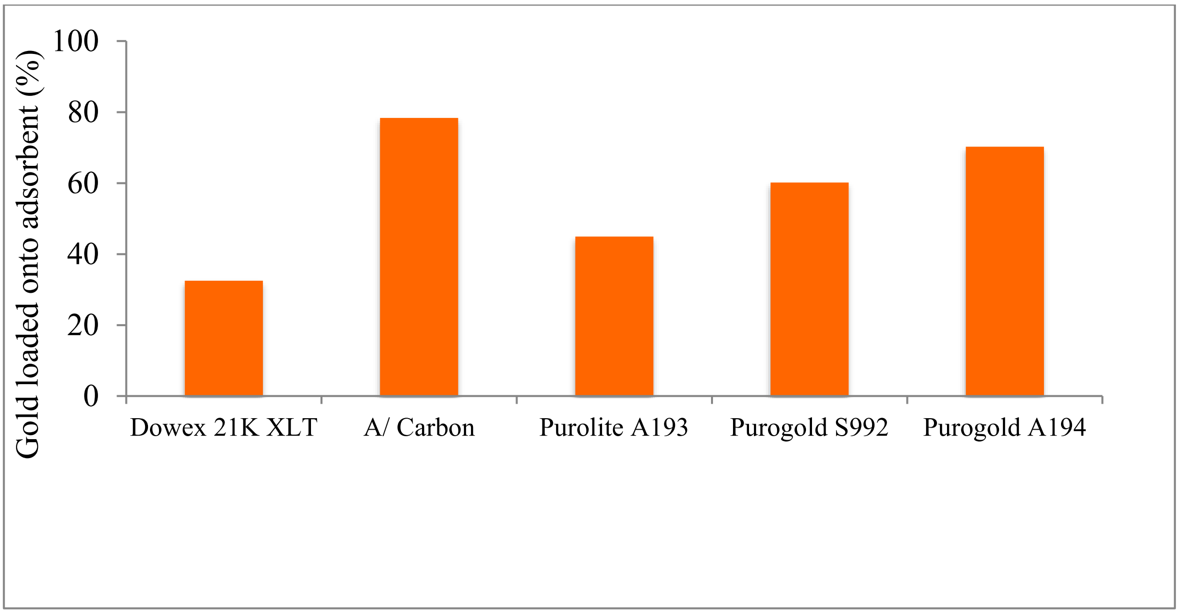


Figure 35. Gold loaded onto adsorbents (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

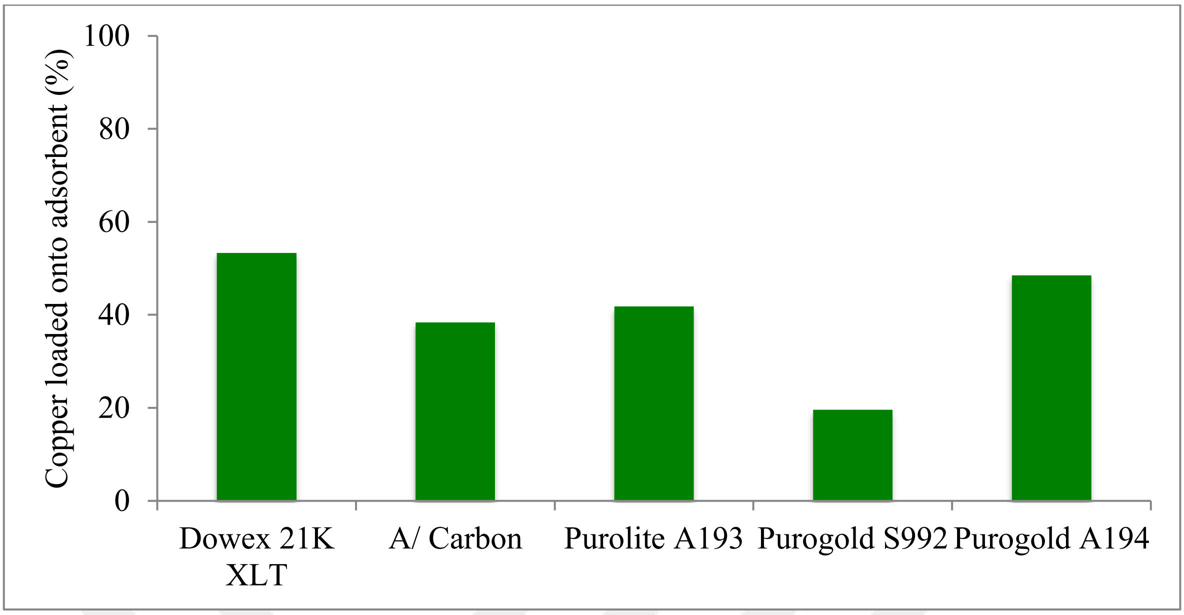


Figure 36. Copper loaded onto adsorbents (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH:10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

Further tests were carried out using the pregnant leach solution produced from cyanide leaching (1.5 g/L NaCN) of the ore which was pretreated by acid leaching (1M H_2SO_4) to remove reactive copper. Following this acid treatment, about 95% of copper dissolved. The liquor contained 26.1 ppm of gold and 62.4 ppm of copper. The sequence of the distribution ratios obtained followed the same trend as for the previous tests (Table 19). Following the initial tests activated carbon, Purogold A194 and Purogold S992 were used as the most promising adsorbents in terms of selectivity and capacity. Figure 37 and Figure 38 show the amount of metals loaded onto adsorbents. Results showed that 96% of gold and only 32% of copper was loaded onto activated carbon. Similar loadings (89% Au and 37% Cu) was also observed for Purogold A194. In comparison Purogold S992 selectively loaded only 10% of copper and 85% of gold. As far as the process economy is concerned, Purogold S992 would be highly preferable since no additional inventory will be required for the copper loading.

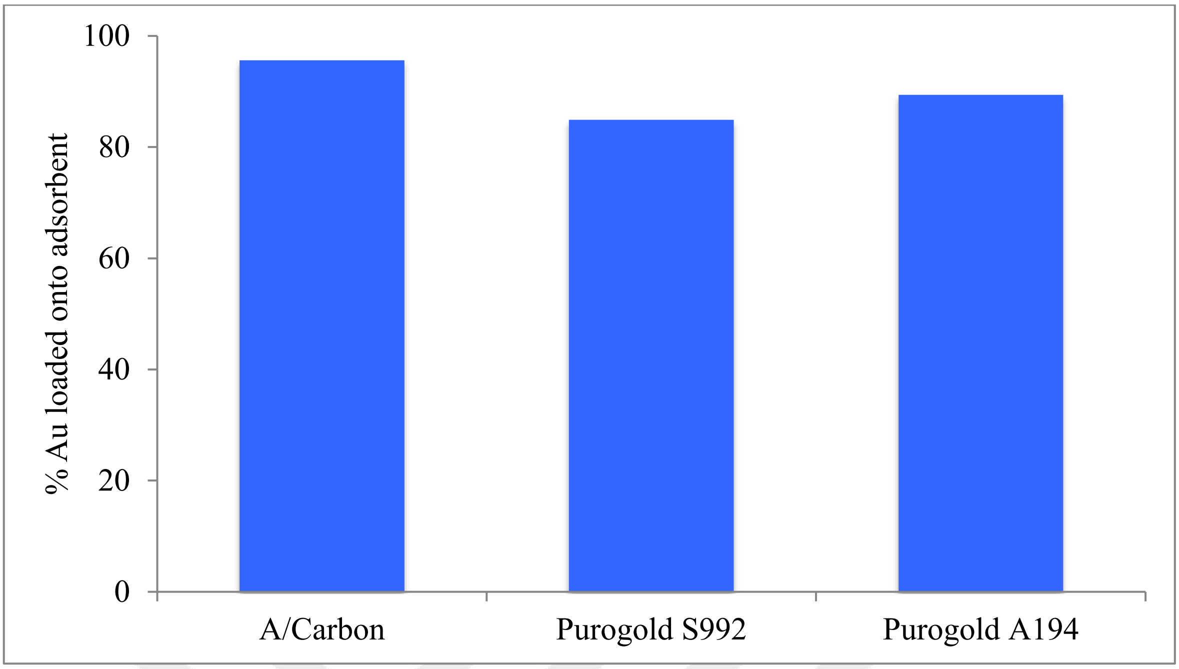


Figure 37. Gold loaded onto adsorbents (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

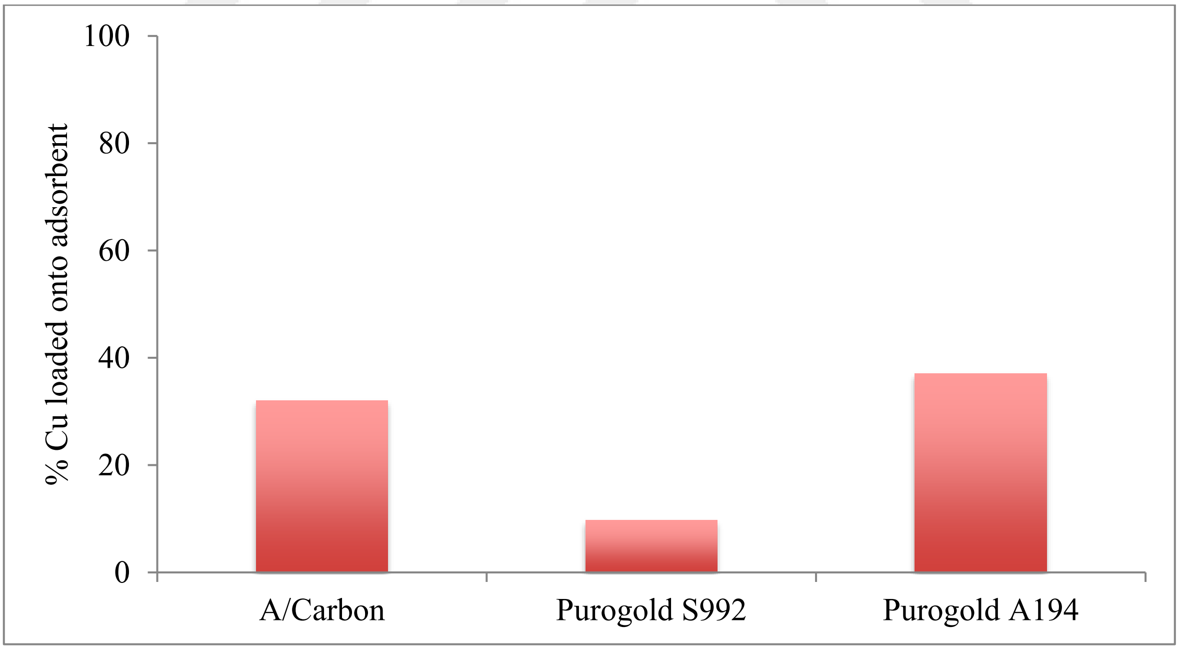


Figure 38. Copper loaded onto adsorbents (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

The adsorption test results are as depicted in Figures 39 to 44. These adsorption tests showed that 24933 g of gold were loaded per ton of activated carbon within 4 hours (Figure 41). During the first hour of adsorption 11292 g/ton that is almost half of the amount of gold loaded onto Purogold S992 resin was observed, at the same interval 19632 g of gold were loaded per ton of activated carbon. These data can simply show how the loading capacity of activated carbon was high. Despite the fact that the amount of gold loaded onto this adsorbent was high, it was not as selective as Purogold S992. The latter loaded only 1104 g/ton of copper whilst 15548 g/ton of copper was loaded onto activated carbon. The extent of adsorption onto strong base anion resin (Purogold A194) was also high as it loaded 23303 g/ton of Au and 18216 g/ton of Cu. Following these results Purogold S992 was the adsorbent, which showed higher selectivity than others. This is consistent with earlier research works done by different investigators (Van Deventer et al., 2012 and 2014), which showed that medium base resin such as Purogold S992 is more selective for gold than copper.

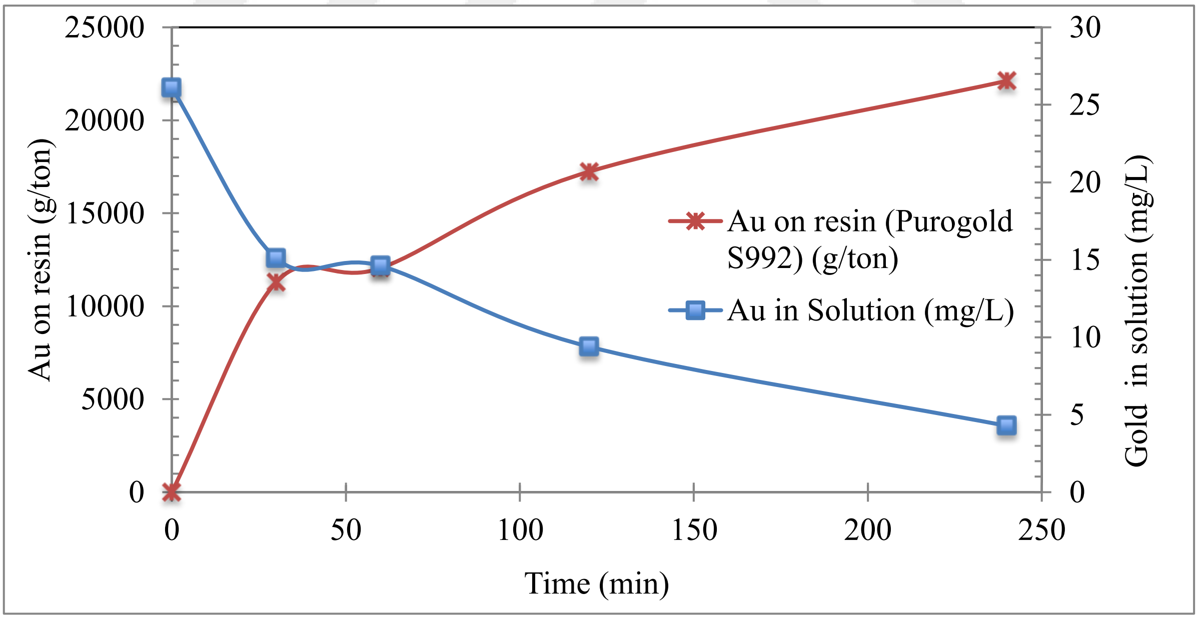


Figure 39. Gold loaded onto purogold S992 (g/ton) and gold in solution (mg/L) (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

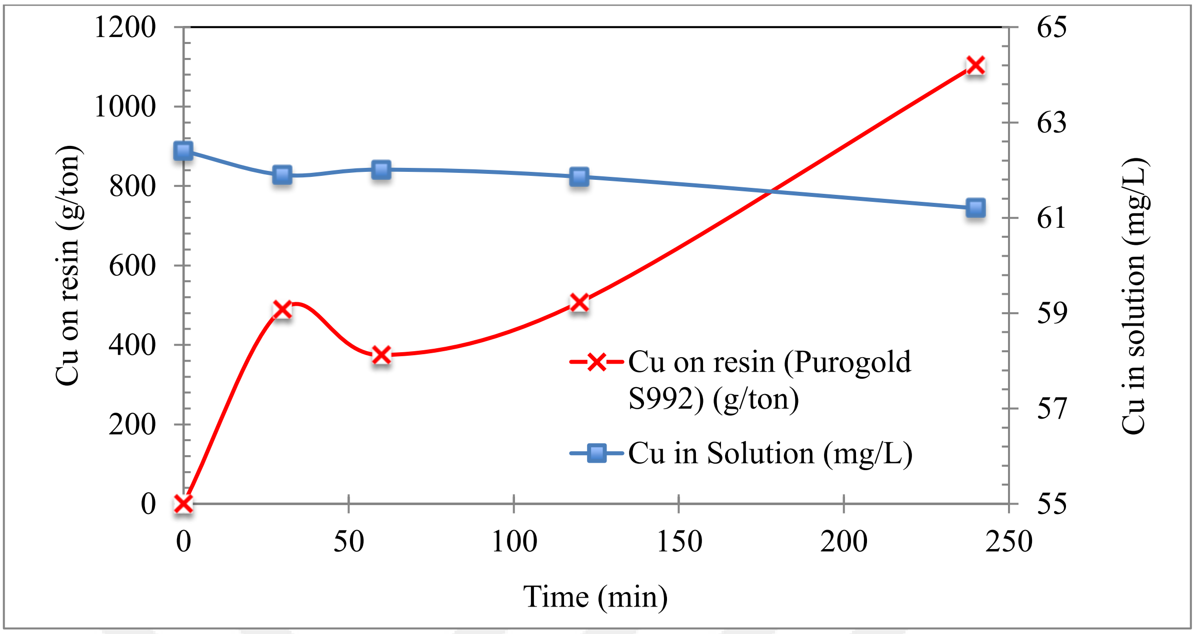


Figure 40. Copper loaded onto Purogold S992 (g/ton) and gold in solution (mg/L) (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

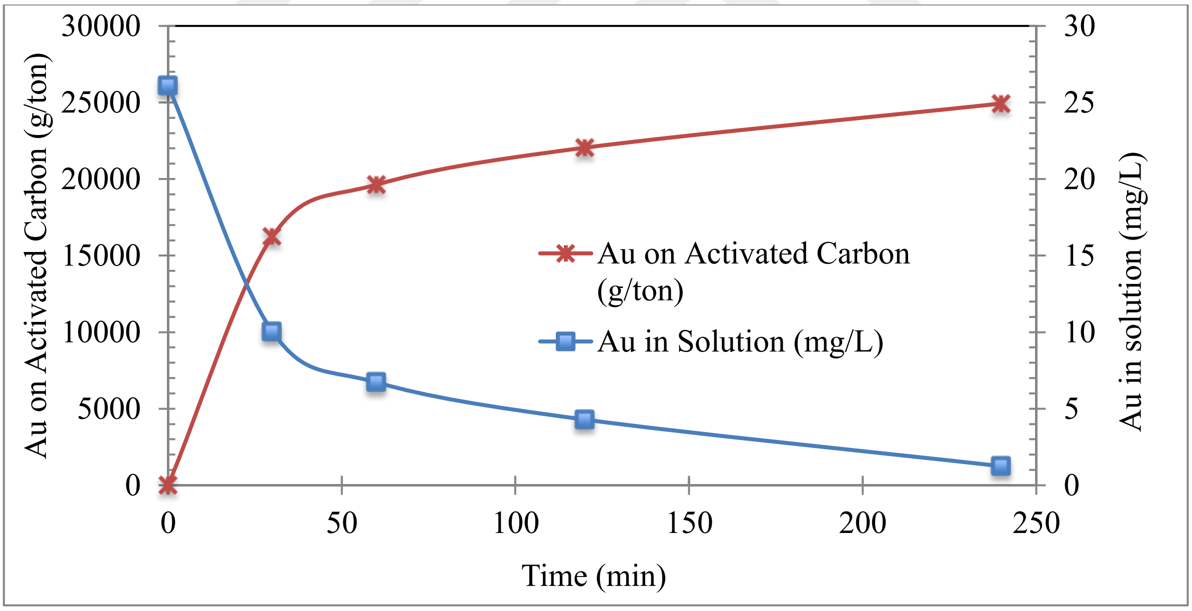


Figure 41. Gold loaded onto activated carbon (g/ton) and gold in solution (mg/L) (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

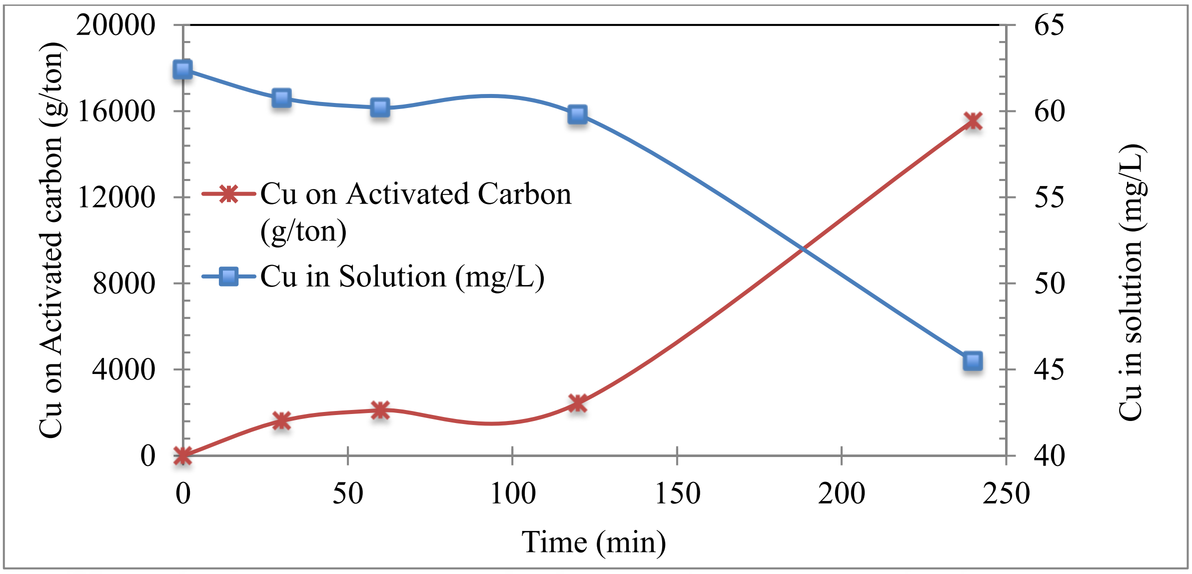


Figure 42. Copper loaded onto activated carbon (g/ton) and copper in solution (mg/L) (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

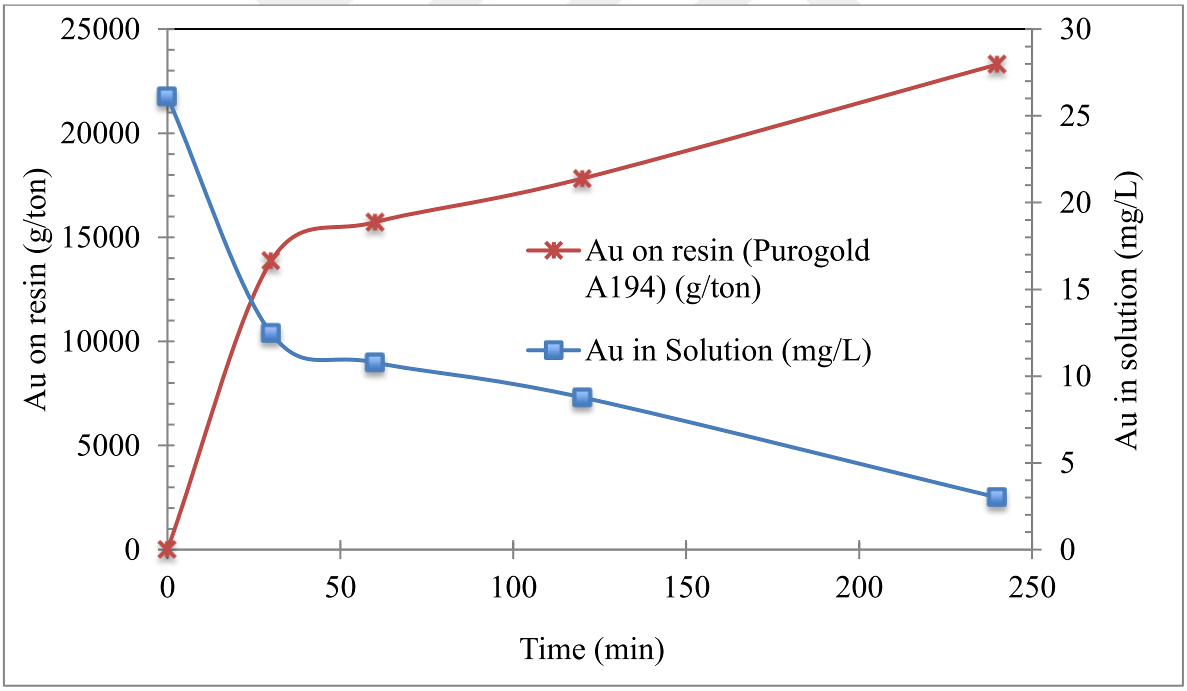


Figure 43. Gold loaded onto Purogold A194 (g/ton) and gold in solution (mg/L) (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

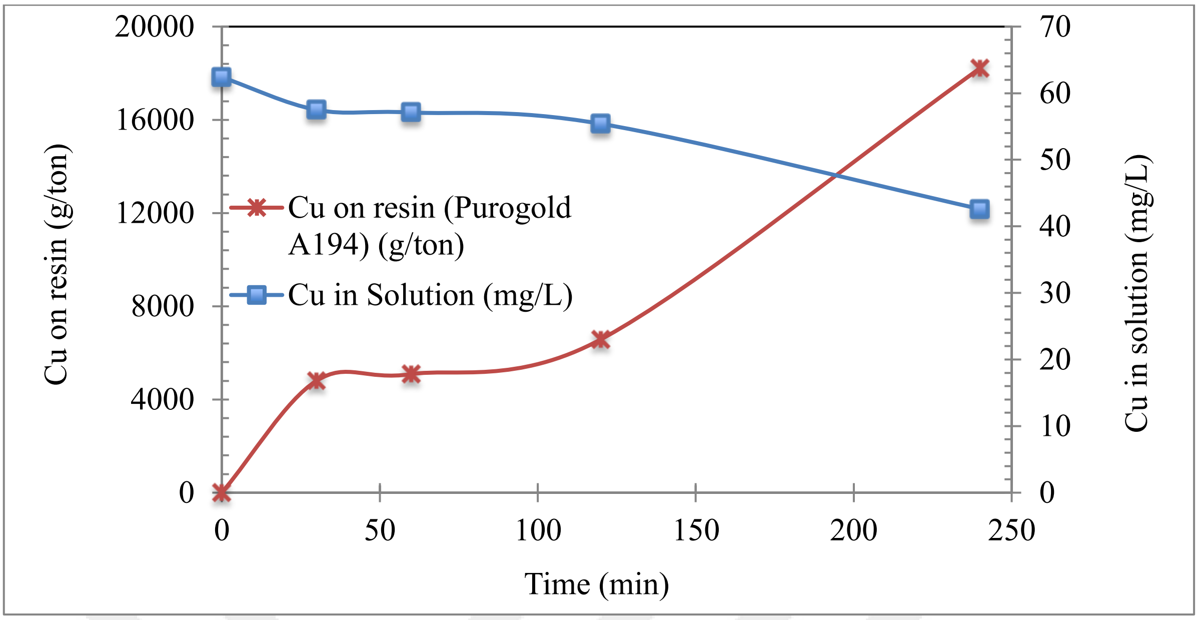


Figure 44. Copper loaded onto Purogold A194 (g/ton) and copper in solution (mg/L) (Conc of Adsorbent: 5 g/L, Solution volume: 50ml, pH: 10.5-11, Stirring speed: 170 rpm, Temperature: 25 °C)

As depicted in Figures 39-44 only Purogold S992 loaded low amount of copper compared to Purogold A194 and activated carbon. Mathematically the coefficient of selectivity or separation factor is inversely proportional to the distribution ratio of copper. Thus, the lower value of distribution ratio of this medium base anion resin (Purogold S992) makes it more selective than other adsorbents used. Several studies (Fleming, 1984; Marsden and House, 2006; Van Deventer et al., 2012) showed that weak base resins possess higher selectivity to gold than strong base resin from cyanide leach solutions bearing a wide variety of cyanide complexes. Table 20 illustrate the distribution ratios of copper and gold and selectivities of the adsorbents tested.

Table 21. Comparison of distribution ratios of copper and gold and selectivity of activated carbon and resins

	D (Au)	D (Cu)	Selectivity (μ)
Activated Carbon	20026.6	341.7	58.6
Purogold S992	5168.4	18	287.1
Purogold A194	7757.2	540	14.4

4.3. Carbon in Leach (CIL) and Carbon in Pulp (CIP) Tests

The dosages of carbon were found by taking the consideration of the solution volume. In the carbon-in-leach process, carbon was introduced at the commencement of the experiment, this allowed both adsorption and leaching to occur at the same time. In CIP process carbon was introduced when leaching process has already finished. Earlier studies (Habashi, 1987; Marsden and House, 2006) had shown that CIL process is effective once employed to treat preg-robbing ores, whilst the CIP is utilized in general scenarios. The percent of Au, Ag and Cu loaded onto activated carbon via CIL are depicted in Figures 45 to 47.

As presented in Figure 45**,** the highest recovery rate of Au onto activated carbon was achieved at 5 g/L NaCN and 5 g/L of activated carbon. This could be due to high cyanide concentration, which limits the adsorption of copper onto carbon as high valence copper complexes such as $Cu(CN)_4^{3-}$ complex increasingly predominates at high cyanide levels (Marsden and House, 2006).

Also the results indicate that there was a linear increase in the recovery rate as the carbon dosage increased. Figures 45-47 below show that activated carbon has low affinity for base metals compared with gold and silver. Marsden and House (2006) reported that the adsorption rate of base metals onto activated carbon is less favored compared to gold and silver. It follows the following trend: $Au(CN)_2^- > Hg(CN)_2 > Ag(CN)_2^- > Cu(CN)_3^{2-} > Zn(CN)_4^{2-} > Ni(CN)_4^{2-} >> Fe(CN)_6^{4-}$.

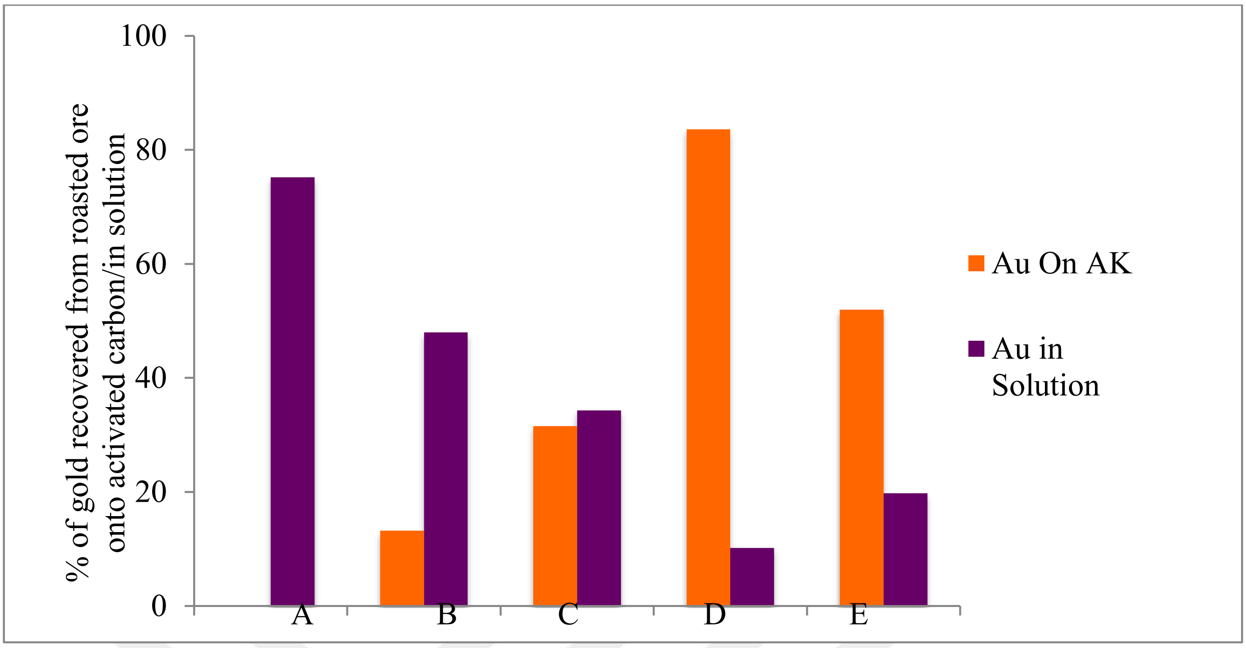


Figure 45. Percentage of gold recovered from roasted ore onto activated carbon/in solution in CIL (A: 0g/L Carbon + 1.5 g/L NaCN, B: 2g/L Carbon + 1.5 g/L NaCN, C: 5 g/L Carbon + 1.5 g/L NaCN , D: 5 g/L Carbon + 5 g/ L NaCN, E: 10 g/L Carbon + 1.5 g/L NaCN)

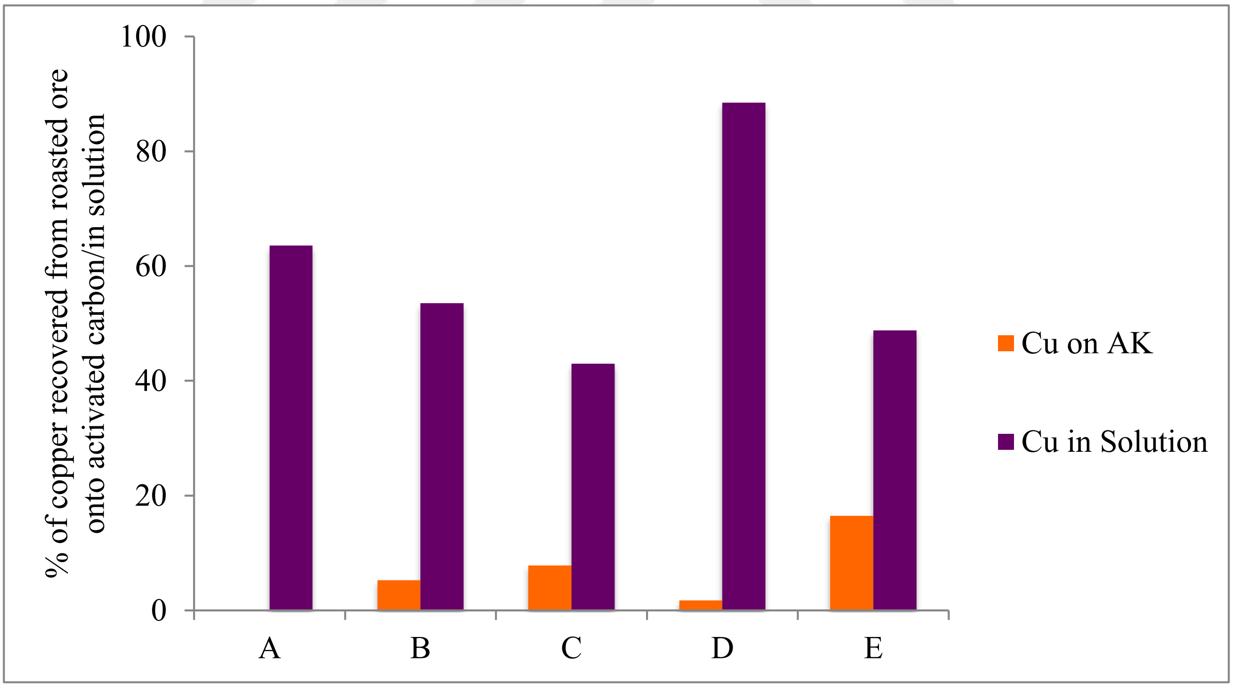


Figure 46. Percentage of copper recovered from roasted ore onto activated carbon/in solution in CIL (A: 0 g/L Carbon + 1.5 g/L NaCN , B: 2 g/L Carbon + 1.5 g /L NaCN, C: 5 g/L Carbon + 1.5 g/L NaCN , D: 5 g/L Carbon + 5 g/L NaCN , E: 10 g/L carbon + 1.5 g/L NaCN)

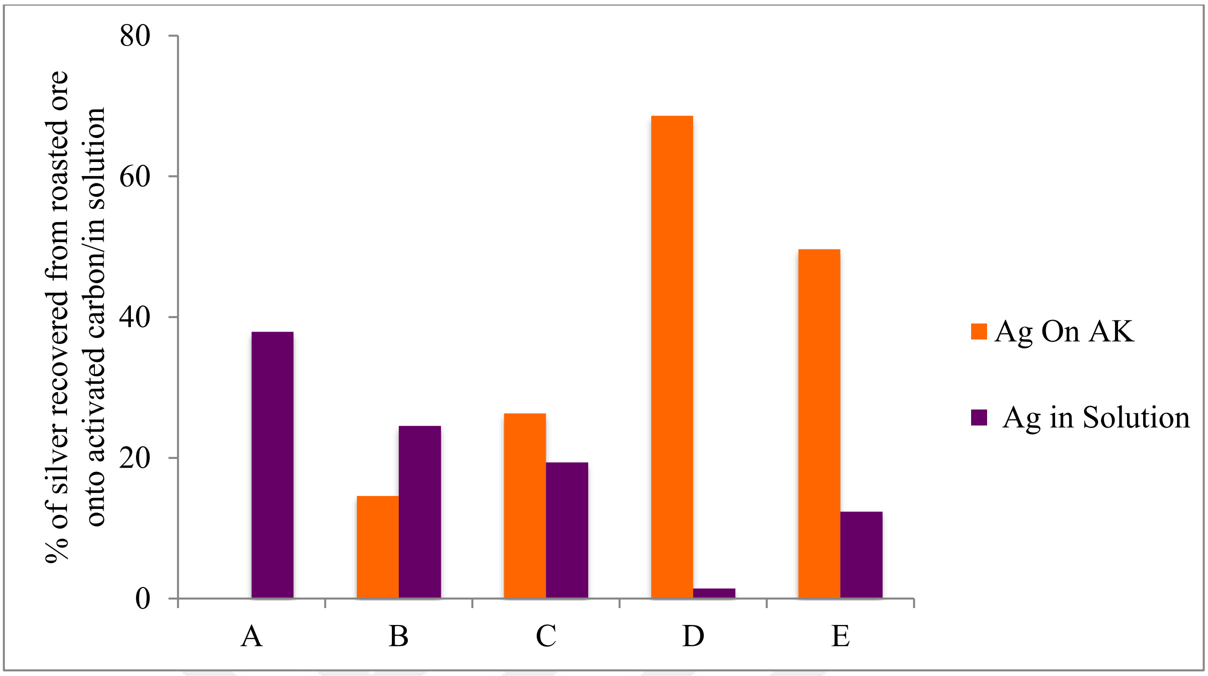


Figure 47. Percentage of silver recovered from roasted ore onto activated carbon/in solution in CIL (A: 0 g/L Carbon + 1.5 g/L NaCN , B: 2 g/L Carbon + 1.5 g/L NaCN, C: 5g/L Carbon + 1.5 g/L NaCN, D: 5 g/L Carbon + 5 g/L NaCN, E: 10 g/L Carbon + 1.5 g/L NaCN).

Accordingly, the amount of metals recovered onto activated carbon followed this trend Au>Ag>Cu. The concentration of carbon in the slurry plays a great role in the whole mechanism of adsorption. Low recoveries achieved at low concentration of carbon could be due to saturation of carbon following the adsorption of gold. Thus, the greatest quantity of the gold remain non adsorbed and get lost as gold that has been leached (Marsden and House, 2006). However, when the concentration of carbon is increased, the gold adsorption/recovery is enhanced. Several researchers pointed out that the gold adsorption kinetics onto carbon are slower in CIL process than in CIP process, this is attributed by the incompletion of the leaching process when the pulp come into contact with activated carbon in the stages of adsorption of CIL and the Au concentration is lower in solution. Extra carbon is needed to match the performance achieved metallurgically in CIP (Nicol et al., 1984; Marsden and House, 2006).

In the CIP tests, the same activated carbon dosages were tested. In these tests carbon was introduced when the leaching process (residence time = 24 hours) was complete. The results showed an increment in the percentage of metals loaded onto activated carbon. As it was also observed in CIL tests, carbon concentration played an essential role in the process of adsorption. For the mass balance calculations, 3 g of sample of leach residue was removed from each vessel at the end of the leaching period (first 24 hours) in order to know the exact amount of feed grade getting into the CIP. This sample was digested by an aqua regia for metal analysis. There was a linear relationship between adsorption (Concentrations of Au, Ag, and Cu in activated carbon) and gold, silver, and copper concentrations in barren solution. The results of these tests are presented in Figures 48 to 53. The rate of gold adsorption onto carbon increased from 66.6% to 98.7% when the carbon dosage was increased from 2 g/L to 10 g/L. This showed the proportionality between the carbon concentration and amount of gold loaded onto carbon.

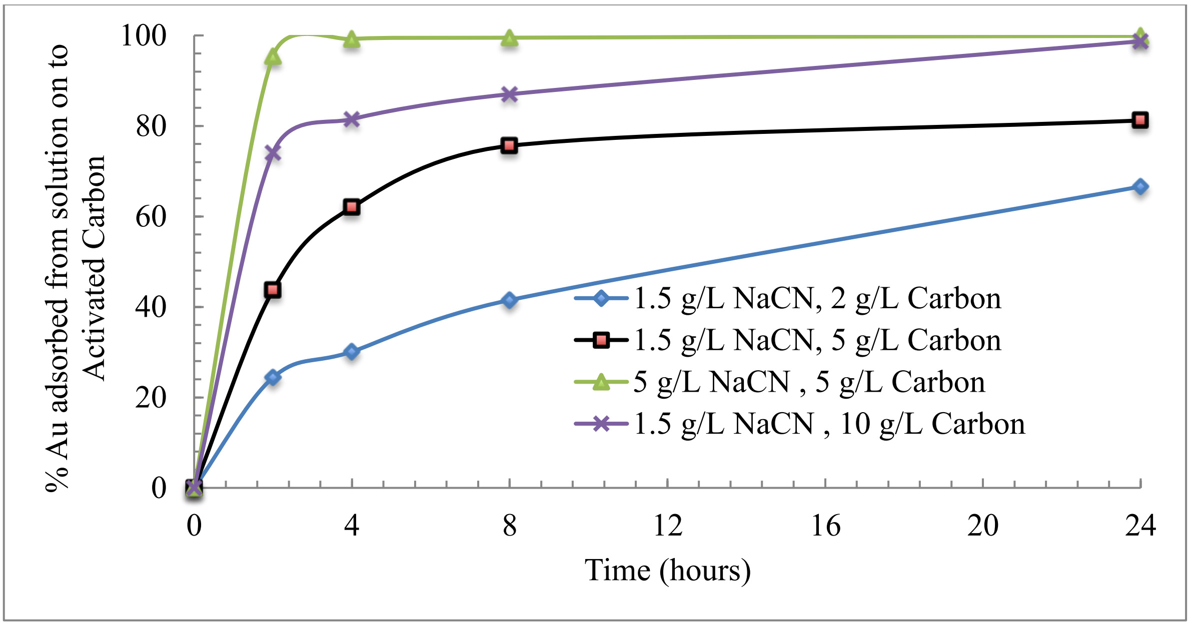


Figure 48 . Gold adsorbed from solution onto activated carbon in CIP (Carbon dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

As far as the percent of gold adsorbed is concerned, over 99% of gold was adsorbed when 5 g/L of carbon was introduced into a liquor, which was leached by 5 g/L NaCN. The initial concentration and final concentration of Au were 27.5 mg/L and 0.032 mg/L. This adsorption level was followed by a solution (25 mg/L) which was produced by 10 g/L of carbon and in leaching by 1. 5 g/L NaCN. After adsorption process, the barren solution contained 0.35 mg/L Au. The general trend of adsorption is illustrated in Figure 49.

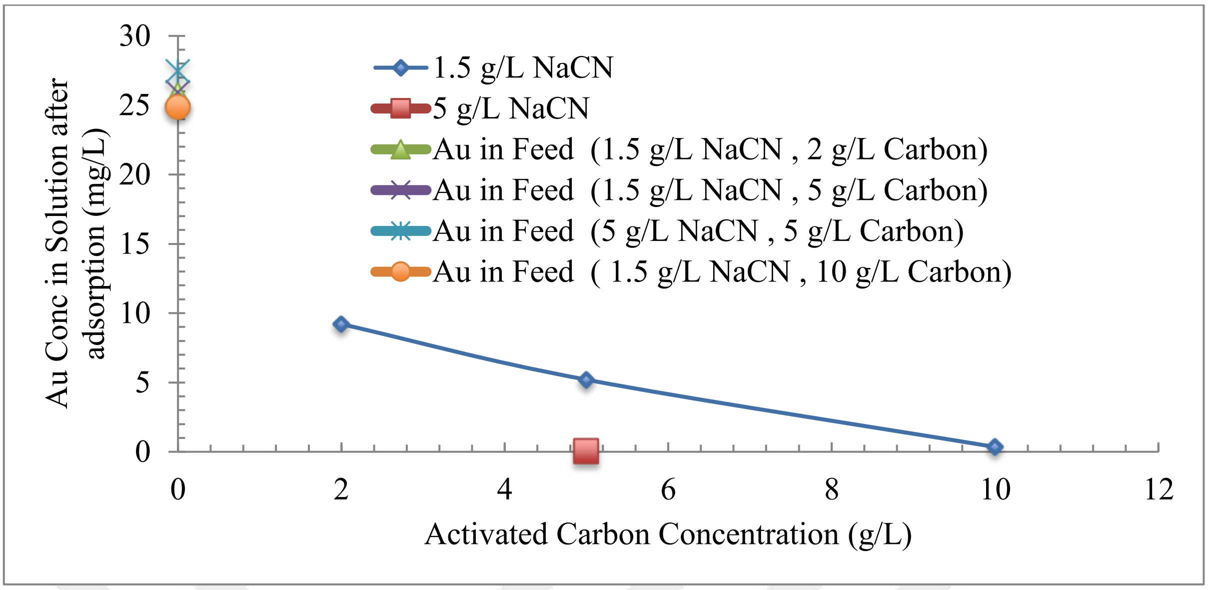


Figure 49. Effect of carbon dosage on the adsorption of gold in CIP

Figure 50 illustrates the percentage of copper adsorbed onto carbon at different carbon dosages. As it was expected, the percentage of copper loaded onto carbon (5 g/L) from the solution generated by 5 g/L NaCN was lower than those attained at different dosages of activated carbon. 3961 mg/L and 3906 mg/L were the initial and final concentration of copper in the solution, indicating that only 55 mg/L of copper was adsorbed onto carbon. The maximum copper recovery with 21.2% was attained at 10 g/L carbon, while the least copper recovery by carbon (5 g/L) was 6.9% which was obtained from the pregnant solution generated by leaching at 5 g/L NaCN. There is also a linear relationship between carbon concentration and percentage of copper loaded onto carbon. When the concentration of carbon was increased from 2 g/L to 10g/L there was a slight increment in the percentage of copper loaded. However this was different for the solution leached by 5 g/L NaCN.

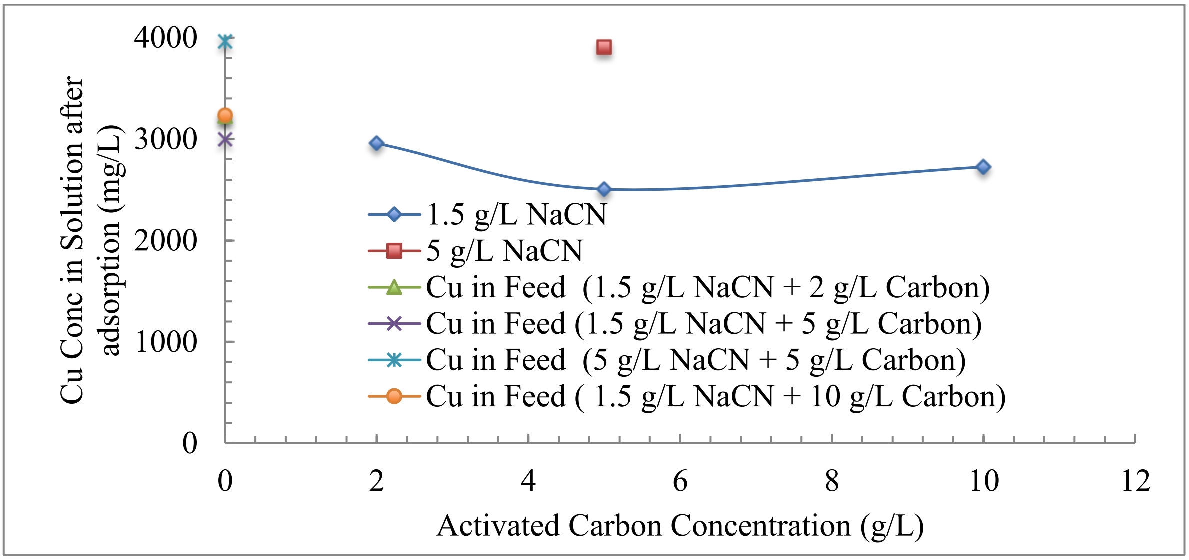


Figure 51 shows the content of metal remaining in the solution following adsorption, the curve obtained here is not as sharp as the one for gold. This shows a slow adsorption level of copper compared to gold. Dai et al., (2010) pointed out that the adsorption process of copper cyanide onto activated carbon initially obeys a first order reaction, however it diverges from it following the change in the mechanism of adsorption. They also showed that as the CN to Cu ratio increases, copper cyanide adsorption onto activated carbon decreases appreciably. However with strong base ion exchange resins, this CN/Cu ratio has little effect on the whole process of copper cyanide recovery (Dai et al., 2010).

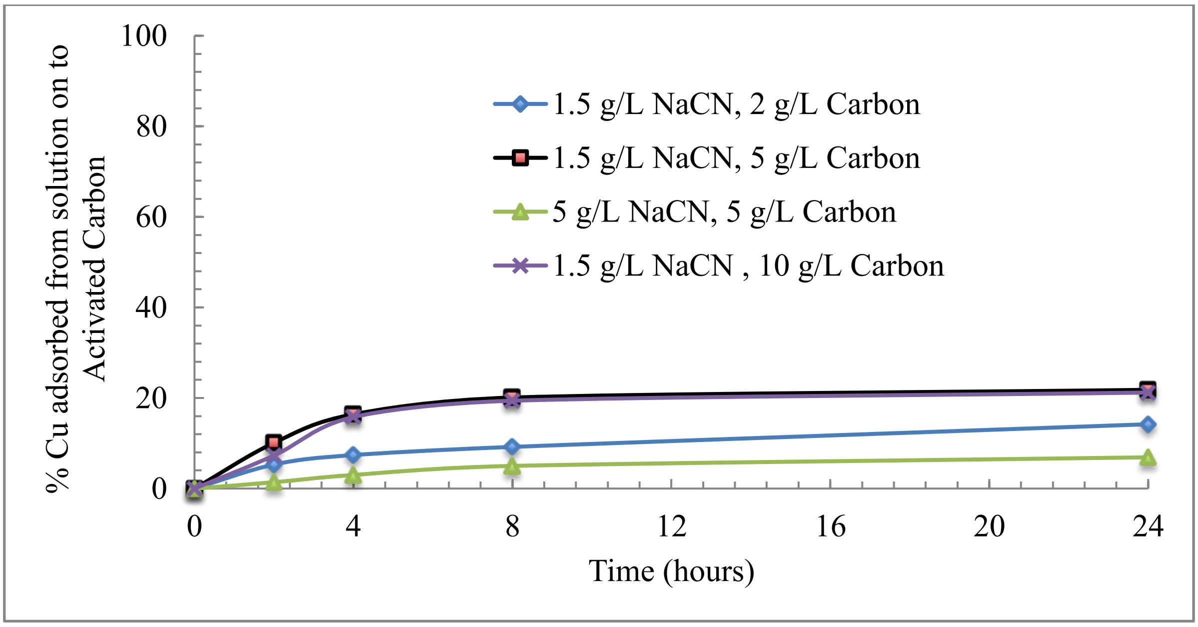


Figure 50. Copper adsorbed from solution onto activated carbon in CIP (Carbon dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

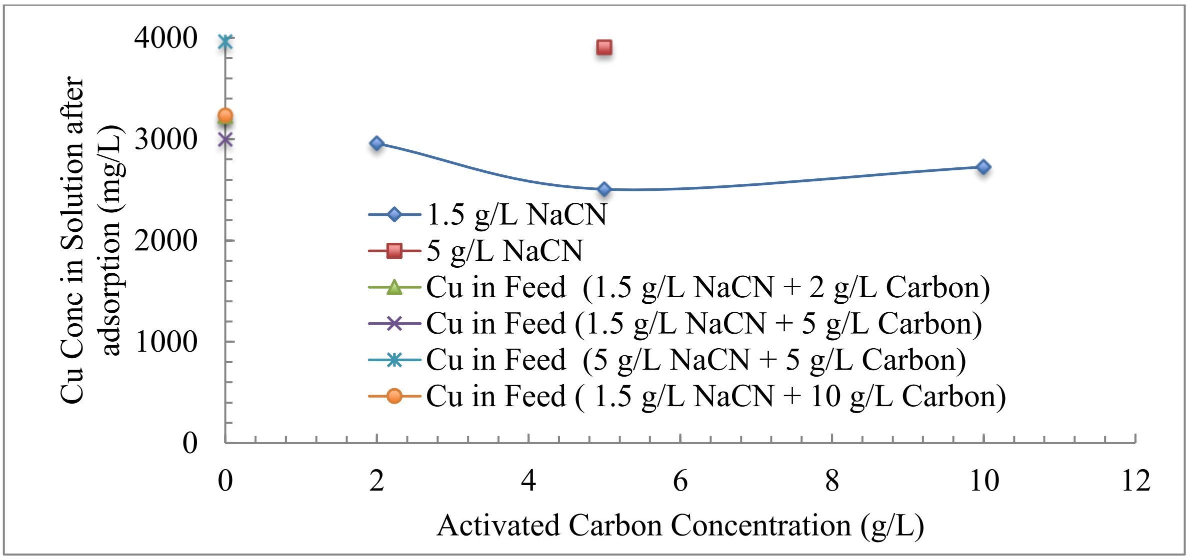


Figure 51. Effect of carbon concentration on the adsorption of copper in CIP

The rate of adsorption of silver onto carbon was also closely examined. It was noticed that the percentage of silver loaded on to carbon increased with an increase in carbon dosage (Figure 52). As in the results observed for gold adsorption, here also the maximum adsorption at 5 g/L carbon was attained from a solution which was leached by 5g/L NaCN. Loaded silver onto carbon was not very far from gold loadings. The results of distribution ratios and selectivity are summarized in Table 22.

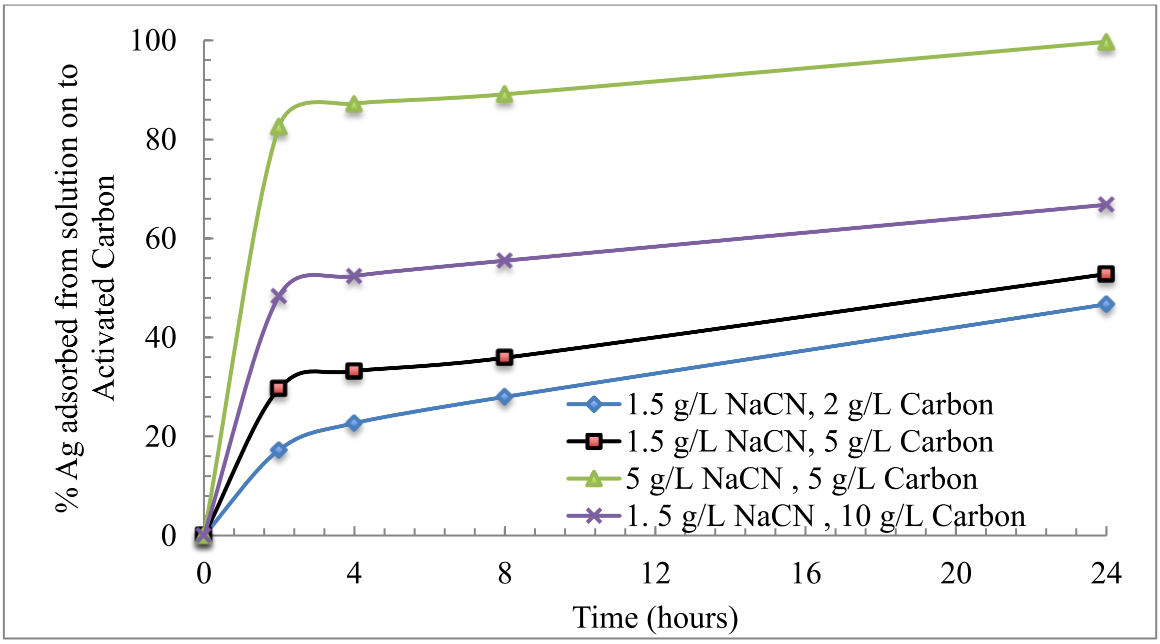


Figure 52. Silver adsorbed from solution onto activated carbon in CIP (Carbon dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

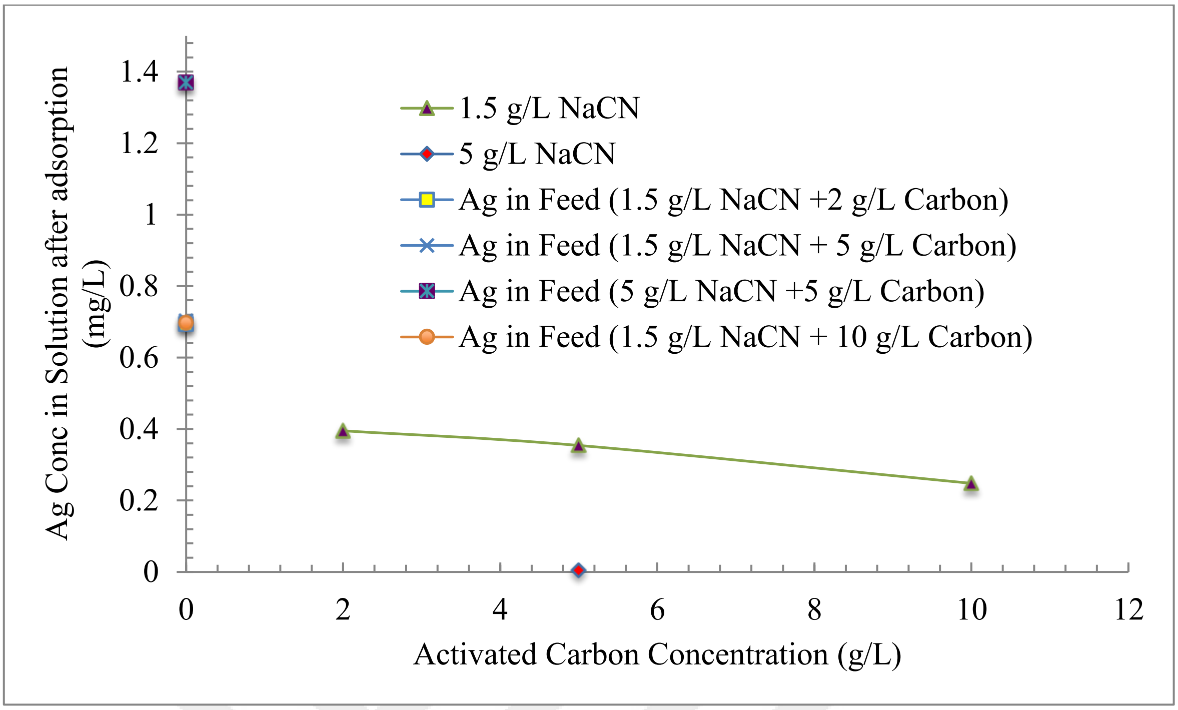


Figure 53. Effect of carbon concentration on the adsorption of silver in CIP

Table 22 . Distribution ratio and coefficient of selectivity for analyzed metals in CIP tests

	D (Au)	D (Cu)	Selectivity(µ)
B	929	75.8	12.3
C	808.2	51.5	15.7
D	171364	13.4	12788
E	7046	24.9	283

Where: B = 1.5 g/L NaCN + 2 g/L carbon   C= 1.5 g/L NaCN + 5 g/L carbon
D = 5 g/L NaCN + 5 g/L carbon   E= 1.5 g/L NaCN + 10 g/L carbon
µ = D(Au)/ D(Cu

4.4. Resin in Leach (RIL) and Resin in Pulp (RIP) Tests

In these set of experiments medium base resin (Purogold S992) and strong base resin (Purogold A194) were used to investigate their efficiency on the whole process of adsorption involving liquors with an appreciable amount of copper. The same dosage of resin was used as the one used in CIL and CIP experiments for comparison. The adsorption results are described as below.

4.4.1. Resin in Leach (RIL) Tests

The RIL tests were undertaken using Purogold S992 and Purogold A194 at pH ranging from 10 to 11. In these tests pH was closely monitored, as Purogold S992 is very sensitive to pH. The above-mentioned resins were introduced at the commencement of the tests to allow leaching and adsorption to take place concurrently. Figure 54 illustrates the pH profile observed during these tests. It can be seen that the highest pH was 11.01 whilst the lowest one was 10.17.

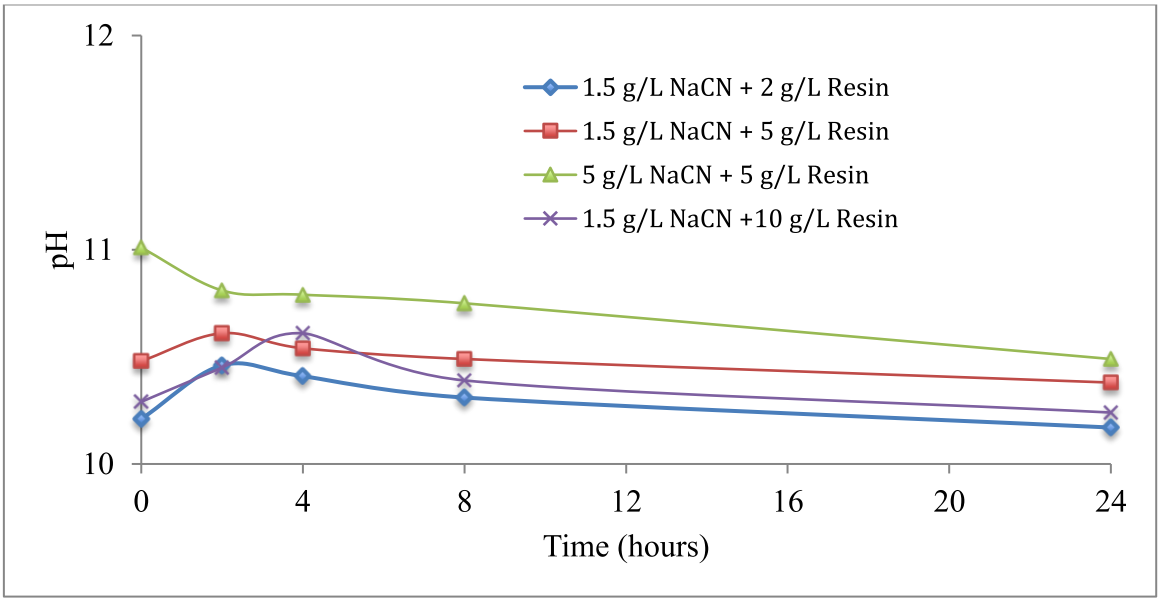


Figure 54. pH profile during the resin in leach (RIL) tests.

Table 23 illustrates the adsorption results obtained by Purogold S992 and Purogold A194 in the resin in leach (RIL) tests. The table shows that the percentage of metals adsorbed onto the above-mentioned resins followed this trend of Au > Ag > Cu. Earlier findings showed the same trend (Marsden and House, 2006; Van Deventer et al., 2012; Van Deventer et al., 2014). In these tests, the highest % of Au adsorbed onto resin was achieved with Purogold A194 from the cyanide leach liquor leached by 5 g/L NaCN and containing 5 g/L of resin. 92.7% of gold was adsorbed onto resins at this cyanide concentration (5 g/L). At this elevated levels of cyanide, the formation of Cu $(CN)_4^{3-}$ and Cu $(CN)_3^{2-}$ are favored and these complexes have an insignificant effect on the process of adsorption.

Table 23 . Percentage of metals loaded onto resins via RIL (1.5 g/L NaCN + 2g/L resin; 1.5 g/L NaCN + 5g/L Resin; 5 g/L NaCN + 5g/L resin; 1.5 g/L NaCN + 10g/L resin)

	Metal (%)	Purogold S992	Purogold A194
1.5 g/L NaCN + 2 g/L Resin	Au	58	62.6
	Cu	3.5	4.64
	Ag	22	29.1
1.5 g/L NaCN + 5 g/L Resin	Au	62.5	75
	Cu	5.32	6.00
	Ag	28.5	33.3
5 g/L NaCN + 5 g/L resin	Au	81.2	92.7
	Cu	2.1	2.9
	Ag	51.1	51.3
1.5 g/L NaCN + 10 g/L resin	Au	70.3	81.25
	Cu	9.2	8,6
	Ag	31.4	37

4.4.2. Resin in Pulp (RIP) Tests

These tests were carried out by the use of the same resins (Purogold A194 and Purogold S992). The resins were added when the leaching process was complete. The results showed that Au concentration in the barren solution decreased with increasing the resin dosage in the range tested (Figures 55-60). For both resins the gold concentration in the solution dropped sharply compared to Ag and Cu (Figures 55-60). However, upon examining the adsorption profiles presented below, the rate of gold adsorption onto resins from solutions containing 5 g/L of resins and leached by 5 g/L NaCN were high. In this test, gold concentration in solution dropped from 25.5 mg/L to 0.051 mg/L for Purogold A194 and from 23.9 mg/L to 0.02 mg/L for Purogold S992. In the same case, copper concentration dropped from 3646 mg/L to 3641 mg/L for Purogold S992 and from 3781 mg/L to 3709 mg/L for Purogold A194. As per activated carbon tests, the extent of adsorption of copper onto resins at 5g/L NaCN is too small compared to that of the adsorption rate of gold. A very slight change of copper concentration in solution was noticed (only 5 mg/L) , when it was treated with Purogold S992 (Figure 58). This may confirm the fact that Purogold S992 is more selective for gold over copper.

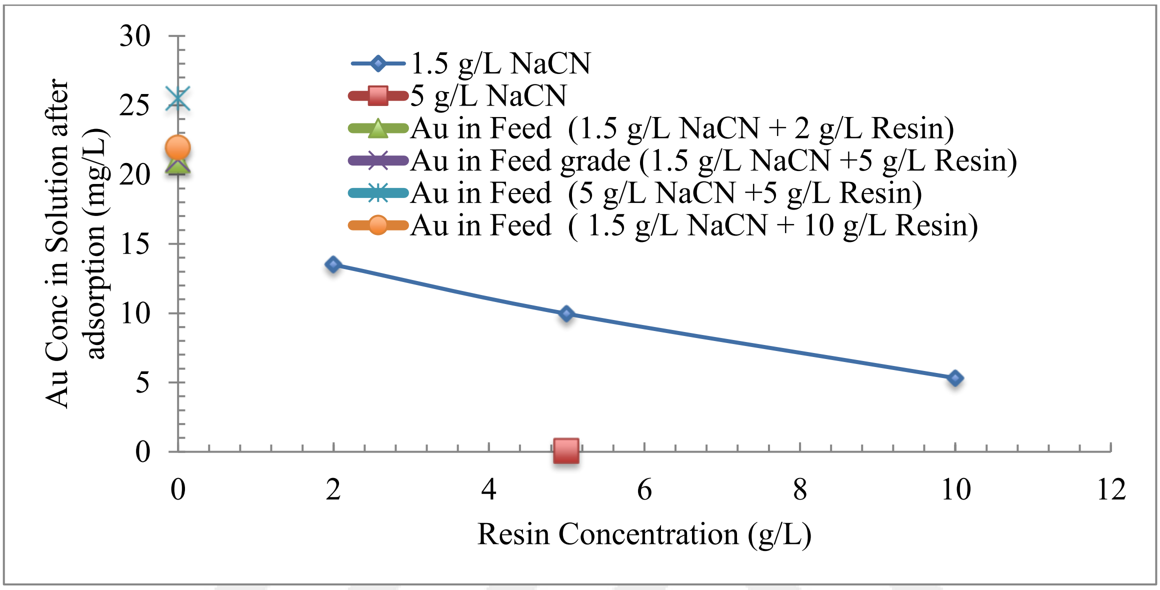


Figure 55 . Effect of resin (Purogold A194) concentration on the adsorption of gold in RIP (Resin dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

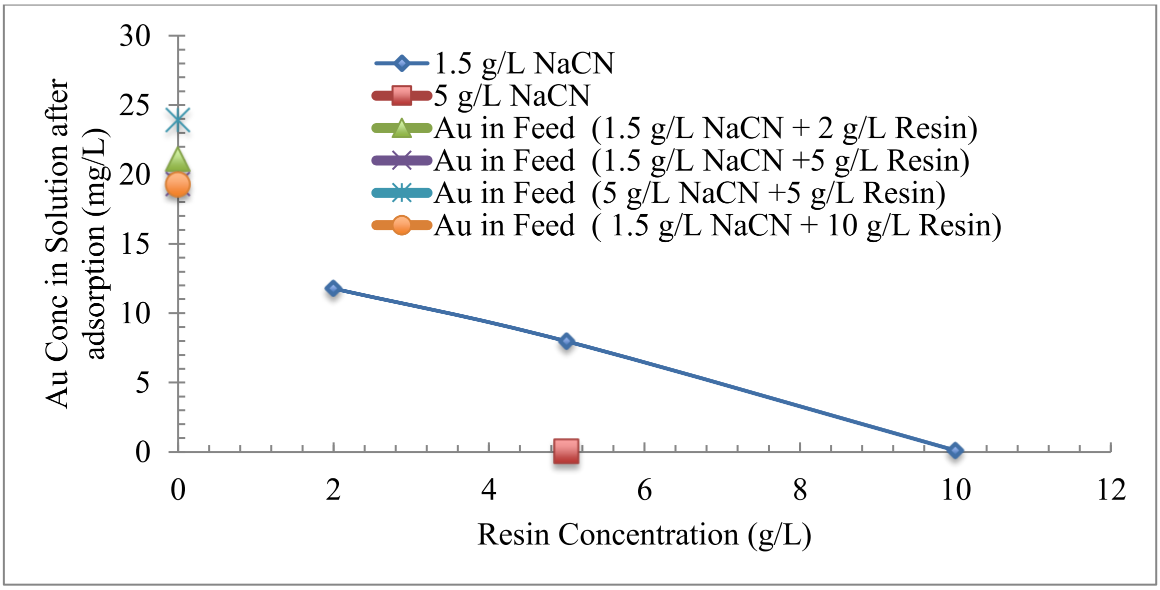


Figure 56 . Effect of resin (Purogold S992) concentration on the adsorption of gold in RIP (Resin dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

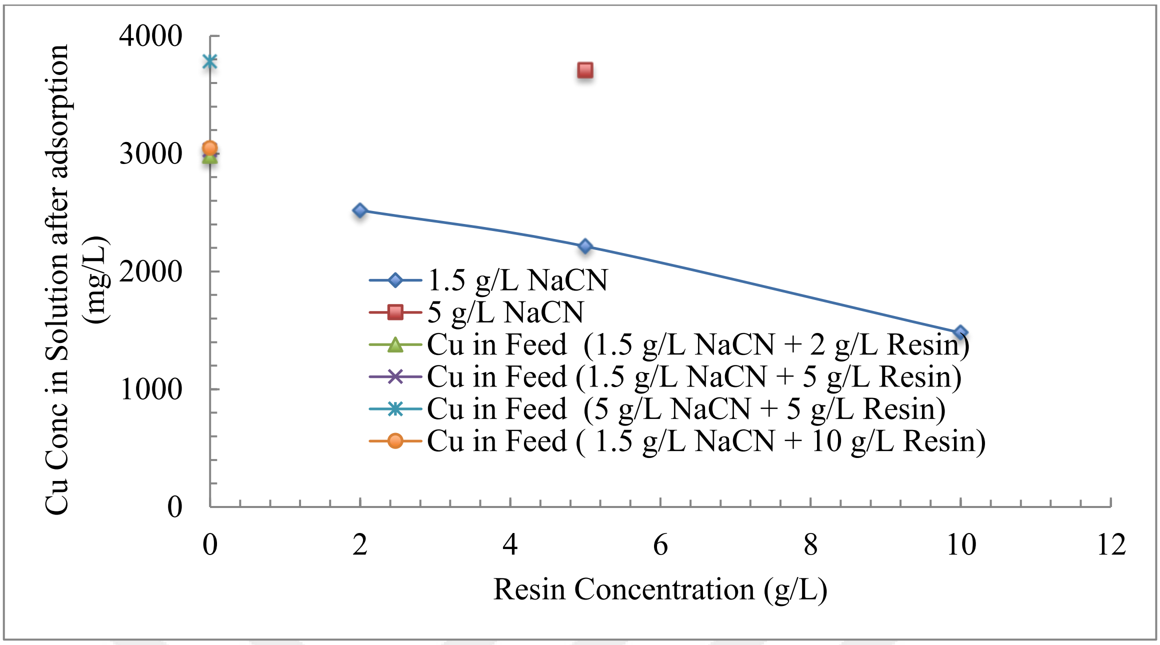


Figure 57 . Effect of resin (Purogold A194) concentration on the adsorption of copper in RIP (Resin dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

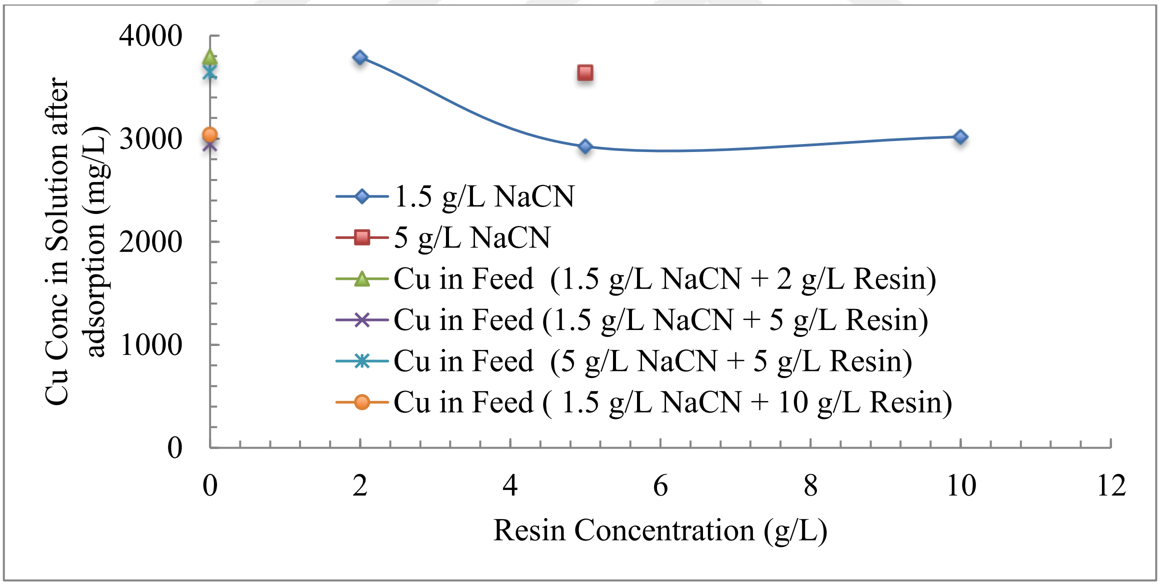


Figure 58 . Effect of resin (Purogold S992) concentration on the adsorption of copper in RIP (Resin dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

The trend of silver adsorption onto resins is depicted in Figure 59 and 60. As the dosage of the resin increased, there was a linear decrease in the concentration of Ag in the barren solution. The adsorption from the solution at 5 g/L (NaCN and resin) was the most extensive for both Purogold S992 and Purogold A194. Only 0.028 mg/L out of 1.517 mg/L remained in the solution at the end of the test when Purogold A194 was used. The barren level of Ag was even lower (0.004 mg/L) for Purogold S992. These results give a point of reflection as silver adsorption onto resins is also favorable.

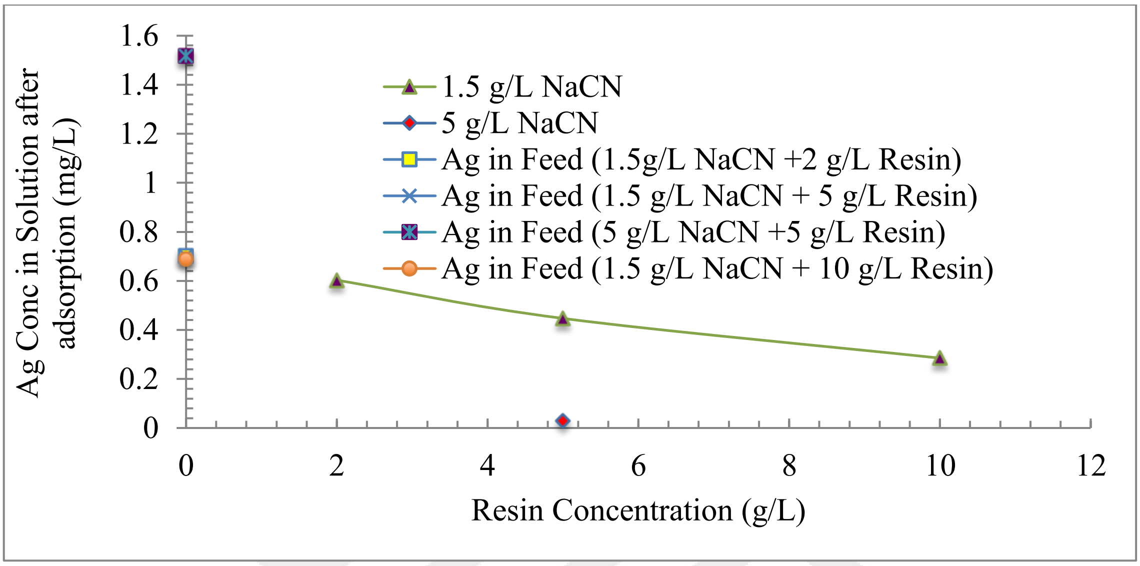


Figure 59 . Effect of resin (Purogold A194) concentration on the adsorption of silver in RIP (Resin dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

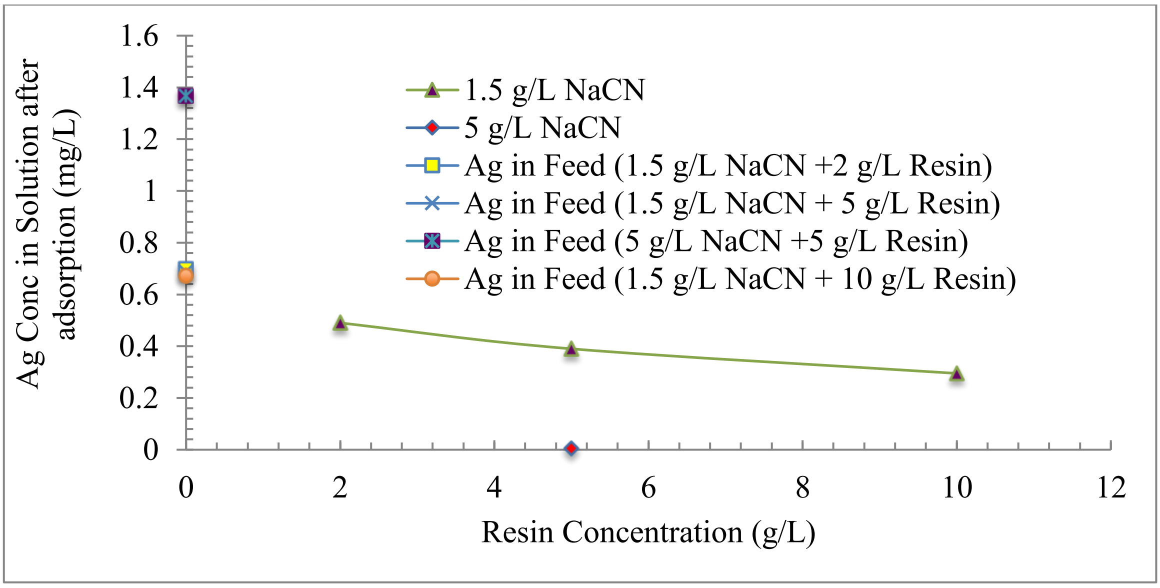


Figure 60 . Effect of resin (Purogold S992) concentration on the adsorption of silver in RIP (Resin dosage: 2-10 g/L, Leaching: 1.5 and 5 g/L NaCN, S/L = 25%)

The distribution ratios for gold and copper and selectivities for Purogold A194 and Purogold S992 are presented in table 24 and 25.

Table 24. Distribution ratios and coefficient of selectivity for analyzed metals in RIP (Purogold A194) tests

	D (Au)	D (Cu)	Selectivity(μ)
B	308	122.3	2.5
C	234.6	85.9	2.7
D	99727	14.5	6878
E	318.2	112.1	2.8

where: B = 1.5 g/L NaCN + 2 g/L resin   C= 1.5 g/L NaCN + 5 g/L resin
D = 5 g/L NaCN + 5 g/L resin   E= 1.5 g/L NaCN + 10 g/L resin
μ = D(Au)/ D(Cu)

Table 25. Distribution ratios and coefficient of selectivity for analyzed metals in RIP (Purogold S992) tests

	D (Au)	D (Cu)	Selectivity(μ)
B	426	31.4	13.6
C	296.9	13.8	21.5
D	238730	10.9	21902
E	18238	6.9	2643

where: B = 1.5 g/L NaCN + 2 g/L resin   C= 1.5 g/L NaCN + 5 g/L resin
D = 5 g/L NaCN + 5 g/L resin   E= 1.5 g/L NaCN + 10 g/L resin
μ = D(Au)/ D(Cu)

5. CONCLUSION

The recovery of Au from a copper-rich gold ore (108 ppm Au, 9.3 ppm Ag, 1.60% Cu, 4.02% Fe, and 926 ppm Zn) was investigated. The ore was found to contain 67.3% SiO_2, 11.51% Al_2O_3, and 6.06% Fe_2O_3. Different tests such as characterization by diagnostic leaching, ultrafine grinding, roasting, acid leaching, cyanide leaching and adsorption tests were performed. Eventually the following conclusions were withdrawn from these studies.

5.1. Direct Cyanide Leaching Tests

The recovery of gold from a refractory copper-rich gold ore using sodium cyanide as a lixiviant was investigated at different cyanide concentrations ranging from 1 g/L to 8 g/L. The dissolution process and the effects of various factors on the cyanide leaching of the ore were explained with particular reference to the literature in an endeavour to delineate the scope of further studies. The results demonstrated that gold can not be readily extracted from the ore. Poor extractions (6.5% to 47.2% Au) were obtained, even at high cyanide concentrations (up to 8 g/L NaCN). These low extractions indicated the refractory nature of the ore. This was apparently linked with physical encapsulation and chemical interference of reactive minerals with leaching.

5.2. Diagnostic Leaching Tests

The copper-rich gold ore (MLG) used in the experiments can be regarded as an ore with complex chemical composition and mineralogy. The presence of copper sulphide minerals induce difficulties in the leaching process and hence the evolution of the novel techniques are inevitable to treat such an ore with high copper content. In this regard, diagnostic leaching of the ore was undertaken to offer an understanding of the refractory comportment of the ore.

Direct cyanidation of the ore with 1.5 g/L NaCN offered an extraction of around 16%. About 84% of unleached gold appeared to possess the character of refractoriness. A series of acid treatment (HCl and HNO_3) improved the extraction of gold to 88.5%. Acid leaching by HCl, which decomposes the carbonates and oxides resulted in 22.6% improvement in gold extraction. Moreover 47.59% of gold recovered at HNO_3+CN stage showed that approximately half of the gold was associated with sulfide minerals. Diagnostic leaching proved to be a significant tool for identifying the refractory phases and potential solutions or strategies for elimination of refractoriness of the ore.

5.3. Pretreatment Tests

The pretreatment of the copper-rich gold ore by ultra-fine grinding, roasting and acid leaching was evaluated to further characterize the refractoriness of the ore. The following conclusions can be drawn from these tests.

Ultra-fine grinding did not enhance the extractions of gold to the desired extent. 54.1% Au was extracted even at the smallest fineness of the ground ore (14.76 µm) and a high cyanide concentration of 8 g/L.

Roasting improved gold recoveries up to 76% at only 1.5 g/L of sodium cyanide. Treatment of the calcine with H_2SO_4 leaching to remove copper interference further enhanced gold recoveries up to 82% at 1.5 g/L NaCN and 96% at 5 g/L NaCN. This proved that treating such an ore requires pretreatment routes such as roasting followed by acid treatment and then cyanidation which can offer stunning recoveries. Alternatively roasting followed by cyanidation at >5 g/L NaCN would offer recoveries ranging from 96% to 100%.

5.4. Carbon in Leach (CIL) and Carbon in Pulp (CIP) Tests

In these experiments activated carbon (Carbon GAC 1240 M-1448) was employed for the adsorption tests. The results induced the following conclusions.

The activated carbon dosage has a direct effect on the adsorption efficiency. As the concentration of carbon increases, the extent of adsorption onto carbon increases. A linear decrease in gold, silver, and copper concentrations in the barren solution occured with increasing the carbon dosages. Au and Ag concentrations on activated carbon increases as the concentration of Au and Ag in solution increases. At low pH level the adsorption of gold and silver is affected positively, but this effect is very limited at high carbon concentrations. Gold is dissolved continously throughout the adsorption process in CIP. High activated carbon concentration and high cyanide concentration increase the dissolution of gold in CIP process.

In both CIL and CIP metal loadings onto activated carbon followed this trend Au > Ag > Cu. Carbon appeared to have high loadings, but its selectivity was interrupted by copper loadings onto it. As far as the chemistry of gold is concerned, $Cu(CN)_2^-$ impede the adsorption of $Au(CN)_2^-$, while $Cu(CN)_3^{2-}$ and $Cu(CN)_4^{3-}$ are weakly adsorbed onto carbon (Marsden and House, 2006). This shows that at high concentration of CN the interference by Cu is reduced, hence the adsorption is enhanced. At 5 g/L NaCN and 5 g/L carbon Au & Ag loadings were high whilst Cu loadings were lower in both tests (CIL & CIP).

5.5. Resin in Leach (RIL) and Resin in Pulp (RIP) Tests

The adsorption rate of metals onto medium base resin and strong base resin was studied by the use of Purogold S992 and Purogold A194 respectively . The primary aim was to investigate the selectivity of the adsorbents for gold over base metals especially copper. From the results obtained the following conclusions can be drawn.

Purogold S992 is a pH sensitive resin. It performs best at pH values between 10 and 10.5, offering low gold loading capacity at pH above 10.5. Purogold A194 is not a pH sensitive resin. Overall, as far as adsorption test results are concerned, the gold loading capacity attained with activated carbon outweighed those obtained with Purogold A194 and Purogold S992. Both adsorbents offered high selectivity for gold over copper. However medium base resin (Purogold S992) loaded less amount of copper and hence its selectivity was higher than the ones for activated carbon and Purogold A194. The trend of selectivity is as follows: Purogold S992> Activated carbon> Purogold A194.

6. PROPOSED FUTURE WORK

Based on the experimental results the following points have been identified for further investigations. High complexity of copper-rich gold ores that will be unearthed in the coming years justify a careful investigation into different choices available at the beginning of the research work. Adsorption tests should include various adsorbents ranging from weak base resin to strong base resin and activated carbons originated from miscellaneous organic precursors. Also new resins with high selectivity should be tested. The cyanide leach liquors must be originated from different pretreatment routes (direct cyanidation, roasting + CN, NH_3 + NaCN, roasting + H_2SO_4 leaching + CN), this will offer a broad range of liquors with different copper concentrations.Various dosage of adsorbents should be applied, this can range from 1 g/L to 25 g/L, also the leach liquors must be treated with different cyanide concentration. Should it be deemed necessary the solid to liquid ratio and oxygen concentration must also be considered. In parallel to this the complexity of elution process of the previously mentioned adsorbents should be evaluated. Taking into considerations the above-suggested points, a friendly process economy may be achieved.

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BIOGRAPHY

Deus Albert Msumange was born on the 8th of June 1992 in Iringa region, situated on SE of Tanzania. Deus started his school journey in 1999 at Kitwiru primary school where he graduated in September 2005. In January 2006, he joined Kantalamba secondary school for his ordinary level studies (middle school). In April 2010 he joined Mzumbe secondary school, one of the best government schools in Tanzania for his high school studies. In September 2012 he was awarded a scholarship offer (Algerian Government Scholarship) via the Ministry of Education and Vocational Training of Tanzania (MOEVT) for his undergraduate studies. In 2016 July, he graduated with a BSc in Geology at Badji Mokhtar Annaba University in Algeria. Later in October 2016, he was awarded a postgraduate scholarship for his master’s studies in Turkey by the Turkish government. He started his MSc in Mining Engineering at the Division of Mineral Processing, Mining Engineering Department of Karadeniz Technical University in September 2017, following a one year of Turkish language preparatory program. The author studied from 2017-2019 as a researcher at Hydromet B&PM research group, which mainly focus on minerals processing and hydrometallurgical extraction of metals from primary and secondary resources. Specifically, Deus worked as a researcher on a project (ID: 7399) funded by KTÜ - BAP, a unit in charge of scientific research projects at Karadeniz Technical University. Deus speaks Swahili, English, Turkish and French fluently.