NICKEL EXTRACTION FROM GÖRDES LATERITES BY HYDROCHLORIC ACID LEACHING

A THESIS SUBMITTED TO
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES
OF
MIDDLE EAST TECHNICAL UNIVERSITY

BY

AHMET GÖVELİ

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR
THE DEGREE OF MASTER OF SCIENCE
IN
MINING ENGINEERING

SEPTEMBER 2006

Approval of the Graduate School of Natural and Applied Sciences

Prof. Dr. Canan Özgen
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science.

Prof. Dr. M. Ümit Atalay
Head of Department

This is to certify that we have read this thesis and that in our opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.

Prof. Dr. M. Ümit Atalay
Supervisor

Examining Committee Members

Prof. Dr. Çetin Hoşten	(METU, MINE)	
Prof. Dr. M. Ümit Atalay	(METU, MINE)	
Prof. Dr. A. İhsan Arol	(METU, MINE)	
Prof. Dr. Cahit Hiçyılmaz	(METU, MINE)	
Prof. Dr. Yavuz Topkaya	(METU, METE)	

I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

Name, Last name: Ahmet GÖVELİ

Signature:

ABSTRACT

NICKEL EXTRACTION FROM GÖRDES LATERITES BY HYDROCHLORIC ACID LEACHING

Göveli, Ahmet
M. Sc., Department of Mining Engineering
Supervisor: Prof. Dr. M. Ümit Atalay

September 2006, 102 Pages

Leaching is the most widely used process for extraction of nickel metal from lateritic ores.

In this study, nickel extraction from Manisa-Gördes region laterites by hydrochloric acid leaching is aimed. The mineralogical analysis of sample showed that hematite, goethite, dolomite, quartz and smectite are the main minerals in the ore. Attrition scrubbing, cycloning and magnetic separation with permroll were used as preconcentration processes but results were unsatisfactory. HCl leaching experiments were conducted both at room temperature and at elevated temperatures. The effects of various parameters such as leaching duration, particle size, concentration of HCl, pulp density, Cl^- concentration and temperature on nickel recovery were examined. The results showed that under the optimised leaching conditions (particle size: 100 % -1 mm, HCl concentration: 3 N, leaching duration: 3 hours, leaching temperature: 100 ^oC, pulp density: 1/30 solid to liquid ratio by volume) it was possible to extract 87.26 % of nickel in the ore.

Keywords: Nickel, Laterite Ore, Hydrochloric Acid, Leaching

ÖZ

GÖRDES LATERİTLERİNDEN HİDROKLORİK ASİT LİÇİ İLE NİKEL KAZANIMI

Göveli, Ahmet
Yüksek Lisans, Maden Mühendisliği Bölümü
Tez Yöneticisi: Prof. Dr. M. Ümit Atalay

Eylül 2006, 102 Sayfa

Liç yöntemi lateritik cevherlerden nikel metalinin elde edilmesinde en yaygın olarak kullanılan yöntemdir.

Bu çalışmada, Manisa-Gördes bölgesinde bulunan lateritik cevherlerden hidroklorik asit liçi ile nikel kazanımı amaçlanmıştır. Yapılan mineralojik analizler sonucunda cevherde bulunan başlıca minerallerin hematit, götit, dolomit, kuvars ve smektit olduğu görülmüştür. Aşındırmalı karıştırma, siklondan geçirme ve permroll ile manyetik ayırma, önkonsantre elde etme yöntemleri olarak kullanılmış ve sonuçlar olumsuz olmuştur. HCl liç deneyleri oda sıcaklığında ve yüksek sıcaklıklarda yapılmıştır. Bu deneylerde liç süresi, tane boyutu, HCl konsantrasyonu, pülp yoğunluğu, Cl^- konsantrasyonu ve sıcaklık gibi parametrelerin nikel kazanımına etkisi incelenmiştir. Sonuçlar, optimum koşullarda (tane boyutu: % 100 -1 mm, HCl konsantrasyonu: 3 N, liç süresi: 3 saat, liç sıcaklığı: 100 ^oC, pülp yoğunluğu: hacimce 1/30 katı/sıvı oranı) HCl liçi sonunda % 87.26 oranında nikel kazanımının mümkün olacağını göstermiştir.

Anahtar Kelimeler: Nikel, Lateritik Cevher, Hidroklorik Asit, Liç

ACKNOWLEDGEMENTS

I would like to express my sincerest gratitude to my supervisor Prof. Dr. M. Ümit Atalay for his kind supervision, guidance, advice, encouragement and patience throughout my thesis work.

I am so greatful to Barış Erdem and Meta Mining Industry Energy Tourism and Trading Ltd. for carrying out the chemical analyses in the experimental part of this study.

And, I want to thank to Dilek Alkaç for her support in the mineralogical analysis of the test sample.

Finally, I feel honored to thank my family for their endless encouragement and moral aid throughout this study.

TABLE OF CONTENTS

CHAPTER

APPENDICES

LIST OF TABLES

TABLES

LIST OF FIGURES

FIGURES

CHAPTER 1

INTRODUCTION

1.1 General Remarks

Today, nickel is produced from two distinct ore types; nickel sulphide deposits and nickel laterite deposits. The great majority of the world's known nickel resources are contained in laterite deposits.

Nickel has been produced from laterite ores for over 100 years beginning with processing of garnieritic ores from New Caledonia. However, until now the world nickel supply has been predominantly from sulfide sources. Going forward, the production of nickel from sulfide ores will remain more or less constant. Most of the expansion in nickel production capacity over the next ten years will come from processing of laterite ores. Thus the capital and operating costs of new laterite projects will have significant impact on the nickel supply and therefore price [1].

Nickel sulphide deposits are primary nickel sources and formed from magmatic processes. Conventional crushing, grinding and flotation are used in order to extract nickel from sulphide ores. The resulting nickel-rich concentrate is pyrometallurgically processed by smelting in furnaces.

Nickel laterite deposits are formed from the weathering of nickel-bearing rocks. Typically, nickel laterites are composed of an upper limonite zone and a lower saprolite zone. These two zones must be treated differently to recover the nickel efficiently, due to the different proportions of iron, magnesium and silica in each zone.

1.2 Objectives of the Thesis

The objective of this research is to find out the optimum conditions to extract nickel metal from Gördes-Manisa region lateritic ore by hydrochloric acid leaching process.

CHAPTER 2

LITERATURE REVIEW

2.1 Nickel

2.1.1 Nickel and Its Properties

Nickel is a transition element that exhibits a mixture of ferrous and nonferrous metal properties [2].

The chemical symbol for nickel is Ni, its atomic number is 28 and atomic mass is 58.71. Nickel is a hard, silvery-white metal with a similar hardness and strength to iron, but more ductile and easier to work and machine. It has the ability to impart its special features and physical properties to other metals. When alloyed with other elements nickel imparts toughness, strength, resistance to corrosion, and various other electrical, magnetic, and heat resistant properties [3].

The physical properties of nickel depend largely on its purity, on the physical state of the metal, and on its previous treatment. The wrought metal is highly malleable, moderately strong, tough, ductile, and highly resistant to corrosion in many media. Its good strength and ductility persist to subzero temperatures. Some important physical properties of nickel are as follows [4]:

- Melting point : 1453 ^{o}C
- Boiling point : 2730 ^{o}C
- Specific gravity (25 ^{o}C) : 8.9

- Volume increase on melting : 4.5 %
- Heat of fusion (mp) : 302 J/g
- Heat of sublimation (25 ^{o}C) : 7317 J/g
- Heat of vaporization (T_{crit}) : 6375 J/g
- Standart entropy : 29.81 J/^{o}K

In its chemical properties nickel resembles iron and cobalt, as well as copper. The +2 oxidation state is much the most important in nickel chemistry, although the +3 and +4 oxidation states are also known. The oxidation of nickel(II) salt solutions with chlorine, bromine, or persulfate in aqueous alkaline solution produces the insoluble nickel(III) oxide, b-NiO(OH). The nickel(IV) oxide $NiO_2 \cdot n\ H_2O$ is prepared by persulfate oxidation of nickel(II) hydroxide. In contrast to cobalt and iron, nickel is normally only stable in aqueous solution in the +2 oxidation state.

Nickel(II) ions form complexes very readily. The green color of most hydrated nickel(II) salts and their aqueous solutions is due to the octahedral $Ni(H_2O)_6^{+2}$ cation. The strong tendency of the nickel(II) ion to form complexes with ammonia, for example $Ni(NH_3)_6^{+2}$, or $Ni(H_2O)_2(NH_3)_4^{+2}$, is utilized in a number of extraction processes.

The most unusual property of nickel is its ability to react directly with carbon monoxide to form a binary carbonyl complex. When carbon monoxide reacts with metallic nickel at 60 °C it forms the volatile nickel tetracarbonyl, $Ni(CO)_4$. This reaction is reversible, with the carbonyl compound decomposing to carbon monoxide and nickel at higher temperatures (180 °C).

$$\mathrm{Ni} + 4\mathrm{CO} \underset{180\ °\mathrm{C}}{\overset{60\ °\mathrm{C}}{\rightleftarrows}} \mathrm{Ni(CO)_4} \tag{2.1}$$

This reaction is the basis for the vapometallurgical refining of nickel. No other metal forms similar carbonyl compounds under such mild conditions at atmospheric pressure.

At moderate temperatures nickel has a high corrosion resistance against air, seawater, and nonoxidizing acids. Nickel is fairly electropositive with a standard electrode potential of – 0.25 V, which is similar to cobalt (– 0.28 V).

$$\mathrm{Ni} \longrightarrow \mathrm{Ni}^{+2} + 2\ e^{-} \quad (E = -\ 0.25\ \mathrm{V}) \tag{2.2}$$

An outstanding property of nickel is its corrosion resistance to alkalis. The metal is therefore frequently used in the production and handling of caustic soda. In contrast, nickel is attacked by aqueous ammonia solutions.

Nickel absorbs hydrogen, especially when finely divided; hydrogen absorption increases with increasing temperature. Defined hydride compounds have not been characterized. Nitrogen is not absorbed by nickel nor does it combine directly with it, although there is evidence for a nitride Ni_3N [5].

2.1.2 Nickel Minerals and Origin of Nickel

Nickel is one of the more common elements in the composition of the earth, but it is sparingly distributed in the earth's crust. Most of the world's known nickel resources occur in minerals such as garnierite and nickeliferous limonite [3].

The most important nickel-containing minerals occurring in nickel deposits are listed in Table 1, together with their chemical compositions. Some are relatively uncommon, and only pentlandite, garnierite, and nickeliferous limonite are of economic significance [6].

Table 1. *Principal Nickel Minerals*

Mineral	Ideal Formula	Nickel Content (%)
Sulfides		
Pentlandite	$(Ni,Fe)_9S_8$	34.22
Millerite	NiS	64.67
Heazlewoodite	Ni_3S_2	73.30
Polydymite	Ni_3S_4	57.86
Siegenite	$(Co,Ni)_3S_4$	28.89
Violarite	Ni_2FeS_4	38.94
Arsenides		
Niccolite	$NiAs$	43.92
Rammelsbergite	$NiAs_2$	28.15
Gersdorffite	$NiAsS$	35.42
Antimonides		
Breithauptite	$NiSb$	32.53
Silicates and Oxides		
Garnierite	$(Ni,Mg)_6Si_4O_{10}(OH)_8$	≤ 47
Nickeliferous limonite	$(Fe,Ni)O(OH).nH_2O$	Low

The bulk of the nickel mined comes from two types of ore deposits:

- Laterites where the principal ore minerals are nickeliferous limonite and garnierite
- Magmatic sulfide deposits where the principal ore mineral is pentlandite

The ionic radius of divalent nickel is close to that of divalent iron and magnesium, allowing the three elements to substitute for one another in the crystal lattices of some silicates and oxides. Nickel sulfide deposits are generally associated with iron and magnesium-rich rocks called ultramafics and can be found in both volcanic and plutonic settings. Many of the sulfide deposits occur at great depth. Laterites are formed by the weathering of ultramafic rocks and are a near-surface phenomenon [2].

2.1.3 Nickel Reserves

2.1.3.1 World Nickel Reserves

Today, nickel is produced from either sulfides or laterites. As given in the Figure 1, approximately 60 % of the world's known nickel resources are in the form of nickel laterites; but notwithstanding the predominance of laterite ore, by far the largest source of nickel production has been from sulfides for economic reasons [1].

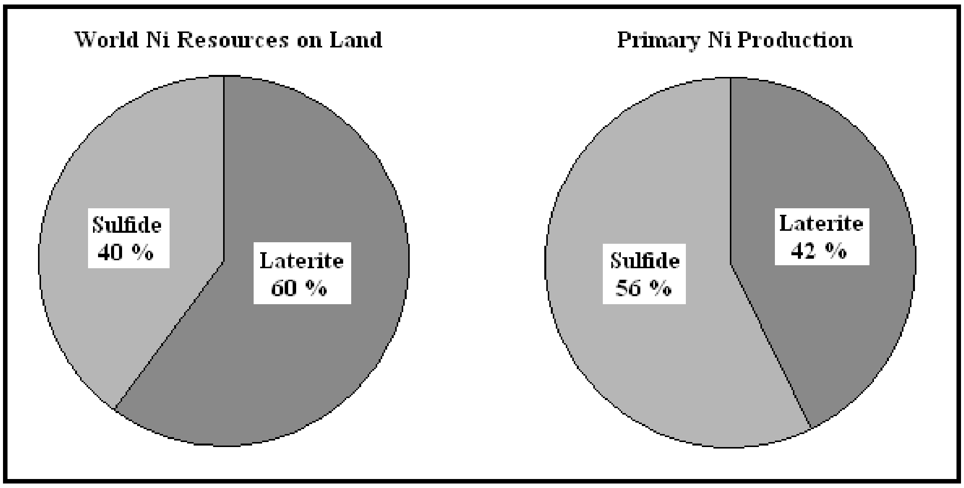


Figure 1. *World Nickel Resources and Production Ratio*

Identified land-based resources averaging 1 % nickel or greater contain at least 140 million tons of nickel metal. The world nickel resources (as metal), calculated from resources grading 1.0 % Ni or higher, according to the countries are given in Table 2 [7].

Table 2. *World Nickel Resources According to Countries*

COUNTRIES	Reserve 1* (tons)	Reserve 2** (tons)
Australia	22,000,000	27,000,000
Botswana	490,000	920,000
Brazil	4,500,000	8,300,000
Canada	4,800,000	15,000,000
China	1,100,000	7,600,000
Colombia	830,000	1,100,000
Cuba	5,600,000	23,000,000
Dominic Republic	720,000	1,000,000
Greece	490,000	900,000
Indonesia	3,200,000	13,000,000
New Caledonia	4,400,000	12,000,000
Philippines	940,000	5,200,000
Russia	6,600,000	9,200,000
South Africa	3,700,000	12,000,000
Venezuela	560,000	630,000
Zimbabwe	15,000	260,000
Other Countries	2,100,000	5,900,000
World Total (rounded)	**62,000,000**	**140,000,000**

* Proven + Probable Reserve   ** Proven + Probable + Possible Reserve

The distribution of world's nickel laterite resources are given in Figure 2 [1]. 70 % of laterite ore resources are limonitic type and 30 % of them are silicates [8].

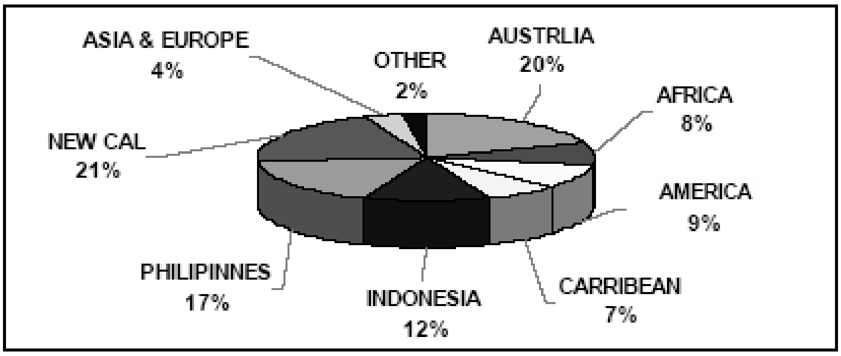


Figure 2. *Distribution of World Nickel Laterite Resources*

2.1.3.2 Turkey Nickel Reserves

Both laterite and sulphide type nickel mineralization are found in Turkey. The main laterite resources in Turkey are in Manisa-Turgutlu-Çaldağ, Manisa-Gördes, Uşak-Banaz, Eskişehir-Mihaliççık-Yunusemre and sulphide resources are in Bitlis-Pancarlı, Bursa-Orhaneli-Yapköydere and Sivas-Divriği-Gümüş [9]. Turkey nickel reserves are given in Table 3.

Table 3. *Turkey Nickel Reserves*

Place	Type	Grade (%)	Proven+Probable Reserve (tons)	Possible Rsv. (tons)	Total (tons)
Manisa Çaldağ*	Laterite	1.14	37,900,000	-	37,900,000
Manisa Gördes**	Laterite	> 1	68,500,000	-	68,500,000
Bursa Yapköy**	Sulphide	1-4	82,000	81,000	163,000
Bitlis Pancarlı**	Sulphide	1.41	-	15,500	15,500
Total			**106,482,000**	**96,500**	**106,578,500**

* Company information
** General Directorate of Mineral Research and Exploration information

General Directorate of Mineral Research Exploration has not carried out a reserve study in Uşak-Banaz, Eskişehir-Mihaliççık-Yunusemre and Sivas-Divriği-Gümüş nickel deposits.

2.1.4 Uses of Nickel

Today, at least 3000 nickel alloys have been identified and more than 80 % of the world's nickel production is used in alloys.

About 69 % of world nickel output is used in the manufacture of stainless steels. When nickel is added to stainless steels their corrosion resistance and strength is considerably increased. Alloy steels containing nickel are therefore widely used in the chemical industry, pipelines to carry seawater and in the highly stressed components for motorcars such as crank-shafts and axles. Nickel is also widely used in the manufacture of coins.

Nickel alloys are able to withstand temperature extremes and are resistant to fracturing under stress. Nickel is used in the storage and transportation of corrosive chemicals and liquefied gases, which require especially low temperature conditions. Many alloys, usually containing more than 50 % nickel, have been developed for high temperature strength in aircraft gas turbines and jet engines.

Nickel also rates as a strategic metal, and is important in steels used for armour plating, gun forgings, shells and bullets. Nickel is also used for catalysing the addition of hydrogen to natural oils i.e. converting the oils into solids which can be used in soap and margarine [3].

The approximate nickel consumption ratios in different fields are given in Table 4.

Table 4. *Nickel Consumption Ratios*

Field	Consumption %
Manufacture of stainless steel	69
Nickel alloys	9
Coating & Plating	8
Iron metallurgical products	7
Battery production	3
Manufacture of Coins	2
Other	2

2.2 Nickel Ores

2.2.1 Sulfide Nickel Ores

2.2.1.1 Definition and Occurence of Sulfide Nickel Ores

Nickel sulphide deposits are formed from magmatic processes and are primary nickel deposits [10]. They are generally associated with iron-and magnesium-rich rocks called ultramafics and can be found in both volcanic and plutonic settings. Many of the sulfide deposits occur at great depth [2]. Nickel is a constituent of the molten rock-magma-and that the nickel bearing sulfides have been derived from it either during or subsequent to the cooling period [11].

Sulfide nickel ores consist principally of nickeliferous pyrrhotite (Fe_7S_8), pentlandite $(Ni,Fe)_9S_8$, and chalcopyrite ($CuFeS_2$). Other minerals which occur in small but significant amounts include magnetite (Fe_3O_4), ilmenite ($FeTiO_3$), pyrite (FeS_2), cubanite ($CuFe_2S_3$), millerite (NiS), heazlewoodite (Ni_3S_2), polydmite (Ni_3S_4), siegenite ($(Co,Ni)_3S_4$) and violarite (Ni_2FeS_4). Sulfide ores typically grade 0.4–2.0 % nickel, 0.2–2.0 % copper, 10–30 % iron, and 5–20 % sulfur. The balance consists of silica, magnesia, alumina, and calcium oxide [12].

Pentlandite is the most common of the sulfide ore minerals of nickel, and it accounts for about three-quarters of the nickel mined in the world. It is almost invariably associated with larger amounts of pyrrhotite, an iron sulfide. Millerite is found in some nickel ores, usually as only a minor constituent. Since it has a nickel content nearly twice that of pentlandite, an amount of it can significiantly sweeten the ore. Heazlewoodite has the highest nickel content of any naturally occuring nickel sulfide or arsenide. Polydmite, siegenite and violarite are members of a continuous linnaeite series and grade into one another by substitution of iron or cobalt for some of the nickel. In most nickel bearing deposits they are very minor constituents if present at all.

Virtually all masses of ultramafic rock are vast storehouses of nickel. The nickel is a tightly held part of the regular framework of atoms that constitute the mineral olivine [11].

Major sulfide ore bodies occur in Canada, the old former Soviet Union, the Republic of South Africa, Australia, Zimbabwe, and Finland [12].

2.2.1.2 Beneficiation of Sulfide Nickel Ores

The low metal content of present-day nickel sulfide ores renders them unsuitable for either direct smelting or direct hydrometallurgical processing. Because the sulfide minerals usually occur as distinct grains in the rock matrix, these ores are amenable to a mechanical upgrading in which much of the rock content can be rejected. The metal content of the ore is concentrated by a physical treatment such as comminution to liberate the metal sulfide grains, followed by froth flotation or magnetic separation to recover a metal-rich concentrate [4].

The resulting nickel-rich concentrate is pyrometallurgically processed by smelting in furnaces to skim off and discard an iron-rich slag to produce a nickel matte. This matte is comprised predominately of nickel and iron with copper and cobalt. The matte is then subjected to further processing to eventually produce nickel metal and other metal byproducts [10].

Most nickel producers, make no attempt to obtain separate nickel, copper, and iron concentrates and produce a bulk concentrate that contains pentlandite, pyrrhotite, and chalcopyrite as smelter feed. In Canada, the major producers separate a large part of the pyrrhotite from pentlandite and chalcopyrite, and Inco further separates the pentlandite and chalcopyrite into separate nickel and copper concentrates.

In response to ever tighter constraints on sulfur dioxide emissions from smelters during the 1970s and 1980s, the Canadian companies have made increased pyrrhotite rejection from smelter feed, and substantial improvements have been achieved. A pyrrhotite concentrate was treated by Inco for many years for nickel and iron oxide recovery. However, environmental constraints on sulfur dioxide emissions have rendered the process obsolete, and pyrrhotite concentrates were largely stockpiled, since none of the known treatment methods that avoid sulfur dioxide production is economic.

Pyrrhotite can be separated from pentlandite and chalcopyrite either by using its ferromagnetic properties or by flotation [13].

One of the more complex nickel concentration process flow sheets is that operated by Inco at its Sudbury operation. In the two primary concentrators, the Clarabelle mill and the Frood–Stobie mill, 50,000 t/d of ore, grading 1.2 % Ni and 1.2 % Cu, is ground to a particle size of 200 μm and treated by magnetic separation and flotation with a sodium amyl xanthate reagent to produce 5,000 t/d of a bulk nickel–copper concentrate and 8,000 t/d of a pyrrhotite concentrate. The two primary concentrates are further upgraded in the Copper Cliff mill. The bulk nickel–copper concentrate is separated by flotation into separate nickel and copper concentrates by using lime and sodium cyanide at 30–35 °C to depress pentlandite and pyrrhotite. Under these conditions about 92 % of the copper is recovered to a concentrate grading 29 % Cu and 0.9 % Ni. About 98 % of the nickel and 92 % of the pyrrhotite are rejected to the separator tails, which grade 13 % Ni and 2.0 % Cu [14].

Falconbridge at its Strathcona mill in the Sudbury basin treats ore containing 1.5 % Ni and 1.1 % Cu by grinding and flotation to produce a nickel–copper concentrate grading 6.7 % Ni and 5.6 % Cu at an average recovery of 83 % Ni and 93 % Cu. A large part of the pyrrhotite content of the ore is rejected directly to the tailings pond. The separation of the nickel and copper in the bulk concentrate is carried out in the matte refining process after smelting [15].

At the BCL nickel–copper mine in Botswana the pyrrhotite/pentlandite ratio in the ore averages about 13 : 1 and the pyrrhotite contains over 30 % of the nickel either in solid solution or as small pentlandite inclusions. Consequently, high nickel recoveries can only be achieved by recovering the nickel from pyrrhotite. This is achieved by producing a bulk pentlandite–pyrrhotite–chalcopyrite concentrate, with a typical composition of 2.8 % Ni and 3.2 % Cu [16].

2.2.2 Lateritic Nickel Ores

2.2.2.1 Definition and Occurence of Nickel Laterites

Nickel laterites are derived from ultramafic rocks and contain economically exploitable reserves of nickel and commonly cobalt. They are found where the ultramafic rocks outcrop over a wide area. The deposits are developed on olivine-bearing ultramafic rocks, mainly dunite and olivine-pyroxene peridotite, and their serpentinized equivalents [17].

The oxidic ores of nickel are formed by a chemical concentration process that occurs as a result of the lateritic weathering of peridotite rock. Peridotite consists mainly of olivine, a magnesium iron silicate containing up to 0.3 % nickel. In many rocks the peridotite has been altered to serpentine, a hydrated magnesium silicate, prior to exposure to weathering. Olivine and serpentine are decomposed by groundwater containing carbon dioxide to form soluble magnesium, iron, and nickel, and colloidal silica. The iron rapidly oxidizes in contact with air and precipitates by hydrolysis to form goethite and hematite, which remain near the surface of the deposit. The dissolved nickel and magnesium, and the colloidal silica, percolate downwards in the laterite deposit, remaining in solution so long as the solution is acidic. When the solution is neutralized by reaction with rock and soil, nickel, silica, and some of the magnesium precipitate as hydrated silicates. An idealized section through laterized deposit is illustrated in Figure 3 [12].

Depth in meters	**Idealized Laterite**	**Approximate Analysis %**			
0	***Overburden***	**Ni**	**Co**	**Fe**	**MgO**
1		< 0.8	< 0.1		
2	***Limonite***	0.8 to 1.5	0.1 to 1.2	40 to 50	0.5 to 5.0
3					
4					
5					
6	***Transition Zone***	1.5 to 1.8	0.02 to 0.1	25 to 40	5 to 15
7					
8	***Saprolite***	1.8 to 3	< 0.02	10 to 25	15 to 35
9					
10					
11	***Unaltered Bedrock***	0.25	< 0.02	5	> 35

Figure 3. *An Idealized Section through Laterized Deposit*

The end result of geological history can be visualized with the help of Figure 3 above, showing a typical laterite deposit. The diagram illustrates the variation of the major chemical components with depth. In this resource two zones are important: limonite and saprolite. The limonite has a distinct, rusty reddish color fading to brown with depth. The cobalt stays with the iron but tends to concentrate with depth. The low iron material is the saprolite, and has the highest nickel grades while being deficient in cobalt. The saprolite is less weathered and tends to be harder than the limonite [10].

Nickeliferrous limonite is a term used in referring to poorly crystalline to non-crystalline nickel-bearing ferric oxides in laterite deposits developed from ultrabasic rocks. Goethite, approximately FeO(OH), is a principle constituent of nickel bearing limonite. The water content of limonite varies widely [11].

2.3 Nickel Recovery Methods from Lateritic Ores

A variety of flowsheets are used to process laterite ores. They generally fall into two categories:

1. *Pyrometallurgical Processes*
2. *Hydrometallurgical Processes*

The distribution of the global resource for nickel laterites is given in Table 5 below from the perspective of the processes employed to extract nickel (pyrometallurgical or hydrometallurgical):

Table 5. *The Distribution of the Global Resource for Nickel Laterites from the Perspective of the Processes Employed*

Process Employed to Extract Ni	Distribution (%)
Pyrometallurgy	39
Hydrometallurgy	61
Total Laterites	**100**

Thus, there is almost twice as much laterite resource that is amenable to hydrometallurgical processing (limonite, nontronite/smectite) as that amenable to pyrometallurgical processing (saprolite, garnierite) [1].

2.3.1 Pyrometallurgical Processes

The energy needed to reduce nickel ores is most commonly provided by heating, in the process called as smelting. The use of intense heat for metal extraction is known as pyrometallurgy. It is a high temperature, high energy operation with good nickel recovery but low cobalt recovery. Smelting is not amenable to processing limonite because the iron content is too high and the nickel grade too low to be economic. Saprolite is more suitable, with lower iron and higher nickel concentrations. In this case, the end product is an iron-nickel alloy known as ferronickel, or if sulfur is added, a high grade nickel matte. Most of the world production achieved from lateritic ores uses high grade saprolite as feed stock, and provides ferronickel directly to manufacturers of stainless steel, by far the greatest end use for the nickel. Laterites suffer a further disadvantage in heat driven processes because of their high natural moisture content. The moisture must be boiled off before the heat treatment becomes effective, and this consumes considerable additional energy.

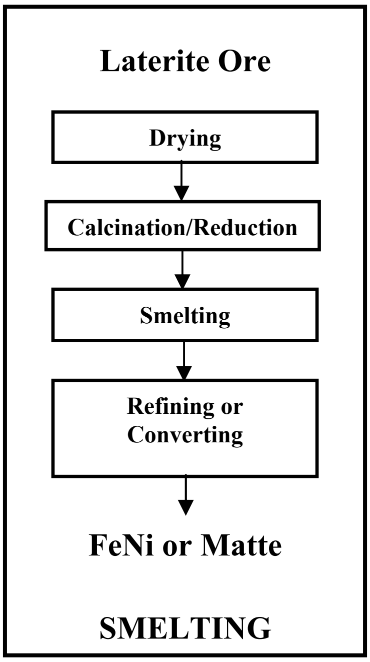


Figure 4. *Generalized Flow Diagram for Smelting Process*

In conventional pyrometallurgical processing, as shown in Figure 4, the ore is dried, calcined (and sometimes reduced) in a rotary kiln and smelted in an electric furnace in the presence of carbon. If matte is the desired product, then sulfur is added to the kiln. The crude metal/matte is further refined to produce the final product [1].

The separation of nickel from the refractory oxides is relatively simple because there are large differences in the free energies of formation of nickel oxide and the gangue components such as silica and magnesia. Adjustment of the reduction conditions permits the complete reduction of nickel oxide while limiting the degree of reduction of iron oxide, but a total separation of nickel from iron by selective reduction is not possible. Two major process routes have been developed to overcome this problem.

In ferronickel production, the nickel oxide and part of the iron oxide are reduced to metal in an electric furnace. The fraction of iron reduced to metal is a function of the reduction potential of the system, so that ores with a low Ni/Fe ratio can be smelted to yield an acceptable ferronickel grade [4].

In nickel matte production, the ore is smelted with a reductant, a flux, and a source of sulfur, which may be gypsum, a sulfide mineral, or elemental sulfur. Most of the nickel and a large portion of the iron are reduced and sulfidized to form a low-sulfur matte phase. Iron removal can then be accomplished by fluxing and elective oxidation of the iron in a converter to produce a nickel matte containing over 75 % Ni, 20–22 % S, and less than 1 % Fe [4]. The matte consisting essentially of nickel, iron and sulfur is charged molten into converters and blown with air. The iron is oxidized preferentially to the nickel, combining with added silica to form a slag. If the amount of sulfur added in the blast furnace is just enough to combine with the nickel as Ni_3S_2, only a small amount of sulfur is eliminated during the blowing operation. Converting is continued until virtually all the iron is removed from the matte. The slag, too high in nickel to discard, is recycled [11].

Several of the world's major nickel laterite smelters are listed in Table 6 together with details of their ore and product nickel grades [4].

Table 6. *Nickel Laterite Smelters with Their Ore and Product Nickel Grades*

Smelter	Ore grade, Ni %	Product	
		Type	Grade, Ni %
SLN, New Caledonia	2.70	Fe-Ni	25
		matte	78
P.T. Inco Indonesia	1.97	matte	79
Pacific Metals, Japan	2.40	Fe-Ni	14-22
Falconbridge, Dominician Republic	1.75	Fe-Ni	38
Cerro Matoso, Colombia	2.90	Fe-Ni	45
Larco, Greece	1.25	Fe-Ni	24-30
Hyuga Smelter, Japan	2.40	Fe-Ni	20-25
Orsk, Soviet Union	1.02	matte	77
Nippon Mining, Japan	2.40	Fe-Ni	20
Glogovac, Yugoslavia	1.32	Fe-Ni	24
P.T. Aneka Tambang, Indonesia	2.25	Fe-Ni	25

In Turkey, there is not any industrial application of a pyrometallurgical nickel extraction process.

Pyrometallurgical processes are energy intensive since all of the free moisture and combined water has to be removed in the process and all of the material has to be first calcined and then melted to form a slag at about 1,600 °C. This requires both hydrocarbon fuels (coal, oil) and electric power.

Recovery of nickel is usually in the range 90-95 % and that of cobalt is around 50 % in this process [1].

2.3.2 Hydrometallurgical Processes

The chemical and mineralogical homogeneity of limonitic nickel ores, allied with the high value of potential byproducts such as cobalt, chromium, and iron, make them ideal feed materials for hydrometallurgical treatment. Most of the nickel in iron-rich laterite ores is contained in goethite (a-FeO·OH), while the cobalt is almost always associated with manganese oxides. The recovery of nickel and cobalt thus essentially consists of separating them from iron and manganese [4].

Caron Process, High Pressure Acid Leaching Process, Enhanced Pressure Acid Leaching Process, Acid Heap Leaching Process and Atmospheric Chloride Leaching Process are the main haydrometallurgical processes and general information about these processes are given in following sections.

2.3.2.1 Caron Process

The Caron process is used for limonitic ores or a mixture of limonite and saprolite. The ore is dried and nickel is selectively reduced (together with cobalt and some iron) to metallic nickel at ~700 °C. The metallics are extracted by leaching in an ammoniacal solution as shown in the Figure 5. Recovery of nickel and cobalt decreases with increasing amount of saprolite since nickel and cobalt are locked in a silicate matrix and are difficult to reduce at this temperature. However, the process can tolerate higher amount of Mg than the PAL (Pressure Acid Leaching) processes [1].

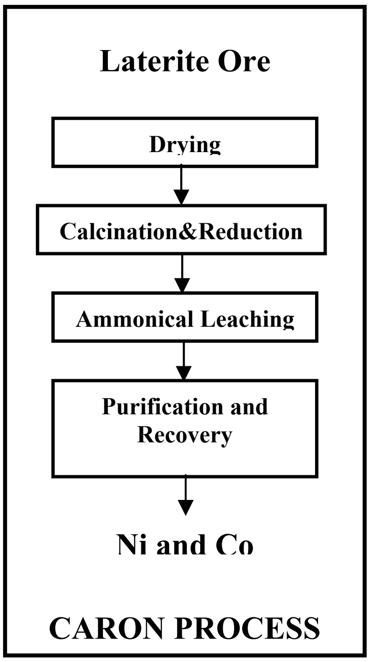


Figure 5. *Generalized Flow Diagram for Caron Process*

The Caron process suffers from several disadvantages: The front-end of the Caron process is pyrometallurgical involving drying, calcining and reduction. These steps are energy intensive.

The back-end is hydrometallurgical requiring various reagents. The nickel and cobalt recoveries are lower than for the smelting processes or the high pressure acid leaching process [1]. Details of a number of operations using the Caron process are given in Table 7.

***Table** 7. Commercial Caron Process Plants*

Operation	**Design Capacity, t/a Ni**	**Feed ore, %**		**Product Type**
		Ni	**Co**	
Nicara, Cuba	22,500	1.40	0.10	NiO sinter
Queensland Nickel, Australia	18,000	1.35	0.11	NiO rondelles, NiS - CoS
Nonoc, Philippines	30,000	1.22	0.10	Ni powder, NiS - CoS
Tocantins, Brazil	5,000	1.60	0.14	Ni cathode

The first commercial application of the process was constructed at Nicaro, Cuba. The ore is first dried to about 5 % moisture and is then selectively reduced in a roaster. The reduced calcine is cooled under nonoxidizing conditions and leached in ammoniacal ammonium carbonate solution to selectively extract nickel and cobalt. The pregnant leach liquor is then boiled to strip ammonia and carbon dioxide, causing the nickel and cobalt to precipitate as basic carbonates. The mixed nickel–cobalt basic carbonate can be calcined to a nickel oxide product or can be refined to nickel powder or cathode. However nickel recovery from the ore had never exceeded 70 %.

The other large Caron plant was built by Marinduque Mining and Industrial Corporation on Nonoc Island in the Philippines. The Nicaro process was adapted to increase nickel and cobalt extractions and to produce hydrogen-reduced powder from the basic nickel carbonate. The Nonoc nickel refinery was commissioned in 1974 and reached a production rate of around 22,000 t/a Ni in 1977, at a nickel recovery of 78 % Ni. Both the Queensland Nickel and the Marinduque nickel operations were designed to recover cobalt separately as a byproduct by precipitating a mixed cobalt–nickel sulfide prior to precipitation of basic nickel carbonate [4].

Another industrial practice is illustrated by the following description of the Queensland Nickel process, which closely resembles the original Nicaro process except for the addition of a cobalt recovery step.

The blended ore (1.35 % Ni, 0.11 % Co, 25 % Fe, 10 % MgO) is first dried in one of three rotary dryers to about 5 % moisture content. The dried ore is then ground in a ball mill and blended with fuel oil as a reductant, before being fed to one of ten roasters, where it is roasted under reducing conditions at 700 – 760 °C with a retention time of 90 min. The ore is heated by hot reducing gas ($CO + H_2$) formed by the combustion of fuel oil in a deficiency of air in the ten combustion chambers on each roaster. The reduction potential of the gas phase is regulated so that most of the nickel and cobalt in the ore are reduced to the metallic state (as a ferronickel alloy) and the iron(III) oxides are reduced to magnetite without significant reduction of the iron oxides to iron(II) oxide or to the metal, as shown in below reactions:

$$NiO + H_2 \rightarrow Ni + H_2O \qquad (2.3)$$

$$3Fe_2O_3 + H_2 \rightarrow 2Fe_3O_4 + H_2O \qquad (2.4)$$

As a result the nickel and cobalt are rendered amenable to ammoniacal leaching while the iron remains as insoluble oxides or silicates. Typically 80 – 85 % of the nickel content of the ore is converted to the metallic form. Any nickel oxide, which is not reduced to metal, is rendered insoluble under the roasting conditions by incorporation into the silicate minerals. Good control of the roasting conditions is therefore essential for optimal nickel recovery in the process.

The reduced ore is cooled in a nonoxidizing atmosphere, to avoid reoxidation of the nickel, and is quenched and leached in ammoniacal ammonium carbonate solution containing 7 % NH_3 and 4 % CO_2. Leaching is carried out in three trains of four mechanically agitated, air-sparged leaching tanks at a pulp density of 20 % solids. The leaching reactions can be represented by the following equation:

$$FeNi + O_2 + 8NH_3 + 3CO_2 + H_2O \rightarrow Ni(NH_3)_6^{2+} + Fe^{2+} + 2NH_4^{+} + 3CO_3^{2-} \qquad (2.5)$$

The iron(II) is further oxidized and precipitates as iron(III) hydroxide:

$$4Fe^{2+} + O_2 + 2H_2O + 8OH^- \rightarrow 4Fe(OH)_3 \qquad (2.6)$$

However, significant losses of cobalt occur by coprecipitation with the iron hydroxide.

The pregnant liquor is separated from the leached ore in a seven-stage countercurrent decantation thickener circuit using ammoniacal solution as the washing medium. The washing efficiency is typically 99 %. Ammonia is recovered from the washed leach residues by steam stripping, and the barren solids are discarded. The pregnant liquor recovered from the wash circuit, which contains 10 g/L Ni and 0.5 g/L Co, was formerly treated with ammonium hydrogen sulfide in a pipeline reactor to precipitate virtually all of the cobalt and about 10 % of the nickel. The mixed sufide product (15 % Co, 40 % Ni) was thickened, washed, and spray dried, and sold for cobalt and nickel recovery elsewhere. Ammonia and carbon dioxide were then stripped from the cobalt-free solution by direct stream injection to precipitate basic nickel carbonate.

Since 1988, the pregnant liquor has been treated by solvent extraction to separate nickel and cobalt. An organic reagent (LIX 87 QN) is used to extract nickel selectively from the pregnant liquor. The nickel is recovered from the solvent by stripping with a more concentrated ammoniacal ammonium carbonate solution. Basic nickel carbonate can then be precipitated from the strip liquor by steam injection to remove ammonia and carbon dioxide [4].

In Turkey, Yüksel, M. studied the nickel recovery from Çaldağ laterites in 1985. Transition and serpentinous type laterites obtained from Çaldağ and a prepared mixture of them were used. Experiments were carried out in two stages. First step was reductive roasting by using CO as reductant and second stage was ammonia-ammonium sulfate leaching. In roasting stage the experimental variables studied were flow rate of reductant gas, roasting time, roasting temperature and amount of additive. The experimental variables studied in the leaching stage were particle size, leaching time and temperature, stirring rate and solid to liquid ratio. The results of the experiment indicated that a nickel recovery of more than 90 % from the transition type ore and from the prepared mixture of transition and serpentinous type ores, with pyrite addition, could be possible but a nickel recovery of more than 60 % from the serpentinous ore was not possible [18].

2.3.2.2 High Pressure Acid Leaching Process (HPAL)

HPAL processes require ores that are predominantly limonitic; in the case of the dry laterites they contain nontronite and/or smectite. In general the ores:

- contain some saprolite
- have lower Mg- usually limited to <4 % (At higher Mg acid consumption is higher)
- require lower Al content (clays are high acid consumers; therefore the Al content should not be too high)

The pressure leaching is carried out either in pachuka tanks or titanium lined autoclaves (all modern plants). Leach temperatures vary in the range 245 to 270 °C. Solid-liquid separation is carried out by Counter-Current Decantation (CCD). There are various ways of purifying the nickel containing solution and separating nickel and cobalt. In modern plants such separation is carried out by solvent extraction (SX). Final products produced are electro-nickel, nickel oxide or nickel briquettes. Some plants produce intermediate materials (mixed sulfides or mixed hydroxides) that are refined elsewhere [1].

The generalized block flow diagram for High Pressure Acid Leaching Process is given in Figure 6.

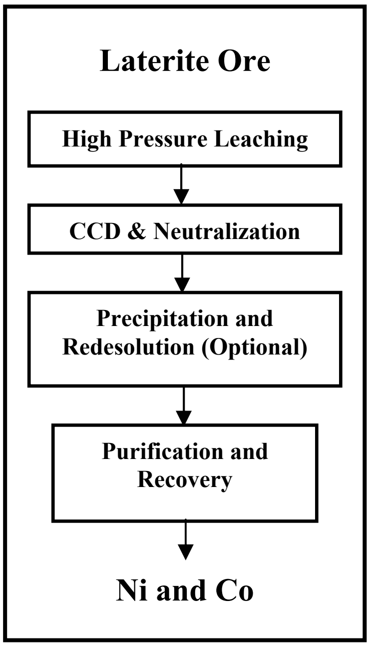


Figure 6. *Generalized Flow Diagram for HPAL Process*

Because of the naturally high moisture content of limonite minerals (20 – 50 % H_2O), it would be advantageous if the ore drying and reduction roasting steps, which together account for over 50 % of the energy consumed in the Caron process, could be avoided, and the ore leached directly. However direct attack by the common mineral acids under ambient conditions is unselective and total dissolution of the ore results, with uneconomically high acid consumption. Fortunately, leaching of the ores in sulfuric acid at high temperature (> 250 °C) and pressure is highly selective since nickel and cobalt are stable as soluble sulfates, while iron(III) sulfate hydrolyzes and precipitates as a hydrated iron(III) oxide. Offsetting the energy savings in this type of process are the increased costs of construction materials capable of withstanding the highly corrosive environment of the high-temperature acid leach.

The applicability of the acid leach process is limited to those ores which do not contain substantial levels of other acid consuming minerals such as magnesia. It was for this reason that Freeport did not select an acid leaching process when seeking a process to treat the Nicaro ores (8 % MgO). However when the same company subsequently developed the low-magnesia (1.7 % MgO) limonitic ore body at Moa Bay, Cuba, the sulfuric acid leach process was selected since it offered much higher (90 % or more) recoveries for both nickel and cobalt, with relatively low acid consumption. Although this process has been applied on a commercial scale only in the one plant at Moa Bay, considerable process development effort was devoted during the 1970s to improving the economics and extending the applicability of the process to higher magnesia content ores [4].

Johnson, J.A., et. al. have been optimised the extraction of nickel from Western Australian laterite ores via sulphuric acid leaching at 250 ^{o}C by the addition of sodium sulphate. Leaching of a nontronitic nickel laterite ore in fresh water, with a sulphuric acid loading of 420 kg/t ore extracted 93 % of nickel from the ore. When limonitic nickel laterite ore was leached in fresh water with an acid loading of 310 kg/t ore, 92 % of the total nickel was extracted. The nickel extractions were optimised at 96 % for the nontronitic ore and 95 % for the limonitic ore, at the above acid loadings, when process water sodium ion levels were increased to 4 g/L and 2 g/L, respectively. Further addition of sodium ion resulted in a gradual decrease in overall nickel extraction, as the leach liquor free acidity decreased. At higher acid loadings, the process water sodium ion levels required for optimum extraction were also higher, so as to prevent dissolution of aluminium [19].

Kar, B.B., et. al. have designed experiments to optimize the sulphatization of a mechanical mixture of chemically pure nickel and iron oxide and a lateritic ore using sulphuric acid. The significance of various parameters and their selectivity for nickel extraction over iron have been established. The interaction among the operating parameters has been studied using statistically designed experiments and the data, thus obtained for the pure system, was correlated with that for the lateritic ore. Regression equations were formulated for both systems and the extraction was represented as a function of response variables. It was observed that the coefficient of temperature was the most dominant, followed by acid concentration. Interestingly, the time factor induces a small effect on extraction [20].

Georgiou, D. and Papangelakis, V.G. carried out experiments using batch pressure leaching techniques. A titanium autoclave equipped with acid injection and sample withdrawal units was employed. Conditions close to the industrial practice were tested: pulp density 30 %, acid to ore ratio 0.2 and temperature ranging from 230 to 270 ^{o}C. Raw limonite and the evolution of the nature of solid products during leaching were characterised using transmission electron microscopy. It was observed that limonite consists of aggregates of needle-like particles of goethite compacted together. Nickel was found to be predominately associated with this phase. During leaching, goethite dissolves continuously liberating nickel whilst iron re-recipitates as dense hematite particles in solution by ex situ precipitation. Nickel was found to exist mainly in the goethite lattice. As goethite dissolves during leaching, nickel is liberated and remains in the aqueous phase. Nickel was also found in magnesium silicate structures highly leachable phase and in manganese particles. High temperature enhances the speed of nickel dissolution. Temperatures as high as 250 to 270 ^{o}C lead to high and rapid extraction and 95 % final extraction. The extent of Ni extraction is not affected in the region of 230 to 270 ^{o}C, but as the temperature increases the reaction becomes much faster. Cobalt was found to exist in the manganese phase. It dissolves rapidly reaching an extraction of 90 % and temperature has no influence in the range of 230 to 270 ^{o}C. Aluminium exists mainly in the goethite and chromite lattice but it is also found in the form of gibbsite or aluminium oxide structures. It dissolves during leaching and re-precipitates as alunite [21].

An industrial application of this process was designed and built by Freeport in 1959 at Mao Bay to produce 22,700 t/a Ni and 2,000 t/a Co in the form of a mixed sulfide concentrate. The concentrate was to be refined to the pure metals in a separate plant which Freeport built at Port Nickel, Louisiana, where a cheap supply of natural gas was available.

The ore, which consists mainly of goethite (a-FeO · OH), is slurried in water and screened to remove low-grade, oversize material, which is discarded. The fines fraction (< 1 mm) that forms the feed to the acid pressure leach averages 1.35 % Ni, 0.15 % Co, 48 % Fe, 1.7 % MgO, 9 % Al_2O_3, 3 % Cr_2O_3, and 4 % SiO_2 in composition. It is leached at 45 % solids pulp density with 98 % sulfuric acid (240 kg H_2SO_4/t dry ore) at about 245 °C and 4.3 MPa, with a retention time of 1–2 h. according to following reactions:

$$NiO + H_2SO_4 \rightarrow NiSO_4 + H_2O \tag{2.7}$$

$$CoO + H_2SO_4 \rightarrow CoSO_4 + H_2O \tag{2.8}$$

The acid is made on-site from elemental sulfur, and the waste heat from the acid plant is used in the leaching circuit. Leaching is carried out in four leaching trains each containing four large vertical autoclaves, which are lead- and brick-lined with titanium internal fittings. Agitation is achieved by injecting high-pressure steam into a draft tube at the bottom of the vessel. Heat is recovered from the autoclave discharge slurry for use in preheating the cold feed slurry. Under these conditions, about 94 % of the nickel and cobalt dissolve, while iron solubility is limited to about 1 g/L.

The leach liquor typically contains 6 g/L Ni, 0.5 g/L Co, 2 g/L Mn, 0.8 g/L Fe, 0.4 g/L Cr, 2.5 g/L Mg, 2 g/L SiO_2, 3 g/L Al, and 30 g/L H_2SO_4. The liquor is separated from the leach residue (0.08 % Ni, 0.01 % Co, and 51 % Fe) by countercurrent decantation (CCD) washing with water in a six-stage thickener circuit. The washed residue is stockpiled for possible future use as iron ore.

The pregnant solution is treated with calcium carbonate to neutralize the acid and precipitate most of the dissolved iron, aluminum, and manganese by hydrolysis. The precipitated sludge is recycled to the wash circuit and joins the leach residue for disposal. The neutralized solution typically contains (g/L): 3.5 – 4.0 Ni, 0.3 – 0.4 Co, 1.0 Mn, 0.3 Fe, 0.4 Cr, 2.5 Mg, 1.0 SiO_2, and 3.0 Al at pH 2.5.

Nickel and cobalt are recovered from solution in 98 – 99 % yield by precipitation at 120 °C with hydrogen sulfide at a pressure of 1.1 MPa in a three-compartment, brick-lined autoclave according to following reaction:

$$NiSO_4 + H_2S \rightarrow NiS + H_2SO_4 \tag{2.9}$$

The barren solution typically contains (g/L): 0.05 Ni, 0.01 Co, 1.0 Mn, and 7 H_2SO_4. The nickel – cobalt concentrate contains 55 % Ni, 5 % Co, 0.7 % Fe, 0.2 % Cu, 1.0 % Zn, and 35 % S [4].

There is not any industrial application of High Pressure Acid Leaching Process in Turkey.

2.3.2.3 Enhanced Pressure Acid Leaching Process

Enhanced Pressure Acid Leaching Process is the combination of Pressure Acid Leach and Atmospheric Acid Leach Processes. This process includes an additional leaching step for saprolite using residual acid from the HPAL step. Saprolite is leached at atmospheric pressure and is a high acid consumer [1].

2.3.2.4 Acid Heap Leaching Process

Heap leaching of metallic ores is a well-proven process, especially in the copper industry, where many millions of tonnes of cathode copper have been produced using solvent-extraction and electro-winning for many years. In recent years the process has been introduced to the production of other metals, especially zinc and nickel.

In its simplest method, the acid heap leach process consists of building a large heap of crushed ore and irrigating the surface of the heap with a dilute solution of acid. As the acid permeates through the heap it dissolves the metal particles in the ore, which are taken into solution. The pregnant solution is collected at the bottom of the heap, usually on an impermeable rubber liner, and pumped away for further treatment, using various chemical processes dictated by the metal being recovered and the chemical constituents of the ore.

Some of the expertise and methodology of heap leach operations will be utilized in developing the heap leach operation for the nickel deposit in Turkey, Manisa, Çaldağ region.

At Çaldağ, the pilot scale project, as shown in Figure 7, envisages that the ore will be crushed and screened at the mine, with the waste material being returned as backfill to the pits. The ore will then be transported to a suitable flat area where the metallurgical operations will be situated. It will be offloaded from the trucks at the plant site where it is crushed further through a two-stage process to a size of less than 6 mm. The crushed ore is stacked on plastic liners, which act as an impermeable membrane to collect and channel the leached solutions to the central collection ponds. The heaps are covered with a network of plastic pipes that drip sulphuric acid onto the crushed ore. The acid dissolves the nickel, cobalt and some other minerals and the solutions flow on to the plastic liners to the collection ponds. Iron is continually removed from the solution using precipitation with limestone. The iron free solution is returned to the heap to increase the nickel levels. A portion of the iron free solution is sent to the metal precipitate section where the nickel and cobalt is precipitated as a mixed hydroxide [22].

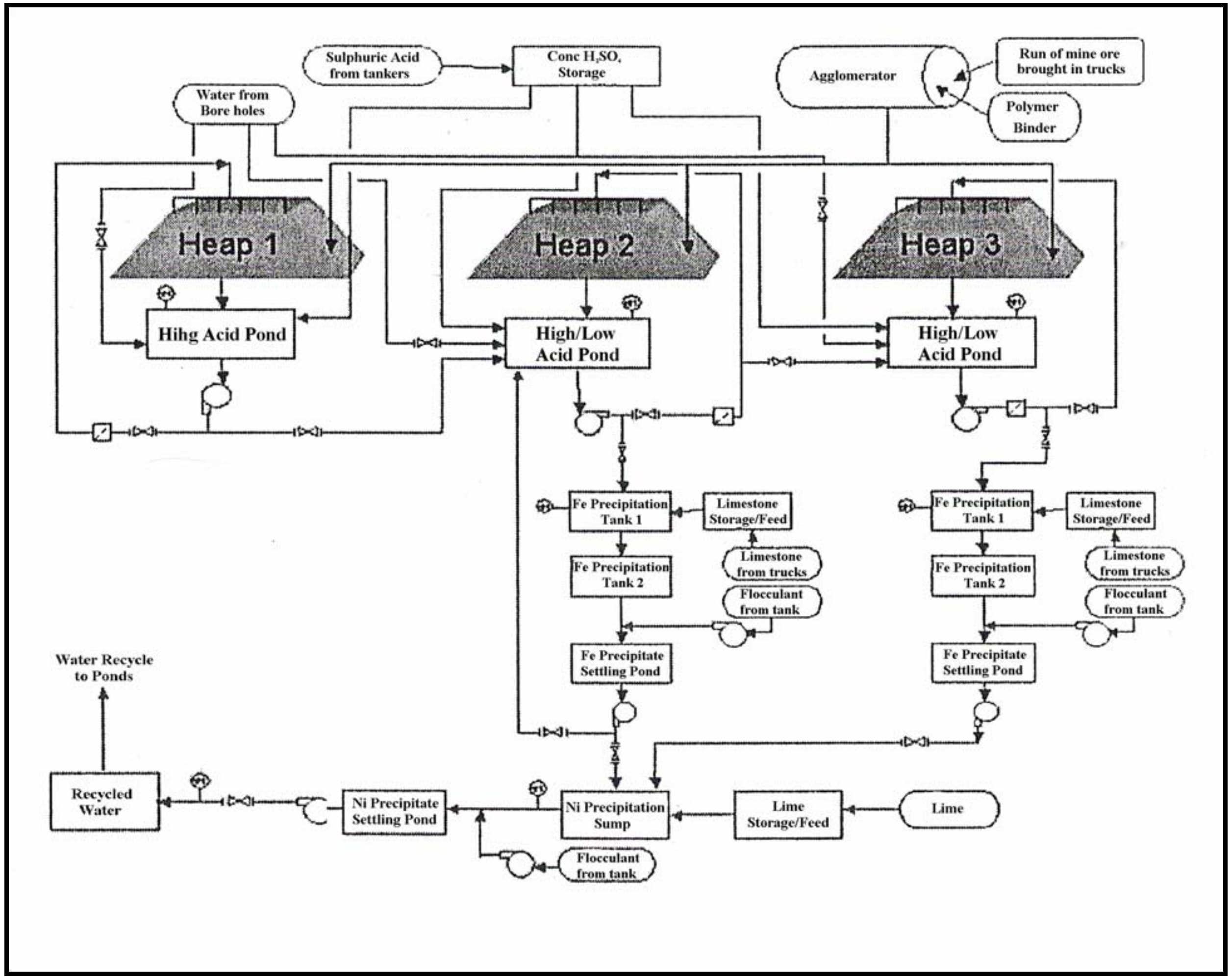


Figure 7. *The Proposed Heap Leach Flow Sheet at Çaldağ, Manisa [23]*

2.3.2.5 Atmospheric Chloride Leaching Process

Atmospheric Chloride Leaching Process is a developing process and can be applied to mixed limonitic and saprolitic ores.

Jaguar Nickel Inc. is developing a nickel-cobalt laterite project in Guatemala, Central America. In this process, a mixed chloride lixiviant is used to recover the nickel and cobalt, while leaving the majority of the iron and magnesium in the leach residue. Subsequent solution purification and mixed nickel/cobalt hydroxide product recovery is by standard precipitation methods, using recycled caustic magnesia, with a final stage of pyrohydrolysis of a portion of the concentrated magnesium chloride brine for recycling both the caustic magnesia and chloride lixiviant [24].

The main unit operations in Atmospheric Chloride Leaching Process, as shown in Figure 8, are:

- Leaching of the ore in a mixture of recycled magnesium chloride solution and recovered hydrochloric acid at 80-105 ^{o}C.
- Solid/liquid separation on a vacuum belt filter. The solids generated during chloride leaching, due to the inherent desiccating properties of the leaching medium, are appreciably more amenable to filtration than those from a PAL/HPAL/EPAL process.
- Purification of the leach filtrate by oxidation with Cl_2 gas (if necessary) and neutralization with recycled MgO. Oxidation of Mn and residual ferrous iron may be necessary, and this can readily be achieved in a chloride medium by chlorine gas with appropriate pH and redox control. This also provides for some or all of the chloride make-up required within the circuit.
- Thickening of the purification slurry and return of the underflow to the end (highest pH region) of the leach. This is done in order to maximize MgO utilization, and to recover any nickel and cobalt co-precipitated.

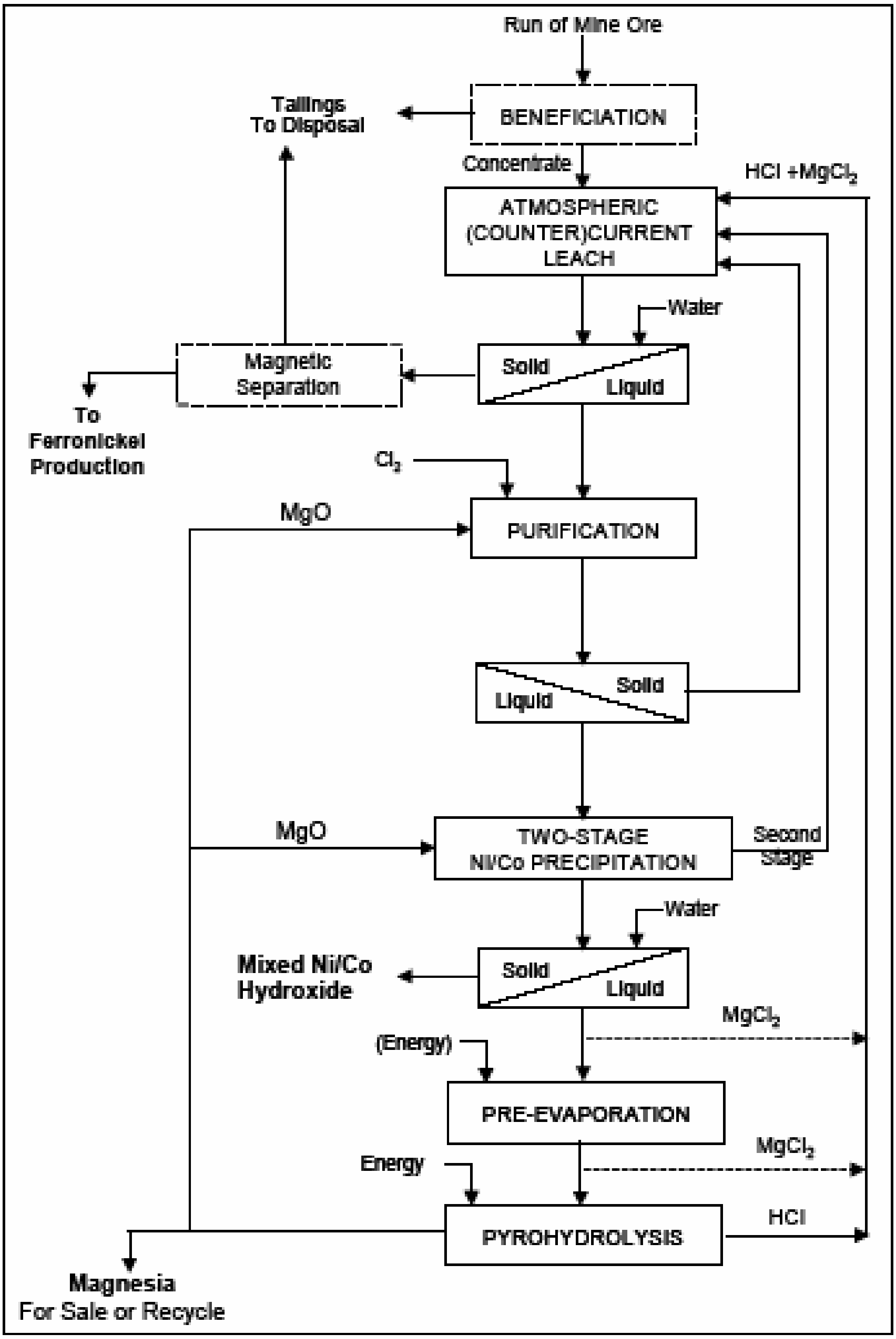


Figure 8. *The Flowsheet for Atmospheric Chloride Leaching Process*

- Precipitation of a mixed nickel/cobalt hydroxide product with recycled MgO.
- Vacuum filtration and washing of the mixed hydroxide product.
- Lixiviant regeneration by modified pyrohydrolysis [25].

According to the the initial laboratory testwork of Harris, G. B., et. al., the use of chloride brine chemistry is effective in not only leaching nickel and cobalt out of the Guatemala laterites, but that the degree of both iron and magnesium leaching can be controlled. Good recoveries of both nickel and cobalt from the non-magnetic component of the ore were achieved. $MgCl_2/HCl$ brine leaching is an effective and efficient method of recovering nickel and cobalt from laterites. The advantages of using this system are considerable in that the degree of iron dissolution can be effectively controlled directly in the leach circuit, thereby eliminating the need for costly purification circuits or pressure hydrolysis. The nature of the atmospheric brine system is such that although some iron dissolves, it subsequently readily hydrolyses and precipitates, always provided that significant ferric chloro anion complexes have not been formed. Another advantage is that in using magnesium chloride as the brine, the amount of magnesium leaching can also be effectively managed [24].

CHAPTER 3

GENERAL INFORMATION ABOUT GÖRDES LATERITES

3.1 Geology

The Gördes deposit is associated with the regional SE Eurasian Ophiolite Belt that stretches from near Belgrade in the north, southeastwards through Albania and Macedonia into Greece and then eastwards through Turkey into Iran.

Within Gördes, a number of geologic studies have been undertaken during the last thirty to forty years. These studies have identified the broad framework of the area whereby the oldest unit has been determined as the Jurassic-Cretaceous aged dolomitic limestones at the basement onto which have been thrust Upper-Cretaceous aged ultrabasic rocks and associated rock types, typically seen as a complex melange. Above these, volcanics have been emplaced, whilst at the top, younger sediments like the Pliyosen aged clayey limestone and clay-stone unconformably overly the whole sequence.

The structure of the area is inherently complex, although the dominant tectonic trend appears to be north-south reflecting the principal thrust direction of the ophiolites over the limestones and dolomites [26].

3.2 Mineralisation

The laterites were formed during the early Tertiary, by intense weathering of Alpine-type ophiolites which normally contain about 0.2 % Ni and 0.3 % Cr in

Turkey. These ophiolites cover extensive areas of Turkey, and thus provide significant potential for the discovery of Ni-rich laterites.

At Gördes, the primary host rock for the laterites is serpentinite which is partially exposed often on the lower slopes across the area.

From preliminary work in the area, the laterites are sandwiched between the serpentinites and the silica cap rocks and appear to have a broad range of thicknesses extending between 2-3 m. and 80 m. Using very limited exposure, a thin saprolitic layer has been noted immediately above the serpentinite contact, developing rapidly upwards into the limonite zone.

The limonite zone exhibits a strong ferruginous weathering profile varying from yellow to dark brown and reddish in colour, all of which contain Ni and Co mineralisation. Close to the silica cap, a stiff hematite zone, dark brown to black in colour, overlies the limonite. High Ni grades are usually associated with this zone, and green garnierite is often seen as the principal Ni-bearing mineral [26].

3.3 Mineralogy

Physically, the nickel laterite mineralisation is soft and soil-like in general and contains discrete rock fragments of hard siliceous and hematite bands. The moisture content varies from 15–20 %.

Deposited nickel-rich zones are composed of limonites with high iron content and saprolites towards the base of the sequence. Nickel is mostly observed within these zones as fine grained goethite, and rarely as lepidocrocite. Another nickel mineral, asbolan, occurs in the lower levels of the zone, generally associated with clay minerals.

From preliminary work, it appears that the Turkish laterites differ from their SE Asia cousins in that they formed under sub-tropical conditions here as opposed to the tropical conditions which acted on SE Asian laterites. With less intense heat and precipitation in Tertiary Turkey, it seems that the lateritisation process was not capable of removing as much of silica and iron as in tropical areas. This has resulted in silica and some of the iron being transported only a very limited distance with the consequent formation of the Fe-silica crusts.

Thus, the resulting laterite deposits are chiefly of limonitic type. However, as the bedrock of fresh ophiolites is reached, a saprolitic horizon is also developed, although the thickness of this zone is as yet unknown [26].

A general view from Gördes laterites is given in Figure 9.

Figure 9. *A General View from Gördes Laterites*

CHAPTER 4

EXPERIMENTAL MATERIALS AND METHODS

4.1 Materials

The ore used in the experiments was the lateritic ore and the materials used in the experiments were hydrochloric acid and magnesium chloride. The characteristics of the lateritic ore and materials are given in following sections.

4.1.1 Lateritic Ore

The lateritic ore used in the experiments was obtained from Gördes district, Manisa, Turkey. The moisture analysis showed that the original moisture content of the sample is 25 %. The density of the ore is 2.95 g/cm^3. A picture of the lateritic ore used as the nickel metal raw material is shown in Figure 10.

Figure 10. *Lateritic Ore Sample*

4.1.1.1 Particle Size Analysis

The sample that was brought to Mining Engineering Department, Mineral Processing Laboratory was under 1 cm. size. In order to determine the particle size distribution of the ore, wet sieve analysis was carried out. Ni, Co and Fe analysis of each size fractions were performed by Meta Mining Industry Energy Tourism and Foreign Trading Ltd. by using X-MET 820 XRF analyzer. The results of these analyses are given in Table 8.

Table 8. *Wet Sieve Analysis Results and Ni, Co and Fe Grades and Distributions of the Ore*

Size (μm)	**Weight %**	**Ni**		**Co**		**Fe**	
		Grade %	**Dist. %**	**Grade %**	**Dist. %**	**Grade %**	**Dist. %**
+ 1168	10.12	0.917	5.04	0.095	7.40	46.08	11.37
- 1168 + 833	3.37	1.141	2.09	0.105	2.73	51.73	4.25
- 833 + 589	3.03	1.218	2.01	0.109	2.54	49.65	3.67
- 589 + 355	6.11	1.327	4.41	0.112	5.27	46.56	6.93
- 355 + 210	5.01	1.634	4.45	0.128	4.94	44.65	5.45
- 210 + 125	4.78	2.184	5.67	0.165	6.07	41.30	4.81
- 125 + 74	2.39	2.566	3.33	0.150	2.76	40.83	2.38
- 74	65.19	2.060	73.00	0.136	68.28	38.47	61.14
TOTAL	**100**	**1.840**	**100**	**0.132**	**100**	**41.02**	**100**

According to the wet sieve analysis of the sample, as it is seen in Table 8, 65.19 % of the ore is under 74 μm and the nickel distribution at this size is 73.00 %.

4.1.1.2 Chemical Composition

The Ni and Co content of the representative test sample were found as 1.84 % and 0.13 % respectively by the chemical analysis carried out by Meta Mining Industry Energy Tourism and Foreign Trading Ltd. The other components of the ore sample was determined by the General Directorate of Mineral Research and Exploration. Table 9 shows the chemical analysis results of the test sample.

Table 9. *Chemical Composition of the Laterite Ore*

Component	**%** or **ppm**
MgO	0.5 %
Fe_2O_3	56.8 %
As	0.69 %
Al_2O_3	4 %
CaO	0.9 %
Cr	2.78 %
SiO_2	20.1 %
MnO	1 %
Zn	420 ppm
Cu	90 ppm
Cl	50 ppm
Loss of Ignition	9.06 %

Chemical analysis results indicated that the ore which was used as the nickel metal source in this study is a limonite type laterite.

3.1.1.3 Mineralogical Composition

The XRD pattern of the ore, plotted in Figure 11, indicated that hematite, goethite, dolomite, moganite and quartz are the minerals in the ore. Smectite and mixed layered clays are also determined during the microscopical examination.

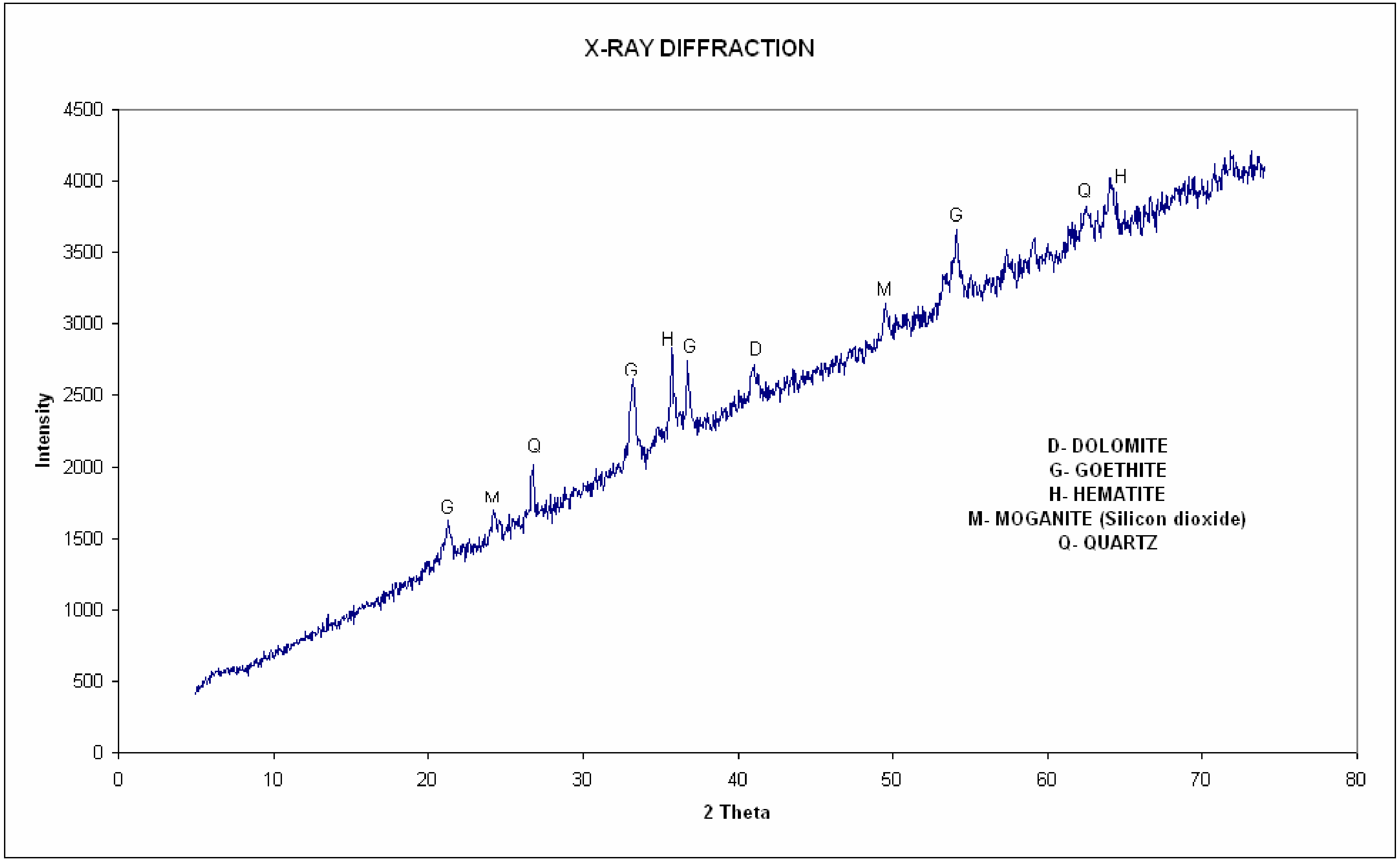


Figure 11. *XRD Pattern of Laterite Ore*

4.1.2 Hydrocholoric Acid (HCl)

Hydrochloric acid is a solution of hydrogen chloride in water. Hydrogen chloride occurs as either a colourless liquid with an irritating, pungent odour or a colourless to slightly yellow gas which is highly soluble in water [27].

In the experiments, 1 N, 2 N, 3 N and 4 N hydrochloric acid solutions prepared with 35 % analytical grade HCl with a density of 1.18 g/cm^3, were used as leach solutions.

The physical properties of hydrochloric acid, such as boiling and melting points, density, and pH depend on the concentration or molarity of HCl in the acid solution [28].

4.1.3 Magnesium Chloride ($MgCl_2$)

The chemical formula for magnesium chloride is $MgCl_2$. It is present in hydrated forms such as $MgCl_2.6H_2O$. It is extracted from seawater and is a source of magnesium for metallurgical processes. Pure magnesium chloride is a white or colorless, odorless and bitter-tasting compound [29].

$MgCl_2.6H_2O$, in this study, was used in order to determine the effect of it to the leaching process by forming a mixed chloride lixiviant medium with HCl.

4.2 Methods

Before agitating leaching tests, the test samples were subjected to some preconcentration processes in order to determine the possibility of decreasing the amount of feed for leaching experiments.

4.2.1 Preconcentration Processes

Attrition scrubbing, attrition scrubbing followed by cycloning and magnetic separation with Permroll are the methods applied for preconcentration of the ore.

4.2.1.1 Attrition Scrubbing

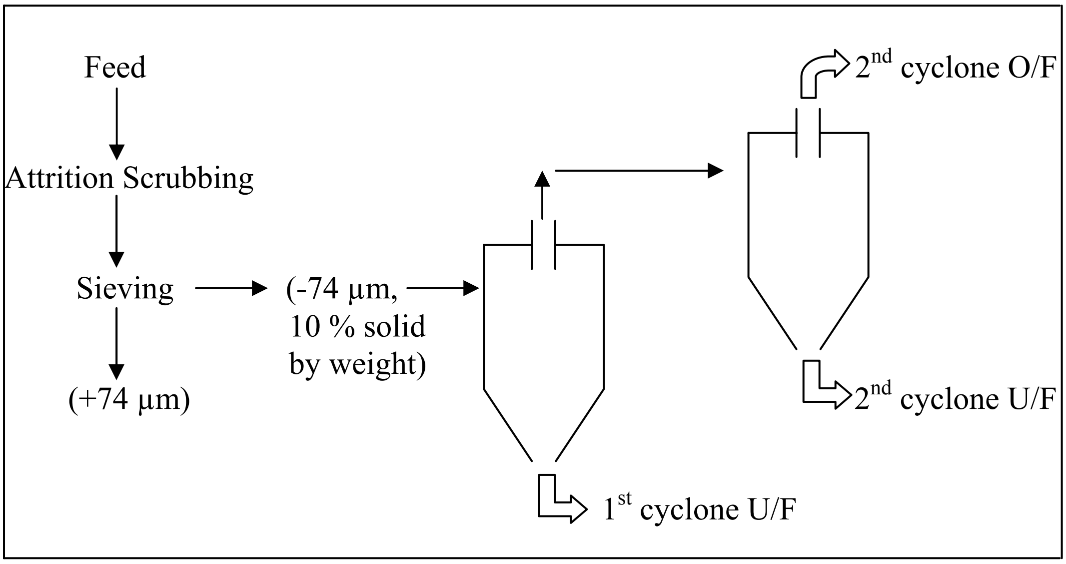
Attrition scrubbing experiments were carried out at two different pulp densities (20 % and 50 % solid by weight) and four different treatment durations (15 min., 30 min., 45 min. and 60 min.) with 250 g. original size samples. At the end of tests, test sample was sieved through 74 µm test sieve and sieve fractions were dried in furnace, weighed and analysed chemically. The conditions of these experiments are given in Table 10.

Table 10. *Conditions of Attrition Scrubbing Experiments*

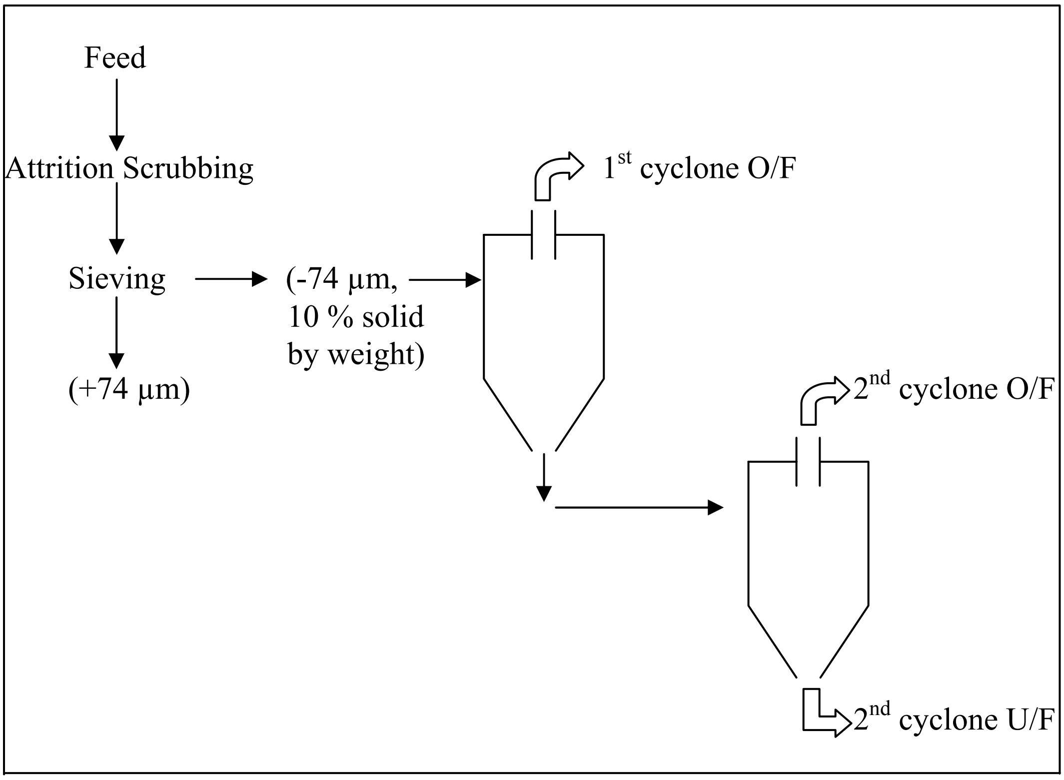
Exp. No	**Solid by weight (%)**	**Mixing duration (min)**
A.1	20	15
A.2	20	30
A.3	20	45
A.4	20	60
A.5	50	15
A.6	50	30
A.7	50	45
A.8	50	60

4.2.1.2 Attrition Scrubbing Followed by Hydrocycloning

These experiments were carried out with the fine fraction of attrition scrubbing tests products which was obtained at optimum conditions (Experiment A.8 conditions). Two different routes were followed. In the first experiment, cyclone overflow, and in the second experiment, cyclone underflow were again treated by hydrocyclones (Cyclone diameter: 24.25 mm. Φ, Feed inlet dimensions: 4.05 mm. x 2.45 mm., Vortex finder: 4.65 mm. Φ, Apex nozzle: 3.30 mm. Φ). Then, the hydrocyclone products were filtered, dried in furnace, weighed and analysed chemically. Figure 12 shows the flowsheet of the applied processes.



a. Recycloning of Cyclone Overflow after Attrition Scrubbing (Exp. B.1)



b. Recycloning of Cyclone Underflow after Attrition Scrubbing (Exp. B.2)

Figure 12. *Flowsheet of Attrition Scrubbing Followed by Hydrocycloning Processes*

4.2.1.3 Preconcentration with Permroll (Magnetic Separation)

In magnetic separation experiment, the test sample was subjected to dry magnetic separation with laboratory type Permroll instrument with 71 mm. roll diameter and magnetic and non-magnetic products were weighed and analysed chemically.

4.2.2 Leaching Processes

Hydrochloric acid (HCl) leaching at room temperature, HCl leaching at room temperature with the addition of magnesium chloride ($MgCl_2.6H_2O$) and HCl leaching at elevated temperatures are the three groups of experiments that were carried out for the extraction of nickel.

4.2.2.1 Hydrochloric Acid (HCl) Leaching Experiments At Room Temperature

These series of experiments were carried out with 50 g. of -1 mm. sized particles in 1,000 ml. beakers at room temperature. Prepared lateritic ore samples were leached with hydrochloric acid solutions with four different HCl concentrations (1 N, 2 N, 3 N and 4 N), where pulp density was 1/30 solid to liquid ratio by volume, for five different treatment durations (0.25 hour, 1 hour, 2 hours, 3 hours and 4 hours). The leaching processes were carried out by a magnetic stirrer for those periods to have homogeneity in solution, to prevent agglomeration and for better solid-liquid contact. The leaching apparatus and set-up are shown in Figure 13. At the end of test, the magnetic stirrer was switched off. The solution was filtered and filter cake was washed. Finally, filter cake (residue) was dried in furnace, weighed and analysed chemically. The test conditions are tabulated in Table 11.

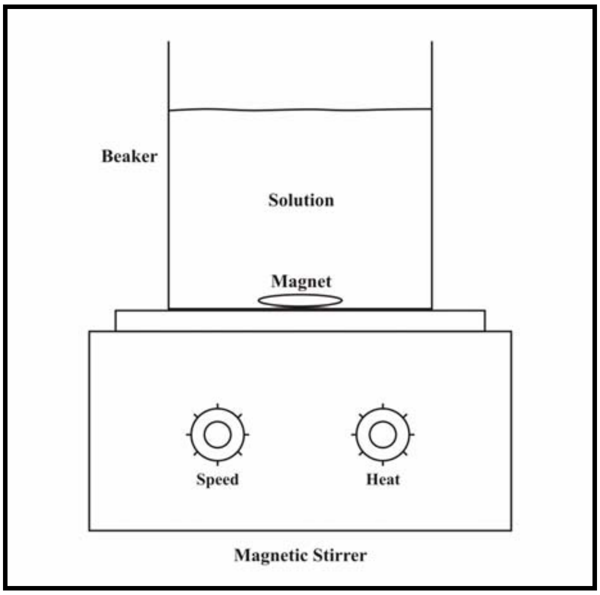


Figure 13. *Apparatus and Set-up for Leaching Experiments at Room Temperature*

Table 11. *Leaching Conditions at Room Temperature*

Exp. No	**HCl Concentration (N)**	**Leaching duration (hr)**
1	1	0.25
2	1	1
3	1	2
4	1	3
5	1	4
6	2	0.25
7	2	1
8	2	2
9	2	3
10	2	4
11	3	0.25
12	3	1
13	3	2
14	3	3
15	3	4
16	4	0.25
17	4	1
18	4	2
19	4	3
20	4	4

4.2.2.2 Hydrochloric Acid (HCl) Leaching Experiments At Room Temperature with Addition of Magnesium Chloride ($MgCl_2$)

These experiments were carried out with 50 g. of -1 mm. sized particles in 1,000 ml. beakers at room temperature for 15 minutes leaching duration. Prepared representative samples were leached with hydrochloric acid solutions with four different concentration values (1 N, 2 N, 3 N and 4 N), where solid to liquid ratio by volume was 1/30. The experiments were carried out at three different total Cl^- levels in leach solution (150 g/lt, 200 g/lt and 300 g/lt) which were obtained by addition of required amount of $MgCl_2.6H_2O$. The leaching processes were carried out by magnetic stirrer for those periods. After the defined duration for the experiments elapsed, the magnetic stirrer was switched off. The solution was filtered, then the filter cake was washed and dried in furnace. Finally, filter cake was weighed and analysed chemically. The test conditions are given in Table 12.

Table 12. *Leaching Conditions at Room Temperature with Addition of $MgCl_2$*

Exp. No	**HCl Concentration (N)**	**Total Cl^- level (g/lt)**
21	1	150
22	2	150
23	3	150
24	4	150
25	1	200
26	2	200
27	3	200
28	4	200
29	1	300
30	2	300
31	3	300
32	4	300

4.2.2.3 Hydrochloric Acid Leaching Experiments at Elevated Temperatures

These experiments were carried out with 25 g. samples with four different particle sizes (-1 mm., -500 microns, -106 microns and -38 microns) in a 250 ml. glass balloon at three different temperatures (50 ^oC, 75 ^oC and 100 ^oC) with four different HCl concentrations (1 N, 2 N, 3 N and 4 N) at different leaching durations. The test conditions are tabulated in Table 13.

Table 13. *Leaching Conditions at Elevated Temperatures*

Exp. No	HCl Con. (N)	Solid to liquid ratio by volume	Particle Size (µm)	Temperature (^oC)	Leaching Duration (hr)
33	2	1/30	-500	25	0.25
34	2	1/30	-106	25	0.25
35	2	1/30	-38	25	0.25
36	2	1/30	-1000	50	0.25
37	2	1/30	-500	50	0.25
38	2	1/30	-106	50	0.25
39	2	1/30	-38	50	0.25
40	2	1/30	-1000	75	0.25
41	2	1/30	-500	75	0.25
42	2	1/30	-106	75	0.25
43	2	1/30	-38	75	0.25
44	2	1/30	-1000	100	0.25
45	2	1/30	-500	100	0.25
46	2	1/30	-106	100	0.25
47	2	1/30	-38	100	0.25
48	2	1/30	-38	50	4
49	2	1/30	-38	50	8
50	2	1/30	-38	75	4
51	2	1/30	-38	75	8
52	2	1/30	-38	100	4
53	2	1/30	-38	100	8
54	3	1/30	-38	100	0.25
55	3	1/30	-38	100	1
56	3	1/30	-38	100	2
57	3	1/30	-38	100	3
58	3	1/30	-38	100	4
59	3	1/30	-38	100	8
60	4	1/30	-38	100	4
61	1	1/30	-38	100	8
62	3	1/12	-38	100	1
63	3	1/7	-38	100	1
64	3	1/30	-106	100	3
65	3	1/30	-500	100	3
66	3	1/30	-1000	100	3
67	3	1/30	-38	50	3
68	3	1/30	-38	75	3
69*	3	1/30	-38	100	3
* Exp. 69 was carried out at 200 g/lt total Cl^- level in the leach solution.					

The leaching set up consisted of an IKA RH basic KT/C hot plate and stirrer, a 250 ml. glass balloon, a condenser and a temperature probe. The condenser was used in order to keep the acid concentration constant in the leach solution. The calculated quantity of hydrochloric acid solution was added to the balloon and after the temperature of the solution increased to desired value, the ore was added into the balloon. At the end of leaching period, the hot plate and stirrer was switched off and the balloon was taken for the hot filtration. Hot filtration was applied to leach solution in order to prevent the recrystallization of nickel in the solution. At the end of filtration, the leach residue was washed with hot water and then the filter cake was dried, weighed and analysed chemically.

The flowsheet of these experiments is shown in Figure 14 and the leaching set-up is shown in Figure 15.

4.3 Chemical Analyses

All chemical analyses in this study were done with the leach residue (filter cake) of experiments.

The chemical analyses were done by Meta Mining Industry Energy Tourism and Foreign Trading Ltd. in Gördes, Manisa by using X-MET 820 XRF analyzer.

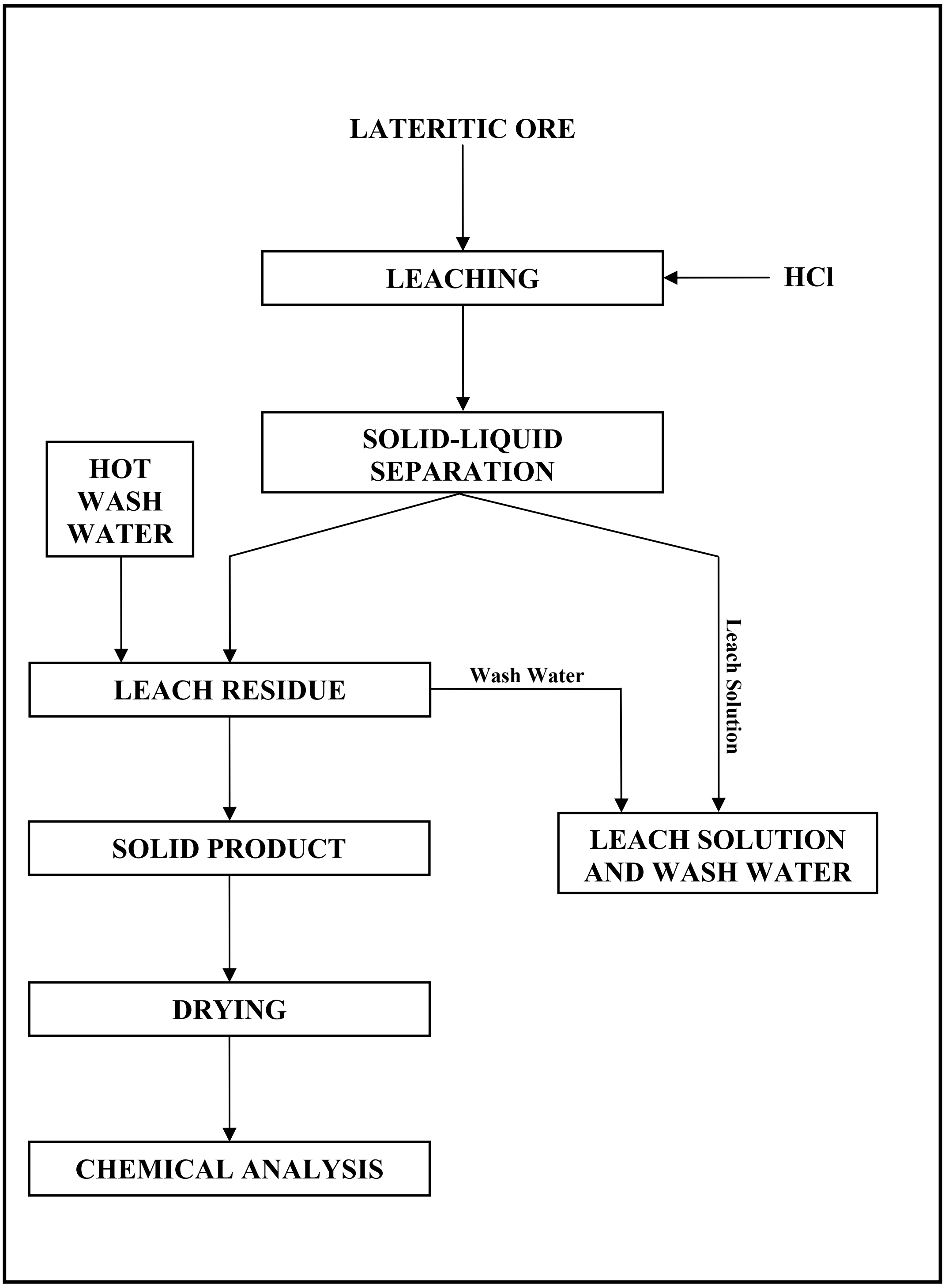


Figure 14. *Flowsheet of the Experimental Procedure*

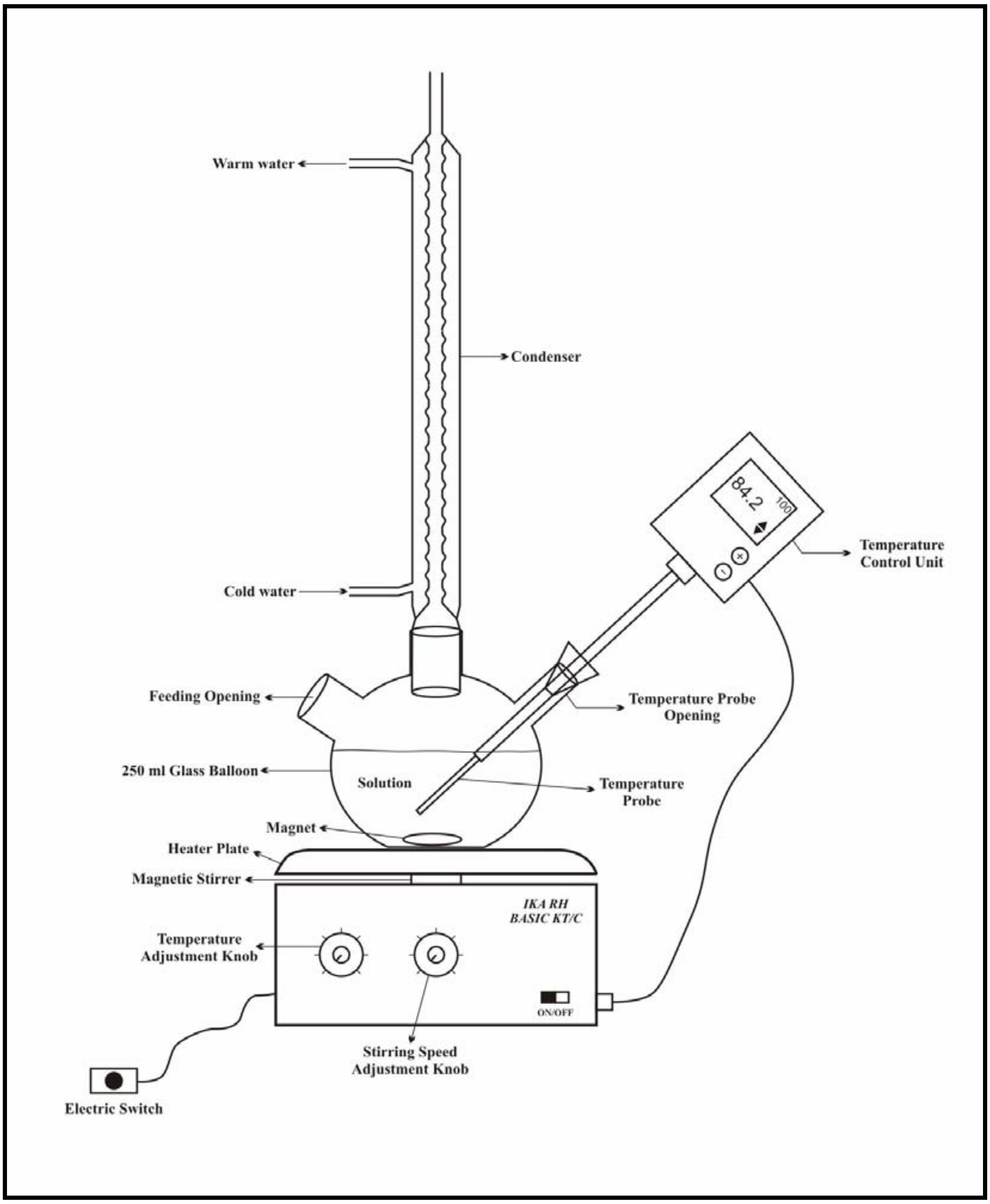


Figure 15. *Set-up for Leaching Experiments at Elevated Temperatures*

CHAPTER 5

RESULTS AND DISCUSSION

5.1 Preconcentration Processes

5.1.1 Attrition Scrubbing

Attrition scrubbing was applied in order to determine the possibility of increasing the nickel content in fine size fraction of the test sample. The pulp density and attrition duration were the variables in the tests. The results of these tests are tabulated in tables given in Appendix A and presented in Figure 16 to Figure 21.

The tests were carried out at two different pulp densities namely 20 % and 50 % solid by weight for the attrition scrubbing durations of 15, 30, 45 and 60 minutes.

As it is seen from Figure 16 and Figure 17, longer test duration increases the amount of fine size fraction of test sample for both pulp densities.

Figure 18 and Figure 19 indicate that the distribution of nickel increases in fine size fraction as the time of attrition scrubbing increases for both 20 % and 50 % solid by weight of pulp.

As it is seen from Figure 20 and Figure 21, attrition scrubbing is more effective for the recovery of nickel in fine size fraction with more dilute pulp and longer attrition scrubbing durations. Although attrition scrubbing resulted in a slight difference in nickel grade of fine size fraction, this difference is not enough to be considered as an efficient preconcentration process.

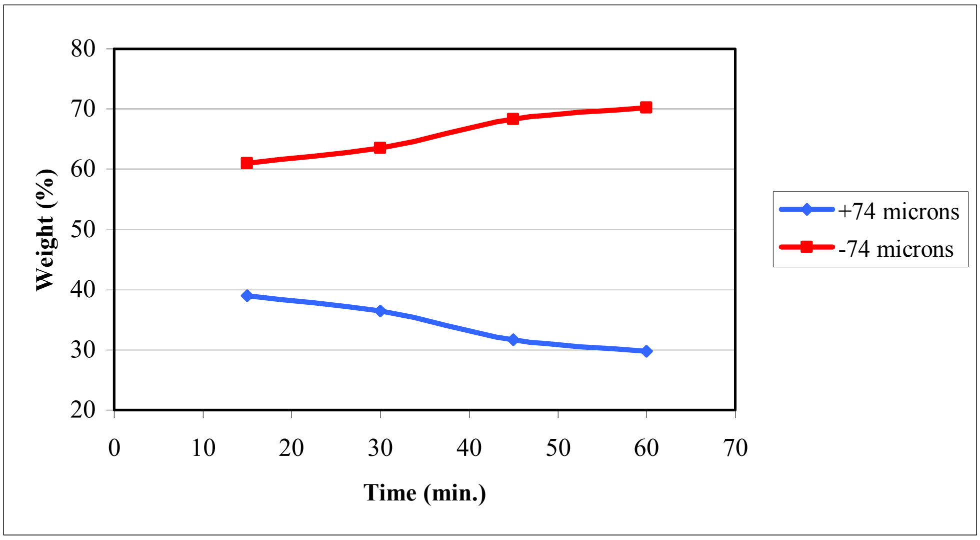


Figure 16. *Particle Size Distribution of Lateritic Ore after Attrition Scrubbing at 20 % Solid by Weight Pulp Density*

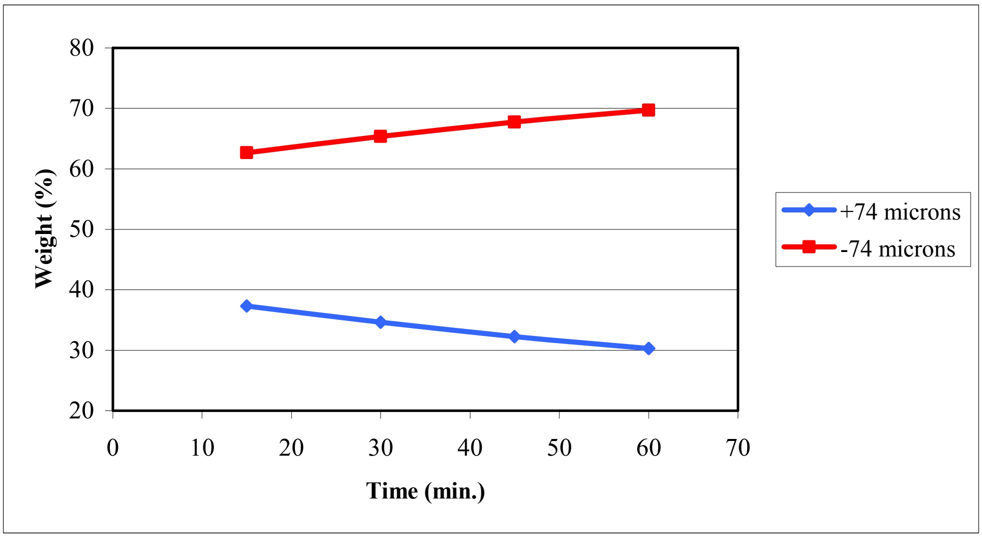


Figure 17. *Particle Size Distribution of Lateritic Ore after Attrition Scrubbing at 50 % Solid by Weight Pulp Density*

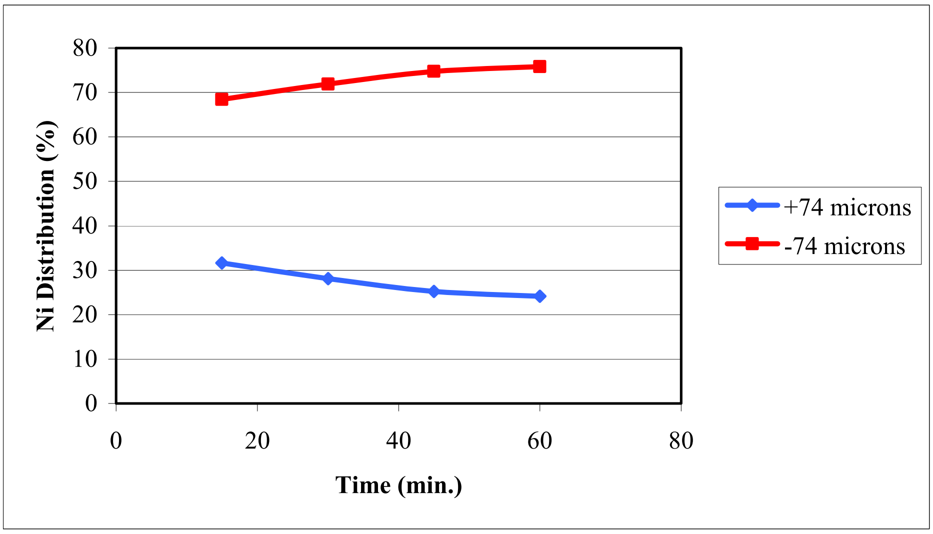


Figure 18. *Nickel Distribution after Attrition Scrubbing at 20 % Solid by Weight Pulp Density*

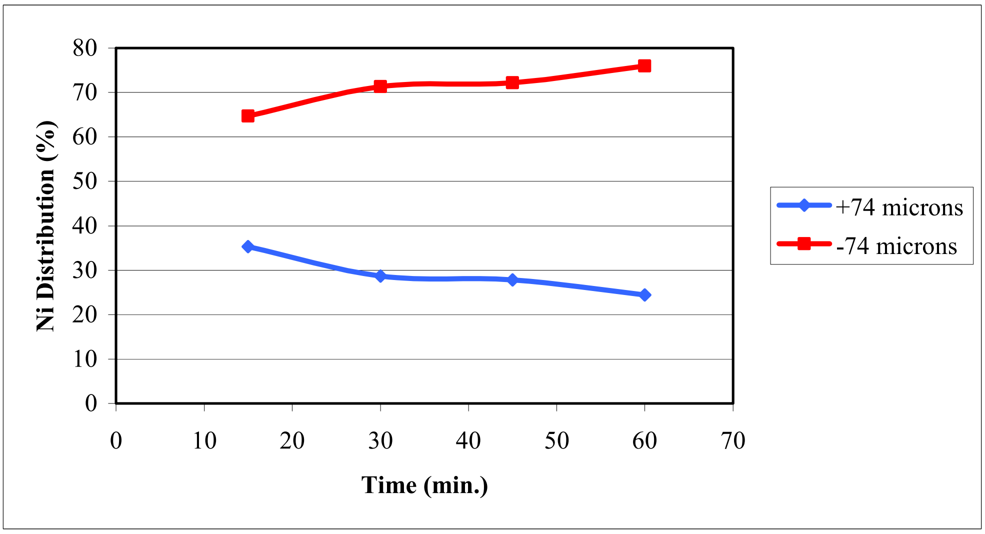


Figure 19. *Nickel Distribution after Attrition Scrubbing at 50 % Solid by Weight Pulp Density*

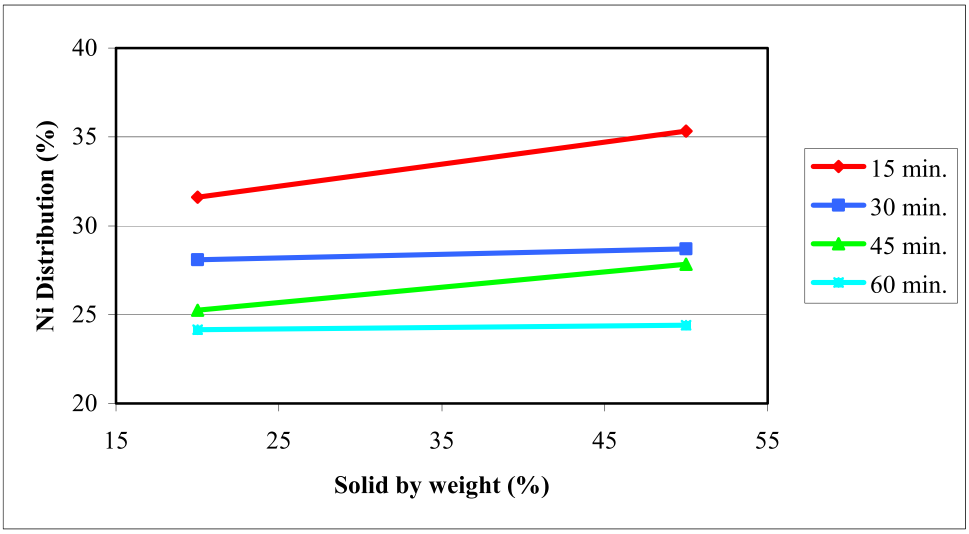


Figure 20. *Nickel Distribution in Coarse Size Fraction (+74 µm) for Different Treatment Durations*

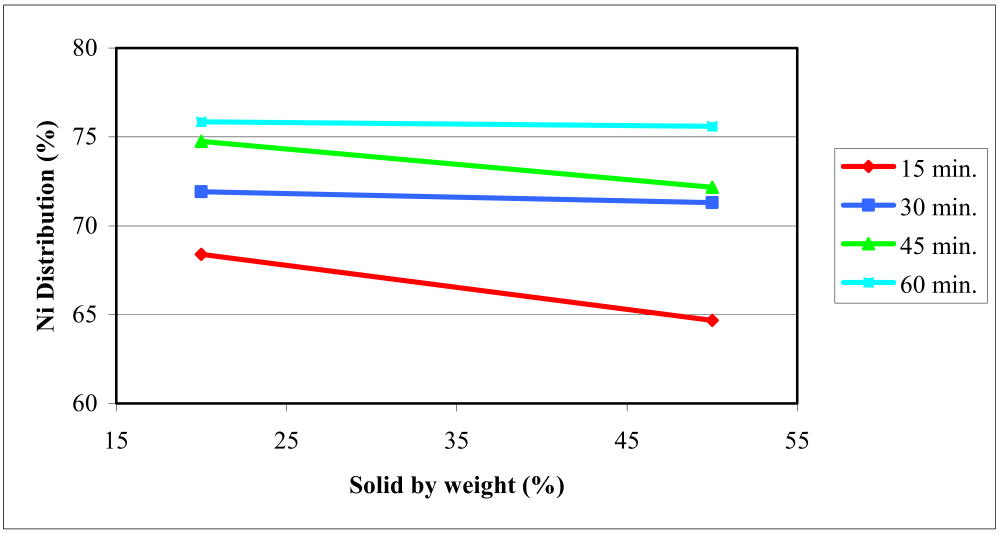


Figure 21. *Nickel Distribution in Fine Size Fraction (-74 µm) for Different Treatment Durations*

5.1.2 Attrition Scrubbing Followed by Hydrocycloning

Two tests were carried out with the -74 µm size fraction of attrition scrubbing test products. After attriton scrubbing at 50 % solid by weight for 60 min. and sieving of pulp, -74 µm size fraction of sample was treated by hydrocylones.

In the first experiment, -74 µm size fraction of the sample obtained by attrition scrubbing was classified by hydrocyclone and the overflow of the first hydrocylone was used as the feed of the next cyclone. The results of this experiment are shown in Figure 22 and tabulated in Table 14.

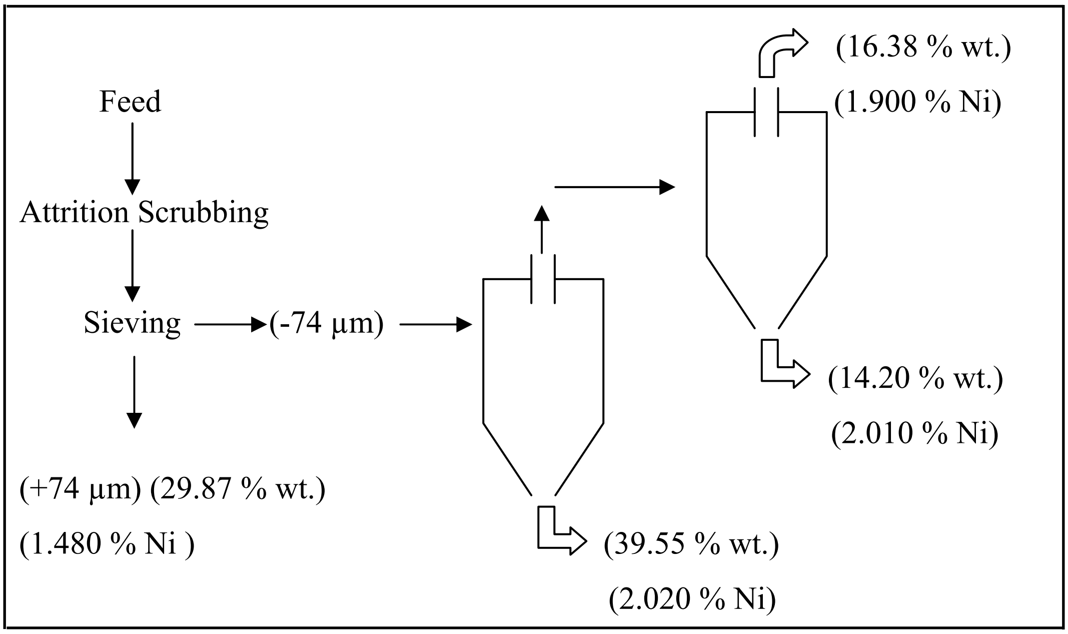


Figure 22. *Recycloning of Cyclone Overflow after Attrition Scrubbing Experiment Results (Exp. B.1)*

***Table 14.** Data and Nickel Distribution Table for Recycloning of Cyclone Overflow after Attrition Scrubbing Experiment (Exp. B.1)*

	Wt (%)	Ni (%)	Wt % x Ni %	Ni Distribution (%)
+74 µm	29.87	1.480	44.21	24.06
1st Cyc. U/F	39.55	2.020	79.89	43.48
2nd Cyc. U/F	14.20	2.010	28.54	15.53
2nd Cyc. O/F	16.38	1.900	31.12	16.94
TOTAL	100	1.838	183.76	100

In the second experiment, -74 µm size fraction of the sample obtained by attrition scrubbing was classified by hydrocyclone and the underflow of the first hydrocylone was used as the feed of next cyclone. The results of this experiment are shown in Figure 23 and tabulated in Table 15.

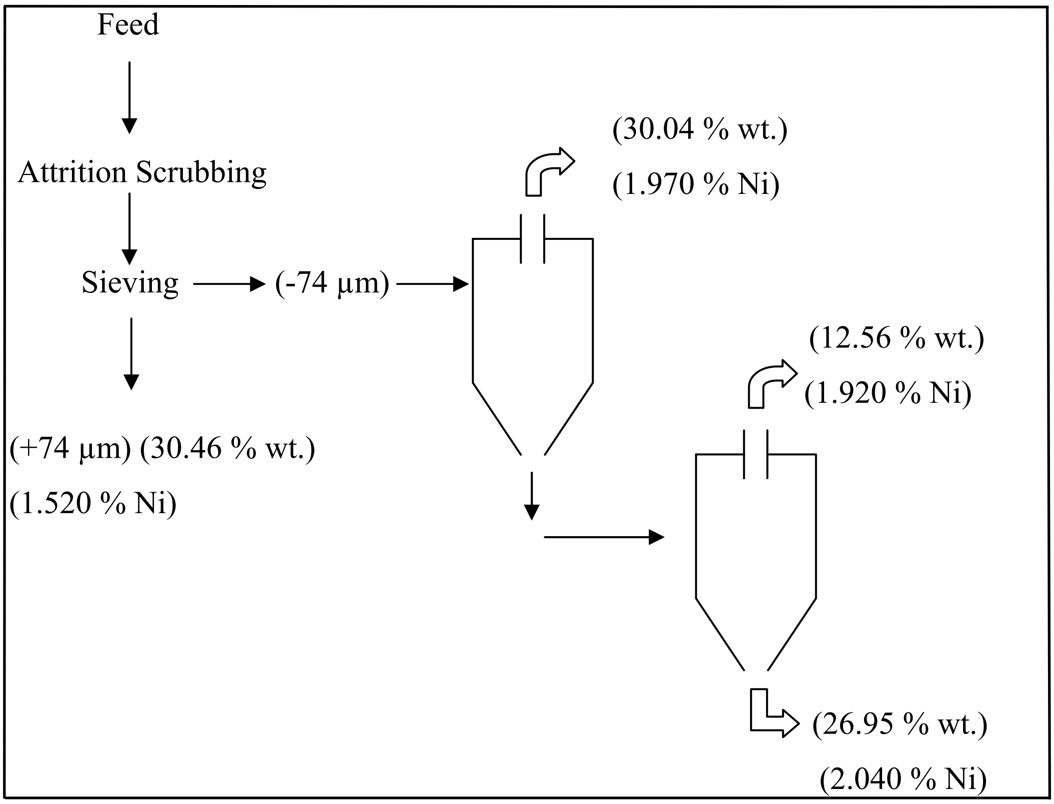


***Figure 23.** Recycloning of Cyclone Underflow after Attrition Scrubbing Experiment Results (Exp. B.2)*

Table 15. *Data and Nickel Distribution Table for Recycloning of Cyclone Underflow after Attrition Scrubbing Experiment (Exp. B.2)*

	Wt (%)	**Ni (%)**	**Wt % x Ni %**	**Ni Distribution (%)**
+74 µm	30.46	1.520	46.30	25.08
1st Cyc. O/F	30.04	1.970	59.18	32.06
2nd Cyc. O/F	12.56	1.920	24.12	13.07
2nd Cyc. U/F	26.95	2.040	54.98	29.79
TOTAL	100	1.846	184.58	100

According to the results above, it is possible to say that hydrocycloning which follows the attrition scrubbing does not produce a preconcentrate with an acceptable nickel grade.

5.1.3 Preconcentration with Permroll

According to the results of magnetic separation experiment, as given in Table 16, nearly 96 % of the ore is obtained as magnetic product. Although most of nickel is distributed in the magnetic fraction of the ore, there is small difference in the grades of magnetic and non-magnetic products. Thus, it is concluded that to obtain a preconcentrate, magnetic separation is not a productive process.

Table 16. *Results of Preconcentration Experiment with Permroll*

Exp. No	**Explanation**	**Weight (%)**	**Ni Grade (%)**	**Wt % x Ni %**	**Ni Distribution (%)**
C.1	Magnetic	95.98	1.862	178.71	96.39
	Non-magnetic	4.02	1.667	6.70	3.61
TOTAL		100.00	1.854	185.41	100.00

5.2 Leaching Processes

5.2.1 Hydrochloric Acid Leaching Experiments at Room Temperature

The nickel extraction values, which were obtained with -1 mm. feed size at room temperature with four different HCl concentrations (1 N, 2 N, 3 N and 4 N) and for five different leaching durations (0.25, 1, 2, 3 and 4 hours) are given in Table 17. During these tests, pulp density was kept constant as 1/30 solid to liquid ratio by volume.

The complete experimental data and calculations are given in Appendix B.

As it is seen from Figure 24 and Figure 25, as leaching duration increases nickel extraction also increases at all HCl concentrations. Furthermore, the increase in the concentration of HCl also results in an increase in nickel extraction for the same leaching period. Maximum nickel extraction value (24.60 %) was obtained with 4 N HCl solution and 4 hours leaching period and this extraction was still very low for an economic process.

Table 17. *Results of HCl Leaching Experiments at Room Temperature*

Exp. No	**HCl Concentration (N)**	**Leaching Duration (hr)**	**Residue Ni %**	**Ni Extraction (%)**
1	1	0.25	1.824	2.40
2	1	1	1.789	4.54
3	1	2	1.741	7.48
4	1	3	1.720	8.79
5	1	4	1.690	10.23
6	2	0.25	1.790	4.41
7	2	1	1.715	7.45
8	2	2	1.687	8.63
9	2	3	1.682	9.98
10	2	4	1.686	11.13
11	3	0.25	1.660	10.54
12	3	1	1.638	11.76
13	3	2	1.619	12.54
14	3	3	1.609	13.88
15	3	4	1.597	14.44
16	4	0.25	1.615	12.57
17	4	1	1.616	14.72
18	4	2	1.579	16.89
19	4	3	1.443	22.71
20	4	4	1.400	24.60
Experimental Conditions				
Feed Size : -1 mm Pulp Density : 1/30 solid to liquid ratio by volume				

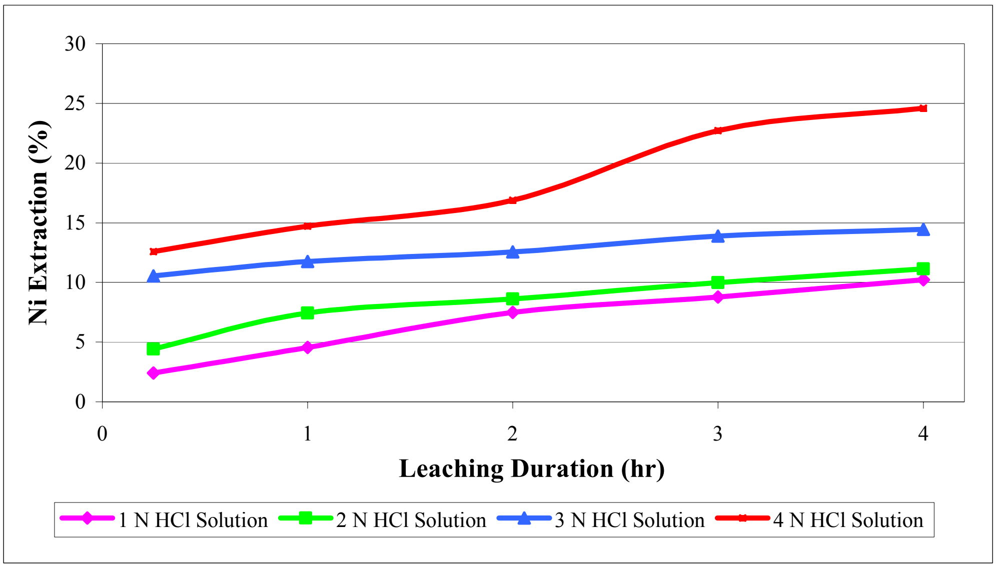


Figure 24. *Extraction of Ni as a Function of Leaching Duration at Different HCl Concentrations*

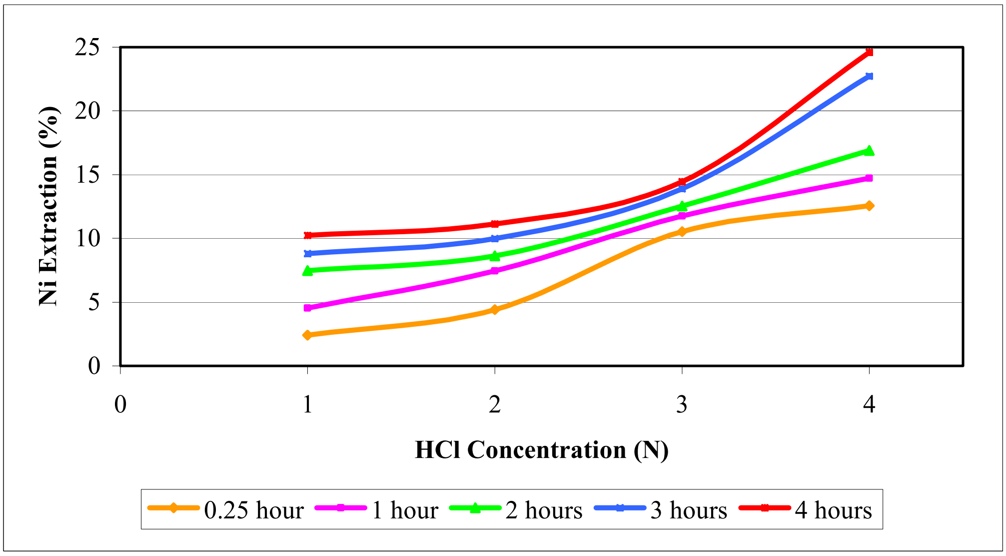


Figure 25. *Extraction of Ni at Different HCl Concentrations at 0.25 hour, 1 hour, 2 hours, 3 hours and 4 hours*

5.2.2 Hydrochloric Acid Leaching Experiments at Room Temperature with Addition of Magnesium Chloride

Harris G.B., et al. and Jansz, J.J.C. state that in concentrated chloride salt solutions the activity of the hydrogen ion, H^+ (or H_3O^+), is significantly increased. In concentrated brines such as magnesium chloride a small amount of acid can achieve much more than a similar amount in more dilute chloride solutions or in sulphate systems [24,30].

According to Harris G.B., et al., provided that the overall chloride concentration is maintained in the leach solution such that $FeCl_4$ does not form to any extent, in which case hydrolysis would be very difficult, then it should be possible to leach out nickel and cobalt from the laterite with minimal co-extraction of iron [24].

Thus, the aim of addition of $MgCl_2$ to the leach solution is to investigate the possibility of increasing the activity of H^+ ion and thus increasing the rate of leaching with minimum extraction of iron.

The nickel extraction values which were obtained with -1 mm. feed size at room temperature with four different HCl concentrations (1 N, 2 N, 3 N and 4 N) and three different total Cl^- levels (150 g/lt, 200 g/lt and 300 g/lt) for 15 min. leaching duration with 1/30 solid to liquid ratio by volume, are given in Table 18.

The complete experimental data and calculations are given in Appendix C.

Table 18. *Results of HCl Leaching Experiments at Room Temperature with Addition of* $MgCl_2$

Exp. No	HCl Concentration (N)	Total Cl^- level (g/lt)	Residue Ni %	Ni Extraction (%)
21	1	150	1.635	2.78
22	2	150	1.464	5.10
23	3	150	1.452	19.30
24	4	150	1.347	22.14
25	1	200	1.496	3.24
26	2	200	1.434	9.79
27	3	200	1.401	20.16
28	4	200	1.273	23.32
29	1	300	1.237	1.02
30	2	300	1.637	1.11
31	3	300	1.256	1.21
32	4	300	1.596	3.44
Experimental Conditions				
Feed Size : -1 mm Pulp Density : 1/30 solid to liquid ratio by volume Leaching Duration : 15 minutes				

As it is seen from Figure 26, as the leach solution concentration increases, nickel extraction increases for the total Cl^- level of 150 g/lt and 200 g/lt. However, it is certain from the same figure that nickel extraction values of the experiments with 300 g/lt total Cl^- level are too low although there is an increase in the extraction with the increase in HCl concentration.

Figure 27 indicates that much more Ni extraction at 200 g/lt Cl^- level was achieved than 150 g/lt Cl^- level for the same HCl concentration. When the total Cl^- level rises to 300 g/lt, nickel extraction shows a sharp decrease.

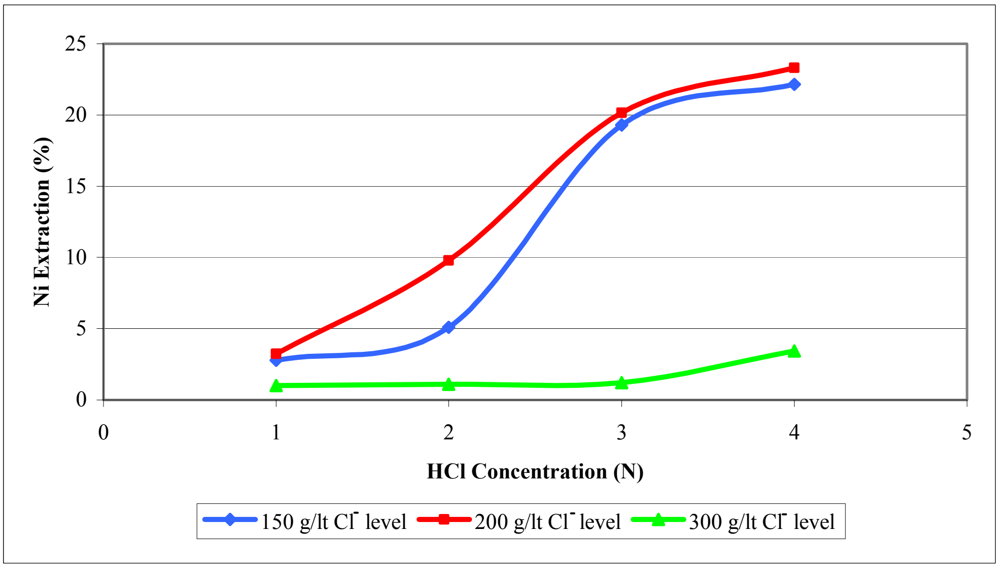


Figure 26. *Extraction of Ni at Different HCl Concentrations with the Addition of $MgCl_2$ to the Solution*

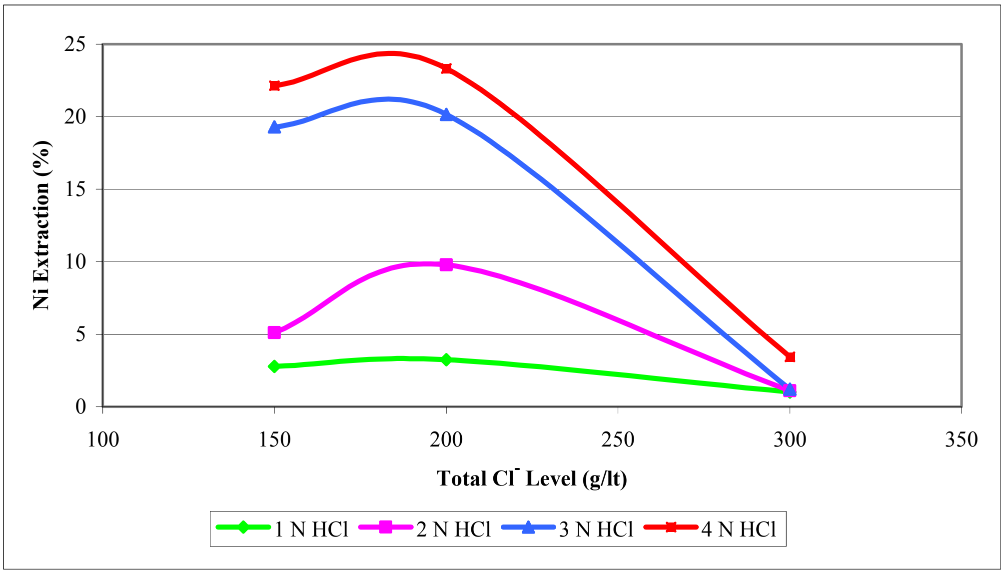


Figure 27. *Extraction of Ni at Different Cl^- Levels in the Leach Solution*

Thus, it is possible to say that increasing the total Cl^- level to 300 g/lt has a negative effect on the extraction of nickel. This may be explained by the statement of Harris G.B., et al. that magnesium chloride has a greater effect on lowering water activity than it does in promoting hydrogen ion activity. Therefore, lower magnesium chloride concentrations have a much greater capacity to retain metals in solution than do higher concentrations [24]. Precipitation of nickel and cobalt hydroxide products with magnesium ion may be the other reason for negative effect of $MgCl_2$ on nickel extraction.

5.2.3 Hydrochloric Acid Leaching Experiments at Elevated Temperatures

Temperature is one of the most important factors affecting the leaching process. In this group of experiments, the temperature, leach solution concentration, particle size, pulp density and leaching time effects on the nickel extraction were examined. The results of these experiments are tabulated in Table 19 and complete experimental data belonging to these experiments and calculations are given in Appendix D.

Table 19. *Results of Leaching Experiments at Elevated Temperatures*

Exp. No	HCl Con. (N)	Solid to liquid ratio by volume	Particle Size (μ)	Temp. (^{o}C)	Leaching Dur. (hr)	Residue Ni %	Ni Extraction (%)
6	2	1/30	-1000	25	0.25	1.790	4.41
33	2	1/30	-500	25	0.25	1.835	6.24
34	2	1/30	-106	25	0.25	1.710	11.30
35	2	1/30	-38	25	0.25	1.705	14.42
36	2	1/30	-1000	50	0.25	1.710	13.01
37	2	1/30	-500	50	0.25	1.640	13.47
38	2	1/30	-106	50	0.25	1.630	16.37
39	2	1/30	-38	50	0.25	1.610	18.73
40	2	1/30	-1000	75	0.25	1.580	21.58
41	2	1/30	-500	75	0.25	1.480	27.00
42	2	1/30	-106	75	0.25	1.440	28.53
43	2	1/30	-38	75	0.25	1.342	32.27
44	2	1/30	-1000	100	0.25	1.460	39.89
45	2	1/30	-500	100	0.25	1.254	45.14
46	2	1/30	-106	100	0.25	1.180	48.58
47	2	1/30	-38	100	0.25	1.070	54.82
48	2	1/30	-38	50	4	1.579	23.59
49	2	1/30	-38	50	8	1.556	26.12
50	2	1/30	-38	75	4	1.201	51.79
51	2	1/30	-38	75	8	1.148	60.74
52	2	1/30	-38	100	4	0.924	70.64
53	2	1/30	-38	100	8	0.736	77.82
54	3	1/30	-38	100	0.25	1.262	59.70
55	3	1/30	-38	100	1	0.965	74.57
56	3	1/30	-38	100	2	0.518	86.86
57	3	1/30	-38	100	3	0.423	90.51
58	3	1/30	-38	100	4	0.395	91.79
59	3	1/30	-38	100	8	0.330	93.87
60	4	1/30	-38	100	4	0.124	97.96
61	1	1/30	-38	100	8	0.920	62.82
62	3	1/12	-38	100	1	1.048	57.99
63	3	1/7	-38	100	1	1.071	52.58
64	3	1/30	-106	100	3	0.573	87.62
65	3	1/30	-500	100	3	0.545	88.25
66	3	1/30	-1000	100	3	0.597	87.26
67	3	1/30	-38	50	3	1.408	33.06
68	3	1/30	-38	75	3	1.020	58.45
69*	3	1/30	-38	100	3	0.234	95.07

* Exp. 69 was carried out at 200 g/lt total Cl^- level in the leach solution.

5.2.3.1 Effect of Temperature

It is well known that temperature is one of the most important factors that affect leaching process. In this study the effect of temperature was investigated for the leaching temperatures of 25 ^{o}C (assumed as room temperature), 50 ^{o}C, 75 ^{o}C and 100 ^{o}C. During the tests, leach solution concentration (2 N), leaching duration (0.25 hour) and pulp density (1/30 solid to liquid ratio by volume) were kept constant. The results of the experiments are given in Table 20 and presented in Figure 28. From these results, it can be concluded that the increase in temperature increases the extraction of nickel for all feed sizes of test sample. 54.82 % nickel extraction was obtained at leaching temperature of 100 ^{o}C for the -38 μm size of feed with 2 N HCl concentration.

Table 20. *Results of Leaching Experiments at Elevated Temperatures with Different Particle Size Fractions*

Experiment No	**Particle Size (μm)**	**Temperature (^{o}C)**	**Residue Ni %**	**Ni Extraction (%)**
6	-1000	25	1.790	4.41
33	-500	25	1.835	6.24
34	-106	25	1.710	11.30
35	-38	25	1.705	14.42
36	-1000	50	1.710	13.01
37	-500	50	1.640	13.47
38	-106	50	1.630	16.37
39	-38	50	1.610	18.73
40	-1000	75	1.580	21.58
41	-500	75	1.480	27.00
42	-106	75	1.440	28.53
43	-38	75	1.342	32.27
44	-1000	100	1.460	39.89
45	-500	100	1.254	45.14
46	-106	100	1.180	48.58
47	-38	100	1.070	54.82
Experimental Conditions				
Leach Solution Concentration : 2 N Pulp Density : 1/30 solid to liquid ratio by volume Leaching Duration : 0.25 hour				

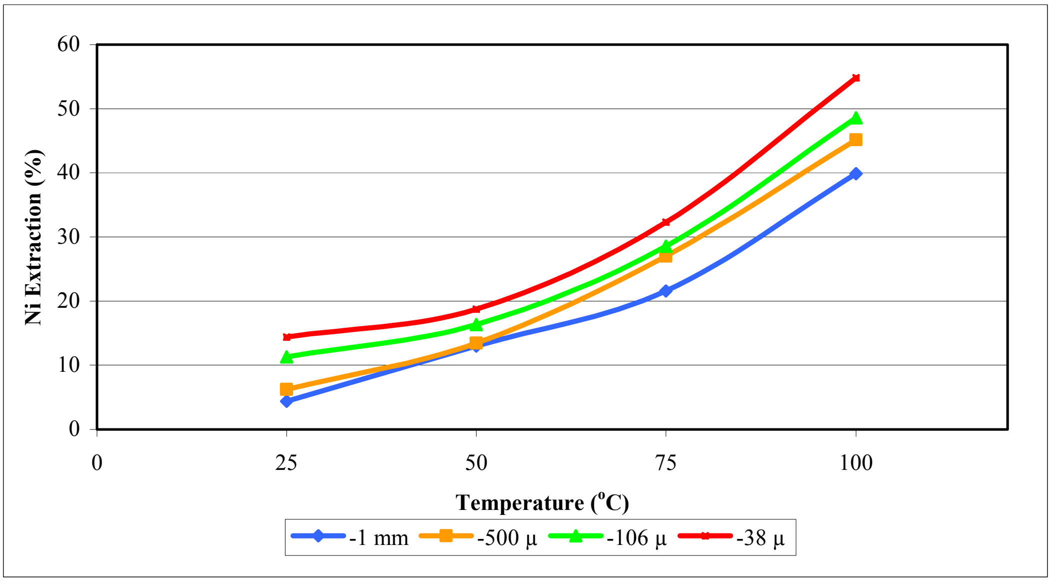


Figure 28. *Extraction of Ni at Different Temperatures at 2 N HCl Concentration*

In Experiments 57, 67 and 68, the effect of temperature on Ni extraction at 3 N HCl concentration was investigated. The results of the experiments are tabulated in Table 21 and presented in Figure 29. From the results, it is verified that the increase in temperature increases the extraction of nickel.

Table 21. *Results of Leaching Experiments at Elevated Temperatures at 3 N HCl Concentration*

Experiment No	Temperature (°C)	Residue Ni %	Ni Extraction (%)
67	50	1.408	33.06
68	75	1.020	58.45
57	100	0.423	90.51
Experimental Conditions			
Leach Solution Concentration : 3 N Particle Size : -38 µm Pulp Density : 1/30 solid to liquid ratio by volume Leaching Duration : 3 hours			

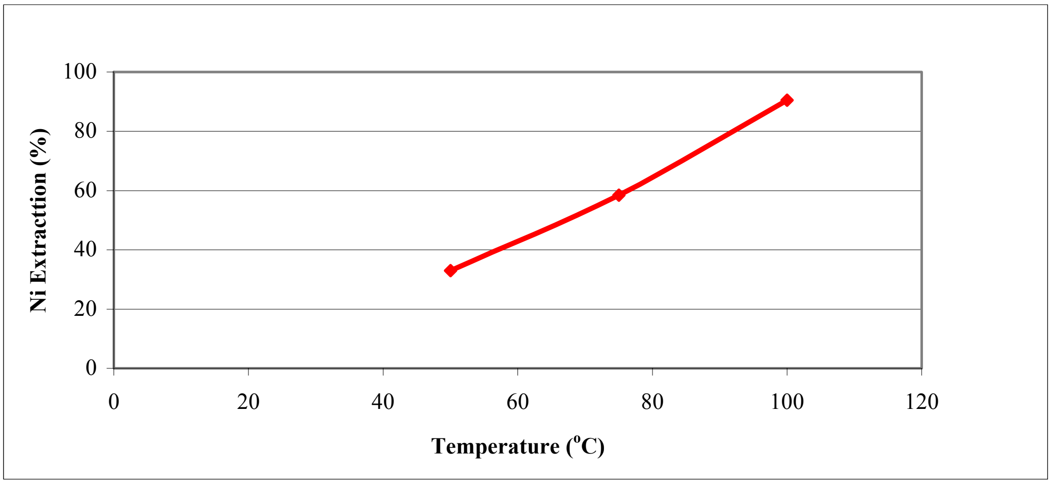


***Figure 29.** Extraction of Ni at Different Temperatures at 3 N HCl Concentration*

5.2.3.2 Effect of Particle Size

The particle size of the ore is an important parameter to be considered in leaching operations due to the fact that it does not only affect the rate of reaction but also causes liberation and exposion of nickel to the hydrochloric acid solution. Therefore, to determine the effect of this parameter on the extraction of nickel, a series of leaching tests were carried out with feed sizes of -500 µm, -106 µm and -38 µm. Finer sizes than 38 µm may not be practical due to the increase in grinding cost and difficulties to be faced during filtration process.

The results are tabulated in Table 20 and plotted in Figure 30 and as it is seen from this figure, nickel extraction increased with decreasing particle size at a constant pulp density (1/30 solid to liquid ratio by volume), leach solution concentration (2 N) and leaching duration (0.25 hour) at all temperatures. Highest extraction of nickel was obtained with the finest feed size of -38 µm. This is an expected result because as the particle size decreased, larger surface area was obtained and much more solid-liquid contact was facilitated.

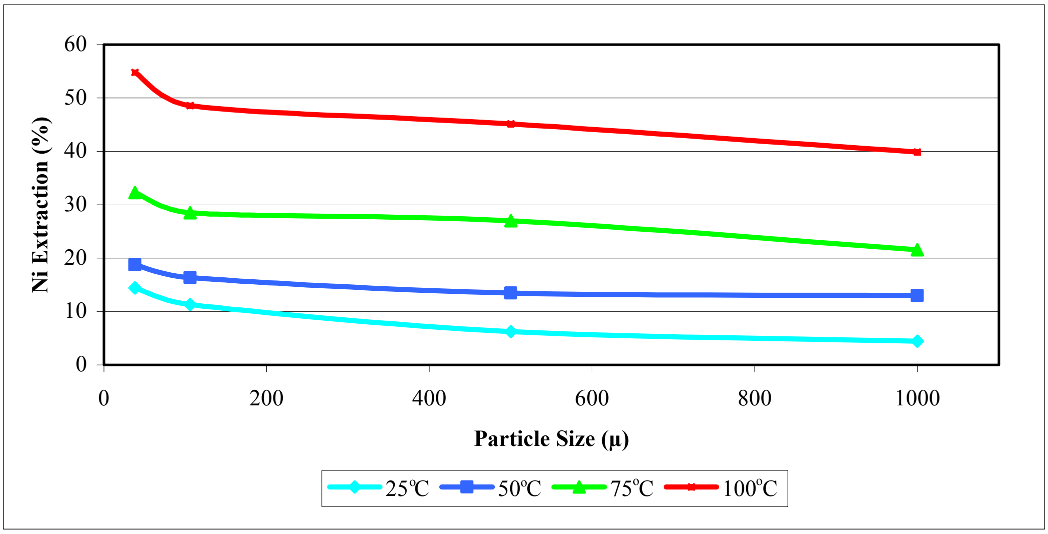


Figure 30. *Extraction of Ni at Different Particle Size Fractions at 2 N HCl Concentration*

In Experiments 57, 64, 65 and 66, the effect of particle size on Ni extraction at 3 N HCl concentration was investigated. The results of the experiments are tabulated in Table 22 and presented in Figure 31. From these results, it can be concluded that at 3 N HCl concentration, the feed size has a small effect on the extraction of nickel. 87.26 % Ni extraction was obtained with the feed size of -1 mm.

Table 22. *Results of Leaching Experiments at Different Particle Size Fractions at 3 N HCl Concentration*

Experiment No	Particle Size (µm)	Residue Ni %	Ni Extraction (%)
57	-38	0.423	90.51
64	-106	0.573	87.62
65	-500	0.545	88.25
66	-1000	0.597	87.26
Experimental Conditions			
Leach Solution Concentration Temperature Pulp Density Leaching Duration	: 3 N : 100 °C : 1/30 solid to liquid ratio by volume : 3 hours		

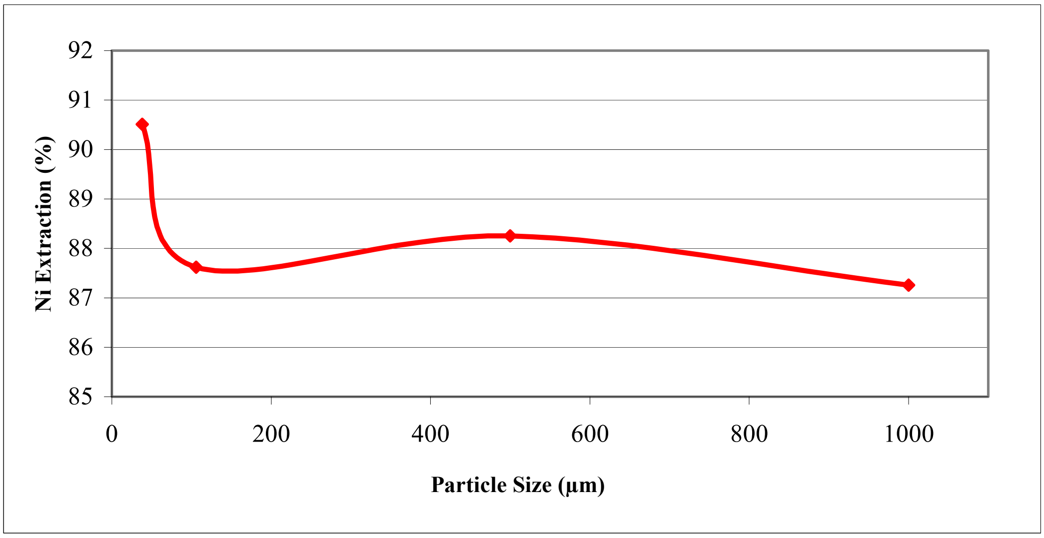


Figure 31. *Extraction of Ni at Different Particle Size Fractions at 3 N HCl Concentration*

5.2.3.3 Effect of Leaching Duration

Leaching duration is another important factor in leaching process. A set of tests was conducted to investigate the effect of leaching duration on nickel extraction. As it is seen from the results tabulated in Table 23 and plotted in Figure 32, nickel extraction increases with increasing leaching duration at all temperatures while other parameters are kept constant (HCl concentration: 2 N, pulp density: 1/30 solid to liquid ratio by volume, particle size: -38 µm).

Table 23. *Results of Leaching Experiments at Different Leaching Durations and Temperatures*

Experiment No	Leaching Duration (hr)	Temperature (^{o}C)	Residue Ni %	Ni Extraction (%)
39	0.25	50	1.610	18.73
43	0.25	75	1.342	32.27
47	0.25	100	1.070	54.82
48	4	50	1.579	23.59
50	4	75	1.201	51.79
52	4	100	0.924	70.64
49	8	50	1.556	26.12
51	8	75	1.148	60.74
53	8	100	0.736	77.82
Experimental Conditions				
Leach Solution Concentration Pulp Density Particle Size	: 2 N : 1/30 solid to liquid ratio by volume : -38 µm			

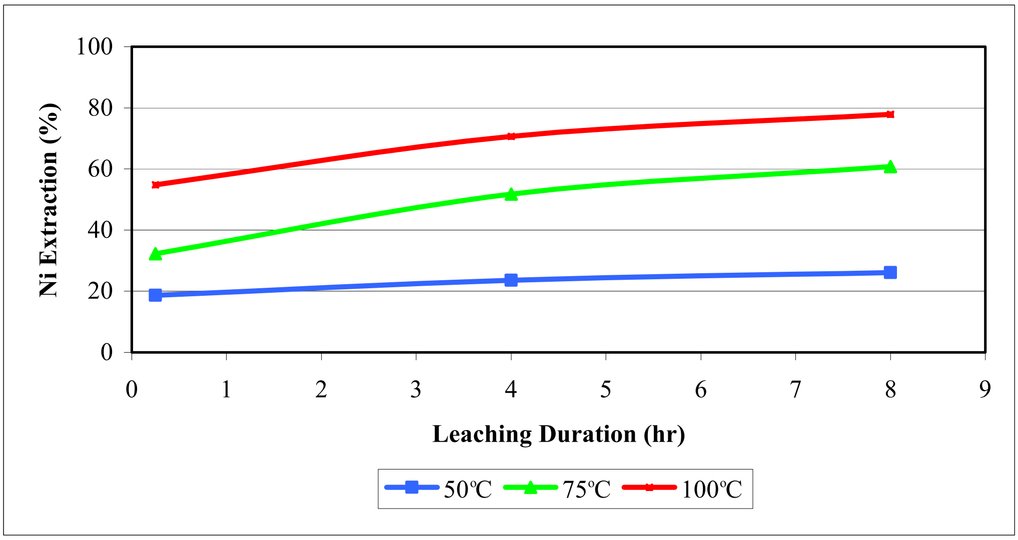


Figure 32. *Extraction of Ni at Different Leaching Durations and Temperatures*

Moreover, according to results given in Table 24 and plotted in Figure 33, similar result was verified that at 3 N HCl concentration nickel extraction increased with increasing leaching duration. A sharp increase in the extraction up to 3 hours and then a slight increase were observed up to 8 hours leaching duration. Thus, it is possible to say that three hours leaching duration will be sufficient for the nickel extraction since 90.51 % of nickel in the ore was extracted in this duration.

***Table 24.** Results of Leaching Experiments at 100 °C and 3 N HCl Concentration at Different Leaching Durations*

Experiment No	Leaching Duration (hr)	Residue Ni %	Ni Extraction (%)
54	0.25	1.262	59.70
55	1	0.965	74.57
56	2	0.518	86.86
57	3	0.423	90.51
58	4	0.395	91.79
59	8	0.330	93.87
Experimental Conditions			
Leach Solution Concentration Pulp Density Particle Size Temperature	: 3 N : 1/30 solid to liquid ratio by volume : -38 µm : 100 °C		

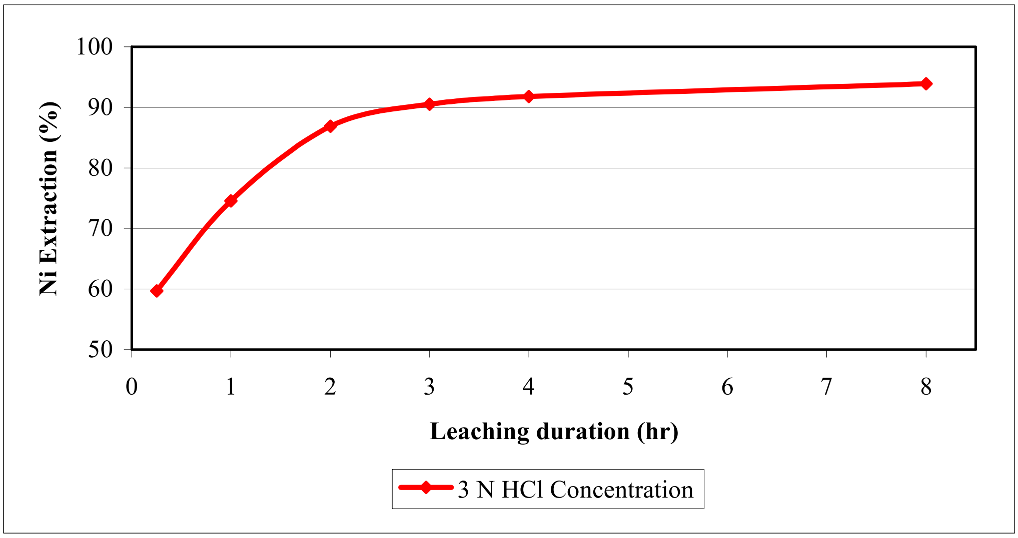


***Figure 33.** Extraction of Ni at Different Leaching Durations*

5.2.3.4 Effect of Leach Solution Concentration

Experiments 52, 58 and 60 were carried out with 2 N, 3 N and 4 N HCl concentrations for 4 hours leaching duration and experiments 61, 53 and 59 were carried out with 1 N, 2 N and 3 N HCl concentrations for 8 hours leaching duration at constant temperature (100 ^{o}C), particle size (-38 µm) and pulp density (1/30 solid to liquid ratio by volume). The variation in nickel extraction percentages after these experiments is tabulated in Table 25 and plotted in Figure 34. It is obvious from the figure that as the concentration of the leach solution increases, Ni extraction increases at both leaching durations of 4 hours and 8 hours.

In Figure 34, it is also seen that the maximum Ni extraction value, 97.96 %, is reached in the experiment 60, which was carried out at 100 ^{o}C temperature with -38 µm particle size feed and 4 N HCl concentration for a leaching duration of 4 hours.

The nearest value to this maximum extraction value was reached in the experiment 59 which was carried out at 100 ^{o}C temperatures with -38 µm particle size and 3 N HCl concentration for a leaching duration of 8 hours. Therefore, it is certain that the concentration of the leach solution has a great importance on decreasing of leaching period, and minimum 3 N HCl concentration is required to obtain economical extraction of nickel at pulp density of 1/30 solid to liquid ratio by volume.

Table 25. *Results of Leaching Experiments at Different leach Solution Concentrations*

Experiment No	**Leaching Duration (hr)**	**Solution Concentration (N)**	**Residue Ni %**	**Ni Extraction (%)**
52	4	2	0.924	70.64
58	4	3	0.395	91.79
60	4	4	0.124	97.96
61	8	1	0.920	62.82
53	8	2	0.736	77.82
59	8	3	0.330	93.87

Experimental Conditions

Temperature : 100 °C
Pulp Density : 1/30 solid to liquid ratio by volume
Particle Size : -38 µm

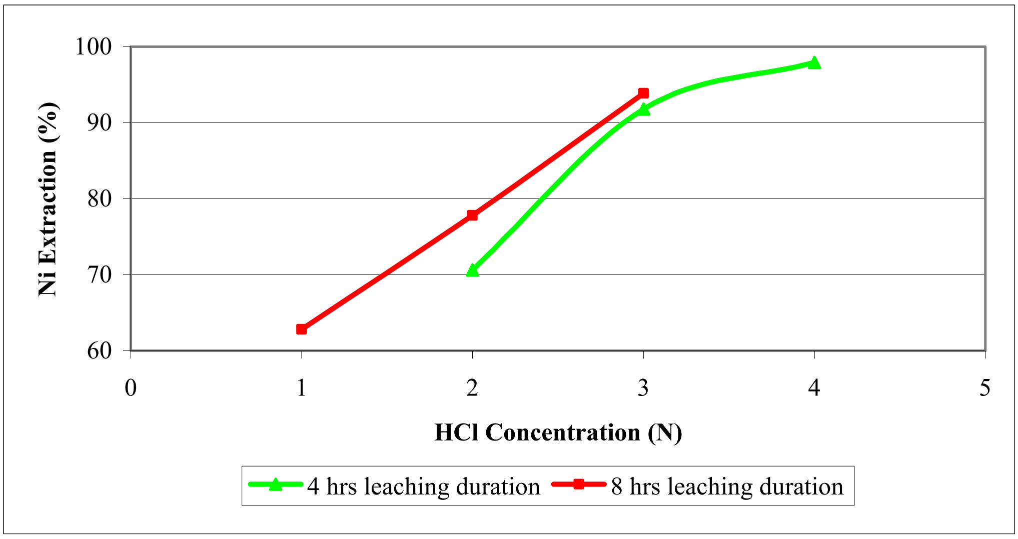


Figure 34. *Extraction of Ni at Different Leach Solution (HCl) Concentrations*

5.2.3.5 Effect of Pulp Density

Pulp density is an important parameter in terms of kinetics of leaching. Therefore, to determine the effect of this parameter on the extraction of nickel, the ore was treated at different pulp densities, which are 1/30, 1/12 and 1/7 solid to liquid ratio by volume. The test results are summarized in Table 26 and plotted in Figure 35. This figure shows that nickel extraction decreases with increasing pulp density. During these tests following parameters were kept constant: leaching temperature: 100 ^oC, HCl solution concentration: 3 N, leaching duration: 1 hour, particle size: -38 μm.

Table 26. *Results of Leaching Experiments at Different Pulp Densities*

Experiment No	Solid to liquid ratio by volume	Residue Ni %	Ni Extraction (%)
55	1/30	0.965	74.57
62	1/12	1.048	57.99
63	1/7	1.071	52.58
Experimental Conditions			
Temperature : 100 ^oC HCl Concentration : 3 N Leaching Duration : 1 hour Particle Size : -38 μm			

It was found that leaching with 1/30 solid to liquid ratio by volume was beneficial and high nickel extraction was obtainable. The decrease in the extraction of nickel when operating at higher pulp densities was due to inadequate acid amount for mineral surfaces.

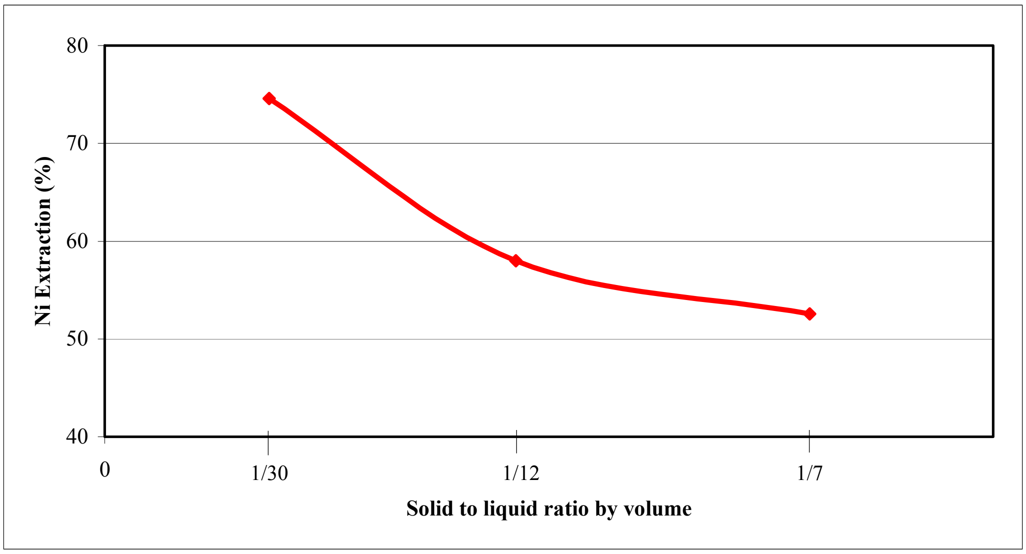


Figure 35. *Extraction of Ni at Different Pulp Densities*

5.2.3.6 Effect of $MgCl_2$ Addition

The effect of $MgCl_2$ addition to the leach solution at elevated temperatures was investigated at the following nickel extraction conditions (HCl concentration: 3 N, leaching duration: 3 hours, leaching temperature: 100 ^{o}C, pulp density: 1/30 solid to liquid ratio by volume, particle size: -38 μm) by increasing the total Cl^- level in the solution to 200 g/lt. According to the results of Experiment 69, tabulated in Table 27, $MgCl_2$ addition increases the Ni extraction to 95.07 % and Co extraction to 90.02 %; however, it increased the Fe extraction to 92.54 %, too.

Table 27. *Results of $MgCl_2$ Addition to the Leach Solution Experiment*

Exp. No	Total Cl^- Level in Solution (g/lt)	Residue Ni %	Ni Extraction (%)	Co Extraction (%)	Fe Extraction (%)
69	200	0.234	95.07	90.02	92.54

Table 28 shows the extraction of Co and Fe metals at the optimum nickel extraction conditions (HCl concentration: 3 N, leaching duration: 3 hours, leaching temperature: 100 ^{o}C, pulp density: 1/30 solid to liquid ratio by volume, particle size: 100 % -1 mm. with a Ni extraction of 87.26 %).

Table 28. *Co and Fe Extractions at the Optimum Nickel Extraction Conditions*

Metals	**Extraction (%)**
Co	81.85
Fe	79.72

As it is seen in Table 28, at the optimum nickel extraction conditions 81.85 % of Co and 79.72 % of Fe were also extracted.

This high extraction of Fe is a disadvantage, because removal of iron from chloride leach solutions does not constitute yet a well established technology. The common methods in dealing with the iron in solution have been either precipitation with a base or solvent extraction [31]. According to Gibson R.W. and Rice N.M., the separation and concentration of the nickel in the HCl leach liquor from magnesium and/or iron can be achieved using solvent extraction with Cyanex 301, Cyanex 302 or Versatic 10 which displayed the best extraction characteristics. Cyanex 301 showed excellent selectivity for Ni but was very difficult to strip except with high concentrations of acid. Cyanex 302 seemed suitable but co-extraction of magnesium was appreciable [32].

During the leaching process, a certain amount of Arsine gas (AsH_3) stripping occurs. At optimized nickel extraction conditions, 24.42 % of Arsenic was found in the leach residue, that is some of 75.58 % was leached and some was stripped as gas into the atmosphere. Arsenic and many of its compounds are especially potent poisons. Elemental arsenic and arsenic compounds are classified as toxic and dangerous for the human health and environment. Therefore, the required precautions must be taken against this problem.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

In this thesis work, it is aimed to extract the nickel metal in the lateritic ore from Gördes-Manisa region by hydrochloric acid leaching process. In the light of experimental test results of the ore, following coclusions can be drawn.

- Applying attrition scrubbing to the lateritic ore changes only the particle size distribution of the ore and the distribution of nickel through the different particle size. Preconcentration of ore was not possible with attrition scrubbing alone and attrition scrubbing followed by cycloning.

- Due to slight differences in nickel grade of magnetic and non-magnetic fractions of the ore, preconcentration of ore with permroll was not satisfactory.

- The results of HCl leaching tests at room temperature were not satisfactory in terms of extraction of nickel metal. Highest nickel extraction at room temperature was 24.60 % for the -1 mm. feed size ore with 4 N HCl solution in 4 hours leaching duration.

- Addition of $MgCl_2$ did not show any significant increase in nickel extraction at room temperature.

- Leaching tests at elevated temperatures resulted in higher nickel extractions.

- Leaching parameters such as temperature, HCl concentration, feed size, pulp density and leaching duration were observed to be critical.

- The optimum leaching parameters for the extraction of nickel from Gördes lateritic ore were 3 N HCl concentration, 3 hours of leaching duration, 100 ^{o}C of leaching temperature, 1/30 solid to liquid ratio by volume pulp density and -1 mm. of feed size.

- At the optimum leaching conditions, 87.26 % nickel extraction was maintained whereas Co and Fe extractions were 81.85 % and 79.72 %, respectively.

- Experimental design techniques are recommended for similar studies in order to decrease the number of experiments.

REFERENCES

[1] Ashok, D. Dalvi, et. al., ***“The Past and the Future of Nickel Laterites”***, PDAC 2004 International Convention, Trade Show & Investors Exchange, March 2004

[2] U. S. Geological Survey, Nickel Statistics and Information, ***“http://minerals.usgs.gov/minerals/pubs/commodity/nickel/index.html”***, retrieved May 29, 2006

[3] Blainey, G., et. al., ***“Nickel”***, Mineral Information Center, The Chamber of Minerals and Energy of Western Australia Inc., 2003

[4] Bailey, J. E., et. al., ***“Ullmann’s Encyclopedia of Industrial Chemistry”***, Wiley-VCH Verlag GmbH & Co., Germany, 2002

[5] Nicholls, D., ***“The Chemistry of Iron, Cobalt and Nickel”***, Pergamon Texts in Inorganic Chemistry, Pergamon Press, Vol. 24, Oxford, 1975

[6] Ogura, Y., Doi, I., ***“Proceedings of International Symposium on Laterite”***, Int. J. Miner. Process., Vol.19, p. 1-4, 1987

[7] “***Nickel Report 2005”***, U.S. Geological Survey, Mineral Commodity Summaries, 2006

[8] Ellias, M., “***Global Lateritic Nickel Resources”***, New Caledonian Nickel Conference, 2001

[9] T.C. Başbakanlık Devlet Planlama Teşkilatı, ***“Dokuzuncu Kalkınma Planı (2007-2013)”***, Madencilik Özel İhtisas Komisyonu, Metal Madenler Alt Komisyonu, Nikel-Kobalt, 2006

[10] Reimann, M.A., et. al., ***"Nickel Laterites, An Increasingly Economic Resource"***, Geostar Metals Inc., December 1999

[11] Boldt, J.R., ***"The Winning of Nickel: Its Geology, Mining, and Extractive Metallurgy"***, pp. 6-8, 14, 391,392, Longmans Canada Limited, Toronto, 1967

[12] Roorda, H.J., Queneau, P.E., ***"Recovery of Nickel and Cobalt from Ilmenites by Aqueous Chlorination in Sea Water"***, Trans. Inst. Min. Metal. (Sec.C: Mineral Process. Extr. Metall), Vol. 82, pp. 79-87, 1973

[13] Toguri, J.M., ***"A Review on the Methods of Treating Nickel Bearing Pyrrhotite with Special Reference to the Sudbury Area Pyrrhotite"***, Canadian Metallurgical Quarterly, Vol. 14, No.4, pp. 323-338, 1975

[14] Kerr, A.N., Kipkie, W.B., ***"Recent Developments at Inco's Copper Cliff Milling Complex"***, TMS-AIME Extractive and Process Metalurgy Meeting, San Diego, CA, 1985

[15] Hoffman, R.R., Kaiura, G.H., ***"Smelting Process Update at Falconbridge Limited-Sudbury Operations"***, 114th TMS-AIME Annual Meeting, New York, 1985

[16] Elliot, B.J., Robinson, K., Stewart B.V. in Sohn H.Y., George, D.E., Zunkel, A.D. (eds), ***"Advances in Sulfide Smelting"***, Proceedings 1983 International Sulfide Smelting Symposium & 1983 Extractive & Process Metallurgy Meeting of TMS of AIME, Vol. 2. Technology and Practice, p.875, San Francisco, California, November 6-9, 1983

[17] Brand, N.W., et. al., ***"Nickel Laterites: Classification and Features"***, AGSO Journal of Australian Geology and Geophysics, Vol. 17, No. 4, pp. 81-88, 1998

[18] Yüksel, M., ***"Study on the Nickel Recovery from Çaldağ Laterites"***, Master of Science Thesis, Middle East Technical University, October 1985

[19] Johnson, J.A., et. al., ***"Optimisation of Nickel Extraction from Laterite Ores by High Pressure Acid Leaching with Addition of Sodium Sulphate"***, *Minerals Engineering*, Vol.18, No.13-14, pp. 1297-1303, 2005

[20] Kar, B.B., et. al. ***"Design of Experiments to Study the Extraction of Nickel from Lateritic Ore by Sulphatization using Sulphuric Acid"***, Hydrometallurgy, Vol. 56, No.3, pp. 387-394, India, 2000

[21] Georgiou, D., Papangelakis, V.G., ***"Sulphuric Acid Pressure Leaching of a Limonitic Laterite: Chemistry and Kinetics"***, Hydrometallurgy, Vol. 49(1-2), pp. 23-46, 1998

[22] European Nickel PLC, Çaldağ Project, ***"http://www.enickel.co.uk/Caldag-Project"***, retrieved May 24, 2005

[23] Malachite Process Consulting Pty. Ltd., ***"Nickel Laterites in Turkey"***, Heap Leaching Scoping Study, April 2004

[24] Harris, G.B., et. al., ***"The Jaguar Nickel Inc. Sechol Laterite Project Atmospheric Chloride Leach Process"***, International Laterite Nickel Symposium, Charlotte, N.C., pp. 219-241, March 2004

[25] Harris, B., Magee, J., ***"Atmospheric Chloride Leaching: The Way Forward For Nickel Laterites"***, Hydrometallurgy 2003; Proceedings of the 5th International Symposium, pp. 501-516, Chesbar Resources Inc., Toronto, Canada, 2003

[26] Meta Mining Industry Energy Tourism and Trading Ltd., Local Geology, ***"http://www.metamaden.com/index_en.htm"***, retrieved June 20, 2006

[27] Australian Government-Department of the Environment and Heritage, HCl, ***"http://www.npi.gov.au/database/substance-info/profiles/48.html"***, retrieved July 14, 2006

[28] Wikipedia-The Free Encyclopedia, Hydrochloric Acid, ***"http://en.wikipedia.org/wiki/Hydrochloric_acid"***, retrieved July 17, 2006

[29] Ezine Articles, Magnesium Chloride, ***"http://www.ezinearticles.com/?Magnesium-Chloride&id=206628"***, retrieved July 18, 2006

[30] Jansz, J.J.C., ***"Estimation of Ionic Activities in Chloride Systems at Ambient and Elevated Temperatures"***, Hydrometallurgy, Vol.11, Issue 1, pp.13-31, 1983

[31] Harris, B., et. al., ***"A New Approach to the High Concentration Chloride Leaching of Nickel Laterites"***, Alta Ni/Co 11 Perth, WA, May 15-17, 2006

[32] Gibson, R.W., Rice, N.M., ***"A Hydrochloric Acid Process for Nickeliferrous Laterites"***, Nickel-Cobalt 97 International Symposium, Technical Program of the 27th Annual Hydrometallurgical Meeting of CIM held in conjunction with the 36th Annual Conference of Metallurgists Laurentian University, Paper 19.5, Sudbury, Ontario, Canada, August 17-20, 1997

APPENDIX A

ATTRITION SCRUBBING EXPERIMENTS DATA AND RESULTS

The complete data of attrition scrubbing experiments are given in Table 29.

Table 29. *Attrition Scrubbing Experiments Data and Results*

Exp. No	**Solid by weight (%)**	**Mixing time (min)**	**Particle Size (µm)**	**Weight %**	**Ni %**
A.1	20	15	+74	38.98	1.490
			-74	61.02	2.060
A.2	20	30	+74	36.45	1.410
			-74	63.55	2.070
A.3	20	45	+74	31.65	1.480
			-74	68.35	2.030
A.4	20	60	+74	29.81	1.500
			-74	70.19	2.000
A.5	50	15	+74	37.34	1.750
			-74	62.66	1.910
A.6	50	30	+74	34.63	1.550
			-74	65.37	2.040
A.7	50	45	+74	32.25	1.580
			-74	67.75	1.950
A.8	50	60	+74	30.31	1.500
			-74	69.69	2.020

The nickel distribution calculations according to chemical analysis results given in Table 29 are tabulated in Table 30 to Table 37.

Table 30. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 20 % Solid by Weight of Pulp for 15 minutes*

	Wt (%)	**Ni (%)**	**Wt % x Ni %**	**Ni Distribution (%)**
+74 µm	38.98	1.490	58.07	31.60
-74 µm	61.02	2.060	125.71	68.40
TOTAL	100	1.838	183.78	100

Table 31. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 20 % Solid by Weight of Pulp for 30 minutes*

	Wt (%)	**Ni (%)**	**Wt % x Ni %**	**Ni Distribution (%)**
+74 µm	36.45	1.410	51.39	28.09
-74 µm	63.55	2.070	131.56	71.91
TOTAL	100	1.830	182.95	100

Table 32. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 20 % Solid by Weight of Pulp for 45 minutes*

	Wt (%)	**Ni (%)**	**Wt % x Ni %**	**Ni Distribution (%)**
+74 µm	31.65	1.480	46.85	25.24
-74 µm	68.35	2.030	138.74	74.76
TOTAL	100	1.856	185.59	100

Table 33. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 20 % Solid by Weight of Pulp for 60 minutes*

	Wt (%)	**Ni (%)**	**Wt % x Ni %**	**Ni Distribution (%)**
+74 µm	29.81	1.500	44.71	24.15
-74 µm	70.19	2.000	140.39	75.85
TOTAL	100	1.851	185.10	100

Table 34. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 50 % Solid by Weight of Pulp for 15 minutes*

	Wt (%)	Ni (%)	Wt % x Ni %	Ni Distribution (%)
+74 µm	37.34	1.750	65.35	32.32
-74 µm	62.66	1.910	119.68	64.68
TOTAL	100	1.850	185.03	100

Table 35. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 50 % Solid by Weight of Pulp for 30 minutes*

	Wt (%)	Ni (%)	Wt % x Ni %	Ni Distribution (%)
+74 µm	34.63	1.550	53.67	28.70
-74 µm	65.37	2.040	133.36	71.30
TOTAL	100	1.871	187.07	100

Table 36. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 50 % Solid by Weight of Pulp for 45 minutes*

	Wt (%)	Ni (%)	Wt % x Ni %	Ni Distribution (%)
+74 µm	32.25	1.580	50.95	27.83
-74 µm	67.75	1.950	132.11	72.17
TOTAL	100	1.831	183.07	100

Table 37. *Data and Nickel Distributions of Attrition Scrubbing Experiment at 50 % Solid by Weight of Pulp for 60 minutes*

	Wt (%)	Ni (%)	Wt % x Ni %	Ni Distribution (%)
+74 µm	30.31	1.500	45.47	24.41
-74 µm	69.69	2.020	140.77	75.59
TOTAL	100	1.862	186.24	100

APPENDIX B

HYDROCHLORIC ACID LEACHING EXPERIMENTS AT ROOM TEMPERATURE DATA AND RESULTS

The data and nickel extraction results of HCl leaching experiments at room temperature are given in Table 38.

Table 38. *Data and Nickel Extraction Results of HCl Leaching Experiments*

Exp. No	**HCl Conc. (N)**	**Leaching Duration (hr)**	**Feed Wt. (g)**	**Residue Wt. (g)**	**Ni %**	**Ni Extraction (%)**
1	1	0.25	50	49.23	1.824	2.40
2	1	1	50	49.09	1.789	4.54
3	1	2	50	48.89	1.741	7.48
4	1	3	50	48.78	1.720	8.79
5	1	4	50	48.88	1.690	10.23
6	2	0.25	50	49.14	1.790	4.41
7	2	1	50	49.65	1.715	7.45
8	2	2	50	49.82	1.687	8.63
9	2	3	50	49.24	1.682	9.98
10	2	4	50	48.49	1.686	11.13
11	3	0.25	50	49.58	1.660	10.54
12	3	1	50	49.56	1.638	11.76
13	3	2	50	49.71	1.619	12.54
14	3	3	50	49.24	1.609	13.88
15	3	4	50	49.29	1.597	14.44
16	4	0.25	50	49.79	1.615	12.57
17	4	1	50	48.55	1.616	14.72
18	4	2	50	48.41	1.579	16.89
19	4	3	50	49.28	1.443	22.71
20	4	4	50	49.55	1.400	24.60

Nickel Extraction Calculations

The formula that is used for the calculation of nickel extraction values is given below:

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{\text{Ni \% in residue} \times \text{Weight of residue}}{\text{Ni \% in feed} \times \text{Weight of feed}} \times 100\right]$$

Experiment 1

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.824 \times 49.23}{1.840 \times 50.00} \times 100\right] = 2.40\,\%$$

Experiment 2

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.789 \times 49.09}{1.840 \times 50.00} \times 100\right] = 4.54\,\%$$

Experiment 3

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.741 \times 48.89}{1.840 \times 50.00} \times 100\right] = 7.48\,\%$$

Experiment 4

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.720 \times 48.78}{1.840 \times 50.00} \times 100\right] = 8.79\,\%$$

Experiment 5

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.690 \times 48.88}{1.840 \times 50.00} \times 100\right] = 10.23\,\%$$

Experiment 6

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.790 \times 49.14}{1.840 \times 50.00} \times 100\right] = 4.41\,\%$$

Experiment 7

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.715 \times 49.65}{1.840 \times 50.00} \times 100\right] = 7.45\,\%$$

Experiment 8

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.687 \times 49.82}{1.840 \times 50.00} \times 100\right] = 8.63\,\%$$

Experiment 9

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.682 \times 49.24}{1.840 \times 50.00} \times 100\right] = 9.98\,\%$$

Experiment 10

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.686 \times 48.49}{1.840 \times 50.00} \times 100\right] = 11.13\,\%$$

Experiment 11

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.660 \times 49.58}{1.840 \times 50.00} \times 100\right] = 10.54\,\%$$

Experiment 12

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.638 \times 49.56}{1.840 \times 50.00} \times 100\right] = 11.76\,\%$$

Experiment 13

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.619 \times 49.71}{1.840 \times 50.00} \times 100\right] = 12.54\,\%$$

Experiment 14

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.609 \times 49.24}{1.840 \times 50.00} \times 100\right] = 13.88\ \%$$

Experiment 15

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.597 \times 49.29}{1.840 \times 50.00} \times 100\right] = 14.44\ \%$$

Experiment 16

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.615 \times 49.79}{1.840 \times 50.00} \times 100\right] = 12.57\ \%$$

Experiment 17

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.616 \times 48.55}{1.840 \times 50.00} \times 100\right] = 14.72\ \%$$

Experiment 18

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.579 \times 48.41}{1.840 \times 50.00} \times 100\right] = 16.89\ \%$$

Experiment 19

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.443 \times 49.28}{1.840 \times 50.00} \times 100\right] = 22.71\ \%$$

Experiment 20

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.400 \times 49.55}{1.840 \times 50.00} \times 100\right] = 24.60\ \%$$

APPENDIX C

HYDROCHLORIC ACID LEACHING EXPERIMENTS AT ROOM TEMPERATURE WITH THE ADDITION OF MAGNESIUM CHLORIDE DATA AND RESULTS

The complete data of HCl leaching experiments at room temperature for 15 minutes leaching duration with the addition of $MgCl_2$ are given in Table 39.

Table 39. *Data and Nickel Extraction Results of HCl Leaching Experiments at Room Temperature with the Addition of* $MgCl_2.6H_2O$

Exp. No	**HCl Conc. (N)**	**Total Cl^- level (g/lt)**	**Feed Wt. (g)**	**Residue Wt. (g)**	**$MgCl_2.6H_2O$ Added (g)**	**Residue Ni %**	**Ni Extraction (%)**
21	1	150	50	54.69	163.93	1.635	2.78
22	2	150	50	59.62	113.10	1.464	5.10
23	3	150	50	51.13	62.28	1.452	19.30
24	4	150	50	53.18	11.45	1.347	22.14
25	1	200	50	59.50	235.51	1.496	3.24
26	2	200	50	57.86	184.69	1.434	9.79
27	3	200	50	52.43	133.86	1.401	20.16
28	4	200	50	55.42	83.04	1.273	23.32
29	1	300	50	73.64	378.68	1.237	1.02
30	2	300	50	55.59	327.86	1.637	1.11
31	3	300	50	72.35	277.03	1.256	1.21
32	4	300	50	55.66	226.21	1.596	3.44

Nickel Extraction Calculations

Experiment 21

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.635 \times 54.69}{1.840 \times 50.00} \times 100\right] = 2.78\,\%$$

Experiment 22

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.464 \times 59.62}{1.840 \times 50.00} \times 100\right] = 5.10\,\%$$

Experiment 23

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.452 \times 51.13}{1.840 \times 50.00} \times 100\right] = 19.30\,\%$$

Experiment 24

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.347 \times 53.18}{1.840 \times 50.00} \times 100\right] = 22.14\,\%$$

Experiment 25

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.496 \times 59.50}{1.840 \times 50.00} \times 100\right] = 3.24\,\%$$

Experiment 26

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.434 \times 57.86}{1.840 \times 50.00} \times 100\right] = 9.79\,\%$$

Experiment 27

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.401 \times 52.43}{1.840 \times 50.00} \times 100\right] = 20.16\,\%$$

Experiment 28

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.273 \times 55.42}{1.840 \times 50.00} \times 100\right] = 23.32\,\%$$

Experiment 29

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.237 \times 73.64}{1.840 \times 50.00} \times 100\right] = 1.02\,\%$$

Experiment 30

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.637 \times 55.59}{1.840 \times 50.00} \times 100\right] = 1.11\,\%$$

Experiment 31

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.256 \times 72.35}{1.840 \times 50.00} \times 100\right] = 1.21\,\%$$

Experiment 32

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.596 \times 55.66}{1.840 \times 50.00} \times 100\right] = 3.44\,\%$$

APPENDIX D

HYDROCHLORIC ACID LEACHING EXPERIMENTS AT ELEVATED TEMPERATURES DATA AND RESULTS

The data of leaching experiments at elevated temperatures is given in Table 40.

Table 40. *Data and Results of Leaching Experiments at Elevated Temperatures*

Exp. No	**HCl Con. (N)**	**Sol. to liq. ratio by vol.**	**Particle Size (µm)**	**Temp. (^{o}C)**	**Leaching Dur. (hr)**	**Feed wt. (g)**	**Residue wt. (g)**	**Residue Ni %**	**Ni Ext. (%)**
33	2	1/30	-500	25	0.25	25	23.50	1.835	6.24
34	2	1/30	-106	25	0.25	25	23.86	1.710	11.30
35	2	1/30	-38	25	0.25	25	23.09	1.705	14.42
36	2	1/30	-1000	50	0.25	25	23.40	1.710	13.01
37	2	1/30	-500	50	0.25	25	24.27	1.640	13.47
38	2	1/30	-106	50	0.25	25	23.60	1.630	16.37
39	2	1/30	-38	50	0.25	25	23.22	1.610	18.73
40	2	1/30	-1000	75	0.25	25	22.83	1.580	21.58
41	2	1/30	-500	75	0.25	25	22.69	1.480	27.00
42	2	1/30	-106	75	0.25	25	22.83	1.440	28.53
43	2	1/30	-38	75	0.25	25	23.21	1.342	32.27
44	2	1/30	-1000	100	0.25	25	18.94	1.460	39.89
45	2	1/30	-500	100	0.25	25	20.12	1.254	45.14
46	2	1/30	-106	100	0.25	25	20.05	1.180	48.58
47	2	1/30	-38	100	0.25	25	19.42	1.070	54.82
48	2	1/30	-38	50	4	25	22.26	1.579	23.59
49	2	1/30	-38	50	8	25	21.84	1.556	26.12
50	2	1/30	-38	75	4	25	18.46	1.201	51.79
51	2	1/30	-38	75	8	25	15.73	1.148	60.74
52	2	1/30	-38	100	4	25	14.61	0.924	70.64
53	2	1/30	-38	100	8	25	13.86	0.736	77.82
54	3	1/30	-38	100	0.25	25	14.69	1.262	59.70
55	3	1/30	-38	100	1	25	12.12	0.965	74.57
56	3	1/30	-38	100	2	25	11.67	0.518	86.86
57	3	1/30	-38	100	3	25	10.32	0.423	90.51
58	3	1/30	-38	100	4	25	9.56	0.395	91.79
59	3	1/30	-38	100	8	25	8.54	0.330	93.87
60	4	1/30	-38	100	4	25	7.58	0.124	97.96
61	1	1/30	-38	100	8	25	18.59	0.920	62.82
62	3	1/12	-38	100	1	25	18.44	1.048	57.99
63	3	1/7	-38	100	1	40	32.59	1.071	52.58
64	3	1/30	-106	100	3	25	9.94	0.573	87.62
65	3	1/30	-500	100	3	25	9.92	0.545	88.25
66	3	1/30	-1000	100	3	25	9.82	0.597	87.26
67	3	1/30	-38	50	3	25	21.87	1.408	33.06
68	3	1/30	-38	75	3	25	18.74	1.020	58.45
69	3	1/30	-38	100	3	25	9.69	0.234	95.07

Extraction Calculations

Experiment 33

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.835 \times 23.50}{1.840 \times 25.00} \times 100\right] = 6.24\,\%$$

Experiment 34

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.710 \times 23.86}{1.840 \times 25.00} \times 100\right] = 11.30\,\%$$

Experiment 35

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.705 \times 23.09}{1.840 \times 25.00} \times 100\right] = 14.42\,\%$$

Experiment 36

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.710 \times 23.40}{1.840 \times 25.00} \times 100\right] = 13.01\,\%$$

Experiment 37

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.640 \times 24.27}{1.840 \times 25.00} \times 100\right] = 13.47\,\%$$

Experiment 38

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.630 \times 23.60}{1.840 \times 25.00} \times 100\right] = 16.37\,\%$$

Experiment 39

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.610 \times 23.22}{1.840 \times 25.00} \times 100\right] = 18.73\,\%$$

Experiment 40

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.580 \times 22.83}{1.840 \times 25.00} \times 100\right] = 21.58\,\%$$

Experiment 41

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.480 \times 22.69}{1.840 \times 25.00} \times 100\right] = 27.00\,\%$$

Experiment 42

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.440 \times 22.83}{1.840 \times 25.00} \times 100\right] = 28.53\,\%$$

Experiment 43

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.342 \times 23.21}{1.840 \times 25.00} \times 100\right] = 32.27\,\%$$

Experiment 44

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.460 \times 18.94}{1.840 \times 25.00} \times 100\right] = 39.89\,\%$$

Experiment 45

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.254 \times 20.12}{1.840 \times 25.00} \times 100\right] = 45.14\,\%$$

Experiment 46

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.180 \times 20.05}{1.840 \times 25.00} \times 100\right] = 48.58\,\%$$

Experiment 47

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.070 \times 19.42}{1.840 \times 25.00} \times 100\right] = 54.82\,\%$$

Experiment 48

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.579 \times 22.26}{1.840 \times 25.00} \times 100\right] = 23.59\,\%$$

Experiment 49

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.556 \times 21.84}{1.840 \times 25.00} \times 100\right] = 26.12\,\%$$

Experiment 50

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.201 \times 18.46}{1.840 \times 25.00} \times 100\right] = 51.79\,\%$$

Experiment 51

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.148 \times 15.73}{1.840 \times 25.00} \times 100\right] = 60.74\,\%$$

Experiment 52

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.924 \times 14.61}{1.840 \times 25.00} \times 100\right] = 70.64\,\%$$

Experiment 53

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.736 \times 13.86}{1.840 \times 25.00} \times 100\right] = 77.82\,\%$$

Experiment 54

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.262 \times 14.69}{1.840 \times 25.00} \times 100\right] = 59.70\ \%$$

Experiment 55

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.965 \times 12.12}{1.840 \times 25.00} \times 100\right] = 74.57\ \%$$

Experiment 56

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.518 \times 11.67}{1.840 \times 25.00} \times 100\right] = 86.86\ \%$$

Experiment 57

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.423 \times 10.32}{1.840 \times 25.00} \times 100\right] = 90.51\ \%$$

Experiment 58

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.395 \times 9.56}{1.840 \times 25.00} \times 100\right] = 91.79\ \%$$

Experiment 59

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.330 \times 8.54}{1.840 \times 25.00} \times 100\right] = 93.87\ \%$$

Experiment 60

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.124 \times 7.58}{1.840 \times 25.00} \times 100\right] = 97.96\ \%$$

Experiment 61

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.920 \times 18.59}{1.840 \times 25.00} \times 100\right] = 62.82\,\%$$

Experiment 62

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.048 \times 18.44}{1.840 \times 25.00} \times 100\right] = 57.99\,\%$$

Experiment 63

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.071 \times 32.59}{1.840 \times 40.00} \times 100\right] = 52.58\,\%$$

Experiment 64

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.573 \times 9.94}{1.840 \times 25.00} \times 100\right] = 87.62\,\%$$

Experiment 65

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.545 \times 9.92}{1.840 \times 25.00} \times 100\right] = 88.25\,\%$$

Experiment 66

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.597 \times 9.82}{1.840 \times 25.00} \times 100\right] = 87.26\,\%$$

$$\text{Cobalt Extraction (\%)} = \left[100 - \frac{0.061 \times 9.82}{0.132 \times 25.00} \times 100\right] = 81.85\,\%$$

$$\text{Iron Extraction (\%)} = \left[100 - \frac{21.18 \times 9.82}{41.02 \times 25.00} \times 100\right] = 79.72\,\%$$

Experiment 67

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.408 \times 21.87}{1.840 \times 25.00} \times 100\right] = 33.06\,\%$$

Experiment 68

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{1.020 \times 18.74}{1.840 \times 25.00} \times 100\right] = 58.45\,\%$$

Experiment 69

$$\text{Nickel Extraction (\%)} = \left[100 - \frac{0.234 \times 9.69}{1.840 \times 25.00} \times 100\right] = 95.07\,\%$$

$$\text{Cobalt Extraction (\%)} = \left[100 - \frac{0.034 \times 9.69}{0.132 \times 25.00} \times 100\right] = 90.02\,\%$$

$$\text{Iron Extraction (\%)} = \left[100 - \frac{7.90 \times 9.69}{41.02 \times 25.00} \times 100\right] = 92.54\,\%$$