BIOLEACHING OF LATERITIC NICKEL ORE USING CHEMOLITHOTROPHIC MICRO ORGANISMS (*Acidithiobacillus ferrooxidans*)

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology
In
Chemical Engineering

By
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Department of Chemical Engineering

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Under the Guidance of

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CERTIFICATE

This is to certify that the thesis entitled "BIOLEACHING OF LATERITIC NICKEL ORE USING CHEMOLITHOTROPHIC MICRO ORGANISMS (*Acidithibacillus ferrooxidans*)" submitted by Sri Soumya Darshan Mishra in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date :-

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Jayesh Doshi

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ABSTRACT

In this study, the recovery of nickel from a low grade ore was attempted employing a chemolithotrophic micro organism, a bacteria, named *Acidithiobacillus ferrooxidans.* The factors studies were pulp density of the ore for leaching and the effect of residence time on leaching of nickel from the ore at a constant total iron. The entire experiment was carried out at room temperature. The objective of the study was thus to calculate the amount of nickel leached or extracted from a low grade ore by bio leaching methods at different pulp densities of the ore as well as at different residence times. The first step in the procedure was the collection and activation of the bacterial strains of *Acidithiobacillus ferrooxidans.* The bacteria were raised in a culture of $9K^+$ media supplied with adequate calculated amount of nutrients and were shaken continuously in a shaker cum incubator to fully activate them. The activity and fully active conditions were determined by Ferrous Iron and Total Iron estimations. Pulp densities of 2%, 5%, 10% and 20% were prepared. For each residence time, 5 conical flasks were allocated for testing samples at 0 hour, 5 days, 10 days and 15 day and a control flask were prepared. Then the samples were analyzed by an Atomic Absorption Spectrophotometer at Regional Research Laboratory, Bhubaneswar for the percentage of nickel extracted from each sample of residence time and different pulp densities. The pH was maintained at around 1.5-2 for each sample for the optimum activity of the bacteria. The data obtained was tabulated and the required graphs were drawn to get the final result. The graphs were plotted between percentage of nickel extracted vs. residence time at various pulp densities and nickel extracted vs. pulp densities at various residence times. From the graphs, it was observed that the maximum nickel extraction was observed for a pulp density of 2% at 15 days. The percentage of nickel extraction decreases with increase in pulp densities for a particular residence time. The percentage of nickel extracted increases with the increase in residence time for a particular pulp density. The percentage of nickel extracted also depends a lot on the type of ore used, modifications made on the ore as well as on the activity of the bacteria. Higher is the activity of the bacteria, more is the extraction of nickel.

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1: INTRODUCTION

1.1: BACKGROUND

Mineral resources of the nation reflect in terms of metal values for economic growth of the country at large. Our natural mineral wealth has been exploited considerably to a greater extent during the past 50 years. With increase in industrialization coupled with population growth, the demand of metals has increased and is likely to go up further in years to come. This has resulted in irreversible impacts on diminishing high-grade ores with simultaneous generation of solid wastes and effluents containing metals. It is thus important to tackle the problem for control of pollution and recovery of metal values in a cost-effective method.

Mineral biotechnology holds greater importance in mineral engineering for the development of economically viable processes for bioremediation of metals, utilization of wastes and low grade ores through biochemical leaching methods, up gradation of ores through bio beneficiation, effluent treatment through bio accumulation and bio precipitation etc. In all these processes the natural ability of microorganisms belonging to various groups has been effectively utilized.

World wide reserves of high-grade ores are diminishing at an alarming rate due to the rapid increase in the demand for metals. However there exist large stockpiles of low and lean grade ores yet to be mined. But the problem is that the recovery of metals from them using conventional techniques is very expensive due to high energy and and capital inputs required. Another major problem is environmental costs due to high level of pollution from these technologies. Environmental standards continue to stiffen, particularly regarding toxic wastes, so costs for ensuring environmental protection will continue to rise.

Biotechnology is regarded as one of the most promising and the most revolutionary solution to these problems, compared to pyrometallurgy or chemical metallurgy. It holds the promise of dramatically reducing the capital costs. It also offers the opportunity to reduce

environmental pollution. Biological processes are carried under mild conditions, usually without adding toxic chemicals. The products of biological processes end up in aqueous solution which is more amenable to containment and treatment than gaseous waste.

1.2: OBJECTIVE

Nickel is a strategic metal of vital importance in many modern industrial and metallurgical applications. Nickel is widely used in a number of alloys, both ferrous and non-ferrous, including high temperature and electrical resistance alloys. Extraction of nickel from low grade ores is the primary objective of bioleaching. Nickel occurs in nature in two forms, namely sulphides and oxides/laterites. Sulphides are the high grade ores whereas oxides/laterites are the low grade ores. About 85% of the total known nickel reserves of the world are associated with the lateritic type of ore, making it significant as the future supply. The continued depletion of high-grade nickel sulphide ores, high cost of fuels and implementation and enforcement of stricter environmental regulations will collectively dictate the future production of nickel from low grade laterite ores. So the objective of bioleaching is to produce nickel from low grade ores with less energy utilization and with an environment friendly process.

1.3: HISTORY

The earliest use of microbial processes for mining occurred long before it was clear that microbes were responsible for the effects observed. At the Rio Tinto (Rd River) mine in Seville, Spain, copper mine workings were rediscovered in 1556. Evidence suggests that the mine used water from the Rio Tinto water containing a very high concentration of ferric ion owing to microbial activity in the area. When the water from this river was irrigated into copper containing deposits, the copper dissolved and later precipitated as smaller deposits. Although the people at that time likely believed this process to be magic, we now know that it was the first recorded use of biomineralization. Leaching of copper was practiced in Norway in the 15^{th} century, in Germany in 16^{th} century and in England in the 18^{th} century. In early 19^{th} century, heap and dump leaching was practiced. In 1947, actual evidence of microbial

leaching was obtained through the pioneer workers Colmer and Hinkel. They isolated pure culture of Thiobacillus Ferooxidans from mine water. This gram negative chemolithotroph could oxidize the sulfide part of minerals to sulphuric acid and ferrous ion to ferric at a very low Ph. The industrial scale bioleaching of copper in heaps has had a chequered 400 year career. Over the past 20 years this technology has blossomed with annualized world copper production from the process increasing from 0.2% to approximately 8-10%. Bioleaching of copper in heaps was first recorded at the Rio Tinto mine in 17^{th} century. The first modern industrialscale copper heap bioleach, producing 14,000tpa, commenced in 1980 at Lo Aguirre in Chile. The first stand-alone mine using copper bioleaching – solvent extraction – electrowinning was the Girilambone Copper Operation (managed by Straits Resources and commissioned 1993) in central NSW, Australia. Now of a total ealized d world copper production of approximately 14 Mtpa, some 4-7% is ealized by some form of bioleaching.

CHAPTER 2

2: THEORITICAL STUDIES

2.1: TYPES OF BIOLEACHING

2.2: MICRO ORGANISMS USED

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2: THEORITICAL STUDIES

2.1: TYPES OF BIOLEACHING

2.1.1: BIOLEACHING:

Basically it is the dissolution of metals from their ores, concentrates and mineral wastes under the influence of microorganisms leading to the yield of metal solution of leach liquor containing metals. Such solutions can be processed through solvent extraction and electrowinning to get highly pure metal or can be processed to get metal salts. Metals such as copper, zinc, uranium, nickel, cobalt, gold etc. can be extracted by the process. 15% copper, 13% uranium and 25% gold are being produced world wide through bioleaching route. Both oxidic and sulphidic ores can be treated by this process.

2.1.2: TYPES OF BIOLEACHING:

- Bench scale bioleaching
- Tank Leaching
- Heap Leaching
- Column Leaching
- Reactor Leaching

2.2: MICRO ORGANISMS USED

For biochemical leaching, both autotrophic and heterotrophic bacterial and fungal species have been used for different ores. Acidophilic bacterial species have been used in refractory gold ore leaching for removal of pyrite matrix. The bacteria belonging to the genus Thiobacillus are aerobic and acidophilic autotrophes which play an important role in the bioleaching of metals from sulphidic minerals. They have been the most extensively studied microorganisms in terms of their physiological and biochemical characteristics. These bacteria derive their energy requirements from oxidation of iron and sulfur compounds. Ferric iron and sulphuric acid produced in the system bring about metal solubilisation. The physiological requirements and the ability of Thiobacillus to oxidize Fe^{2+} and S determine the bioleaching efficiency.

The ability of microorganisms to leach and mobilize metals from solid materials comprises of three principles namely

- Redox reactions
- Formation of organic or inorganic acids
- Excretion of complexing agents

In the process both autotrophic and heterotrophic microorganisms tested for metal removal or substrate degradation are species of Thiobacillus, Bacillus, Pseudomonas, Sulpholobus, Leptospirillum, Acidophillum, Cyanobacteria, Aspergillus, Penicillium, Rhizopus, Streptomyces etc.

Specifically, a consortium of microorganisms namely, Acidithiobacillus ferrooxidans, Acidithiobacillus thiooxidans, Leptospirillum ferrooxidans, Sulpholobus spp. And thermophilic bacteria including Sulpholobus hermosulphidoxidans and Sulpholobus brierleyi are known to be involved in bioleaching. Anaerobes would also be found in leaching areas.

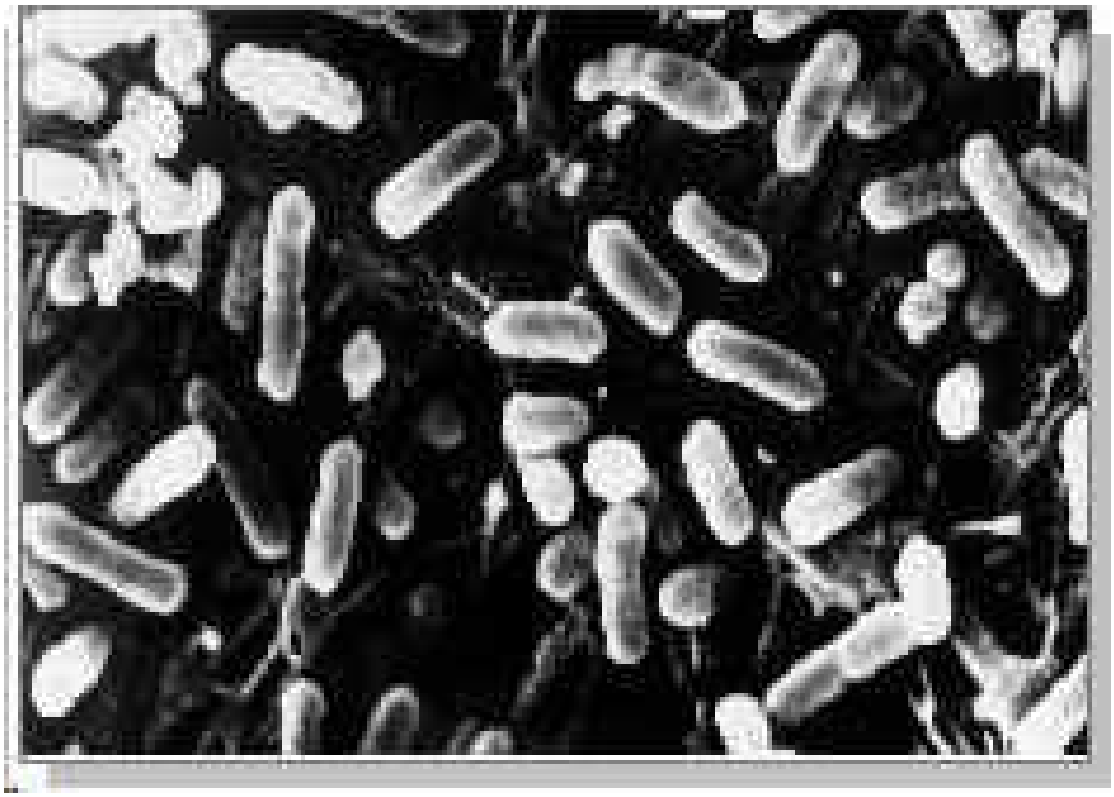

Fig 2.1- BACTERIA USED IN BIOLEACHING PROCESSES.

TABLE 2.1- Micro organisms, processes and areas of application.

MICROORGANISMS	PROCESS	AREA OF APPLICATIONS
Bacteria of the genera Thiobacillus and Leptospirillum	Oxidation of sulphide minerals, S and Fe^{2+} at pH 1.4-3.5 and temperature 5-35^{o}C.	Dump, underground and tank leaching of metals from sulphides and mixed ores and concentrates from wastes of pyrometallurgical industries, desulphurization of coal
Thermophillic bacteria similar to Thiobacilli	Same at pH 1.1-3.5 Temperature 30-55^{o}C	Same as above
Thermophillic bacteria of the genus Sulphobacillus	Same at pH 1.1-5.0 Temperature 20-60^{o}C	Same as above
Acidophillic bacteria of genera Sulpholobus and Acidianus.	Same at pH 1-5 Temperature 45-96^{o}C	Same as above
Organotrophic microorganisms and their metabolites (Fungi, Bacteria, Yeast, Algae)	Destruction of sulphide minerals and aluminium silicates, reducing and oxidation of manganese, solubilising gold and biosorption of metal.	Extraction of metals from carbonates and silicate ores and rocks, leaching of gold and bacterial biomass and metabolites in ore floatation and selective extraction of metals from solution.

2.3: BIOLEACHING MECHANISMS

A generalized reaction can be used to express the biological oxidation of a mineral sulphide involved in leaching:

$MS + 2O_2 \text{-----} \rightarrow MSO_4$

Where M is a bivalent metal.

There are two mechanisms by which bioleaching takes place viz. direct and indirect.

The direct mechanism involves the physical contact of the organism with the insoluble sulphide and is independent of the indirect mechanism. In the direct mechanism, the bacteria can directly oxidize the sulphide moiety of the metal sulphide and elemental sulphur as per the following equations:

$S^{2-} + 2O_2 \text{---}^{\text{T.ferrooxidans}}\text{---------} \rightarrow SO_4^{2-}$

$Fe^{2+}\text{----------}^{\text{T.ferrooxidans}}\text{----} \rightarrow Fe^{3+} + e^-$

$S + H_2O + 3/2\ O_2\text{-----}^{\text{Bacteria}}\text{--} \rightarrow H_2SO_4$

In the direct mechanism, of leaching by bacteria, intimate contact and adhesion o the mineral takes place prior to the enzymatic attack by the organism. The direct mechanism is inferred from the scanning electron micrographs which demonstrate bacterial adhesion on the mineral surfaces. The direct mechanism is further confirmed by the leaching of synthetic sulphides free of iron, where only the direct attack of the bacteria can lead to leaching.

CuS (Covellite) + $2O_2 \text{---} \rightarrow CuSO_4$

ZnS (Sphalerite) + $2O_2 \text{---} \rightarrow ZnSO_4$

The indirect mechanism involves the ferric-ferrous cycle. An important reaction mediated by Acidothiobacillus ferrooxidans is:

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

Ferric sulphate is a strong oxidizing agent capable of dissolving a wide range of metal sulphide minerals. Leaching brought about by ferric sulphate is termed indirect leaching because it proceeds in the absence of both oxygen and viable bacteria. This mode is responsible for leaching several minerals:

$CuFeS_2$ (chalcopyrite) $+ 2Fe_2(SO_4)_3 \rightarrow CuSO_4 + 5FeSO_4 + 2S$

FeS_2 (pyrite) $+ Fe_2(SO_4)_3 \rightarrow 3FeSO_4 + 2S$

$UO_2 + Fe_2(SO_4)_3 + 2H_2SO_4 \rightarrow UO_2(SO_4)_{4-3} + 2FeSO_4 + 4H^+$

$4FeS_2 + 15O_2 + H_2O \xrightarrow{Bacteria} 2Fe_2(SO_4)_3 + 2H_2SO_4$

$4CuFeS_2 + 17O_2 + 2H_2SO_4 \xrightarrow{Bacteria} 4\,CuSO_4 + 2Fe_2(SO_4)_3 + 2H_2O$

$2Cu_2S + 5O_2 + 2H_2SO_4 \xrightarrow{Bacteria} 4CuSO_4 + 2H_2O$

$CuS + 2O_2 \xrightarrow{Bacteria} CuSO_4$

$S^{2-} + 8Fe^{3+} \rightarrow S^{6+} + 8\,Fe^{2+}$

$S^{2-} + 2Fe^{3+} \rightarrow S + 2Fe^{2+}$

The bacteria present can again reoxidise the ferrous iron and elemental sulphur to sulphuric acid:

$Fe^{2+} \xrightarrow{T.ferrooxidans} Fe^{3+} + e^-$

$S^{2-} + 2\,Fe + 2\,Fe^{3+} \rightarrow S + 2Fe^{2+}$

$2S + 3O_2 + 2H_2O \rightarrow 2H_2SO_4$

This sulphuric acid maintains the pH at levels favourable to the growth of bacteria and also helps in the effective leaching of oxide minerals.

CuO (Tenorite) $+ 2H_2SO_4 \rightarrow CuSO_4 + H_2O$

$UO_3 + 3H_2SO_4 \rightarrow UO_2(SO_4)_{4-3} + H_2O + 4H^+$

2.3.1: Autotrophic leaching mechanisms:

Three proposed mechanisms for the action of Acidithiobacillus ferrooxidans on sulphide minerals are as follows:

- The indirect mechanism in which the bacteria oxidizes ferrous ions in the bulk solution to ferric ions that leach the minerals.
- The indirect contact mechanism in which the bacteria oxidize ferrous ions to ferric ions within the layer of bacteria and exopolymeric materials and the ferric ions within this layer leach the minerals.
- The direct contact mechanism in which the bacteria directly oxidize the minerals by biological means without any requirements for ferric or ferrous ions.

For sulfidic ore leaching, Thiobacillus ferooxidans and Thiobacillus thiooxidans were widely used organisms.

Groudev reported the mixed culture of Thiobacillus ferrooxidans and Thiobacillus thiooxidans could extract more nickel during bioleaching of nickel sulfide than that of individual when used alone.

Wanda et al. reported that 57% of nickel extract from sulfidic ore by using Thiobacillus ferrooxidans in 9K medium after 30 days of leaching.

Nickeliferrous limonite constitutes by far the largest known terrestrial reserves of nickel and cobalt. They are also major future sources of chromium and iron.

Wood (1985) studied the mineralogical parameters influencing the acid bacterial leaching of sulfidic ores. It included chiefly the mode of nickel occurrence, permeability of the ore, the quantities of acid consuming gangue in the ore etc.

Sukla and Das (1986) reported the leaching behaviour of Sukinda lateritic nickel ore with sulphuric acid. Mineralogically, the Sukinda ore contains goethite, quartz, chromites and manganese in oxide form in the iron matrix and not as a separate mineral. Since nickel is intimately associated with iron in the laterite, any process that recovers nickel from laterite will therefore be likely to recover iron. They also reported that at about $360^{o}C$ goethite decomposes to hematite.

Sukla and Das (1987) reported on extraction of cobalt from Sukinda laterites by reduction roast and ammonia leaching. Pressure leaching with sulphuric acid and atmospheric acid leaching is associated with goethite.

2.3.2: Heterotrophic Leaching Mechanisms:

Among the heterotrophic bacteria, members of genus Thiobacillus and pseudomonas have been found to be effective in the leaching of non-sulfidic mineral ores. Fungi of the genus penicillium and aspergillus have also been used in the mineral leaching.
As reported, microorganisms are able to mobilize metals by the following:

- Formation of organic acid
- Oxidation or reduction reaction
- Extraction of complexing agents
- Chelates formation

Acid production:

Glucose/Sucrose--$^{Fungus/microorganism}$----→ Citric/Oxalic/Glutanic acid

Acid Leaching:

$NiO + 2H^{+}$----→ $Ni^{2+} + H_2O$

Complexation/ Chelation of nickel with citric acid:

$Ni^{2+} + H_3$ (Citrate) ------→ Ni (Citrate) + $3H^{+}$

Precipitation of Oxalic acid:

$Ni^{2+} + HO_2C.CO_2H$----------→ Ni $(O_2C.CO_2)_3 + 2H^{+}$

Organic acids however, occupy a central position in the overall process and supply both protons and metal complexing organic acid anion.
Mc.Kenzie (1987) studied on the solubilisation of nickel, cobalt and iron from laterites by means of organic chelating acids at low pH.
Alibhai (1991) reported about 55-60% of nickel and cobalt extraction from Greek lateries when strains of indigenous penicillium sp. and Aspergillus niger were used for bioleaching.

Bioleaching of Greek non-sulfidic nickel ores using microorganisms associated leaching process has been reported by Tzeferis (1991). They developed two bioleaching techniques such as

- Leaching in the presence of microorganisms
- Chemical leaching at high temperature ($95^{o}C$)

Hey extracted to the extent of 55-60 % nickel using first technique and the nickel recovery in the second technique were in the range of 70-72%.

Sukla (1995) reported on increased stability of Aspergillus niger in nickel bioleaching. They achieved 95% nickel leaching with ultrasound pretreated strains of Aspergillus niger in 14 days as compared to 92% nickel leaching after 20 days with untreated Aspergillus niger.

Sukla et al. (1995) have reported the use of filamentous fungus Penicillium for bioleaching of Sukinda lateritic nickel ore. Under optimum conditions, the fungus could leach a maximum of 12.5% nickel.

2.4: BACTERIAL LEACHING TECHNIQUES

The two major techniques used in leaching are percolation and agitation leaching. Percolation leaching involves the percolation of a lixiviant through a static bed, whereas agitation leaching involves finer particle sizes agitated in a lixiviant. Due to the large-scale operations involved in bacterial leaching, percolation leaching is preferred commercially.

The principal commercial methods are in situ, dump, heap and vat leaching. In situ leaching involves pumping of solution and air under pressure into a mine or into ore bodies made permeable by explosive charging. The resulting metal-enriched solutions are recovered through wells drilled below the ore body. Three types of ore bodies are generally considered for in situ leaching surface deposits above the water table, surface deposits below the water table and deep deposits below the water table.

Dump leaching involves uncrushed waste rock which is piled up. These dumps generally contain about 0.1-0.5% Cu, too low to recover profitably by conventional procedures. Some of these dumps are huge, containing in excess of 10 million tones of waste rock.

Heap leaching requires the preparation of the ore, primarily size reduction, so as to maximize mineral-lixiviant interaction and the laying of an impermeable base to prevent lixiviant loss and pollution of water bodies. Essentially, both dump and heap leaching involve the application of lixiviant to the top of the dump or heap surface and the recovery of the metal laden solution that seeps to the bottom by gravity flow. The dilute sulphuric acid sprinkled on top percolates down through the dump, lowering the pH and promoting the growth of acidophilic microorganisms. The acid run-off is collected at the bottom of the dump, from where it is pumped to a recovery station. Copper is extracted from the acid run-off by cementation or solvent extraction or electrowining. All the above processes are essentially uncontrolled from a biological and engineering standpoint. Beside these processes are slow in nature and require long periods to recover a portion of the metal.

Vat leaching as currently applied to oxide ores involves the dissolution of crushed materials in a confined tank. More controls can be brought in for enhanced recovery by the use of bioreactors, though necessarily these involve higher costs. However for ore concentrates and precious metals they are being considered actively.

2.5: METALS EXTRACTED BY BIOLEACHING

2.5.1: Bioleaching of Copper:

Biological copper leaching is practiced in many countries including Australia, Canada, Chile, Mexico, Peru, Russia and the U.S.A. Copper recovery from bioleaching accounts for about 25% of the world copper production. Following the initial isolation of Acidithiobacillus ferrooxidans from coal mine water in 1947, studies quickly disclosed its presence in copper-leaching operations. Acidithiobacillus ferrooxidans is also found in the Malanjkhand Copper Mines in Madhya Pradesh, India.

The physical configurations of bioleaching operations world-wide for copper are mostly uniform. Typically copper ore mined from open pits is segregated; higher grade metal is concentrated to produce feed for smelting, while the lower grade ore is subjected to leaching. The ore is piled on an impermeable surface until a dump of suitable dimension forms. After the top is leveled, leach solution is flooded or sprayed onto the dump. A copper dump represents a complex and heterogeneous microbiological habitat. It contains solids ranging in size from boulders to fine sand and includes material of complex mineralogy. Bacterial colonization occurs in the top 1 meter or so. The temperature may

reach 90°C in the interior of the dump and supports a range of thermophillic microorganisms, which are often anaerobic, or microaerophillic. In these regions, indirect leaching by ferric sulphate also prevails. The exterior of the dump is at ambient temperature and undergoes changes in temperature reflecting seasonal and diurnal fluctuations. Many different microorganisms have been isolated from copper dumps, some of which have been studied in the laboratory. These include a variety of mesophilic, aerobic iron and sulphur oxidizing microorganisms, thermophilic iron and sulphur oxidizing microorganisms, and anaerobic sulphate reducing bacteria. Some are heterotrophic bacteria, which indirectly affect metal solubilisation by affecting the growth and activity of metal solubilising bacteria. Others are protozoa, which interact with and prey on different types of bacteria. Leach solutions enriched with copper exit at the base of the dump and are conveyed to a central recovery facility. In most large-scale operations the leach solution, containing 0.5-2 g/l copper is pumped into large cementation units containing iron scrapings for cementation and then electrolysis. A typical large dump may have an operating life of over 10 years.

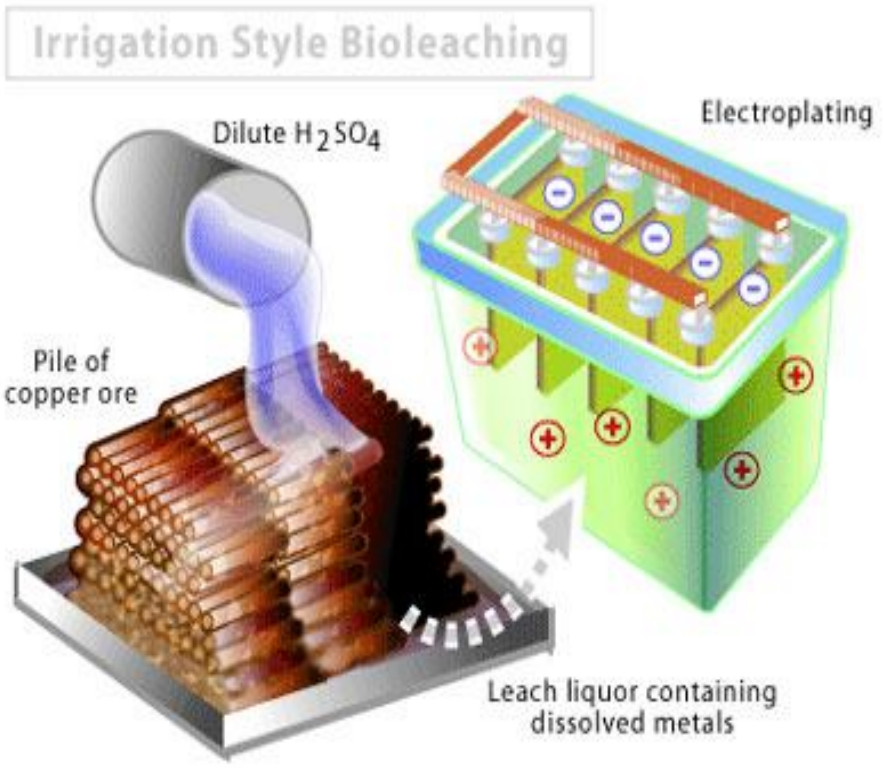

Fig 2.2- COPPER BIOLEACHING PROCESS.

2.5.2: Bioleaching of Uranium*:*

Uranium leaching proceeds by the indirect mechanism as Acidithiobacillus ferrooxidans does not directly interact with uranium minerals. The role of Acidithiobacillus ferrooxidans in uranium leaching is the best example of the indirect mechanism. Bacterial activity is limited to the oxidation of pyrite and ferrous iron. The process involves periodic spraying or flooding of worked-out stops and tunnels of underground mines with lixiviant. Another method in use for uranium extraction is vat-leaching. Bioleaching has also been used successfully to obtain uranium from waste gold ore.

2.5.3: Bioliberation of Gold:

Iron- and sulphur- oxidizing acidophilic bacteria are able to oxidize certain sulphidic ores containing encapsulated particles of elemental gold., resulting in improved accessibility of gold to Complexation by leaching agents such as cyanide. Bio-oxidation of gold ores is a less costly, less polluting alternative to other oxidative pre-treatments such as roasting and pressure oxidation.

Recently bio-oxidation of gold ores has been implemented as a commercial process, and is under study worldwide for further application to refractory gold ores. Technology developed by K. A. Natarajan and co-workers at the Indian Institute of Science is being applied at the Hutti Gold Mines, Karnataka, India for extraction of gold. Bio-oxidation involves treatment with Acidithiobacillus ferrooxidans to oxidize the sulphur matrix prior to cyanide extraction. Commercial exploitation has made use of heap leaching technology for refractory gold ores. Refractory sulphidic gold ores contain mainly two types of sulfides: pyrite and arsenopyrite. Since gold is usually finely disseminated in the sulfide matrix, the objective of biooxidation of refractory gold ores is to break the sulfide matrix by dissolution of pyrite and arsenopyrite.

2.5.4: Phosphate Solubilisation:

Vast quantities of rock phosphate available in India are not being utilized owing to their low grade. On the other hand, our agricultural lands require phosphatic fertilizers for higher crop yield. Reports on microbial rock phosphate solubilization are available in literature. This ability of microorganisms to utilize insoluble rock phosphates has a potential in agricultural applications. Therefore, opportunities do exist to isolate and

identify such organisms and for optimization of process parameters for utilization of low-grade rock phosphates.

2.5.5: Sulfide Leaching:

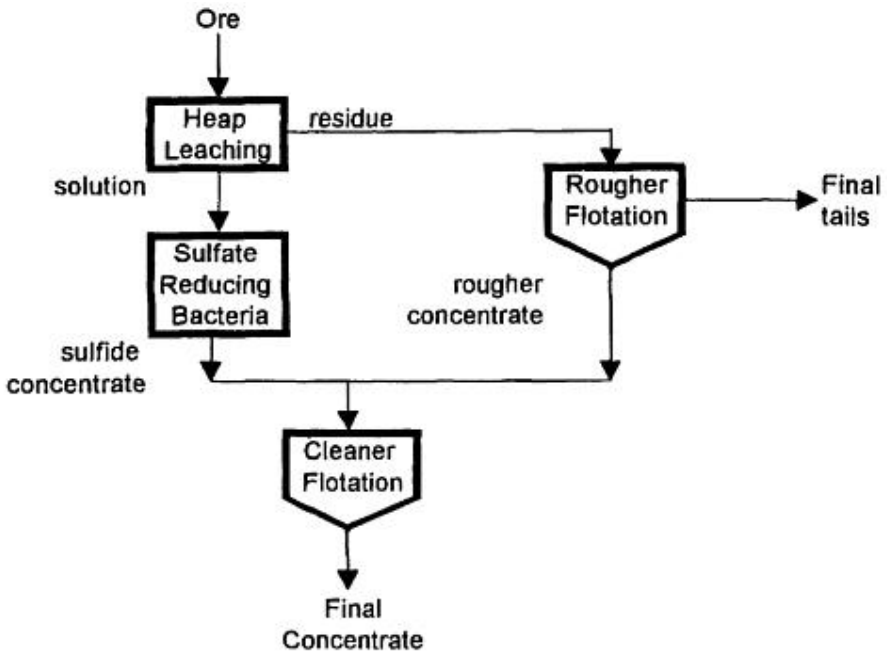

Fig. 2.3- Sulfide Leaching

2.5.6: Nickel ore Leaching:

Nickel ore deposits are of two types:

- Sulfide
- Oxide (Lateritic)

The sulfide ores have been the major source of nickel till date. The laterites are the non-sulfidic ores, highly weathered material rich in secondary oxides of iron, aluminium or both and nearly devoid of bases and primary silicates and may contain abundant quartz and kaolinite. Laterite often contains minor amounts of nickel, cobalt and chromium.

Non-sulfidic ores such as oxides, carbonates and silicates contain no energy source for the microorganisms to utilize. Bioleaching of non-sulfidic ores and minerals may be used for the recovery valuable metals from low grade ores and minerals as well as for the beneficiation of mineral raw materials, recovery of metal from wastes and heavy metal detoxification of soils and solid residues.

Extraction of nickel and cobalt from low grade laterite ores constitutes one of the expensive processes, due to the low grade pf metals present in the ore. The mineralogical concentration and distribution of nickel and cobalt within the ore matrix inhibit the application of beneficiation processes to concentrate the ores. The importance of the low grade laterite ore to the future supply of nickel and cobalt becomes obvious when one considers that 85% of the nickel reserves and a greater proportion of cobalt reserves are in laterite ores .

Lateritic nickel ore, or ores produced by the weathering of parent rock, constitute 75% of the world's nickel reserves and it is necessary to utilize these for the extraction of metal values. The complexity of the ores has led to the development of a variety of possible extraction techniques. Four of these namely matte smelting, productions of ferronickel, sulfuric acid leaching at elevated pressures and reduction followed by ammonia leaching are in commercial operation. Pyrometallurgical methods for the production of nickel require a large amount of energy whereas hydrometallurgical methods need less energy but sophisticated technology. Therefore it has become necessary to develop new hydrometallurgical methods. In this respect, the microbial leaching techniques for extraction of metal values are worth mentioning. Ores that are subjected to leaching process of lateritic ores are:

- Saprolite
- Weathered Saprolite
- Limonite
- Nontronite

These ores represent the various layers in the lateritic bedrock. The limonite consists of mainly goethite, a hydrated iron oxide such as alpha-FeO(OH), $HFeO_2$, or $Fe_2O_3.H_2O$. This continues to a nontronite rich zone. Saprolite is the next layer, which is distinguished because it is rich in magnesium silicate.

Table 2.2- Bioleaching Operations and Effective Micro organisms

Bioleaching operations	**Effective microorganisms**
Manganese leaching	Pseudomonas, Alcaligenes, Aeromonas, Clostridium
Phosphorous leaching	Aspergillus, Hyphomicrobium
Silica leaching	Bacillus, Acetobacter, Streptococcus
Iron and other base metal leaching	Aspergillus, Penicillium, Mucor, Pseudomonas, Bacillus, Micrococcus, Rhodopseudomonas, Enterobacter, Candida, Escherichia

2.6: GROWTH KINETICS AND EFFECTS OF PHYSICOCHEMICAL PARAMETERS ON THE GROWH OF Thiobacillus ferrooxidans

There is evidence that the growth of T.ferrooxidans and oxidation of ferrous iron are tightly coupled. A direct relationship between ferrous iron oxidation and O_2 uptake/ CO_2 fixation has been shown. It was also found that the rate of ferrous iron oxidation by T.ferrooxidans was directly related to concentration of nitrogen. It is also proved that the presence of toxic metals could produce a similar effect. Many studies show that the growth of T. ferrooxidans and its ability to oxidize metal ions are dependent on pH, temperature, and concentrations of ferrous

and ferric ions. In addition the availability of oxygen and CO_2 are the other important factors which influence the metabolism of the bacteria.

2.6.1: pH

With ferrous ion as the energy source, T.ferrooxidans grows in an environment with pH between 1.0-6.0. Lag periods are observed before the activity of the bacteria is increased with a pH value of 1.2. pH values in the range of 1.5-3.5 did not influence the growth of bacteria but values less than 1.5 or greater than 3.5 affected the growth. The activity of T. ferrooxidans is also reported to be independent of pH in the pH range of 1.9-2.4. pH values between 1.0 and 4.0 inhibited growth strongly. Bacteria must be equipped with an efficient mechanism that allows them to grow with fluctuating pH values. Whatever be the mechanism, we must maintain the cytoplasmic pH at a constant value. With acidophile, resistance to pH has been south in the structural and compositional peculiarities of the cell wall or the cytoplasmic membrane or both. The Thiobacilli contains huge amounts of cyclopropane fatty acids. It has been suggested that these constituents within the cell membrane function by increasing rigidity and decreasing membrane permeability. T.ferrooxidans has a remarkable ability to adapt to different environmental conditions and to produce spontaneous phenotypic variants. Recent genetic studies suggest that this is due to the transposition of mobile DNA sequences.

2.6.2: Temperature

Most strains of T.ferrooxidans characterized with respect to temperature are mesohilic with temperature optima between 30^oC-40^oC. The optimum temperature is said to be pH dependent showing a lower optimum temperature with decreasing pH. The maximum temperature for bacteria to grow is also pH dependent, being 45^oC over the pH range of 2.5-3.5 and 35^oC at a pH of 1.5. The discrepancy between the optimal values for temperature as well as pH, suggests the possibility that different strains, as employed in different studies exhibit optimum conditions that are strain dependent. The optimal values may be derived in terms of growth rate, oxidation rate of ferrous iron etc. The relatively low and stable ambient temperatures in underground mines employing biohydrometallurgical processes in temperate

climates have generated an interest in psychrophilic or psychotrophic strains of T.ferooxidans. Psychotropic members of this population were numerically superior to mesophilic members and the former had wide temperature ranges for growth. The relationship of growth and iron oxidizing ability of T. ferrooxidans with temperature has usually been correlated with an Arrhenius equation. The values of activation energy calculated on the basis of ferrous iron oxidation in growing cultures of bacteria are usually higher than those which are the results of short term measurements of oxidation rate in the presence of cell suspension.

2.6.3: Concentration of ferrous iron:

The growth of T.ferrooxidans and its ability to oxidize is significantly influenced by the concentration of ferrous iron. It is reported that a decline in the growth of the bacteria due to the exhaustion of ferrous iron in the substrate. It is reported that increasing the ferrous iron concentration up to 5.6 kg/m^3 enhanced the oxygen uptake rate by employing higher concentrations of ferrous iron resulted in lower oxidation rates. Higher concentrations of ferrous iron showed an inhibitory effect on growth. The Monod equation is the most widely used expression to correlate the growth of T.ferrooxidans as a function of ferrous iron concentration. The values for maximum specific growth rate and substrate saturation constant have been determined.

2.6.4: Carbon Dioxide and Oxygen:

T.ferrooxidans is a chemoautotrophe and as such an obligate aerobe with a strict requirement for CO_2 as its source of carbon for growth. In the view of limited solubility of CO_2 at the pH required for the optimum growth of T.ferrooxidans cultures could be predicted to become limited by CO_2 stability. It has been shown that the limitation of CO_2 in growing cultures of T.ferrooxidans shifts the exponential kinetics of ferrous iron oxidation to linear relationship. The importance of CO_2 availability in achieving optimal growth rates and maximum cell yield has been shown. Significantly higher cell yields were obtained as the level of CO_2 were increased although growth rate remains unaffected. The level of CO_2 that supports the

maximal rate of cell growth is in the range of 7-8%. An increase beyond 8% results in the inhibition culture growth. Microbial growth relates to substrate utilization, the electron transfer efficiencies and the free energies of the microbial reactions. It was reported that O_2 is not a limiting substrate for bacterial growth in experiments carried out at 30^oC in 500 ml flasks containing 200 ml broth shaken at 240 rpm. It has been reported that oxygen becomes a limiting substrate when the concentration of dissolved oxygen is less than 0.29 mg/l and T.ferrooxidans does not grow in cultures with dissolved oxygen concentrations less than 0.2 mg/l.

2.6.5: Applied Potential:

Recent studies have indicated that the yield of T.ferrooxidans can be significantly increased through electrochemical means. It was demonstrated that enhanced yields could be attained due to the in situ electrochemical reduction of soluble ferric iron in the growth medium. The effect of applied D.C potentials both in the +ve and –ve range on the activity and growth of T.ferrooxidans has also been investigated. Application of +ve potentials up to 1000 mv in an acidic medium inhibited bacterial activity because of electrolytic generation of nascent oxygen in the absence ferrous iron. Employing –ve potentials in the range -500 to -1000 mv to bacterial culture containing ferric iron promoted the activity and growth of T.ferrooxidans through electrochemical conversion of ferric iron to ferrous iron.

Fig 2.4- TANKS USED FOR INDUSTRIAL SCALE BIOLEACHING.

2.7 STUDY OF THE AAS.

2.7.1: ATOMIC ABSORPTION SPECTROPHOTOMETRY (AAS)

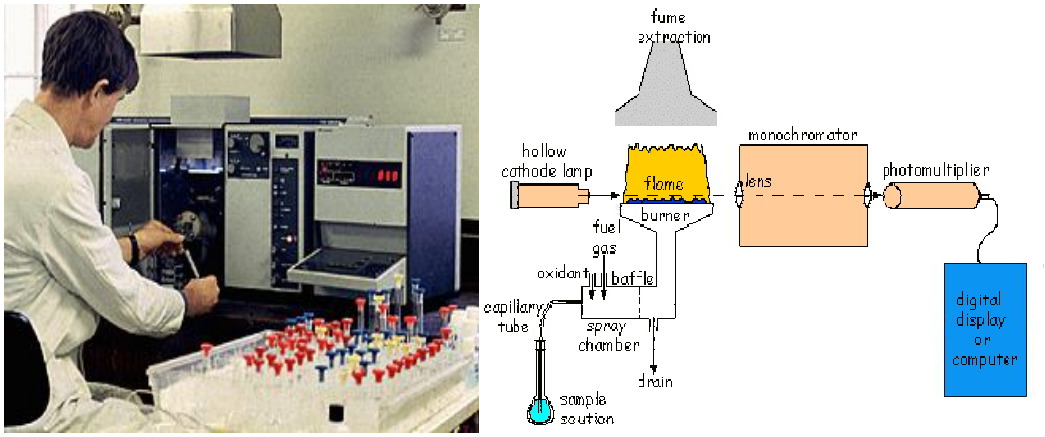

Fig. 2.5- Atomic Absorption Spectrophotometer.

Atomic absorption spectrophotometry (AAS) is an analytical technique used to measure a wide range of elements in materials such as metals, pottery and glass. Although it is a destructive technique (unlike ED-XRF), the sample size needed is very small (typically about 10 milligrams - i.e. one hundredth of a gram) and its removal causes little damage. The

sample is accurately weighed and then dissolved, often using strong acids. The resulting solution is sprayed into the flame of the instrument and atomised (see schematic diagram). Light of a suitable wavelength for a particular element is shone through the flame, and some of this light is absorbed by the atoms of the sample. The amount of light absorbed is proportional to the concentration of the element in the solution, and hence in the original object. Measurements are made separately for each element of interest in turn to achieve a complete analysis of an object, and thus the technique is relatively slow to use. However, it is very sensitive and it can measure trace elements down to the part per million level, as well as being able to measure elements present in minor and major amounts.

CHAPTER 3

3: EXPERIMENTAL SET-UP

3.1: FLOW SHEETS

3.2: EXPERIMENTAL PROCEDURE

3.3: PREPARATION OF INDICATORS

3: EXPERIMENTAL SET-UP

3.1: FLOW SHEETS

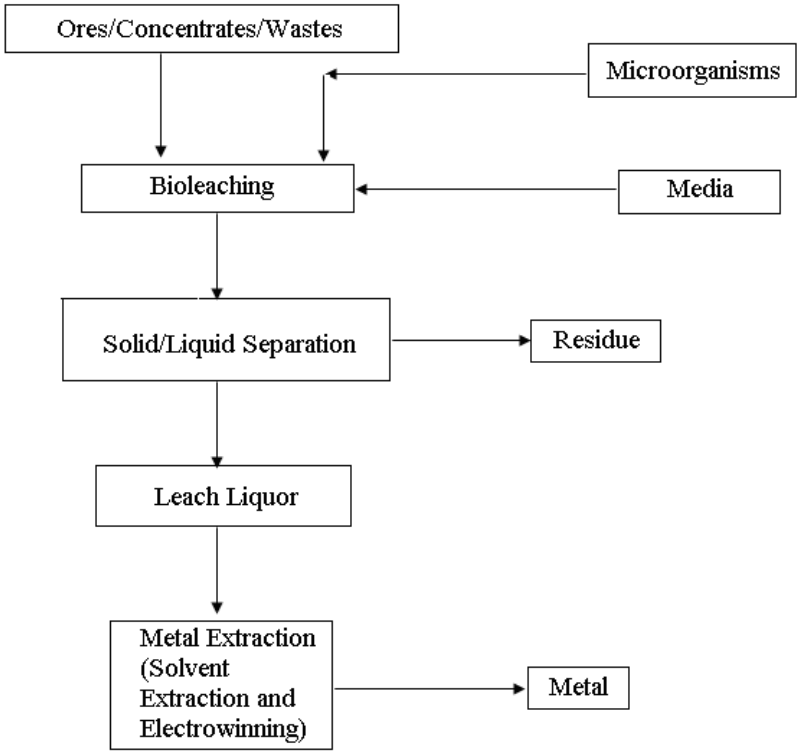

Fig. 3.1- A generalized flow sheet of bioleaching process.

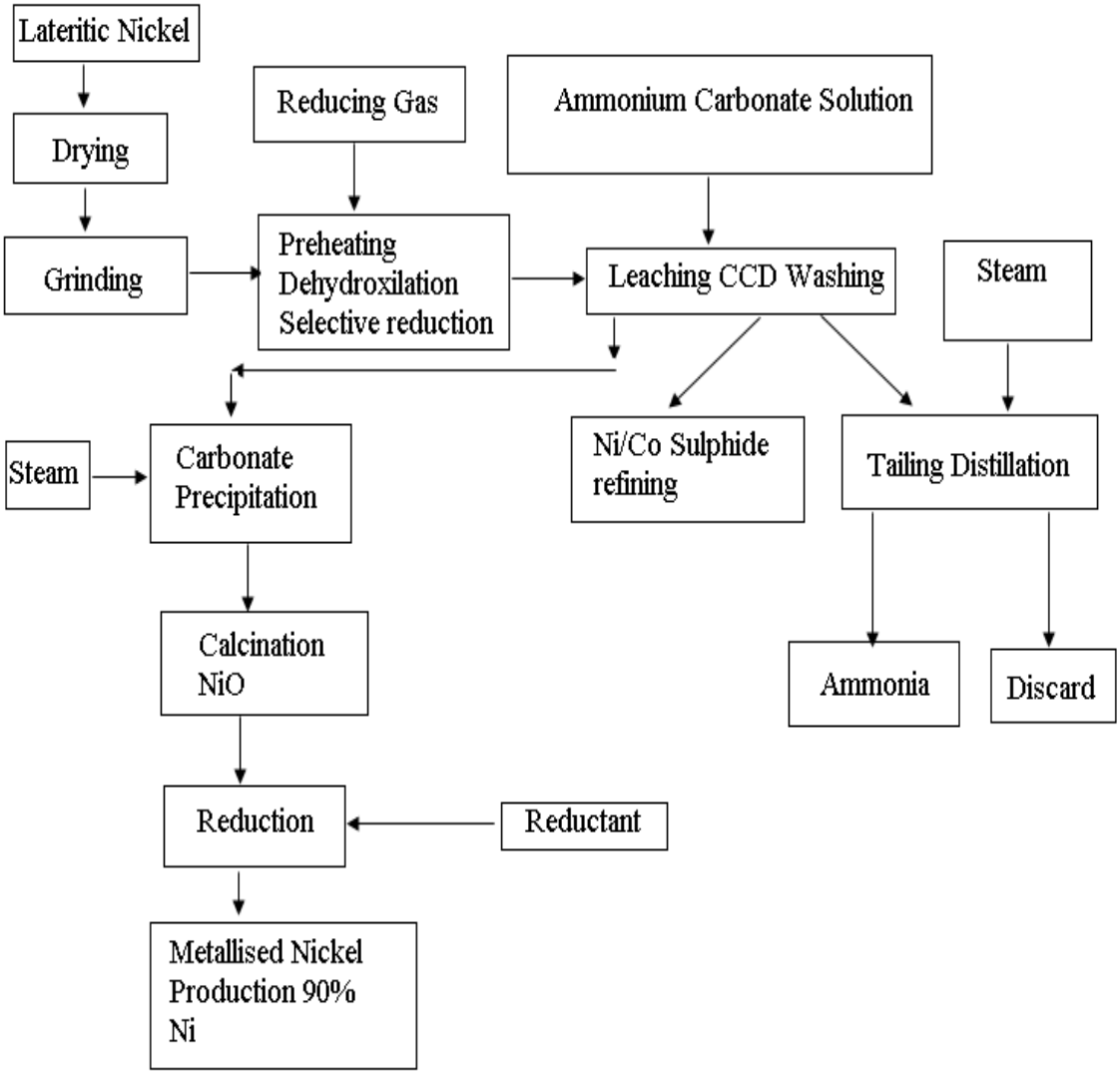

Fig. 3.2- Flow Sheet of non-biotic leaching of nickel ore (Caron process).

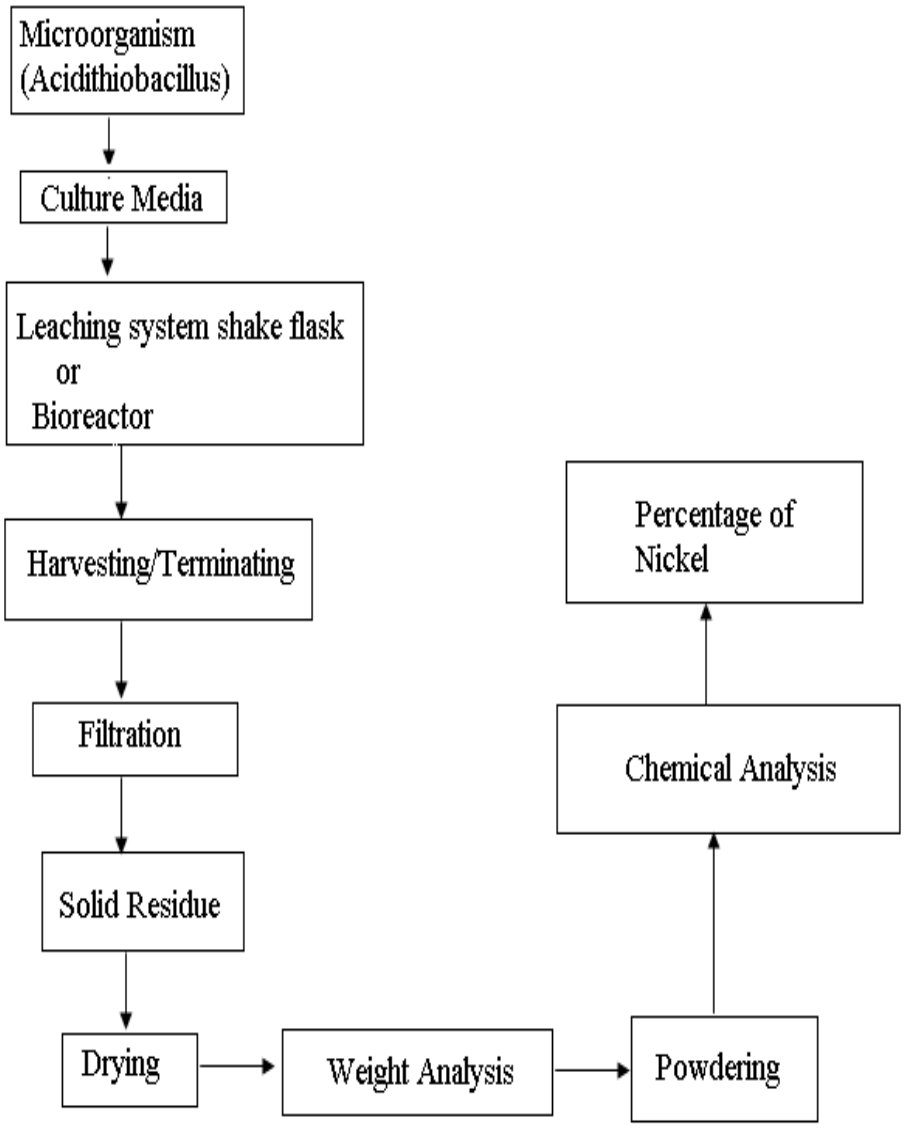

Fig. 3.3- Flow Sheet of Experimental Studies using Acidithiobacillus.

3.2: EXPERIMENTAL PROCEDURE

3.2.1: ORE ANALYSIS:

The lateritic nickel ores so obtained are reported to have nickel associated with goethite matrix and cobalt occurred primarily in manganese mineral phases. The ore is a complex, soft, and agglomerate of highly porous fine particles of very high surface area. The ore is grinded and sieved to different sizes ranging from +44 to –350 BSS and is used for study of nickel leaching. The raw ore is treated with some chemical to get better leaching.

3.2.2: PROCEDURE OF ORE ANALYSIS:

To know the percentage of nickel in the lateritic ore, chemical analysis in the following procedure is to be done. Lateritic nickel ore of 1g is taken and added to 50 ml of concentrated hydrochloric acid in a beaker. Then the mixture is heated in fume cupboard until the residue turns white. Then the mixture is cooled and filtered and kept in a clean conical flask which is washed prior to it with distilled water several times and the volume was made up to 250 ml by adding distilled water. Only 10 ml of the filtrated solutions was pipetted out into a 100 ml volumetric flask. The volume was made up to 100 ml by adding further distilled water. This solution is regarded as 10 times dilution. Then the diluted samples are to be taken for Atomic Absorption Spectrophotometer (AAS) analysis.

3.2.3: MINERALOGICAL ANALYSIS:

Mineralogical analysis of the lateritic nickel ores are analyzed using high resolution synchrotron based X-ray Diffractometer and by optical microscopy. The lateritic nickel ore will reveal the presence of goethite, a hydrated iron oxide (alpha-FeOOH) and the leached residues will show some jarosite peaks. This step is just required to demonstrate the structure of the mineral sample.

3.2.4: MICROBIOLOGICAL ANALYSIS: Laboratory stock cultures of Acidithiobcillus ferrooxodans strain are used for experiments. This organism is strictly aerobic, gram negative rod, flagellated and chemoautotrophic, which derives its energy for metabolism from the oxidation of inorganic iron and reduced sulfur compounds. This bacterium is non-sporulating rod (0.4 - 0.5 micrometer wide and 1 - 2 micrometer long.) in nature with round ends usually

occurring in single or in pairs. This organism has the ability to achieve optimum growth under strongly acidic conditions.

The strain is grown in $9K^+$ medium of Silverman and Lundgren containing $(NH_4)_2SO_4$ (3 g/l), KCl (1 g/l), $MgSO_4.7H_2O$ (0.5 g/l), $FeSo_4.7H_2O$ (44.2 g/l). The pH of the medium is adjusted to 1.5-2 with dilute sulfuric acid. The bacterial growth is assessed by monitoring the iron oxidation rate and also by the appearance of a reddish brown colour in the medium due to the formation of ferric iron. The genus *Acidithiobacillus* represents a versatile group of chemolithotrophic organisms. The organism most studied is *Acidithiobacilus ferrooxidans.* Thiobacilli are members of the division of *Proteobacteria* close to junction between the beta and gamma sub-division.

3.2.5: $9K^+$ MEDIUM PREPARATION AND INOCULATION OF *Acidithiobacillus ferrooxidans* STRAIN:

500 ml conical flask was taken with 300 ml of distilled water in it which was previously autoclaved and the media constituents were poured into it. The concentrations of the chemicals for the media are given below in g/l. The appropriate amounts for 300 ml was calculated and added to the distilled water. pH of the solution was found to be approximately 3.5 now. The pH meter so used was standardized by using pH buffer tablets of standard pH like 4, 7, 9. Then the pH was adjusted to 2 with dilute sulphuric acid using the pH meter. For the control flask, the entire procedure is same but 10 ml of mercuric chloride ($HgCl_2$) was added as bacteriocide. Then 10 ml of active culture of *Acidithiobacillus ferrooxidans* with approximately 2.8x10^6 cells/ml were added to the other flask and the both the flasks were kept in rotary shaker cum incubator at 30^oC-35^oC and speed was adjusted to 130rpm-150 rpm. 2 ml of sample was taken from each flask to estimate the ferrous and total iron concentration for zero hr. analysis. Simultaneously, ferrous and total iron estimation was done at regular intervals to see the generation time and activity of the strain.

Table 3.1- $9K^+$ MEDIUM COMPOSITION.

COMPOSITION	*CONCENTRATION (g/l)*
Ammonium Sulfate	3.0
Dipotassium Hydrogen Phosphate	0.5
Potassium Chloride	0.1
Magnesium Sulfate	0.5
Ferrous Sulfate	44.0

3.2.6: FERROUS IRON ESTIMATION:

2ml of sample was pipetted into a 250ml conical flask. Then 2ml of orthophosphoric acid is added to the flask. Then 4-5 drops of BDAS(Barium diphenyl amine sulphonate) indicator is added and the flasks were rinsed with distilled water. Then it was titrated against 0.1n potassium dichromate till blue-violet coloration occurs. Three concurrent readings were taken. The difference in readings is noted.

3.2.7: TOTAL IRON ESTIMATION:

2ml of sample (bacteria+media) was pipetted into a 250ml conical flask. Then 4-5 ml of concentrated HCl was added and the sample was heated to boiling in an electric heater in a fumigation chamber. Then stannous chloride indicator was added dropwise until the yellow coloration disappears. This was followed by cooling the solution under running water from a water tap. Then 2-3 drops of saturated solution o mercuric chloride was added till the white precipitate occurs. Then 2-3 drops of orthhophosphoric acid and 4-5 drops of BDAS indicator was added. Then the flasks were rinsed with distilled water. Then the solution was titrated against 0.1 N potassium dichromate till silky blue violet colouration occurs. 3 concordant readings are to be taken. The difference in burette readings are to be noted down.

3.2.8: FORMULA FOR CALCULATION:

Concentration in g/l = (Burette Reading) * (Strength of $K_2Cr_2O_7$) * 55.85/ (Volume Taken)
Strength of $K_2Cr_2O_7$ = 0.1N
Volume Taken = 2 ml

3.2.9: SHAKE FLASK STUDIES:

3.2.9.1: Microorganisms used in the leaching experiments:

The bacterial strain *Acidithiobacillus ferrooxidans* used for the experimental purpose is a laboratory culture and was provided by the Regional Research Laboratory, Bhubaneswar.

3.2.9.2: Adaptation of microorganisms:

Adaptation was carried out for obtaining the efficient strains for increased extraction of nickel from treated lateritic nickel ore. Before the leaching experiments are to be performed, the organisms are to be initially adapted to the ore at 2%, 5%, 10% and 20% pulp densities to improve the tolerance of the strain and hence leaching efficiency increases.

3.2.9.3: Bioleaching of the treated lateritic nickel ore using different pulp densities:

For bioleaching of the treated lateritic nickel ore, studies were conducted using the four types of ore with four pulp densities i.e. 2%, 5%, 10% and 20%. The residence time are noted to be 0 hour analysis, 5 days, 10 days and 15 days. 500 ml of $9K^+$ media was prepared. It was divided equally in five conical flasks, each conical flask containing 100 ml of the media. One conical flask among them is kept as control. 10 ml innoculum is added in each flask. Innoculum consists of fully activated bacteria. No innoculum is added to the control flask. Around 10 grams of mercuric chloride ($HgCl_2$) is added as bacteriocide. For analyzing at variable pulp density, 2 grams, 5 grams, 10 grams and 20 grams ore are added to different flasks. The flasks are shaken properly to mix them well. The contents separate into a top layer and a bottom layer. 2 ml of sample are taken from the top layer to be analyzed in the Atomic Absorption Spectrophotometer for percentage of nickel extracted. We must also take care of the fact that all the apparatus must be washed with distilled water before use in the experiment to prevent contamination. The control flask contains a pulp density of 2%.

3.2.9.4: Analyzing the percentage of nickel extracted:

The percentage of nickel extracted was analyzed using an Atomic Absorption Spectrophotometer. The leached solution was kept undisturbed to get two distinct layers. 2 ml

of the solution from the top layer is taken in a 25 ml round-bottomed flask. A few drops of strong acid like sulphuric acid was added and the volume was made up to 25 ml. It was then kept in an AAS to get the mean values from which the percentage of nickel extracted can be calculated using the following formulae.

$$X = \frac{(\text{MEAN} * \text{DILUTION FACTOR} * \text{STOCK SOLUTION}) * 100}{(\text{WEIGHT OF THE ORE}) * 10\text{^}6}$$

$$\text{PERCENTAGE OF NICKEL EXTRACTED} = (X * 100) / (\text{NICKEL \% IN ORE})$$

Here dilution factor is taken to be 12.5 for a stock solution of 25 ml of sample.

3.3: PREPARATION OF INDICATORS

3.3.1: BDAS INDICATOR:

Barium Diphenyl Amine Sulphonate or BDAS indicator was prepared by adding 0.5-0.6 grams of Barium Diphenyl Amine Sulphonate to 250 ml of distilled water and heated till all BDAS was dissolved. Then it was cooled and filtered.

3.3.2: MERCURIC CHLORIDE INDICATOR:

Mercuric Chloride ($HgCl_2$) was taken in an amount of 10 grams and added in 250 ml distilled water and heated upto boiling. When all the $HgCl_2$ particles were dissolved, the solution was cooled and filtered to get the indicator.

3.3.3: STANNOUS CHLORIDE INDICATOR:

6.25 grams stannous chloride ($SnCl_2$) was taken in a beaker. 12.5 ml of conc. HCl was added along with 12.5 ml of distilled water. Then 2-3 tin granules were added to complete the indicator.

3.3.4: POTASSIUM DICHROMATE SOLUTION/INDICATOR:

4.904 grams of purely dry $K_2Cr_2O_7$ was taken and dissolved in 100 ml distilled water. Then it was diluted 100 times or 1000 times to get 0.1N or 0.01N $K_2Cr_2O_7$ solution.

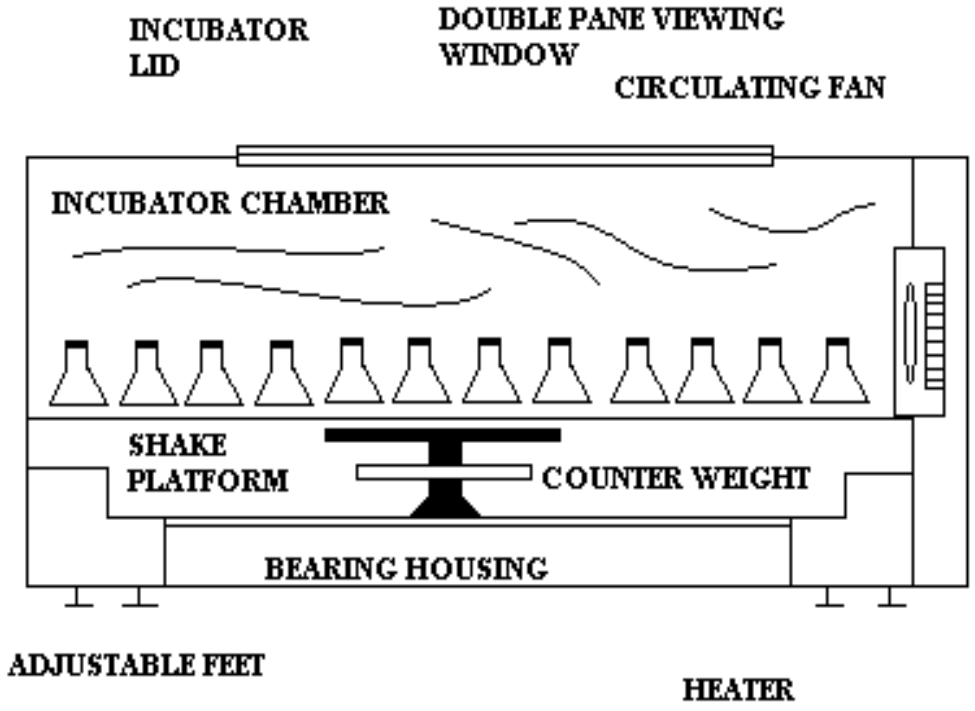

Fig. 3.4- SCHEMATIC DIAGRAM OF SHAKE FLASK EXPERIMENTS IN SHAKER.

CHAPTER 4

4: OBSERVATIONS AND TABULATIONS

4.1: ORE ANALYSIS

4.2: FERROUS IRON AND TOTAL IRON ESTIMATION

4.3: SHAKE FLASK STUDIES

4: OBSERVATIONS AND TABULATIONS

4.1: ORE ANALYSIS

Table 4.1- ORE ANALYSIS

NICKEL	0.57%
COBALT	0.32%
IRON	45.78%
CHROMIUM	4.04%
MANGANEESE	0.35%
ACID INSOLUBLES (AI)	7.74%

4.2: TOTAL IRON AND FERROUS IRON ESTIMATION

4.2.1: FERROUS IRON ESTIMATION AT 2% PULP DENSITY

Table 4.2- FERROUS IRON ESTIMATION AT 2% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K_2Cr_2O_7$ CONSUMED	Fe^{2+} IRON
1	0 DAYS	50	47.1	2.9	8.098
2	0 DAYS	47.1	44.1	3	8.378
3	0 DAYS	44.1	41.1	3	8.378
4	0 DAYS	41.1	38.1	3	8.378
5	5 DAYS	50	47.1	2.9	8.098
6	5 DAYS	47.1	44.3	2.8	7.82
7	5 DAYS	44.3	41.4	2.9	8.098
8	5 DAYS	41.4	38.5	2.9	8.098
9	10 DAYS	50	47.5	2.5	6.981
10	10 DAYS	47.5	45.1	2.4	6.702
11	10 DAYS	45.1	42.7	2.4	6.702
12	10 DAYS	42.7	40.3	2.4	6.702
13	15 DAYS	50	48.2	1.8	5.027
14	15 DAYS	48.2	46.3	1.9	5.306
15	15 DAYS	46.3	44.4	1.9	5.306
16	15 DAYS	44.4	42.5	1.9	5.306

4.2.2: TOTAL IRON ESTIMATION AT 2% PULP DENSITY

Table 4.3- TOTAL IRON ESTIMATION AT 2% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K2Cr2O_7$ CONSUMED	TOTAL IRON
1	0 DAYS	50	47	3	8.378
2	0 DAYS	47	44.2	2.8	7.819
3	0 DAYS	44.2	41.2	3	8.378
4	0 DAYS	41.2	38.2	3	8.378
5	5 DAYS	50	47.3	2.7	7.54
6	5 DAYS	47.3	44.4	2.9	8.098
7	5 DAYS	44.4	41.5	2.9	8.098
8	5 DAYS	41.5	38.6	2.9	8.098
9	10 DAYS	50	47.2	2.8	7.819
10	10 DAYS	47.2	44.5	2.7	7.54
11	10 DAYS	44.5	41.7	2.8	7.819
12	10 DAYS	41.7	38.9	2.8	7.819
13	15 DAYS	50	47.3	2.7	7.54
14	15 DAYS	47.3	44.6	2.7	7.54
15	15 DAYS	44.6	41.8	2.8	7.819
16	15 DAYS	41.8	39.1	2.7	7.54

4.2.3: FERROUS IRON ESTIMATION AT 5% PULP DENSITY

Table 4.4- FERROUS IRON ESTIMATION AT 5% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K_2Cr_2O_7$ CONSUMED	Fe^{2+} IRON
1	0 DAYS	50	47	3	8.378
2	0 DAYS	47	44	3	8.378
3	0 DAYS	44	40.8	3.2	8.936
4	0 DAYS	40.8	37.8	3	8.378
5	5 DAYS	50	47	3	8.378
6	5 DAYS	47	44.1	2.9	8.098
7	5 DAYS	44.1	41.2	2.9	8.098
8	5 DAYS	41.2	38.3	2.9	8.098
9	10 DAYS	50	47.5	2.5	6.981
10	10 DAYS	47.5	44.8	2.7	7.54
11	10 DAYS	44.8	42.3	2.5	6.981
12	10 DAYS	42.3	39.8	2.5	6.981
13	15 DAYS	50	47.9	2.1	5.864
14	15 DAYS	47.9	45.9	2	5.585
15	15 DAYS	45.9	43.9	2	5.585
16	15 DAYS	43.9	41.9	2	5.585

4.2.4: TOTAL IRON ESTIMATION AT 5% PULP DENSITY

Table 4.5- TOTAL IRON ESTIMATION AT 5% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K2Cr2O_7$ CONSUMED	TOTAL IRON
1	0 DAYS	50	47	3	8.378
2	0 DAYS	47	44	3	8.378
3	0 DAYS	44	40.8	3.1	8.657
4	0 DAYS	40.8	37.8	3	8.378
5	5 DAYS	50	47	3	8.378
6	5 DAYS	47	44.1	3	8.378
7	5 DAYS	44.1	41.2	2.9	8.098
8	5 DAYS	41.2	38.3	3	8.378
9	10 DAYS	50	47.3	2.7	7.54
10	10 DAYS	47.3	44.6	2.7	7.54
11	10 DAYS	44.6	41.8	2.8	7.819
12	10 DAYS	41.8	39.1	2.7	7.54
13	15 DAYS	50	47.4	2.6	7.261
14	15 DAYS	47.4	44.8	2.6	7.261
15	15 DAYS	44.8	42.3	2.5	6.981
16	15 DAYS	42.3	39.7	2.6	7.261

4.2.5: FERROUS IRON ESTIMATION AT 10% PULP DENSITY

Table 4.6- FERROUS IRON ESTIMATION AT 10% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K_2Cr_2O_7$ CONSUMED	Fe^{2+} IRON
1	0 DAYS	50	47	3	8.378
2	0 DAYS	47	44	3	8.378
3	0 DAYS	44	40.8	3.2	8.936
4	0 DAYS	40.8	37.8	3	8.378
5	5 DAYS	50	47.1	2.9	8.098
6	5 DAYS	47.1	44.3	2.8	7.819
7	5 DAYS	44.3	41.4	2.9	8.098
8	5 DAYS	41.4	38.5	2.9	8.098
9	10 DAYS	50	47.4	2.6	7.261
10	10 DAYS	47.4	44.8	2.6	7.261
11	10 DAYS	44.8	42.3	2.5	6.981
12	10 DAYS	42.3	39.7	2.6	7.261
13	15 DAYS	50	47.9	2.1	5.864
14	15 DAYS	47.9	45.8	2.1	5.864
15	15 DAYS	45.8	43.6	2.2	6.144
16	15 DAYS	43.6	41.5	2.1	5.864

4.2.6: TOTAL IRON ESTIMATION AT 10% PULP DENSITY

Table 4.7- TOTAL IRON ESTIMATION AT 10% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K2Cr2O_7$ CONSUMED	TOTAL IRON
1	0 DAYS	50	47	3	8.378
2	0 DAYS	47	44.2	2.8	7.819
3	0 DAYS	44.2	41.2	3	8.378
4	0 DAYS	41.2	38.2	3	8.378
5	5 DAYS	50	47.1	2.9	8.098
6	5 DAYS	47.1	44.1	3	8.378
7	5 DAYS	44.1	41.1	3	8.378
8	5 DAYS	41.1	38.1	3	8.378
9	10 DAYS	50	47.3	2.7	7.54
10	10 DAYS	47.3	44.7	2.6	7.261
11	10 DAYS	44.7	42.1	2.6	7.261
12	10 DAYS	42.1	39.5	2.6	7.261
13	15 DAYS	50	47.6	2.4	6.702
14	15 DAYS	47.6	45.2	2.4	6.702
15	15 DAYS	45.2	43	2.2	6.144
16	15 DAYS	43	40.6	2.4	6.702

4.2.7: FERROUS IRON ESTIMATION AT 20% PULP DENSITY

Table 4.8- FERROUS IRON ESTIMATION AT 20% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K_2Cr_2O_7$ CONSUMED	Fe^{2+} IRON
1	0 DAYS	50	47	3	8.378
2	0 DAYS	47	44	3	8.378
3	0 DAYS	44	40.8	3.2	8.936
4	0 DAYS	40.8	37.8	3	8.378
5	5 DAYS	50	46.9	3.1	8.657
6	5 DAYS	46.9	43.9	3	8.378
7	5 DAYS	43.9	40.9	3	8.378
8	5 DAYS	40.9	37.9	3	8.378
9	10 DAYS	50	47.2	2.8	7.819
10	10 DAYS	47.2	44.4	2.8	7.819
11	10 DAYS	44.4	41.8	2.6	7.261
12	10 DAYS	41.8	39	2.8	7.819
13	15 DAYS	50	47.8	2.2	6.144
14	15 DAYS	47.8	45.7	2.1	5.864
15	15 DAYS	45.7	43.5	2.2	6.144
16	15 DAYS	43.5	41.3	2.2	6.144

4.2.8: TOTAL IRON ESTIMATION AT 20% PULP DENSITY

Table 4.9- TOTAL IRON ESTIMATION AT 20% PULP DENSITY

SL NO.	DURATION	IBR	FBR	$K2Cr2O_7$ CONSUMED	TOTAL IRON
1	0 DAYS	50	47	3	8.378
2	0 DAYS	47	44	3	8.378
3	0 DAYS	44	40.9	3.1	8.657
4	0 DAYS	40.9	37.9	3	8.378
5	5 DAYS	50	47.3	2.7	7.54
6	5 DAYS	47.3	44.4	2.9	8.098
7	5 DAYS	44.4	41.5	2.9	8.098
8	5 DAYS	41.5	38.6	2.9	8.098
9	10 DAYS	50	47.6	2.4	6.702
10	10 DAYS	47.6	45.3	2.3	6.423
11	10 DAYS	45.3	42.9	2.4	6.702
12	10 DAYS	42.9	40.5	2.4	6.702
13	15 DAYS	50	47.8	2.2	6.144
14	15 DAYS	47.8	45.6	2.2	6.144
15	15 DAYS	45.6	43.5	2.1	5.864
16	15 DAYS	43.5	41.3	2.2	6.144

4.3: SHAKE FLASK STUDIES

4.3.1: ZERO HOUR ANALYSIS

Table 4.10- NICKEL EXTRACTION DATA AT ZERO HOUR

PULP DENSITY	MEAN VALUE FROM AAS	% NICKEL EXTRACTED
2%	0.427	1.171%
5%	0.929	1.019%
10%	1.570	0.861%
20%	2.253	0.618%
CONTROL	0.338	0.927%

4.3.2: 5 DAYS ANALYSIS

Table 4.11- NICKEL EXTRACTION DATA AFTER FIVE DAYS

PULP DENSITY	MEAN VALUE FROM AAS	% NICKEL EXTRACTED
2%	0.598	1.639%
5%	1.205	1.321%
10%	2.274	1.247%
20%	3.425	0.939%
CONTROL	0.417	1.143%

4.3.3: 10 DAYS ANALYSIS

Table 4.12- NICKEL EXTRACTION DATA AFTER TEN DAYS

PULP DENSITY	MEAN VALUE FROM AAS	% NICKEL EXTRACTED
2%	0.733	2.009%
5%	1.645	1.804%
10%	3.001	1.645%
20%	4.325	1.186%
CONTROL	0.458	1.255%

4.3.4: 15 DAYS ANALYSIS

Table 4.13- NICKEL EXTRACTION DATA AFTER FIFTEEN DAYS

PULP DENSITY	MEAN VALUE FROM AAS	% NICKEL EXTRACTED
2%	1.106	3.032%
5%	2.358	2.586%
10%	4.089	2.242%
20%	5.321	1.459%
CONTROL	0.640	1.754%

CHAPTER 5

5: RESULTS AND CONCLUSIONS

5.1: GRAPHICAL ANALYSIS

5.2: THEORITICAL CONCLUSIONS

5.3: SAMPLE CALCULATIONS

5: RESULTS AND CONCLUSIONS

5.1: GRAPHICAL ANALYSIS

5.1.1: FERROUS IRON CURVES

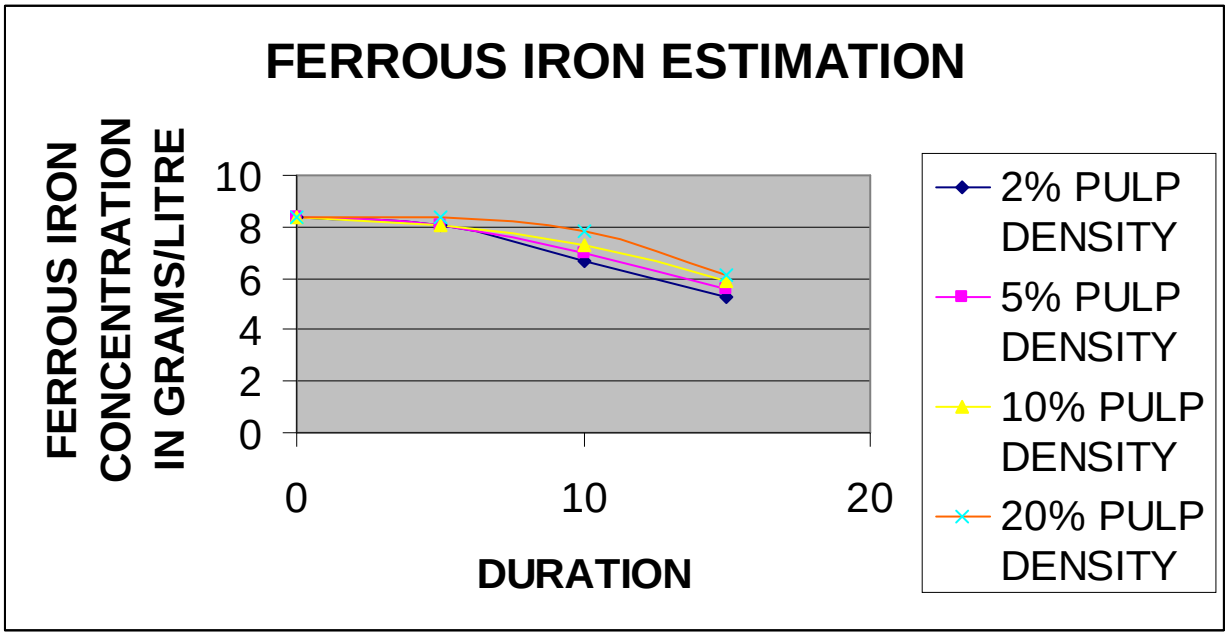

Fig. 5.1: Ferrous iron vs. residence time

5.1.2: TOTAL IRON CURVES

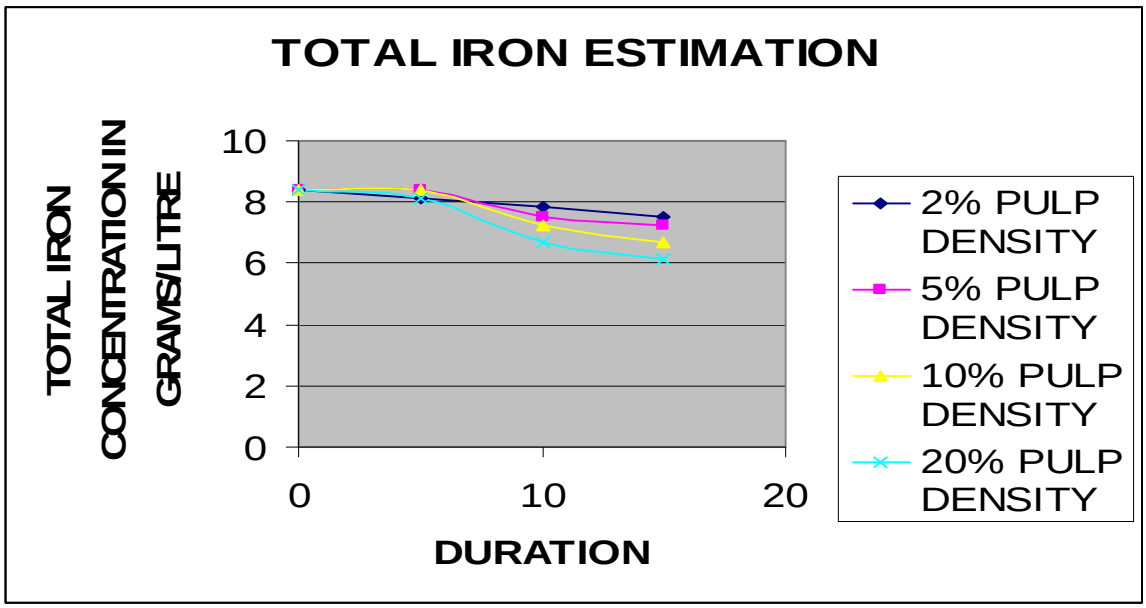

Fig. 5.2: Total iron vs. residence time

5.1.3: NICKEL EXTRACTION CURVES

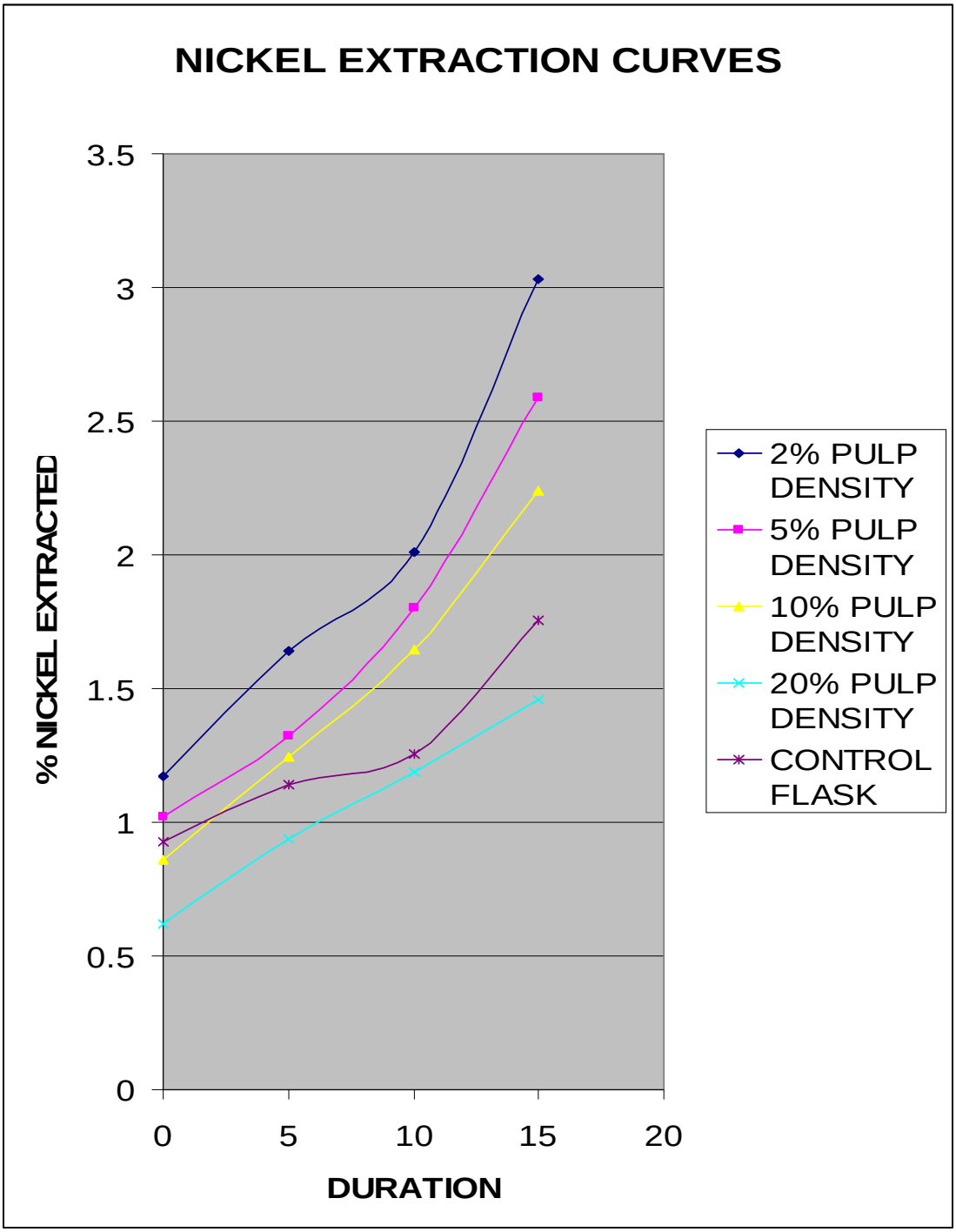

Fig. 5.3: % Nickel Extracted vs. Residence Time

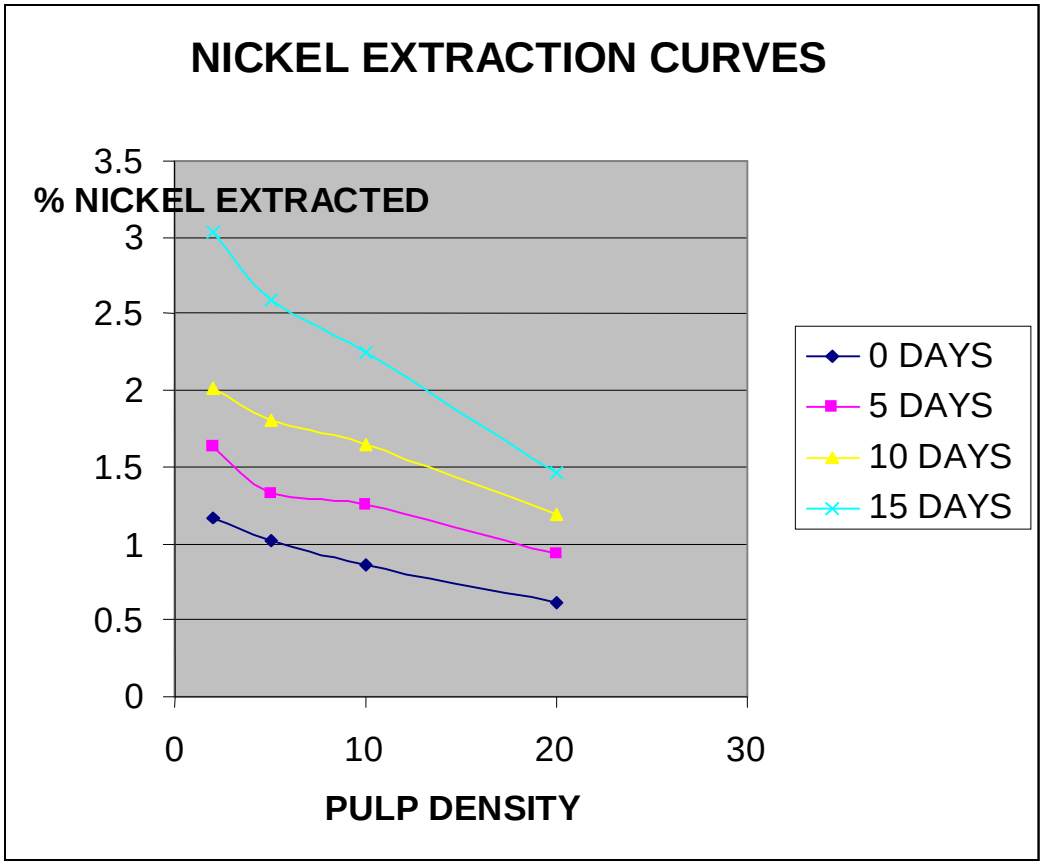

Fig. 5.4: % Nickel Extracted vs. Pulp Density

5.2: THEORITICAL CONCLUSIONS

From the tables, the corresponding graphs were drawn. The trends of the percentage of nickel extracted with change in pulp density as well as the duration were noted. It was observed that the percentage of nickel extraction increases with the duration or residence time for any fixed pulp density. On the other hand, the extraction of nickel decreases with the increase in pulp density. So the maximum nickel is extracted when the pulp density is 2% and the residence time is 15 days. Moreover the activity of the bacteria also has a major role to play in the maximum extraction of nickel. As we can see from our tables, the ferrous iron concentration had not reached 0 after a time period of 15 days. So, it gives an indication that the bacteria used for leaching was not fully active then. But we can agree on the fact that to get a

maximum leaching of nickel, we must use a fully active bacteria, low pulp density and high residence time.

5.3: SAMPLE CALCULATIONS

For ferrous iron estimation and total iron estimation, for 10% pulp density at 10 days residence time, burette reading was 2.6. Strength of $K_2Cr_2O_7$ used was 0.1 N and volume taken was 2 ml.

So, ferrous iron and total iron estimations are:

(2.6 * 0.1 * 55.85) / 2 = 7.261 grams/liter

For a pulp density of 2% and a residence time of 5 days, the mean value from the AAS was found to be 0.598. So according to the formula,

X = (0.598*12.5*25*100)/(2*10^6) = 0.00934375

% NICKEL EXTRACTED = (0.00934375/0.57) * 100 = 1.639%

Similarly, we can proceed for all other observations.

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