

**OPTIMIZATION OF BENEFICIATION AND REDUCTION  
PROCESS PARAMETERS OF LOW GRADE MANGANESE  
ORE FOR THE PRODUCTION OF MANGANESE IRON ALLOY**

A thesis submitted in partial fulfillment of the requirements for the award of degree of  
**Master of Technology in Material Engineering**

By

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**Exam Roll: M4MAT19026**

**Registration No: 140917 of 2017-18**

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**Jadavpur University**

**Kolkata – 700032**

**2019**



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**2019**

# *Declaration of Originality and compliance of academic ethics*

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*I hereby declare that this thesis contains literature survey and original researchwork by the undersigned candidate, as part of his “**Optimization Of Beneficiation And Reduction Process Parameters of Low Grade Manganese Ore for Production of Manganases Iron Alloy**” studies. All information in this document have 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.*

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*Thesis Title: **Optimization of Beneficiation and Reduction Process Parameters of  
Low Grade Manganese Ore For Production of Manganese Iron Alloy***

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# *Certificate*

This is to certify that the entitled “*Optimization of Beneficiation and Reduction Process Parameters of Low Grade Manganese Ore for Production of Manganese Iron Alloy*” has been carried out by *Mr. Arka Lahiri* (Exam Roll: M4MAT19026, Registration No: 140917 of 2017-18 ) under my guidance and supervision and accepted in partial fulfillment for the degree of Master of Technology in Material Engineering from Jadavpur University. To the best of our knowledge the contents of this thesis nor any part thereof have not been previously submitted for the award of any degree or diploma.

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*Signature of Examiners*

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*Date : Arka Lahiri*

# *Abstract*

*The demand of manganese has been increasing on a global scale which has put importance on the raw materials of steel industry as manganese plays an important role in carbon steel production. Apart from the metallurgical and steel sector, modern battery industry and electronic industry uses magnetic materials which cannot be developed without manganese or processed manganese. The growing demand for manganese has made the need for beneficiation of low grade manganese ore even more essential as the high grade ores are depleting. For the purpose of beneficiation, gravity separation techniques like jigging and tabling were used. The main aim was to produce Mn-Fe alloy with increased percentage of manganese. After reduction, Magnetic separation was done so as to increase the Mn / Fe ratio in the non magnetic part. For the raw ores and after the beneficiation methods, XRF as well as XRD analysis were done. Effect of the various parameters were studied on the gravity separation techniques to optimize the various parameter levels for highest separation.*



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# *Chapter 1*

## *Introduction*

# **1. INTRODUCTION**

Manganese occurs as silvery grey in colour and is very hard and brittle in nature. It is always available in combination with iron, laterite and other minerals. Manganese in alloy form is an essential input in steel making and steel is one of the most important key indicators in the industrial economy of any country. In the recent years the trade volume of manganese ore has grown world over and also in India. Presently India is one of the major importers of manganese ore in the world.

Manganese ore and its alloys play an important role both in the metallurgical and non-metallurgical industries. Metallurgical applications probably account for 97 to 98 per cent of the total manganese ore consumption while the rest is being used in the manufacture of dry cell batteries and chemicals. It may be pointed out that steel, no matter what grade or type, cannot be made and shaped without the appropriate amount of manganese in it; and there is no satisfactory substitute for manganese. It is estimated that about 95 per cent of the total manganese ore is ultimately consumed in the production of iron and steel either directly as ore in the blast furnaces or as ferro-manganese and metal in steelmaking.[1]

Taking into consideration the relevant factors in the Indian situation, namely manganese being an essential ingredient for steelmaking, limited reserves of proved manganese ore, low recovery of saleable ore and progressive growth of steel production of the country, the installation of suitable beneficiation including agglomeration facilities to beneficiate the low grade manganese ore fines and agglomerate them and also to improve the lump recovery, is considered absolutely necessary for the conservation of the country's limited reserves.

## **1.1 PRODUCTION AND STOCKS**

The production of manganese ore was 2153 thousand tonnes during 2015-16 which has increased to 2393 thousand tones during 2016-17. There were 142 reporting mines during 2016-17 as against 146 during 2015-16. Besides, manganese ore production was reported by seven mines of iron ore, three mines of laterite and one each of quartz and limestone in 2016-17 as against six mines of iron ore, one each of laterite limestone and quartz in 2015-16.[2]

State / District	No. of mines	Production By Grade: Mn Content					Total	
		MnO <sub>2</sub>	Above 46%	35%-46%	25%-35%	Below 25%	Quantity	Value
<b>India</b>	<b>142(12)</b>	<b>27950</b>	<b>228188</b>	<b>554264</b>	<b>1174530</b>	<b>408250</b>	<b>2393182</b>	<b>16026609</b>
Public Sector	24	535	158254	345283	503181	44659	1051912	9059571
Private Sector	118(12)	27415	69934	208981	671349	363591	1341270	6967038
<b>Andhra Pradesh</b>	<b>23</b>	<b>-</b>	<b>-</b>	<b>11914</b>	<b>118400</b>	<b>102174</b>	<b>232488</b>	<b>770941</b>
Vizianagaram	23	-	-	11914	118400	102174	232488	770941
<b>Gujarat</b>	<b>2</b>	<b>-</b>	<b>-</b>	<b>-</b>	<b>-</b>	<b>43059</b>	<b>43059</b>	<b>20581</b>
Panchmahal	1	-	-	-	-	38487	38487	18295
Vadodara	1	-	-	-	-	4572	4572	2286
<b>Jharkhand</b>	<b>4</b>	<b>-</b>	<b>-</b>	<b>-</b>	<b>504</b>	<b>6</b>	<b>510</b>	<b>3562</b>
Singhbhum(West)	4	-	-	-	504	6	510	3562
<b>Karnataka</b>	<b>9(1)</b>	<b>-</b>	<b>-</b>	<b>16858</b>	<b>182301</b>	<b>62215</b>	<b>261374</b>	<b>1189857</b>
Ballari	1(1)	-	-	16858	165966	46992	232516	1067011
Chitradurga	4	-	-	-	-	7654	7654	15512
Davangere	2	-	-	-	16335	4069	20404	106258
Tumkur	2	-	-	-	-	800	800	1076
<b>Madhya Pradesh</b>	<b>42(7)</b>	<b>-</b>	<b>141578</b>	<b>106875</b>	<b>299858</b>	<b>99821</b>	<b>648132</b>	<b>5051691</b>
Balaghat	31(1)	-	131502	39308	265444	21766	508020	4350874
Chhindwara	4	-	10076	17567	10366	2732	41241	486248
Jabalpur	6(6)	-	-	-	-	75323	75323	135496
Jhabua	1	-	-	-	23548	-	23548	79073
<b>Maharashtra</b>	<b>19</b>	<b>535</b>	<b>27411</b>	<b>288708</b>	<b>273351</b>	<b>14296</b>	<b>229962</b>	<b>2011564</b>
Bhandara	2	535	2888	188947	181969	-	374339	3237373
Nagpur	17	-	24543	99761	91382	14296	229962	2011564
<b>Odisha</b>	<b>31(4)</b>	<b>27115</b>	<b>59199</b>	<b>129909</b>	<b>294825</b>	<b>76463</b>	<b>587511</b>	<b>3667350</b>
Kendujhar	17(2)	25139	59037	81631	165780	20084	331671	1791890
sundargarh	14(2)	1976	162	48278	129045	56379	235840	1875460
<b>Rajasthan</b>	<b>1</b>	<b>-</b>	<b>-</b>	<b>-</b>	<b>2545</b>	<b>-</b>	<b>2545</b>	<b>7567</b>
Banswara	1	-	-	-	2545	-	2545	7567
<b>Telangana</b>	<b>5</b>	<b>300</b>	<b>-</b>	<b>-</b>	<b>2746</b>	<b>10216</b>	<b>13262</b>	<b>66123</b>
Adilabad	5	300	-	-	2746	10216	13262	66123

**Table 1.1: Gradewise Production of Manganese Ore.2016-2017 (By Sector / State / District)**  
[2]

In all, 71 producers reported production of manganese ore in 2016-17. About 69% of the total production was reported by 12 mines including 2 associate mine, each producing more than 40,000 tonnes per annum, while 18% was contributed by 13 mines (including one associate associate mine) each falling in the production range of 20,001 to 40,000 tonnes..As regards

gradewise composition of production in 2016-17, 66% of the total production was of lower grade (below 35% Mn), 23% of medium grade (35-46% Mn) and 10% was of high grade (above 46% Mn). Production of manganese dioxide was 27,950 tonnes (1%) during the year as against 20,161 tonnes (1%) in the previous year. Madhya Pradesh being the leading manganese ore producing State accounted for 27% of the total production in 2016-17. Next in the order of production were Maharashtra and Odisha (25% each). The remaining production was reported from Andhra Pradesh, Gujarat, Jharkhand, Karnataka, Rajasthan and Telangana .

Name	Location of mine	
	State	District
MOIL Ltd	Madhya Pradesh Maharashtra	Balaghat Bhandara Nagpur
The Sandur Manganese and Iron ores Ltd	Karnataka	Ballari
RBSSD & FN Das	Andhra Pradesh	Vizianagaram
MangilalRungta	Odisha	Kendujhar
Orissa Manganese & Minerals Ltd	Odisha	Sundargarh
S.K. Sarawagi & Co.	Andhra Pradesh	Vizianagaram
Aryan Mining & Trading Corpo. Private Ltd.	Odisha	Sundargarh
Tata Steal Ltd	Odisha	Kendujhar Sundargarh

**Table 1.2: ] Principal Producers of Manganese Ore.2016-2017[4]**

<b>RANK</b>	<b>COUNTRY</b>	<b>PRODUCTION (IN TONES)</b>
	WORLD	18,500,000
	OTHER COUNTRIES	400,000
1	SOUTH AFRICA	6,200,000
2	AUSTRALIA	3,000,000
3	CHINA	2,900,000
4	GABON	1,800,000
5	BRAZIL	1,000,000
6	INDIA	950,000
7	MALAYSIA	400,000
8	UKRAINE	390,000
9	KAZAKISTAN	390,000
10	GHANA	390,000
11	MEXICO	240,000
12	MYANMAR	100,000

**Table 1.3: Manganese Ore Production Around the World. [3]**

## **1.2 INDUSTRIAL USE OF MANGANESE**

Manganese alloys are the largest produced ferro-alloys in the world with a share of about 41% of the global production of ferro-alloys. For production of one tonne of ferro-manganese, about 2.6 tonnes of manganese ore, 0.5 tonne of reductant and 3 MWh of electricity inputs are required.

### **1. Ferro-manganese**

The total production of various types of manganese alloys (high carbon ferro-manganese, medium carbon ferro-manganese and low carbon ferro-manganese) was about 4.72 lakh tonnes in 2015-16 (up to February 2016), as per Monthly Statistics of Mineral Production (February, 2016). It is to be noted that the data coverage is partial and does not reflect the actual production.

### **2. Silico-manganese**

Silico-manganese is a combination of 60-70% Mn, 10-20% silica and about 20% carbon. As per Monthly Statistics of Mineral Production (March, 2016), production of silico-manganese was reported at 2,69,920 tonnes in 2015-16 from 2,49,691 tonnes in 2014-15. It is to be noted that the data coverage is partial and does not reflect the actual production. The major factor driving the production of manganese alloys is high production growth of low nickel austenitic stainless steel. India is emerging as the largest producer of this steel where manganese is added substituting the expensive nickel.

### **3. Dry Battery**

Manganese dioxide is used for manufacturing dry cell batteries in which it functions as a depolariser of hydrogen. Consumption of manganese dioxide ore in Dry Battery Industry was 14,600 tonnes in 2015-16, [excluding Electrolytic Manganese Dioxide EMD]. The demand was met through imports, supported by indigenous production of manganese dioxide and EMD. Dry battery Industry also consumes EMD along with natural manganese dioxide ore. The only one plant of 1,000 tonnes per year capacity producing EMD is owned by MOIL and is located in Bhandara district.

## **Iron & Steel**

Iron & Steel Industry was the second major consumer of manganese ore wherein manganese ore is used directly as a blast furnace feed. Manganese is essential to iron and steel production by virtue of its sulfur-fixing, deoxidizing, and alloying properties. Small amounts of manganese improve the workability of steel at high temperatures by forming a high-melting sulfide and preventing the formation of a liquid iron sulphide at the grain boundaries.

Mn alloys are mostly used in steelmaking and foundry activities. Some 30 % of the manganese used today in steelmaking is still used for its properties as a deoxidant and a sulphide former. In this last case it combines with sulphur avoiding the formation of iron sulphides, which sulphides are low melting point phases which become liquid at hot rolling temperatures and which, consequently, generate surface cracking. The other 70 % of the manganese is used purely as an alloying element. Steels usually contain from 0.2 % to 2 % Mn depending on grades as Mn is the cheapest alloying element among those which enhance some key mechanical properties like strength and toughness. In the specific case of stainless steel it can substitute expensive nickel in some austenitic grades. There are two main families of Mn alloys called ferro-manganese (FeMn) and silico-manganese (SiMn). Silico-manganese adds additional silicon which is a stronger deoxidizer and which also helps to improve some mechanical properties of steel. In each family carbon is controlled and is lowered when producing “refined” grades. Nitrogen, Boron, Titanium, Phosphorus are elements which can be controlled depending on requested specification.

### **1.3 GRADE DESIGNATION**

Manganese ores and manganese bearing iron ores shall be classified as follows: a) Manganese ore b) Ferruginous Manganese ore c) Siliceous Manganese ore d) Manganiferrous iron ore e) Manganese ore (chemical grade).

#### **Classification Requirements**

- For the purpose of this standard, ore containing 35 percent manganese or more shall be classified as manganese ore.
- Manganese ores shall be designated as -manganese ore (Chemical grade), ferruginous manganese ore or siliceous manganese ore depending upon  $\text{MnO}_2$ , Fe and silica content, respectively.

- Ores containing low manganese but high Fe shall be designated as manganiferrous iron ores.
- Details of the different grades are mentioned in the table below.

Type of Ore	Constituents (%)				
	Mn	Fe	SiO <sub>2</sub>	MnO <sub>2</sub>	Fe <sup>+</sup> Mn
Manganese ore	35 & above	-	-	-	-
Ferruginous Manganese ore	<35 & Up to 25	<23 & Up to 13	-	-	48(min.)
Siliceous Manganese ore	<35 & Up to 25	-	20(min.)	-	-
Manganiferous iron ore	<25 & Up to 10	<48 & Up to 30	-	-	55(min.)
Manganese ore (chemical grade)	-	8(max.)	-	72(max)	-

**Table 1.4: Classification of Manganese Ore[3]**

[Indian standard - Classification of manganese ore, ferruginous manganese ore, siliceous manganese ore, dioxide manganese ore and manganiferrous iron (First revision)]

In this thesis Manganese ore collected from TATA Steel was subjected to Jigging and Tabling depending on the size range for the purpose of beneficiation. In both the processes, the optimum parameter levels for separation were found out. After beneficiation and separation of the gangue from the ore, the samples with highest separation were subjected to reduction in Inconel in a Tube furnace. Reductions were carried out at different temperatures and for different time intervals. The samples from both the upgradation methods with highest E.O.R were further taken for magnetic separation followed by smelting. Smelting would yield Iron Manganese alloy in the final product.



*Chapter 2*

*Scope of Work*

## 2. SCOPE OF WORK

The availability of Iron Manganese alloy or Ferro-manganese grade Mn ore is limited in India while the demand and the requirement for this grade is to increase many times in the coming years. There is high demand for manganese in different industries, especially in steel industry and owing to this high demand there is a need for high supply of usable manganese. The demand for Manganese in various sectors is expected to increase by many folds and may reach upto 30 million tons by 2020. Considering the changed situation, increasing demand by the growing domestic production of steel coupled with limited reserves, it has become imperative to adopt beneficiation process to upgrade the low grade manganese ore to achieve higher recovery. The concentrates can be agglomerated for further processing. It may be pointed out that unless timely action is taken, the availability of high grade ores required for ferro-manganese production in future may pose problem. And thus it has become essential to explore the low grade Manganese ore reserves. The compelling factors for the early setting up of beneficiation and agglomeration plants are:

- a) Increasing domestic demand and additional requirements for the expansion of the existing steel plants and setting up of new plants.
- b) The present resources are getting depleted rapidly and thus the existing resources should be handled in such a way so as to increase the life of the present resources.
- c) Although there is high grade manganese available in our country but its high time that measures are taken to exploit low grade manganese deposits which are present in large quantities in our country.

Ores contain a certain percentage of gangue. So if the gangue could be separated from the lumpy ores, manganese content is expected to increase. Also from the fines if the concentrate could be separated out by beneficiation methods, it would be expected to increase the manganese percentage in the concentrate samples. By varying the different parameter levels during jigging and tabling or any other beneficiation method, the optimized parameter levels could be found out from the characterization data which yields maximum manganese content amongst the given methods and their respective levels.

Reduction of manganese ores are strongly affected by the reduction temperature, time and reductant percentage. Carbothermal or Aluminothermic reduction can be carried out and the composition of manganese ore helps us to calculate the stoichiometric amount of reductant required. The quality of reduction can be analyzed with the help of extent of reduction and calculating the degree of metallization. The characterization of the reduced samples can further help us determine the phases present.

Reduction followed by magnetic separation would help to separate the magnetic phases from the non magnetic phases. This will in turn help to increase the Mn/Fe ratio. The Non magnetic parts can be further subjected to smelting so as to produce Iron-Manganese Alloy or Ferromanganese.

*Chapter 3*

*Methodology*

### **3. METHODOLOGY**

#### **3.1 GENERAL CHARACTERIZATION OF RAW MATERIALS**

- After the initial crushing in primary and secondary crusher and pulverizing, some of the sized ore samples was crushed and sieved below 75  $\mu\text{m}$  for performing the general characterization. The samples were characterized by X-Ray Diffraction (XRD) for phase analysis, wavelength dispersive X-Ray Fluorescence for finding out the composition.
- Proximate and Ultimate analysis of boiler grade coal was carried out.
- Thermo gravimetric analysis of the boiler grade coal was performed by Pyris diamond thermo Gravimetric/Differential Thermal Analysis (TG/DTA, Perkin Elmer, Singapore) in Nitrogen atmosphere.

#### **3.2 BENEFICIATION METHODS**

##### **3.2.1 JIGGING**

- The beneficiation of the low grade manganese ore lumps was done using Jigging method. Jigging was carried out for the lumps for three sizes +5/16, +4, -5 to +7. For each of these three sizes, 3 motor speeds were selected viz 1100 rpm, 1450 rpm, 1800 rpm.
- Initially 100 grams of the samples were subjected to the jigging. Each of the experiments was carried out for 15 minutes. The wet samples were then collected from the bed and dried by heating in a heater at 100 degree celsius for one hour. The dried samples were then weighed and the percentage weight loss was then found out.

##### **3.2.2 TABLING**

- The beneficiation of the low grade manganese ore fines was done using Tabling method.
- For Tabling, 2 size range (+25 to +150 and -150 to +200), 2 volume flow rates (4 L/min and 6 L/min) and 2 angles ( $4^\circ$  and  $8^\circ$ ) were selected and for each of the experiments, concentrate and tailings were separated.
- Weights of all the collected samples of concentrates and tailings were found out.

### **3.3 CHARACTERIZATION OF JIGGING AND TABLING SAMPLES**

- Samples of jigging and the concentrate samples of tabling were reduced in size and sieved below 75 $\mu$ m and prepared for wavelength dispersive X-ray Fluorescence (WDXRF).
- From the results of the XRF, the best samples with highest manganese content after beneficiation were found out for the two methods selected for beneficiation purpose, jigging and tabling.

### **3.4 REDUCTION OF THE BEST SAMPLES**

- The samples with maximum manganese percentage from jigging and tabling were selected for reduction.
- The samples were reduced under two variables: 2 temperatures : 1100 and 1200<sup>0</sup>C, 2 times : 60 min and 90 min.
- The samples were subjected to reduction with boiler grade coal in tube furnace in a closed environment using an inconel as in direct reduction process.
- After all the reductions were carried out, extent of reduction (EOR) was calculated.
- Chemical analysis was then carried out to evaluate the degree of metallization of the samples reduced under optimized conditions.

### **3.5 MAGNETIC SEPARATION**

- The samples with the highest E.O.R were selected for magnetic separation. Two samples were selected each from reduced jigging samples and tabling samples.
- A hand magnet of 20 mT was used for separation purpose. The magnetic phases got attracted by the magnet and the non magnetic phases were separated.
- Weights of the magnetic and non magnetic parts were noted and the weight percentage of magnetic and non magnetic parts was calculated.

### **3.6 SMELTING**

- After magnetic separation and characterization of the jigging and tabling samples, the nonmagnetic parts of the samples were further taken for smelting. Since both the tabling and jigging samples after the reduction contained primarily MnO phase and

some Fe phase so all the samples were mixed together and subjected to smelting so as complete the reduction of MnO and produce Fe-Mn alloy as product .

- The non-magnetic part was mixed homogeneously with BaO<sub>2</sub> and dried at 373K for 2 hours in a closed oven to drive away the absorbed and combined water
- The dried mixture was mixed with aluminium powder, 2% BaO<sub>2</sub> and 3% CaO. The final mixture was poured in a clay crucible. The whole material was heated at 873K for 30 minutes in Raising hearth furnace and temperature should be lower than the fusion point of aluminium (923K) in order to prevent the boiling up the charge before the final mix.
- To start the process, magnesium ribbon was used as an igniter. The magnesium ribbon of 3 feet long was taken for the generation of heat up to 2573 K which helps to melt the sample and allows the reaction to occur. 8 pieces of 0.5 inch long are cut and mixed with the pre heated sample. The rest of the ribbon was burnt at one end and slowly put into the crucible for burning the sample and produce Iron Manganese alloy.

The detailed methodology of the work is given in the flowchart in figure 3.1:

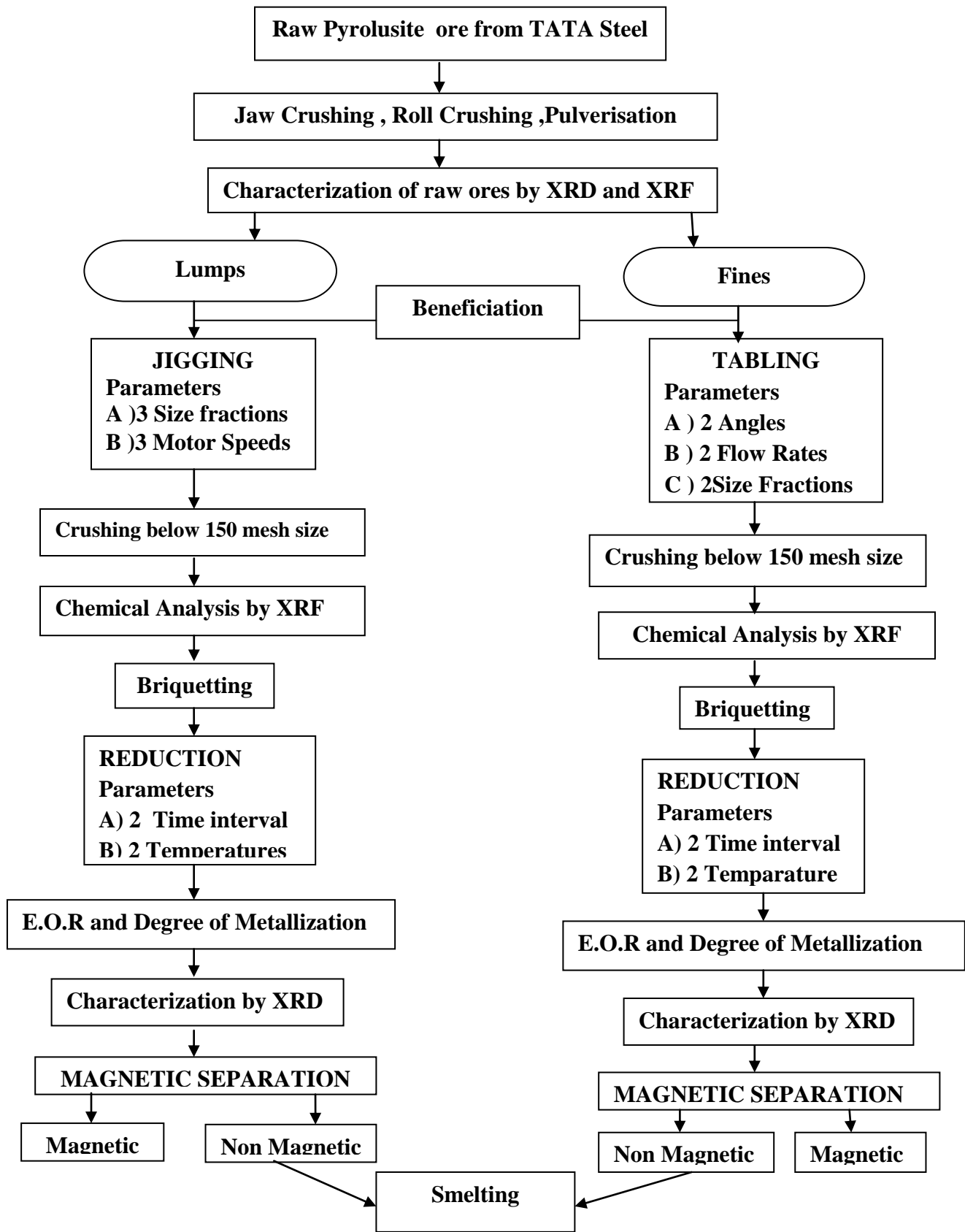


Fig 3.1:Flowchart of Methodology



*Chapter 4*

*Literature Review*

#### 4.1 DIFFERENT MANGANESE ORES

Manganese ores occur in almost all rocks including granite, limestone and clay. The minerals containing manganese in commercially important quantity include the oxides, the magnates, the carbonates, the silicates and other sources of metal being manganiferous iron ores, manganiferous zinc ores and manganiferous silver ores. Of the true manganese ores, the most important for metallurgical purposes are the oxides. The important manganese minerals, its composition, specific gravity, hardness and manganese content are given below in table.

Mineral	Color	Specific Gravity	Hardness (Mohs' Scale)	Composition	Mn Content
Pyrolusite	Black	4.8-5.6	2.0-2.5	MnO <sub>2</sub>	63.2%
Manganite	Steel-grey	4.2-4.4	3-4	Mn <sub>2</sub> O <sub>3</sub> ·3H <sub>2</sub> O	60.4%
Braunite	Brownish black	4.7-4.9	6-6.5	Mn <sub>2</sub> O <sub>3</sub> +SiO <sub>2</sub>	69.6%
Psillomelane	Steel-grey	3.7-4.7	5-6	MnO <sub>2</sub> +MnO+BaO+K <sub>3</sub> O+ <sup>^</sup> H <sub>2</sub> O	45-60%
Rhodochrosite	Pink	3.3-3.6	3.5-4.5	MnCO <sub>3</sub>	47.8%
Rhodonite	Pink	3.5-3.7	5.5-6.5	MnO-SiO <sub>2</sub>	41.9%
Hausmannite	Brownish black	4.7-4.8	5-5.5	Mn <sub>3</sub> O <sub>4</sub>	72.0%
Manganese glance		3.9-4.1	3.5-4	MnS	63.2%
Wad or "bog manganese"		3.0-4.3	1-6	Impure earthy mixture of hydrous manganese oxide	5.5%

**Table 4.1 : Common Manganese Minerals and its Properties[3]**

Description of the different manganese ores are given below [3] :

- a. **MANGANITE**-Manganite is a mineral composed of manganese oxide hydroxide,  $\text{MnO}(\text{OH})$ , crystallizing in the monoclinic system (pseudo-orthorhombic). Crystals of manganite are prismatic and deeply striated parallel to their length; they are often grouped together in bundles. The color is dark steel-grey to iron-black, and the lustre brilliant and submetallic. The streak is dark reddish brown. The hardness is 4, and the sp.gravity is 4.3.
- b. **BRAUNITE** - Braunite is a silicate mineral containing both di- and tri-valent Mn ore with the chemical formula  $\text{Mn}^{2+}\text{Mn}^{3+}_6[\text{O}_8|\text{SiO}_4]$ . Common impurities include iron, calcium, boron, barium, titanium, aluminium, and magnesium. It occurs as impregnation and massive bodies and characterized by black to brownish black colour streak. Braunite forms grey/black tetragonal crystals and has a Mohs hardness of 6 - 6.5.
- c. **PSILLOMELANE**-It is a group name for black manganese oxides including hollandite and romanechite. Psilomelane consists of hydrous manganese oxide with variable amounts of barium and potassium. Psilomelane is erroneously, and uncommonly, known as black hametite, despite not being related to true hematite, which is an iron oxide. The mineral is readily distinguished from other hydrous manganese oxides (manganite and wad) by its greater hardness 5 to 6; the specific gravity varies from 3.7 to 4.7. The streak is brownish black and the fracture smooth. Owing to its amorphous nature, the mineral often contains admixed impurities, such as iron hydroxides. It is soluble in hydrochloric acid with evolution of chlorine gas.
- d. **RHODOCHROSITE**-Rhodochrosite is a manganese carbonate mineral with chemical composition  $\text{MnCO}_3$ . In its (rare) pure form, it is typically a rose-red color, but impure specimens can be shades of pink to pale brown. It streaks white, and its Mohs hardness varies between 3.5 and 4. Its specific gravity is between 3.5 and 3.7.
- e. **RHODONITE**-Rhodonite is a manganese inosilicate,  $(\text{Mn}, \text{Fe}, \text{Mg}, \text{Ca})\text{SiO}_3$  and member of the pyroxenoid group of minerals, crystallizing in the triclinic system. It commonly occurs as cleavable to compact masses with a rose-red color often tending to brown because of surface oxidation. Rhodonite crystals often have a thick tabular habit, but are rare. It has a perfect, prismatic cleavage, almost at right angles. The hardness is 5.5–6.5, and the specific gravity is 3.4–3.7; luster is vitreous, being less frequently pearly on cleavage surfaces.

- f. HAUSMANNITE- It is a complex oxide of manganese containing both di- and tri-valent manganese. It belongs to the spinel group and forms tetragonal crystals. Hausmannite is a brown to black metallic mineral with Mohs hardness of 5.5 and a specific gravity of 4.8.
- g. BOG -MANGANESE- In general this cannot be considered as a true manganese ore. It is a mixture of oxides of Mn with oxides of cobalt and copper and sometimes silver. It is amorphous earthy softy and friable. It is not so valuable as pyrolusite or psillomelane.
- h. PYROLUSITE- Pyrolusite is a mineral consisting essentially of manganese dioxide and is important as an ore of manganese .The sp.gravity is about 4.8. It the chief ore of manganese.

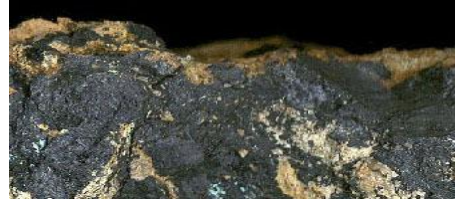
Pictures of the different ores are given in the figures below:



**Fig 4.1(a) :Manganite Fig 4.1(b) : Braunite**



**Fig 4.1(c) :Rhodonite Fig 4.1(d) : Rhodochrosite**



**Fig 4.1(e) :Hausmannite****Fig 4.1(f) : Psilomelane**



**Fig 4.1(g) :Pyroslusite**

## **4.2 COMMON UNIT OPERATIONS FOR BENEFICIATION OF MANGANESE ORE**

In recent years, there is a lot of development taking place in manganese ore processing across manganese ore producing countries in the world. The emphasis is on development of cost effective flow sheets to beneficiate the low-grade manganese ores to produce concentrates suitable for sinter making that is to be used for manganese-alloys. Some of the development features in the processing equipment side are use of heavy media cyclone, jigging, innovations in spiral concentrator, Floatex Density Separator, hydro-cyclone, stub-cyclone, autogenous grinding, column flotation, high gradient magnetic separators (HGMS), fine size screening, etc. Some of the common unit operations applicable for manganese ore processing are discussed below [4] :

#### **4.2.1 WASHING & WET SCRUBBING**

This process is very primitive and widely used in lumpy manganese ore processing to dislodge soft & friable clay, shaly materials, etc. adhered to the lumpy ore. The scrubbing practice is also helpful in hard and porous manganese lumps, which are invariably cavity/pores filled with goethite/limonite or lateritic clayey material, for their substantial elimination.

#### **4.2.2 GRAVITY CONCENTRATION**

This technique is deployed if valuable & heavy iron minerals are free from associated light gangue minerals or waste rock. The common manganese minerals have usually high, specific gravity (Pyrolusite: 4.8-5.6, Manganite: 4.2-4.4, Braunite: 4.7-4.9, Psillomelane: 3.7-4.7, Hausmannite: 4.7-4.8) as compared to the most commonly associated gangue minerals like, quartz & chert (2.65), calcite/limestone (2.70-2.75), clay/shale (2.65), gibbsite (2.67).

Effectiveness of gravity concentration depends on proper feed preparation which includes, crushing, screening & grinding to liberation size and also to ensure feed of a proper size to a particular unit operation (machine), removal of slimes which affects the separation efficiency of the machine as it enhances viscosity of the pulp and hampers proper sizing of crude fractions before subsequent treatment.

**4.2.2.1 HEAVY-MEDIA SEPARATION (HMS) :** In heavy-media separation (also called sink-and-float separation), the medium used is a suspension in water of a finely ground heavy mineral (such as magnetite or arsenopyrite) or technical product (such as ferrosilicon). Such a suspension can simulate a fluid with a higher density than water. When ground ores are fed into the suspension, the gangue particles, having a lower density, tend to float and are removed as tailings, whereas the particles of valuable minerals, having higher density, sink and are also removed. The magnetite or ferrosilicon can be removed from the tailings by magnetic separation and recycled.

The process is used for coarse ore in the size range of -50+3 mm . Media loss is the largest single cost factor in HMS. Feed for the HMS should be hard & compact hematite with non-porous gangue mineral and are thoroughly washed to clean slimes before its use. In general, porous ores accounts for substantial loss of media (extent of 1 to 1.5 kg/t of ore treated) as cavities/pores of ore is filled up with the medium.

**4.2.2.2 HEAVY-MEDIA CYCLONE (HMC):** The process is used for fine ore. The cyclone type separator utilizes centrifugal as well as gravitational forces to make separation between ore and gangue minerals. The centrifugal force makes it possible to bring about separation at a specific gravity lower than that required in the conventional separator. Ferrosilicon in water is used as a media in cyclones. A parting density of 3.2 (max.) can be maintained successfully.

**4.2.2.3 JIGGING:** In the process called jigging, a water stream is pulsed, or moved by pistons upward and downward, through the material bed. Under the influence of this oscillating motion, the bed is separated into layers of different densities, the heaviest concentrate forming the lowest layer and the lightest product the highest. Important to this process is a thorough classification of the feed, since particles less than one millimetre in size cannot be separated by jigging.

**4.2.2.4 FLOATEX DENSITY SEPARATOR:** This is a very high capacity hindered settling classifier and is designed to treat particles below 1 mm size. It can be used for concentration, pre-concentration or compartmentalization of particles of different specific gravity mineral.

**4.2.2.5 CYCLONES:** Cyclones comprehend the entire gamut of devices from hydro-cyclone, stub cyclone and heavy media cyclone (HMC) which offer a wide array of benefits. They offer the cutting edge technology for cost reduction. The use of cyclones with their simple construction, smaller size and low cost, high capacity per unit area, flexibility in operation, can be used for pre-concentration of iron ore mineral below 3 mm size. These cyclones would play an increasingly important role in Indian manganese ore industry either as classifier/dewatering devices or as a concentrator (stub cyclone).

### **4.2.3 MAGNETIC CONCENTRATION**

The common manganese mineral in the country are feebly-magnetic. Hence, separation of valuable magnetic manganese minerals from that of non-magnetic associated gangue minerals like quartz, amphiboles, barite, apatite, calcite etc., can be achieved by exploiting the difference in magnetic properties. The magnetic separators are classified into low and high intensity/gradient magnetic separators that can be operated wet or dry circuit.

**Low Intensity Separators (LIMS):** These separators are designed with a magnetic intensity of 1200 gauss in the separation zone to recover highly magnetic jacobsonite minerals from feebly magnetic manganese ore.

**High Intensity Separators:** These separators are designed with a magnetic intensity of 7000 to 20,000 gauss in the separation zone to recover feebly magnetic minerals of manganese from those of non-magnetic silicate gangue. Dry separators (DHIMS) can be used for concentrating manganese ore, but the ore should be bone dry and sized. Wet high intensity magnetic separators (WHIMS) are generally used for treating manganese ores at finer sizes.

#### **4.2.4 FROTH FLOTATION**

Froth flotation is a process for selectively separating hydrophobic materials from hydrophilic. This is used in mineral processing, paper recycling and waste-water treatment industries. It has been described as "the single most important operation used for the recovery and upgrading of sulfide ores". The development of froth flotation has improved the recovery of valuable minerals, such as copper- and lead-bearing minerals. Along with mechanized mining, it has allowed the economic recovery of valuable metals from much lower grade ore than previously. The process of flotation is applicable for fine liberation mesh mineral for removal of gangue mineral rejected for value addition of manganese ore are siliceous and ferruginous gangue and phosphorus impurities. Siliceous gangue includes quartz, shale, clay, feldspar and other host rock impurities, while ferruginous impurities accounts for limonite/goethite & hematite mineral. Presence of apatite minerals accounts for phosphorus constituents.

Siliceous gangue constituent can easily be rejected at various sizes deploying conventional gravity concentration technique of HMS, Jig, Spiral, Table etc., followed by high intensity magnetic separation (dry/wet).

Alumina contributed by softer clay and harder altered feldspar. Removal of clay by impaction or compaction activities also accounts for loss of pyrolusite along with clay minerals in finer fractions. To this extent it is expected that some manganese losses will occur during the process of mechanical washing physical processes. The possible applicable processes identified for manganese ore are classification, washing, hand sorting or HMS at coarser size followed by jig, spiral or table of finer size fraction.

Ferruginous gangue are difficult to process as both the iron and manganese bearing minerals have similar physical properties like specific gravity and magnetic susceptibility even after liberation and they cannot be separated by simple physical beneficiation techniques.

Thus, such ore requires conversion of iron bearing minerals of limonite/goethite and hematite to magnetite by reduction roasting, which then can be separated by low intensity magnetic



separation at which the manganese minerals are non-magnetic. High phosphorous manganese ores contain invariably the apatite as main phosphorus bearing mineral that can either be floated with fatty acid collector or subjected to WHIMS for its rejection in the non-magnetic fraction.

### **4.3 NEED FOR PROCESSING**

Mineral, consuming industries demand manganese ores in a tailor made format i.e., meeting physical, chemical and in very rare cases mineralogical stipulations. Manganese ore is invariably used for metallurgical processes (iron making in blast furnace as well as manganese-based alloys making). In the Indian context, the common gangue associated with manganese ores are metallic minerals, slag forming minerals and deleterious sulphur & phosphorus bearing mineral. In many cases, presence of these impurities beyond desired limits render the ore unsuitable for use in iron or manganese alloy making (blast furnace/EAF). The common gangue associated with manganese ore and its adverse effect in manganese alloy making is detailed below [4]:

#### **4.3.1 METALLIC GANGUE**

Generally, these impurities report along with manganese metal during production process. Iron is the most common gangue contributed by iron minerals like hematite, goethite, limonite and manganese mineral like jacobsonite. The other metallic gangue contributors are Pb, Zn, Cu, W, Ni, Ti & Ag compound and always present in minor trace amounts. Except zinc which is readily volatilized, remaining impurities are retained in the metal by a process of reduction during smelting. Reducing efficiency of iron oxides during ferro alloy production is around 95% whereas that of manganese oxide is 70 to 75%. This means iron oxides are the first to reduce during smelting of ferromanganese which ultimately dictates the grades (%Mn) of manganese-based ferro alloys. Hence, higher Mn/Fe ratio or lower iron content is preferred.

#### **4.3.2 SLAG FORMING GANGUE**

The slag forming constituents in the ore bring down its value. Acid insoluble are contributed by this type of gangue and these may be basic such as lime, magnesia & barite and acidic as silica or alumina. Lesser silica means higher Mn/SiO<sub>2</sub> ratio. Lesser silica results in less slag formation during ferromanganese production resulting in less manganese loss in slag and reduced electricity consumption. The power consumption for ferromanganese production depends on the slag volume and temperature of slag which in turn depend on silica content of the manganese ore used. It is therefore important to have low silica in manganese ore for

ferromanganese smelting. Every 1% increase in silica content increases manganese ore requirement by 60 kg and power consumption by 60 kw/tonne ferromanganese produced.

### **4.3.3 PHOSPHORUS & SULPHUR GANGUE**

Sulphur is less dangerous than phosphorus since in normal furnace operation the sulfur passes away (into slag combining with manganese or lime) and only traces enter the alloy, whereas phosphorus passes completely to the alloy. The sulfur is contributed by sulfides including base metal. Phosphorus is contributed by discrete mineral phase like apatite and also chemically combined with manganese as a separate mineral i.e. Gorexite. The presence of arsenic is also harmful for certain industrial use of manganese. The manganese-based alloys are used as de-oxidizers in steel making. Phosphorus present in manganese alloys which is inherited from manganese ore gets dissolved in steel, results in cold shortness i.e., development of cracks in steel during cold working.

### **4.3.4 MANGANESE CONTENT**

The cost structure of the manganese alloy production indicates that manganese ore dominates the cost component to the tune of 40%, thereby emphasizing the need for availing the benefits of using the high-grade ores. For the same manganese units input for alloy production, the low-grade ore requirement is about two-times that of high-grade ores and electrical consumption is one and half times. In addition, the performance of low grade ore in alloy production is substantially inferior to that of high-grade ores. It increases input cost, results in inferior quality end product and increases slag production thereby making the slag disposal an issue. On the other hand, the slag resulting from high grade ores is much rich in MnO content (35-38%) which is used for silicomanganese production. Thus, the higher grade ores significantly reduce the cost, slag and improve the grade of the alloy products which requires up-gradation of manganese content. Ferromanganese grade ore reserves are limited in the country. Acute shortage is being faced by the ferromanganese industries due to limitations of domestic manganese ore supplying companies. This has resulted in steep rise in the prices of manganese ore due to large demand supply gap. The grade of the manganese ores has deteriorated globally with manganese content falling and phosphorous and silica increasing alarmingly even in high-grade manganese ores.

In the present Indian scenario, manganese input is predominantly through manganese alloy in iron & steel making and hence value-addition of BF and mixed grade ore (>25%Mn) in

the country is all the more necessary. Besides, optimum utilisation of locked-up valuables in low-grade, sub-grade/marginal-grade ore resources and fines are essential for survival of the industry and growth which can be achieved only through implementation of appropriate beneficiation technology.

#### **4.4 BENEFICIATION METHOD TO BE ADOPTED**

In this thesis we have chosen two beneficiation routes by two gravity separation methods jigging and by tabling. So we will focus on these two methods and the possible parameters that can be varied in the experiments.

##### **4.4.1 JIGGING**

Jigging is a process of ore concentration carried out in any fluid whose effectiveness depends on differences in specific gravity of granular mineral particles. It consists of separation of the particles into layers of different specific gravities followed by the removal of the separated layers. Jig concentration is different from other types of gravity concentration such as heavy media separation, where the separation is done directly. Stratification of bed particles in a jig concentration is achieved gradually, and the separation into products is realized after a certain time. Though jigs are simple in operation, the jigging process as a whole is rather complicated and strongly influenced by several interrelating factors (interacting variables or parameters). These factors are directly associated with one or more of the subsystems into which the jigging process can be divided. The principal subsystems are the jig itself and the drive unit providing a pre-designed stroke pattern, as well as, feeding, feed distribution, evacuation of strata, and conveyance from jig mechanisms.

In the paper “Physicochemical processing of low grade ferruginous manganese ores” [5] the authors focused on the developing of a cost effective physicochemical beneficiation process for low grade ferruginous manganese ores to recover a valuable iron bearing by-product (Fe Oxalate or Mn Ferrite) and manganese ore concentrate. It was found the one molarity solution of oxalic acid can leach five times more Fe than Mn from ferruginous manganese ores during treatment at temperature 333 K for 2 h

“Optimization of jigging process using statistical technique” [6] was carried out for jigging studies of a synthetic binary mixture of quartz and coal were carried out. The variables studied were the particle size, bed thickness, number of strokes per minute, and water level. The jigging studies of a synthetic binary mixture of quartz and coal were carried out. The variables studied were the particle size, bed thickness, number of strokes per minute, and water level. Experiments were carried out using 24 full factorial design. The main and interaction effects on quartz recovery were evaluated using Yates' analysis. The optimum jigging condition was found by the method of steepest ascent.

In the paper “Effective Beneficiation of Low Grade Iron Ore Through Jigging Operation”[7]. The samples were subjected to jigging in a laboratory Denver mineral jig and Harz jig. The operating variables used to determine the effectiveness of jigging include, particle size, velocity of water and amplitude. Recovery of iron values and the separation efficiency were assessed by determining mineralogy and the percentages of Fe in the jig concentrate and tailings by wet chemical and X-ray fluorescence (XRF) techniques.

The **second mechanism** of separation is ‘Hindered Settling’ which occurs during the pulsation stroke and leading into suction stroke. After the initial differential acceleration, the fluid flow velocity during the pulsation stroke increases and the Drag force ( $F_D$ ) increases. If the percent solids in a system are high such as particle bed in a jig, hindered settling is prevalent over free settling. In hindered settling the particle experiences resistance due to increased turbulence, particle particle interactions and particle wall interactions. Such settling enhances separation based on particle density rather than particle size.[8]

In the free settling of mineral particles in a liquid, the falling particles are at a distance from each other so that no particle is affected by its neighbor. In hindered settling, the concentration of particles is sufficiently high so that each particle is affected by its proximity to other particles in the suspension. It is a phenomenon where particles of mixed sizes, shapes and densities in a crowded mass, yet free to move along themselves, are sorted in a rising current of water, the velocity of which is much less than the free-falling velocity of the particles but yet fast enough so the particles are in motion. This is the condition normally encountered in mineral concentration processes. The concentration of particles in the fluid thus imparts an apparent density to the composite fluid or suspension greater than that of the liquid alone, resulting in a buoyant effect on the larger particles. Particle shape affects the settling rate of both coarse and fine particles. The general effect is to reduce their settling velocities and the effect is greater for

coarse particles and for those settling under hindered settling conditions than for fine particles or free settling ones.[9]

The final mechanism of separation is 'Consolidation Trickling' wherein the suction stroke pulls in fine, heavy particles through the void spaces between the void spaces of larger particle bed on top of the screen. Consolidation Trickling separates particles mainly based on size. The size ratio (i.e. ratio of fine to coarse particles,  $d_f/d_c$ ) determines the particle size limit for consolidation trickling.

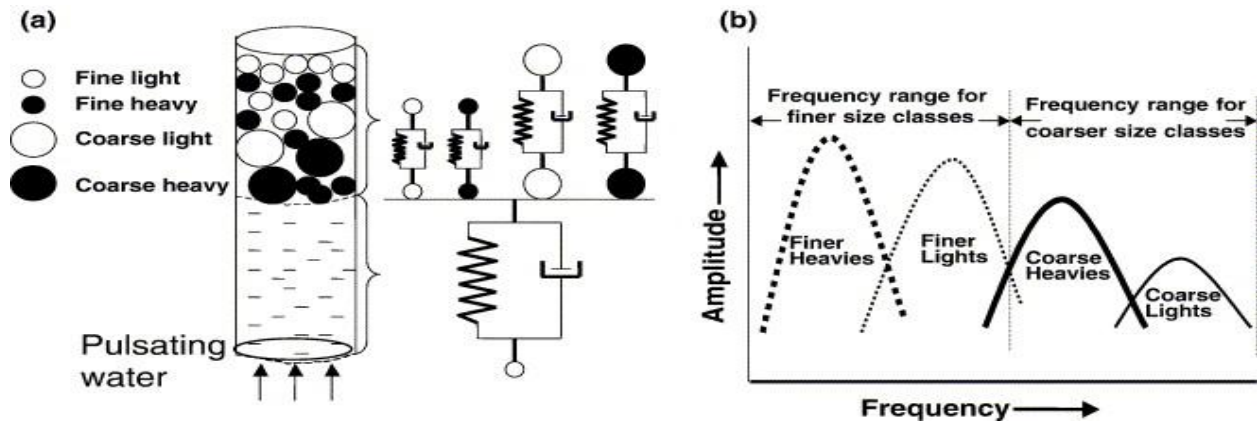
Jigging is a very complex process because of the various interrelating variables and parameters. Large number of variables makes it difficult to develop a mathematical model for jigging. Here we will focus on some of the major parameters in jigging.

**A) Pulsation rate (frequency) and stroke length** – These are the two main parameters because they control the time of each stroke. For a plunger type jig, the magnitude and direction of fluid flow is given by a sinusoidal function. The fluid flow in positive direction gives pulsation stroke and fluid flow in negative direction gives suction stroke. The time required for each pulsation or suction stroke is given by the equation,  $T = 1 / [2(N/60)]$  where N is total number of pulsations per minute and T is time of a single pulsation or suction stroke. Pulsation rate plays a major role in determining the velocity of water. The stroke length determines the maximum velocity of water and it is defined as the distance travelled by the plunger between the highest and lowest position. Consider a feed with large size feed with large density difference between light and heavy particles. For such feed low pulsation rate with high stroke length would be required to take advantage of the hindered settling. Again if we consider a feed with finer size where the density difference is lot narrower, here higher pulsation rate would be required with smaller stroke length to increase separation from differential acceleration.

Thus pulsation rate and stroke length should be optimized to obtain good separation. [8]

**B) Role of jig frequency** - Consider a system where particles and the water are represented by independent pairs of springs and dashpots. The natural frequency of these four types of particles depends on their respective spring stiffness and mass where, natural frequency =  $\sqrt{k/m}$  of the particle, k and m are the spring stiffness and mass of the particle. The segregation in the jig bed is due to the frequency of the particles. Analysis suggest that particles of coarser size due to higher voidage around them moderately contribute to bed expansion (gets fluidized less due to low drag force). Thus coarser particle are more stiff than fines and thus have greater natural

frequency than fines. Amongst coarser or fine particles, lighter particles always will have higher frequency.



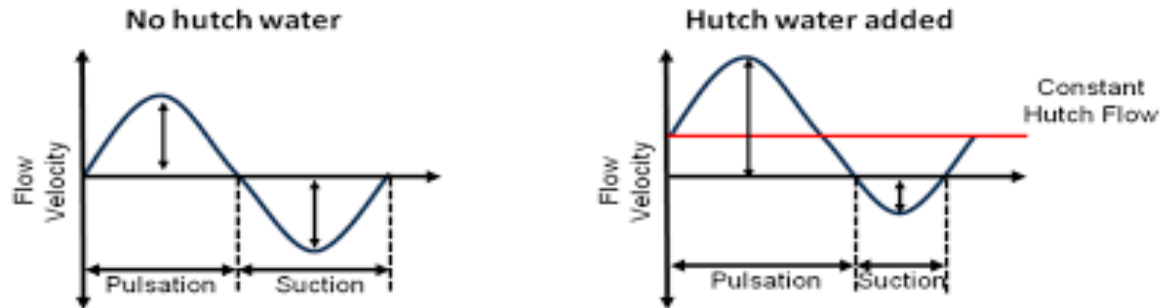
**Fig 4.2 a: Effect of Size and weight of particles on stiffness[8]**

**Fig 4.2 b: Variation of frequency with amplitude depending on size and weight of particles [8]**

Thus lighter particles in any coarser size class are likely to resonate at higher frequencies and get separated easily from their heavier counterpart in response to a particular frequency of pulsation. Such situation will lead to density classification with higher separation efficiency of the coarser size class. If the applied jig frequency is close to the natural frequency of the coarser light particles, coarser feed shows higher separation efficiency. Frequency of water pulsation helps in segregating the coarser lights to the top of the jig bed.

Therefore in a jigging system first, frequency of water pulsation is optimised based on the particle size, and next, the amplitude of water pulsation is adjusted to achieve the desired maximum water velocity[8]

**C ) Increasing upward flow by water addition (water velocity) –** In jigging it is common practice to add some water which help in fluidizing the particles especially for coarse narrowly sized particles which compact tightly during the suction stroke preventing separation by consolidation trickling. Addition of water increases the magnetude and duration of the pulsation stroke by the velocity of water by which water is rising through the jig bed and decreases the magnetude and duration of suction stroke.[8]



**Fig 4.3 : Effect of Hutch water on flow velocity[8]**

Thus increase in duration of pulsation stroke means decrease in pulsation rate which would indicate increased stroke length. Two main results from different studies on the dependence on water velocity are:

First, the separation efficiency increases with increase in maximum water velocity and reaches a maximum value and then decreases; Second, for best separation the coarser feed requires a higher maximum water velocity. The latter finding essentially relates the role of maximum water velocity with feed characteristics.

#### **D) Role of feed characteristics on particle separation**

**1) Effect of volume fraction-** Volume fraction is defined as the ratio of volume of that particle size to the total volume of all particle sizes in the feed. Size ratio and volume fraction of the particle types together control voidage in any binary system. Voidage controls drag force and consequently the particle segregation starts during fluidization. Three different feed samples were prepared in the same size range with three sizes present in each feed but the quantity of one size was always more than the other two sizes in each feed.

According to the research by B.K.Mishra and B.Adhikary, December 1999, the test was carried out at different amplitudes with frequency set at optimum level. Results show that feed sample with highest proportion of fines show much better separation efficiency at lower amplitude of jig. The fine dominated feed has less voidage and therefore it experiences higher drag and gets fluidized at lower amplitude. This creates a fluidization environment suitable for particle segregation for all size classes including the finer size class. Feed-B and C require higher amplitude because they have lesser proportion of fines.

**II ) Effect of size of particle** – For the above test with the feed samples prepared it could be seen that separation efficiency depends on the top size of each feed sample which controls the maximum water velocity of the of the jig. So the particle of the top size class decides the maximum water velocity for optimum separation. A feed sample with majority fines would require much lower maximum water velocity than a sample with majority coarser particles for optimum separation.[8]

Jig operates in a cycle which may be considered consisting of four stages, namely, inlet, expansion, exhaust, and compression. In the inlet stage the bed lifts up en masse. Near the end of the lift stroke the particles at the bottom of the bed start falling resulting in the loosening of the bed which, in turn, causes its expansion or dilation. During the final stages of jig cycle, the particles resettle through the fluid, and the bed collapses back to its original volume. The main forces that act in fluid dynamics are drag force and buoyant force.[10] The pulsation and suction is repeated to bring about stratification with respect to specific gravity across the bed height. The jigging is a separation process in which a mineral bed is pulsated by a current of water resulting in stratification of mineral particles of different specific gravity. Thus it exploits the stratification of particulate matter under the influence of hydrodynamic and gravity forces. Jig offers economic advantages over heavy media separators owing to its simple design and operational features. [11]

#### **4.4.2 TABLING**

Research was done on “Influence of Shaking Table Process Parameters On Concentration Of Chromite Plant Tailings”[12]. In this paper they have selected a typical chromite beneficiation plant tailing of Sukinda region has investigated by using wet shaking table for the effective utilisation of the natural resource. It was observed that as the wash water flow rate increases, there is an increase in the grade of the concentrate fraction at lower level of feed flow rate and at higher level of feed flow rate and vice versa. As the wash water flow rate increases, the transport of the gangue minerals to the tailing fraction increases which in turn improves the grade of the concentrate fraction. The higher grade of the concentrate fraction is obtained at higher level of both deck tilt angle and feed flow rate. It was also noted that at lower level of deck tilt angle, as the feed rate increases there is decrease in the quality of the concentrate fraction but at higher deck tilt angle, it was vice versa.[12]



Research was done on “ The Shaking Table Concentrator The influence of Operating conditions and Table parameters on mineral separation- The development of a mathematical model for normal operating condition”[13]. They worked on the effects of several variables on the performance of a shaking table have been investigated on a laboratory-scale Wilfley table using a synthetic mixture to represent a typical industrial metallic ore. The following main conclusions were drawn from the study:(i) Feed pulp density should be kept below 35% solids.(ii) A minimum level of wash-water is required to effect separation.(iii) There is an optimum deck inclination for any given material.(iv) Increasing a particular density and size fraction of the feed, causes a corresponding increase in the band width of this material at its discharge point.[14]

Shaking tables are widely used in commercial mines but have found little use by small-scale miners due to their relatively high cost [15]. They utilize both particle density and to a lesser extent particle size to fractionate fine ore samples (<1mm) into products, middlings and waste fractions (Mintek 2016). That is, if a milled ore is introduced into this system, it can be expected to undergo natural sorting wherein high-density particles sink to the bottom and lighter ones float to some extent [16]

The shaking or Wilfley table consist of a substantially plane surface (the deck), inclined slightly from the horizontal and shaken with an asymmetrical motion in the direction of the long axis. Asymmetrical motion means, the stroke of the table is faster in one direction and slower in opposite direction. Usually the back- stroke is faster compared to forward stroke[17]. The wash water flows over the table at right-angle to the direction of jog. Longitudinal cleats or riffles are fixed on the table surface in the direction of the table movement. The feed is introduced through the feed box at the upper corner of the table. As the feed hits the deck, it is fanned out by combination of differential motion transversely flowing water. The jolts cause the heavier particles to work down of form the bottom layer and travel in the through formed by the riffles. The lighter gangue materials are thrown into suspension and discharge over the edge of the table opposite the feed box by the flowing wash water. The heavier minerals finally arrange themselves on the smooth unrifled proportion of the table when they encounter the full force of the wash water. The middlings are selected in that corner of the table which is intermediate between concentrate and tailing. The reciprocating (to and fro) speed of the riffle table is usually

200-300 strokes/ minutes with amplitude of 12-15mm. the coarser feed requires larger stroke length.[18]

The points of the compass are used for clarity in describing the operation of the Wilfley shaking table. The feed is screened to <3mm and fed into a small hopper above the north-east corner of the shaking table, where it is mixed with clean water. The resulting slurry is introduced to the north-east corner of the shaking table and begins to spread southwards as a thin film. The feed fan outs towards the edge of the table, allowing the operator to see exactly what is happening, and to decide where to subdivide the fan into distinct streams each dominated by a particular mineral. The shaking motion has a slow westward stroke and rapid return eastward stroke – often with a bump. This induces settled particles to crawl in a juddering manner westward along the table with the thin film of slurry. The shaking is usually very rapid with a frequency of 4 to 5.5 strokes per second. The shaking displacement is usually half to one inch to-and-fro. A set of low riffles aligned east-west guide the heavies ever westward to fall off the south-west corner of the table into a hopper as a continuous discharge. Meanwhile, a spray bar introduces clean ‘wash water’ along the north edge of the table, sending a thin film of clean water southward to encounter the riffles and the westward flowing slurry. The wash water mixes with the slurry and overrides the riffles taking the lighter particles with it to spill over the southern edge as a continuous discharge of tailings. They worked on the mechanism of concentration on the shaking table and tried to obtain the theoretical model describing the velocity of a fluid in depth and time when in contact with a symmetrically reciprocating surface.[19]

Under idealized conditions particles segregate into four groups:-

- (i) Light-large
- (ii) Large- heavy
- (iii) Small- light
- (iv) Small- heavy

From research [13] the effects of several variables on the performance of a shaking table have been investigated on a laboratory-scale Wilfley table using a synthetic mixture to represent a typical industrial metallic ore. The following main conclusions could be drawn from the study:

- (i) Feed pulp density should be kept below 35% solids.
- (ii) A minimum level of wash-water is required to effect separation.
- (iii) There is an optimum deck inclination for any given material.

(iv) Increasing a particular density and size fraction of the feed, causes a corresponding increase in the band width of this material at its discharge point.

Particle shape and size distribution also play important roles as well. Some of the parameters that can influence the grade of the concentrates are briefly explained below:

(i) An increase in the wash water flow rate increases the grade of the concentration fraction at both lower and higher feed flow rates. As wash water flow rate increases, the transport of gangue minerals to the tailings fraction increases which in turn improves the grade of the concentration fraction.[12]

(ii) Higher grade of the concentrate fraction is obtained at higher level of both deck tilt angle and feed flow rate. With a lower level of deck tilt angle, an increase in the feed rate decreases the quality of the concentrate fraction [12]

Advantages of the shaking table :(i) Highly selective, with high upgrading ratio if used correctly. (ii) Able to see separation and make adjustments. [17]

Disadvantages of the shaking table:(i) Low capacity, large floor area requirements.(ii) Require frequent operator attention, checking and adjustments. (iii) Feed should be sized. [17]

#### **4.5. AGGLOMERATION**

Research was done on “Agglomeration And Prereduction Of Ores ” has elaborated on the various agglomeration processes .The success of metallurgical processes greatly depends on the particle size of raw materials used. The size requirements differ from process to process and the desired sizes may range from tenths of millimeters to tens of centimeters. The desired size can be obtained by crushing or grinding if the input material is too coarse, or agglomeration if the input material is much finer. Agglomeration is a process of size ,enlargement; briquetteing,, sintering and pelletising being the three common processes used in the metallurgical industries[20]

Manganese ore beneficiation in India produces a large amount of fines.The concentrate produced by the beneficiation of low grade manganese ore fines cannot be directly used in the smelting

furnaces because of its fineness. So to make the concentrate suitable for furnace use agglomeration is required. The detrimental effect of fines depend on the chemical composition and the size of fines .[21]

**(i) PELLETISING:**

For pelletising, the fines are properly moisturised and made to rotate in a drum or disc with or without the use of a binder to produce spherical balls. The moist balls (green pellets) are hardened to impart strength by firing at temperatures above 1000°C. The advantage of pelletising over sintering is that the pellets have much higher strength and more uniform size. The finely ground manganese ore concentrate is amenable to pelletising and the pellets produced are physically and chemically superior to lump ore. But pelletising of manganese ore is not economically viable, because of higher capital investment and higher operating cost due to fine grinding required for balling and fuel oil required for induration

**(ii) BRIQUETTING:**

It is possible to agglomerate the manganese ore fines/concentrates by briquetting process, but the briquettes produced by using a number of binders, namely molasses, hydrated lime, bentonite, sodium silicate, portland cement etc and their mixtures are not suitable for use as feed material for the smelting furnaces, because of not having adequate heat resistance and handling properties. In the steel industry for agglomeration purposes, these techniques play a vital role.[22]

**(iii) SINTERING:**

In sintering the agglomeration of ore particles is achieved by surface fusion as a result of burning of a solid fuel (Coke fines) added to the charge mix, Sintering is likely to be more economical as it does not require any binder or fine grinding of raw materials. Operating experience in a number of commercial sintering plants spread all over the world has established that the sinter produced from mixtures of manganese ore fines/ concentrates, coke breeze and fluxes is physically and chemically suitable for ferro-manganese production. Further, it may be pointed out that the inclusion of sinter as part of the charge in the production of ferro-manganese considerably improves the operating conditions and performance of the smelting furnaces.

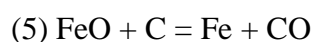
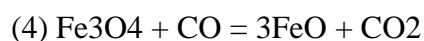
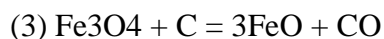
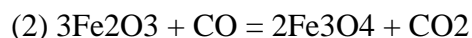
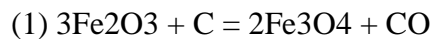
Sintering is the agglomeration technique of fine materials to produce clusters by incipient fusion at higher temperature. In spite of economic importance of agglomeration pre-treatment, its successful application to complex, low grade Ni laterite ores and various other ores is limited.[23]

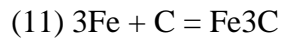
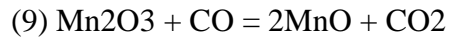
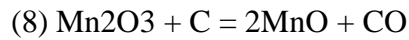
Advantages of sintering are:

- (i) Gainful utilization of manganese ore fines.
- (ii) Improved charge permeability in furnace.
- (iii) Considerable power saving per tonne of manganese.
- (iv) Reduced explosions due to reduction in oxygen level in charge.[24]
- (v) Reduced pollution as fines are major land pollutants.

#### **4.6 REDUCTION**

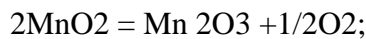
Research was done on “Carbothermic Reduction of Ferruginous Manganese Ore for Mn/Fe Beneficiation: Morphology Evolution and Separation Characteristic”[25]. The results demonstrated that the ferruginous manganese ore reduction can be described as follows: plate-like Fe-Mn symbiotic phase decomposition → granular MnO phase formation → fine metallic iron formation → aggregation of metallic iron → boundary development between Fe and Mn phases.[25] In this paper the authors worked on the beneficiation of Fe and Mn elements from a ferruginous manganese for Mn/Fe Beneficiation: Morphology Evolution and Separation Characteristic ore via carbothermic reduction. The reduction of  $\text{MnO}_2$  and  $\text{Fe}_2\text{O}_3$  follow the reactions and equations mentioned below:[26]





Iron oxides can be reduced to metallic iron above 800 °C. Iron carbide also forms at temperatures higher than 800 °C. The  $\text{Mn}_2\text{O}_3$  transforms to  $\text{MnO}$  spontaneously at this experimental condition. However, elemental manganese forms until the temperature reaches higher than 1400 °C. Therefore, except for reaction 10, the others listed above are actually taking place in the carbothermic reduction process .

According to some results, the most abundant forms of the manganese oxides are  $\text{MnO}_2$  ,  $\text{Mn}_2\text{O}_3$  ,  $\text{Mn}_3\text{O}_4$  and  $\text{MnO}$ . These compounds dissociate during heating.[27]



Therefore, different oxide phases are formed dependent on the temperature and partial oxygen pressure. Reduction of manganese oxides is considered in two steps. The first step is the reduction of oxygen-rich oxides to  $\text{MnO}$  and the second one is the reduction of  $\text{Mn}$  to metallic manganese. Reduction starts with the transformation of  $\text{MnO}_2$  into  $\text{Mn}_2\text{O}_3$  and  $\text{Mn}_2\text{O}_3$  into  $\text{Mn}_3\text{O}_4$  at temperatures over 450°C, then these two phases are reduced by either carbon or carbon monoxide in the system of  $\text{Mn-C-O}$ .

The reduction of  $\text{MnO}$  with carbon monoxide can only be achieved at temperatures over 1430°C at which the ratio  $P_{\text{CO}}/P_{\text{CO}_2}$  is 7400. Since the reduction, if done with carbon monoxide, can only be achieved in the abundance of carbon, at temperatures over 1430°C and at an extremely high carbon monoxide pressure, the reduction of  $\text{MnO}$  with carbon monoxide can not be accomplished in many industrial applications. For this reason, reduction of  $\text{MnO}$  with solid carbon or iron carbide, manganese carbides are also formed.

During ferromanganese production, some parameters such as temperature, time, and charge basicity affect manganese recovery. A previous study showed that an increase in these parameters increased the extent of the carbothermal reduction process, thus increasing manganese recovery [28].

The main role of the carbonaceous reducing materials in the production of ferroalloys is to reduce the oxide compounds such as MnO in the mineral ore, either by the Boudouard reaction or by direct reduction. As it was reported that the Boudouard reaction involving carbon monoxide (Eq. 1) is less important in the reduction of manganese ore, it was replaced by carbon (direct) reduction in the manganese ore smelting process [29]. Thus, the selection of carbonaceous reducing materials plays an important role in ferromanganese production due to its function in direct reduction.



Coke, which is commonly used as a reductant in ferromanganese production, is expensive. Using coal rather than coke as a reductant reduces cost but creates a pollution problem [30]. Today, there is still less information about the use of coal, especially bituminous coal, as a reductant, especially for smelting the medium-grade manganese ore to produce ferromanganese.

#### 4.7 MAGNETIC SEPARATION

After reduction roasting process some of the magnetic mineral compounds will turn into nonmagnetic, or vice versa.  $\text{Fe}_2\text{O}_3$  (paramagnetic) will turn into  $\text{Fe}_3\text{O}_4$  (magnetic), and  $\text{MnO}_2$  (paramagnetic) will turn into  $\text{Mn}_2\text{O}_3$  (paramagnetic),  $\text{Mn}_3\text{O}_4$  (magnetic) and  $\text{MnO}$  (diamagnetic). Thus the main objective of the magnetic separation in the next process is to separate the magnetic iron ( $\text{Fe}_3\text{O}_4$ ) from the manganese oxide as the non-magnetic compound for improving its Mn/Fe ratio.

The magnetic portions need to be separated out of the sample and non magnetic portion is smelted for ferromanganese production.

## 4.8 SMELTING

. The smelting process of this low-grades manganese ore conducted for 60 minutes in three phase submerged arc furnace. The current rate was 1000 A and the pouring temperature was 1500°C.(BambangSuharno et al.). The smelting was carried out in an electric arc furnace.For the reaction  $\text{MnO} + \text{C} = \text{Mn} + \text{CO}$  to take place the temperature required is higher than 1400°C. Thus very high temperature is needed to enhance the formation of metallic Mn from MnO.Limestone was used as flux in the smelting process.Melting point of MnO is 1950 degree Celsius thus for smelting of the pyrolusite ore temperature should be very high.



*Chapter 5*  
*Materials*  
*and*  
*Methods*

## **5. INTRODUCTION**

In order to achieve accurate and precise data it is necessary to use standard equipments. Different kinds of instrument are used in the present study for specific purpose and standard calibrated systems are used for analysis. In this chapter an overview of experimental setup, methods, sample preparation and data collection methods are given.

### **5.1 SAMPLE PREPARATION UNIT**

The ores that are collected from the fields are at first hammered to reduce their size followed by primary crushing in a Jaw Crusher and then secondary crushing in a Roll Crusher. The product from the secondary crusher was then sieved in a vibrating sieve shaker whereby the different size fractions were obtained. The overview of the different equipments used in the sample preparation unit are mentioned below.

#### **5.1.1 JAW CRUSHER**

A jaw crusher uses compressive force for breaking of particle. This mechanical pressure is achieved by the two jaws of the crusher of which one is fixed while the other reciprocates. A jaw or toggle crusher consists of a set of vertical jaws, one jaw is kept stationary and is called a fixed jaw while the other jaw called a swing jaw, moves back and forth relative to it, by a cam or pitman mechanism, acting like lever. The volume or cavity between the two jaws is called the crushing chamber. The movement of the swing jaw can be quite small, since complete crushing is not performed in one stroke. The inertia required to crush the material is provided by a flywheel that moves a shaft creating an eccentric motion that causes the closing of the gap. Jaw crushers are classified on the basis of the position of the pivoting of the swing jaw

1. Blake crusher-the swing jaw is fixed at the upper position
2. Dodge crusher-the swing jaw is fixed at the lower position
3. Universal crusher-the swing jaw is fixed at an intermediate position

A picture of the instrument is given in figure 5.1



**Fig 5.1 : Jaw Crusher**

### **5.1.2 ROLL CRUSHER**

This is an important class of intermediate comminution machine in the intermediate range of size reduction. Crushing rolls consists of pair of heavy cylindrical rolls revolving towards each other so as to nip a falling ribbon of rock and discharge it crushed below rolls. The two rolls are heavy and rigid ones. The material is cast steel and wear resisting. Both the rolls are positively driven towards each other by motors. The heavy rolls turn on parallel horizontal plane having the roll centres at the same height separated by a distance. The feed caught between the rolls are broken by compressive force and drop down below. The rolls turn towards each other at the same speed. They have narrow faces but have large diameter so that they can nip moderately large lumps. A picture of this instrument is given in figure 5.2



**Fig 5.2 : Roll Crusher**

### **5.1.3 VERTICAL SEIVE SHAKER**

This is the most important method of sizing the mineral particles. This is widely used to determine the efficiency of size reduction operations and also used as a yardstick for assessing the fineness of a ground product. As sieve analysis has been the most important method of size

analysis it has become pertinent to discuss about the standard screens or sieves used worldwide for the purpose. Three major sizes of ore particles, coarse, semi-coarse & fine, are screened by this equipment and they are used for experimental purpose. Two set of aperture size are applied for screening. Sizes of -1inch (25.4 mm) to + 4 mm are used for coarse and semi-coarse screening and size of 60 mesh (250  $\mu\text{m}$ ) to 300 mesh (53  $\mu\text{m}$ ) are used for fine screening. Each set is containing of 5-10 sieves with square apertures conforming BSS standard. Each set is completed with a lid and receiver of suitable diameter (10 cm). The sieves are fitted in the shaker frame in increasing order of aperture size, with the receiver (pan) at the bottom most position. During operation, the material lying on the sieves is subjected to a combined rotary and tapping motion which causes the finer particles to trickle down and arrange themselves on the various decks as per their size. A picture of the instrument is shown in figure 5.3.



**Fig 5.3 : Vertical Seive Shaker**

#### **5.1.4 WEIGHING BALANCE**

A weighing scale is a device for measuring weight. Balances measure the mass of an object. In many industrial and commercial applications, scales and balances to determine the weight or mass of things ranging from feathers to loaded tractor – trailers. The basic principle is that weight is laid on one side of the beam. The result is that the coil attached to the other side of the

beam tries to move out of the magnetic field of the magnet . The capacity of the balance is 1000 gm and the readability is 0.01gm. The measuring pan of weighing balance (0.1 mg or better) is inside in a transparent enclosure with doors to suppress the effect of dust and fluctuation due to air & current in the room during operation. This enclosure is often called a draft shield. A picture of the instrument is shown in figure 5.4.



**Fig 5.4 : Weighing Balance**

## **5.2 BENEFICIATION UNIT**

The samples after crushing in to various size fractions were subjected to beneficiation . The lumps were subjected to Jigging and the fines were separated in different size ranges for Tabling.

### **5.2.1 JIGGING MACHINE**

Jig concentrators are devices used mainly in the mining industry for mineral processing, to separate particles within the ore body, based on their specific gravity (relative density). The particles would usually be of a similar size, often crushed and screened prior to being fed over the jig bed. There are many variations in design; however the basic principles are constant: The particles are introduced to the jig bed (usually a screen) where they are thrust upward by a pulsing water column or body, resulting in the particles being suspended within the water. As the pulse dissipates, the water level returns to its lower starting position and the particles once again settle on the jig bed. As the particles are exposed to gravitational energy, those with a higher specific gravity (density) settle faster than those with a lower count, resulting in a concentration

of material with higher density at the bottom, on the jig bed. The particles are now concentrated according to density and can be extracted from the jig bed separately. In the mining of most heavy minerals, the denser material would be the desired mineral and the rest would be discarded as floats (or tailings). A picture of the instrument is given in figure 5.5

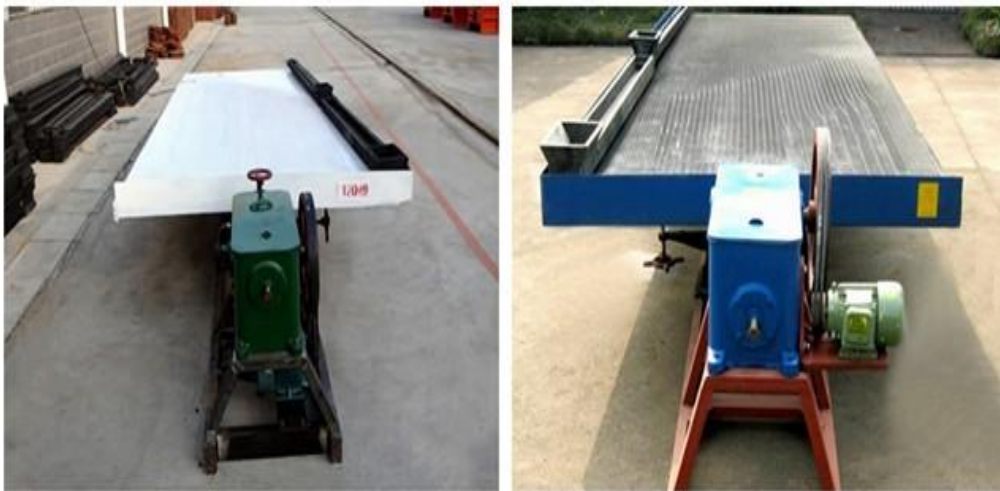


**Fig 5.5 : Jigging Machine**

### **5.2.2 SHAKING TABLE**

A gravity table is a mechanized gold pan, that operates with a high degree of efficiency and continuously. The table is comprised of a deck, in somewhat of a rectangular shape, covered with riffles (raised bars running perpendicular to the feed side of the table), mounted in a near flat position, on a supporting frame that allows the table to slide along the long axis of the table. The mechanism is attached to the table, and it moves the table along the long axis a distance adjustable between ½" and 1" and then back to the starting position between 200 and 300 times per minute. This reciprocal movement is faster on the reverse stroke than it is on the forward stroke. This shaking movement helps transport the concentrates or heavy material to the concentrate end of the table. A very important operating variable of a shaking table is the tilt adjustment. Normally, the feed side is lower, and the concentrate end is higher on the table, which creates an upward slope where the heavy material will ascend, while the light density material will not, and consequently, flow over the riffles. The tailing (low density) side is nearly level. Feed is introduced to the feed box in a slurry at 25% solids, and is distributed along the feed side of the table by the feed trough. Additional wash water is added at the end of the feed

trough, and distributed along the remainder of the length of the table by a wash water launder. About 75% of the water is added to the feed and the remaining 25% added in the wash water launder. this will normally maintain a good water balance. The amount of wash water is another important variable in operating concentrating tables. Too much will wash the concentrate. The gravity concentrating tables remove the high density material from the low density material, since the high density material will reside behind the riffles and allow the low density material to flow over the top of the riffles with the wash water, to the tailings discharge. A figure of the instrument is given in figure 5.6



**Fig 5.6 : Shaking Table**

### **5.3 CHARACTERIZATION UNIT**

Characterization techniques are used for raw ores as well as for treated ores .Phase , chemical , morphological analysis are the major analysis along with some other analysis. The details of the various instruments used for analysis are mentioned below.

#### **5.3.1. X-RAY DIFFRACTOMETER**

X-ray diffraction (XRD) is one of the most important non-destructive tools to analyze all kinds of matter—ranging from fluids, to powders and crystals. X-ray diffraction techniques are used for the identification of crystalline phases of various materials and the quantitative phase analysis subsequent to the identification. The Bragg equation,  $n\lambda = 2d\sin\theta$  is one of the keystones in understanding X-ray diffraction. In this equation,  $n$  is an integer,  $\lambda$  is the characteristic

wavelength of the X-rays impinging on the crystallize sample,  $d$  is the interplanar spacing between rows of atoms, and  $\theta$  is the angle of the X-ray beam with respect to these planes. When this equation is satisfied, X-rays scattered by the atoms in the plane of a periodic structure are in phase and diffraction occurs in the direction defined by the angle  $\theta$ .

In the present study phase analysis is carried out by X-ray diffraction spectrometer with Bragg-Barentano geometry (RigakuUltima III, Japan) of raw ore and several treated products(oxidized, reduced). The equipment is based on this equation Here ' $d$ ' is the spacing between diffracting planes, ' $\theta$ ' is the incident angle, ' $n$ ' is any integer, and ' $\lambda$ ' is the wavelength of the beam. Cu ' $K\alpha$ ' ( $\lambda$ -1.54) is the source of x-ray with operating current and voltage of 40 kV and 30mV respectively. The scan is continued from 20 to 90 degree with 5°/min. Samples are crushed and fined by mortar & pastel about to 5 gm and ultrafine sample are spread over in a quartz holder and then put in the holding position of the equipment. Data is collected after comparing the positions and intensities of peaks with those of the corresponding standards by PDF database (PCPDF-WIN software, JCPDS -International Center for Diffraction Data). A picture of the instrument is shown in figure 5.7



**Fig 5.7: X RayDiffractometer**



## **5.4 CHEMICAL ANALYSIS APPARATUS**

### **5.4.1 SCANNING ELECTRON MICROSCOPE – ENERGY DISPERSIVE X-RAY SPECTROMETER**

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of .The signals used by a scanning electron microscope to produce an image result from interactions of the electron beam with atoms at various depths within the sample. Various types of signals are produced including secondary electrons (SE), reflected or back-scattered electrons (BSE), characteristic X-rays and light (cathodoluminescence) (CL), absorbed current (specimen current) and transmitted electrons. Secondary electron detectors are standard equipment in all SEMs, but it is rare for a single machine to have detectors for all other possible signals.electrons. For SEM- EDX analysis the finely powdered sample is taken on carbon tape which is mounted in a sample holder. For polished sample first of all a little thicker sections of ore is cut by a rock cutter and then the sections are mounted on glass slides by araldite. Now the sections are polished well by different methods and stages of dry and wet polishing until a stainless highly glossy surface is made. Then the glass slide is cut to size (made smaller) to be fit in the sample holder of SEM. Before analysis the sample is coated with conductive material (palladium) with JEOL JFC -1600 Auto Fine Coater. The morphological study is investigated by JEOL JSM- 8360 Scanning Electron Microscope (Japan). Surface morphology has significant characteristic to study the samples in the present study. The morphological characteristics are generally changed after various treatments of the raw ore. Elemental mapping of a selected location and point analysis are carried out to know the mineral composition and presence of various elements in the grain. Energy dispersive X-ray spectroscopy (EDX) is an analytical technique used for the element analysis or chemical characterization of a sample. It is the measurement of X-rays emitted during electron bombardment in an electron microscope (SEM) to determine the chemical composition of materials on the micro- and nanoscale. By determining the energies of the X-rays emitted from the area being excited by the electron beam, the elements present in the sample are determined (qualitative analysis). The rate of detection of these characteristic X-rays is used to measure the amounts of elements present. If the electron beam is restored over an area of the sample then EDX systems can also acquire X-ray maps showing spatial variation of elements in the sample. The elemental mapping of a specific polished section and fine sample is carried out along with

SEM on OXFORD Instrument model no 7582 (England) with INCA software. A picture of the instrument is shown in figure 5.8



**Fig 5.8: Scanning Electron Microscope**

#### **5.4.2 WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE SPECTROMETER (WDXRF)**

XRF (X-ray fluorescence) is a non-destructive analytical technique used to determine the elemental composition of materials. XRF analyzers determine the chemistry of a sample by measuring the fluorescent (or secondary) X-ray emitted from a sample when it is excited by a primary X-ray source. Each of the elements present in a sample produces a set of characteristic fluorescent X-rays ("a fingerprint") that is unique for that specific element, which is why XRF spectroscopy is an excellent technology for qualitative and quantitative analysis of material composition. X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that is excited by bombarding with high-energy X-rays or gamma rays. The phenomenon is widely used for elemental analysis and chemical analysis. Quantification of elemental or major oxide by WDXRF is well known non destructive method for ore, mineral and rock analysis. In the present study raw ore and host rock are analyzed by this equipment to have the knowledge of chemical composition. The sample for Wave length dispersive X-ray fluorescence (WDXRF) measurement is prepared as press pellets on boric acid

support. 2.5 gm of finely powdered (-200 mesh size) representative sample is mixed thoroughly with one to two drops of 10% solution of methyl acrylate (Perspex) binder in acetone and allowed to dry. The sample is then pressed over 5 gm boric acid by a hydraulic press (Herzog) at a pressure of 200 kN. A uniform bead of 27 mm diameter is obtained in this process. The X-ray tube is end window type with a Rhodium anode as a target for the generation of X-ray. The XRF spectrometer is used MagiX 2424 model of PAN analytical, which is a sequential wavelength dispersive X-ray . A picture of the instrument is given in figure 5.9



**Fig 5.9: Wavelength dispersive x-ray fluorescence spectrometer (WDXRF)**

## **5.5 THERMAL ANALYSIS APPARATUS ( TGA )**

Thermo gravimetric analysis (TGA) is a thermal analysis technique which measures the change in weight of the ore as a function of temperature and time, in a controlled environment. This is very useful to investigate the thermal stability of ore, its behaviour and phase transition in different atmospheres (e.g. inert or oxidizing). It is suitable to use, with all types of solid materials, including organic or inorganic materials. Differential thermal analysis (DTA) is a calorimetric technique, recording the temperature and heat flow associated with thermal transitions in a material. The behaviour of raw samples at high temperature in inert atmosphere

and oxidation reactions of the samples are investigated by using Pyris Diamond Thermo gravimetric /Differential Thermal Analysis (TG / DTA, Perkinelmer, Singapore). The  $\alpha$ -alumina powder is used as a reference material. The ground ore and a reference sample are weighed accurately and then placed inside the sample and reference cells, respectively. The experiment is carried out under oxygen atmosphere by purging 125ml/min dry oxygen. The data is collected using a PC-based data. A picture of the instrument is shown in figure 5.10



**Fig 5.10: TG/DTA**

## **5.6 TUBE FURNACE**

Reduction experiments are carried out in a conventional resistance furnace (up to 1623K) containing silicon carbide as heating element with PID controller. Size of alumina tube is 50 mm outer diameter (O.D) and 40 mm inner diameter (I.D) with length (L) 700mm. Heating zone in the furnace is around to 10 cm. Alumina boat with length around to 100 mm is used for sample holding in isothermal heat treatment. The samples are heat treated isothermally in the temperature range of 873 to 1473 K in air. Ten grams (approx) of sample in the size range - 18.75+12.5mm along with stoichiometric amount of coal are taken and transferred into an alumina crucible. At first the temperature is raised to desire temperature and quickly the boat is inserted carefully in the heating zone. The temperature is calibrated and standardized by using a Pt/Rh thermocouple. The furnace has closed environment (closed with cork on both ends). After the experiment the sample is cooled in the furnace to room temperature. A picture of the instrument is shown in figure 5.11



**Fig 5.11: Tube Furnace**

### **5.7 RAISING HERTH FURNACE**

Raising Hearth Furnace (maximum temperature capacity of 1873 K) is used for the purpose of pre-treatment as well as smelting of ore. It has platform mounted frame structure with prominent furnace section with its opening at the bottom. The change of required temperature is controlled by computer controlled programme. The furnace can be closed by upward moving hearth, raising the platform into the heating cavity by manual and mechanical means. The image of Raising Hearth Furnace is given in figure 5.12 [31]



**Fig 5.12 : Raising Hearth Furnace**

*Chapter 6*

*Results*

*and*

*Discussions*

## 6. RESULTS AND DISCUSSIONS

### 6.1 CHARACTERIZATION OF RAW MATERIAL

#### 6.1.1 PYROLUSITE

The raw material used in the experiment is Pyrolusite ore collected from Tata Steel. The general characterization tests of the raw material were performed as given below.

##### 6.1.1.1 WDXRF ANALYSIS

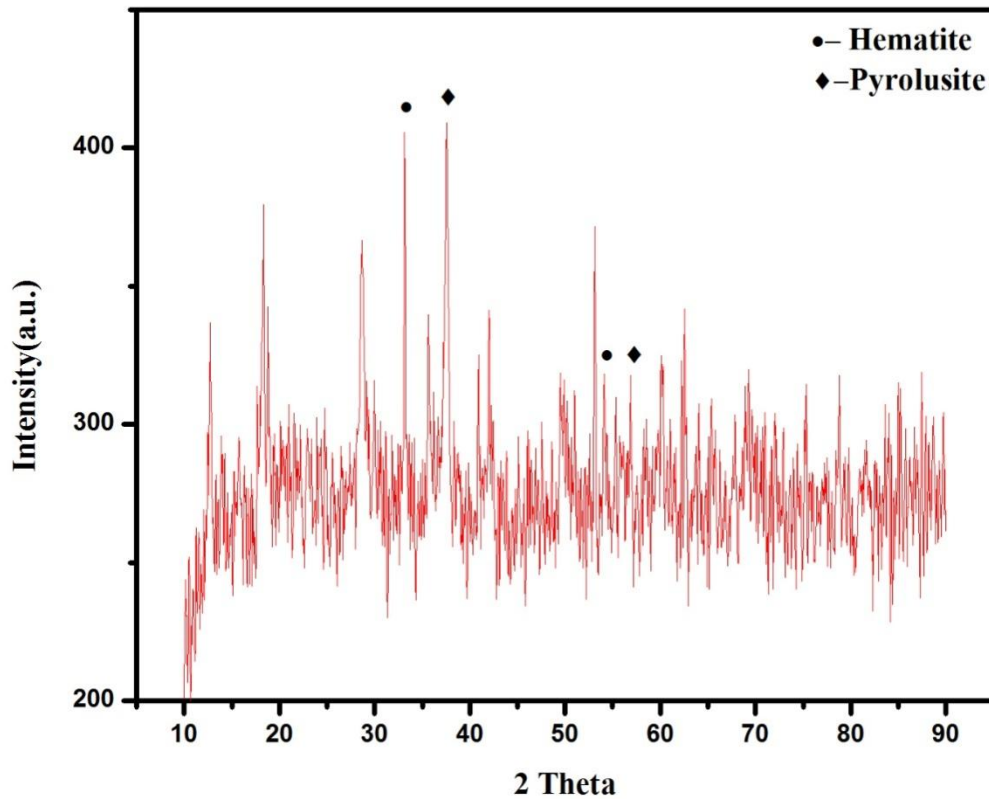
Some of the raw sized ores used in the experiment were crushed below 75  $\mu\text{m}$  and subjected to wavelength dispersive X-Ray Fluorescence. The result of the test is given in the table 1 below:

Element	Mass percentage
Total Manganese	38.061
Total Iron	15.132
SiO <sub>2</sub>	3.766
MgO	0.144
P	0.045
Na <sub>2</sub> O	0.072
K <sub>2</sub> O	1.298
CaO	0.142
TiO <sub>2</sub>	0.142
Al <sub>2</sub> O <sub>3</sub>	2.718
BaO	0.480
S	0.002
S-Blend	0.100

**Table 6.1: WDXRF results of Pyrolusite sample**

### 6.1.1.2 X-RAY DIFFRACTOMETER ANALYSIS

Phase Analysis of the Pyrolusite sample was carried out by X-Ray diffraction spectrometer with Bragg-Barentano geometry (RigakuUltima III, Japan),the results of the experiment are given in the figure below:



**Fig 6.1: XRD result of iron ore sample**

From the figure it was found that Pyrolusite(  $Mn_2O$  ) and Haematite (  $Fe_2O_3$  ) are the predominant phases.

### 6.1.2 COAL

Boiler grade coal was used in these experiments .This coal was supplied by Vizag Steel Plant along with ultimate analysis of coal.TG-DTA, proximate analysis and XRD of the coal was performed for its characterization.



### 6.1.2.1 PROXIMATE ANALYSIS

The results of the proximate analysis of the coal are given in table 6.2.

( % )	BOILER COAL
Fixed Carbon	28.08
V.M	28.31
Moisture	7.40
Ash	36.21

**Table 6.2: Proximate Analysis of Boiler Grade Coal**

### 6.1.2.2 ULTIMATE ANALYSIS

The data of proximate analysis was further refined by Ultimate analysis(LecoTruSpec) of coal which was supplied by Vizag Steel Plant and is given in table 6.3

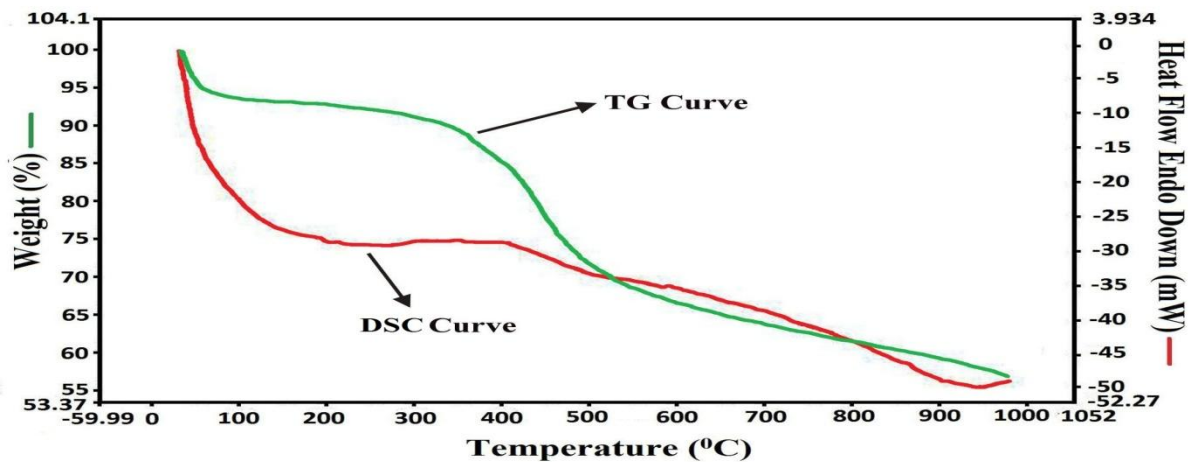
SI No.	PARAMETERS	SAMPLE TAGGED AS BOILER GRADE COAL
1	Total Moisture % ( as received basis )	2
2	Analytical moisture	7.30
3	Volatile matter % ( air dry)	29.13
4	Ash % ( Air Dry )	33.05
5	Fixed Carbon % ( Air dry )	30.52
6	Sulphur ( Dry Air )	0.561

<b>7</b>	<b>Ultimate Analysis</b>	
	<b>Dry Basis</b>	
	<b>% Carbon</b>	50.9
	<b>% Hydrogen</b>	3.47
	<b>% Nitrogen</b>	0.803

**Table 6.3: Ultimate Analysis of Coal**

### 6.1.2.3 TG/DTA ANALYSIS FOR BOILER GRADE COAL

The TG/DTA study of boiler grade coal is essential for the design of the coal gasification part of the project, as it is necessary to know its gasification properties. The green line is the TG curve and the red line the DSC curve. The nature of weight loss conforms to the data from the proximate analysis of the boiler coal. The graph shows weight loss upto 110 °C, which is obviously the expulsion of the moisture ( 7-8 % ) in the coal. The major weight loss from (400 to 550)°C is the removal of the volatile matter ( 25-30 % ). This is the single most important step during gasification. The following weight loss at a very slow rate may occur due to soot formation or mild oxidation by the trace amount of oxygen present in nitrogen which is used as a shielding gas in the experiment. The TG/DTA curve is given in figure 6. 2



**Fig 6.2 : TG/DTA of Boiler Grade Coal**

## 6.2 JIGGING OF MANGANESE ORE

The beneficiation of the low grade manganese ore lumps was done using Jigging method. The different parameters and their respective levels for the jigging process have been given in table 6.4. The two parameters selected were size range and motor speed (r.p.m). The collected ore samples were broken down into suitable size ranges by hammering followed by primary crushing using jaw crusher.

PARAMETERS	DIFFERENT LEVELS		
SIZE FRACTION	+5/16	+4	-5 - +7
MOTOR SPEED ( RPM )	1100	1450	1800

**Table 6.4: Parameters and their levels for jigging**

Jigging was carried out for the lumps for three size ranges +4, -5 to +7, +5/16. For each of these three sizes, 3 motor speeds were selected 1100 rpm, 1450 rpm, 1800 rpm. Initially 100 grams of the samples were subjected to the jigging as mentioned in the table below. Each of the experiments was carried out for 15 minutes. The wet samples were then collected from the bed and dried by heating in a heater at 100 degree celsius for one hour. The dried samples were then weighed and the final weights are given in the table 6.5. The percentage weight loss was then found out according to the formula used in the table 6.5. The highest separation in terms of percentage weight loss was found to be 4.94 percent for -5 to +7 size range at 1450 rpm followed by 4.17 percent for +5/16 size at 1100 rpm . For the +4 size highest separation was found to be 2.285 percent at 1450 rpm. The separations in terms of percentage weight loss are given for all the samples in table 6.5.

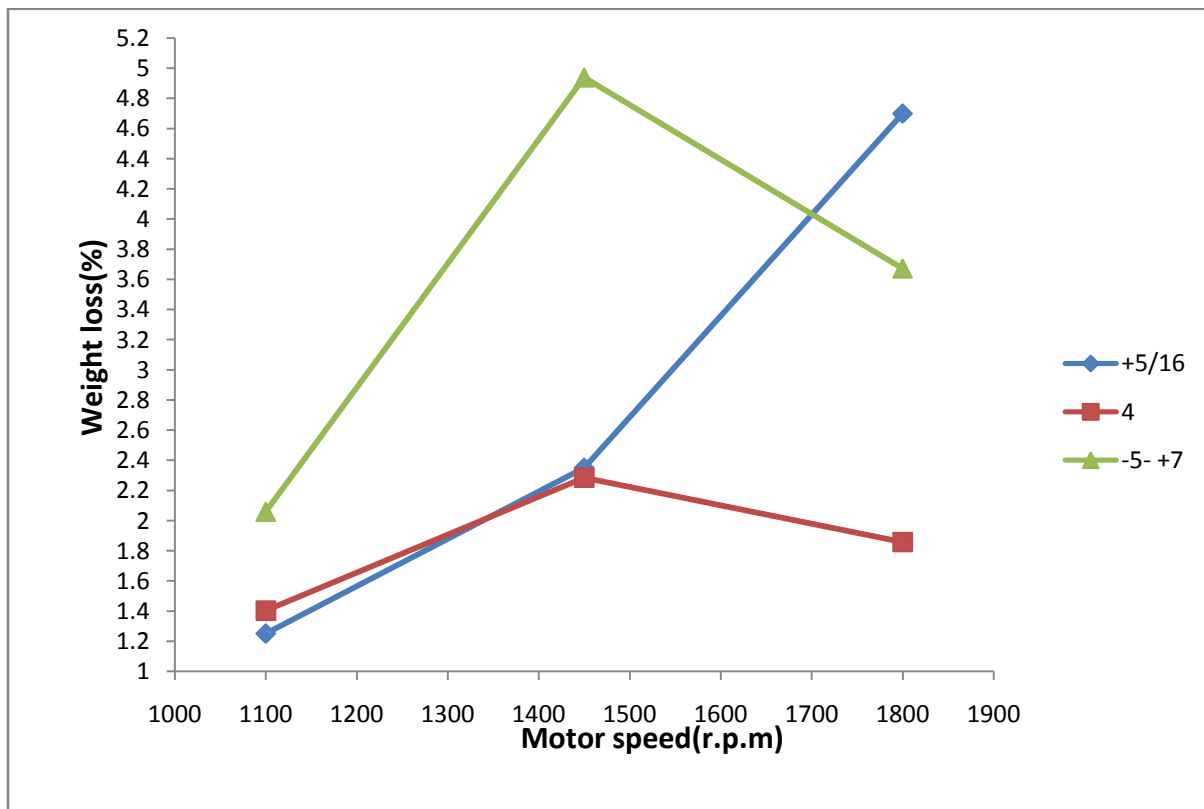
SIZE FRACTION	MOTOR SPEED IN RPM	INITIAL WEIGHT $W_{\text{INITIAL}}$ (gm)	FINAL WEIGHT $W_{\text{FINAL}}$ (gm)	PERCENTAGE WEIGHT LOSS $(W_{\text{INITIAL}} - W_{\text{FINAL}}) / W_{\text{INITIAL}} * 100$ (%)
A ) + 5/16	A ) 1100	100	98.75	1.25
	B ) 1450	100	97.65	2.35
	C ) 1800	100	95.30	4.70
B ) + 4	A ) 1100	100	98.59	1.40
	B ) 1450	100	97.71	2.28
	C ) 1800	100	98.14	1.85
C ) -5 to +7	A ) 1100	100	97.94	2.06
	B ) 1450	100	95.06	4.94
	C ) 1800	100	96.32	3.67

**Table 6.5: Initial weight, final weight and percentage weight loss in jigging**

The data was plotted on graph with motor speed ( r.p.m ) along X-axis and weight loss ( % ) along Y-axis as given in figure 6.3. It is observed that for the smallest size -5 to +7 the weight loss reached maximum at 1450 rpm and with further increase of speed to 1800 rpm, the weight loss decreased. For the next higher size +4 it is observed to follow a similar trend with maximum weight loss due to separation at 1450 rpm and with further increase of speed to 1800 rpm the

separation decreased. For the biggest size +5/16 it is found to follow a different trend .The weight loss was minimum at 1100 rpm and with increase of the motor speed to 1450 rpm the weight loss further increased and reached a maximum at 1800 rpm. With the increase in motor speed, the frequency and amplitude of water pulsation increases and thus it allows better separation for the bigger size fraction.

The separation increases for the smaller sizes till an optimal motor speed and decreases with higher speeds. For the lighter particle size it first increases with motor speed but after attaining an optimum separation it decreases .This is probably due to reversing back of some of the low weight materials at very high motor speeds.



**Fig 6.3: Plot of separation of different size ranges against motor speed**

### 6.3 TABLING OF MANGANESE ORE

The beneficiation of the low grade manganese ore fines was also done using Tabling method. The different parameters and their respective levels for the tabling process have been given in table 6.6. The three parameters selected were angle , volume flow rate and size range .The collected ore samples were broken down into suitable size ranges by hammering followed by primary crushing using jaw crusher. The crushed samples were then reduced to fines by breaking them down in a pulverizer.

PARAMETERS	DIFFERENT LEVELS	
Angle ( degree )	4	8
Volume flow rate ( Lit/ min)	4	6
Size range	+25 - +150	-150 - +200

**Table 6.6: Parameters and their levels for tabling**

Tabling was carried out for the two angles chosen viz. 4 degree and 8 degree . For each of the two angles two volume flow rates of 4 lit/min and 6 lit/min were taken. Again for each of these two volume flow rates two size ranges of +25 - +150 and -150 - +200 were taken . Initially 150 gm of the size ranges were subjected to tabling. The wet samples after tabling were then collected from the two collectors containing concentrates and mixture of middling and tailing. The collected samples were then dried by heating in a heater at 100 degree celsius for one hour. The dried samples were then weighed and the final weights of both the concentrates and the mixture of middling and tailing are given in the table 6.7. The separation of the concentrate and middling tailing mixture was found out from the weight ratio as given in table 6.7. For the tabling process with parameters set at 4 degree angle , volume flow rate of 4 Lit/min and +25 to +150 size range maximum amount of concentrate was collected which was found to be 108 gms and also highest weight ratio among all the experiments 3.8 for this sample .

For the experiment with parameters set at 4 degree angle , volume flow rate of 6 Lit/min and -150 to +200 size range the amount of concentrate was found to be 107 gms and the weight ratio was 3.68. For the experiment with parameters set at 8 degree angle , volume flow rate of 6 Lit/min and +25 to +150 size range the amount of concentrate was 105 gms and the weight ratio was 3.5 and for the experiment with parameters set at 8 degree angle , volume flow rate of 4 Lit/min and -150 to +200 size range the amount of concentrate was 91gms and the weight ratio was 3.03 . The separation for the tabling was found in terms of weight ratio and not in terms of concentrate weight .Thus for some of the other results like the experiments for the two size ranges at 8 degree , 4 lit/min although the concentrate weight for the +25 - +150 size range ( 94 gms ) was higher than the -150 - +200 size range (91 gms ) but the weight ratio for the -150 - +200 range was higher ( 3.03 ) than that of +25 - +150 size range. Thus at 8 degree angle and 4lit/min flow rate the -150 - +200 size range had better separation.

ANGLE	VOLUME FLOW RATE	SIZE FRACTION		WEIGHT	WEIGHT RATIO= (Wt. OF CONC.) / (Wt. OF MIDDLING AND TAILING)
4 degree	4 litres / min	+25 to +150	Concentrate	108	3.8
			Middling and tailing	28	
		-150 to +200	Concentrate	96	3.31
			Middling and tailing	29	
	6 litres / min	+25 to +150	Concentrate	94	3.03
			Middling and tailing	31	
		-150 to +200	Concentrate	107	3.68
			Middling and tailing	29	
8 degree	4 litres / min	+25 to +150	Concentrate	94	2.54
			Middling and tailing	37	
		-150 to +200	Concentrate	91	3.03
			Middling and tailing	30	
	6 litres / min	+25 to +150	Concentrate	105	3.5
			Middling and tailing	30	
		-150 to +200	Concentrate	98	3.37
			Middling and tailing	29	

**Table 6.7: Different parameters , their levels and weight of concentrate and middling / Tailings in Tabling**



From the results of tabling the following observations were made:

(1) On increasing the volume flow rate from 4 litres/min to 6 litres/min for the samples of -150 to +200 size range it was found that weight ratio increased from 3.31 to 3.68 for the 4 degree samples and from 3.03 to 3.37 for the 8 degree samples. Similarly for the +25 to +150 range sample at 8 degree angle the weight ratio increased from 2.54 to 3.5 on increasing the volume flow rate . But for the +25 to +150 range sample at 4 degree it was found that on increasing the volume flow rate the weight ratio decreased from 3.8 to 3.3.

Thus on increasing the volume flow rate, the weight ratio increased for all the samples except for the one.

(2) On increasing the angle from 4 degree to 8 degree for the samples of size range -150 to +200 the weight ratio decreased from 3.31 to 3.03 for the volume flow rate of 4 litres/min and decreased from 3.68 to 3.37 for the volume flow rate of 6 litres/min. Similarly for the samples of size range +25 to +150 it was found that weight ratio decreased from 3.8 to 2.54 on increasing the angle for the volume flow rate of 4 litres/min . But for the +25 to +150 size range at volume flow rate of 6 litres/min the weight ratio increased from 3.03 to 3.5.

Thus on increasing the angle ,the weight ratio decreased for all the samples except for one sample.

(3) Size fraction and size range selected for the experiment was to have no such effect on the weight ratio and hence on the separation . This could be probably due to dominant nature of the two other factors i.e, angle and volume flow rate. Also the samples selected were fines for the tabling process and the two size ranges selected i.e. +25 to +150 and -150 to +200, were close enough to have any profound impact on the separation performance.

#### **6.4 CHARACTERIZATION OF JIGGING AND TABLING SAMPLES**

X- Ray Fluorescence( XRF ) was performed for all the samples of jigging and tabling and the samples with maximum Mn content were selected for further analysis. The maximum Mn content for jigging was found for the sample of +4 size fraction at 1450 rpm and for tabling highest Mn content was found for the sample of size range +25 to +150 at 4° angle and volume

flow rate of 4L/ min . The results of XRF for the best separation samples in jigging is given in table 6.8 and for tabling is given in table 6.9 respectively.

<b>SIZE RANGE</b>	<b>MOTOR SPEED (RPM)</b>	<b>PERCENTAGE WEIGHT LOSS (%)</b>	<b>MANGANESE CONTENT (%)</b>
+4	1450	2.285	42.843

**Table 6.8: Sample with highest Manganese recovery after Jigging**

<b>ANGLE (DEGREE)</b>	<b>VOL FLOW RATE (LIT / MIN)</b>	<b>SIZE RANGE</b>	<b>MANGANESE CONTENT (%)</b>
4	4	+25 to +150	41.793

**Table 6.9: Sample with highest Manganese recovery after Tabling**

### **6.5 `REDUCTION OF SAMPLES**

The samples with highest manganese content from jigging and tabling were selected for reduction. For each of the two samples, two time intervals and two temperatures were selected. The different parameters and their levels are given in table 6.10

<b>PARAMETERS</b>	<b>DIFFERENT LEVELS</b>	
<b>Time ( mins )</b>	60	90
<b>Temperature ( °C )</b>	1100	1200

**Table 6.10: Parameters and their levels for Reduction**

### 6.5.1 EXTENT OF REDUCTION ( E.O.R)

A total of eight reductions were carried out with the parameter levels given in table 6.11 and the extent of reduction of each of the eight samples were found out. The samples from Jigging and Tabling results corresponding to the highest Extent of Reduction were selected for further analysis. The results of reduction with EOR are given in table 6.11 .( Calculation given in Appendix I and Appendix II ):

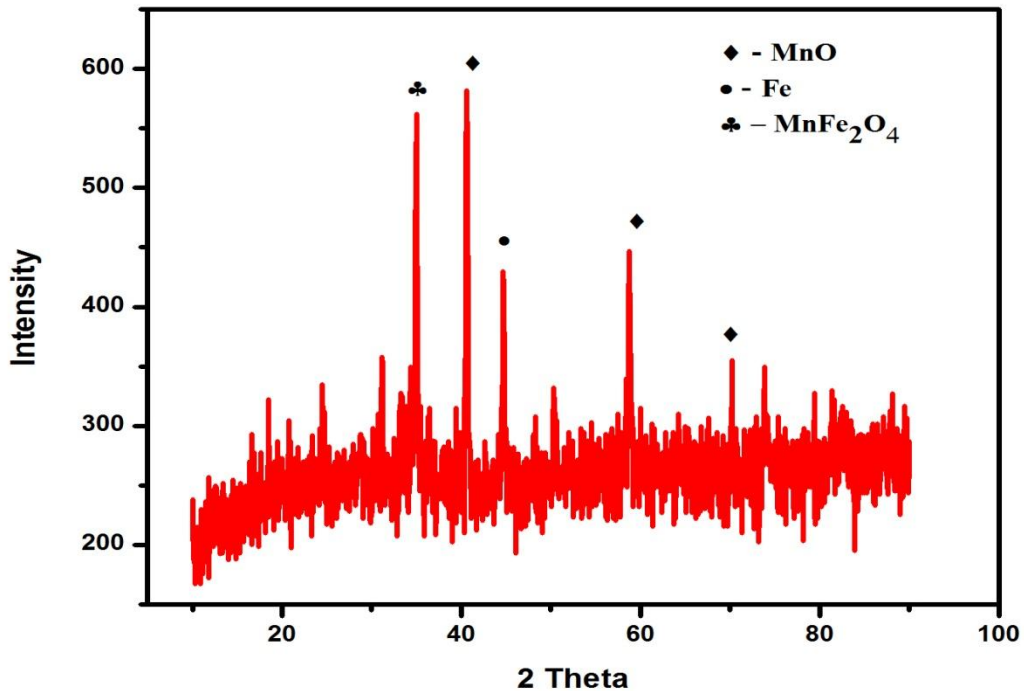
	<b>Time ( mins )</b>	<b>Temperature ( °C )</b>	<b>Initial Weight ( <math>W_i</math> )</b>	<b>Final Weight ( <math>W_f</math> )</b>	<b>Loss in weight = ( <math>W_i - W_f</math> )</b>	<b>Loss in Weight Percent ( % )</b>	<b>Extent of Reduction ( % )</b>
<b>Jigging</b>	<b>60</b>	1100	9.73	7.58	2.15	22.12	69.30
		1200	10.20	8.10	2.10	20.58	64.48
	<b>90</b>	1100	9.79	7.65	2.14	21.86	68.49
		1200	10.32	8.45	1.87	18.15	56.86
<b>Tabling</b>	<b>60</b>	1100	9.59	7.41	2.18	22.73	73.12
		1200	9.42	7.27	2.14	22.81	73.37
	<b>90</b>	1100	9.69	7.74	1.95	20.18	64.91
		1200	8.50	6.587	1.91	22.54	71.51

**Table 6.11: Extent Of Reduction of Reduced Samples**

## 6.6 CHARACTERIZATION OF REDUCED SAMPLES

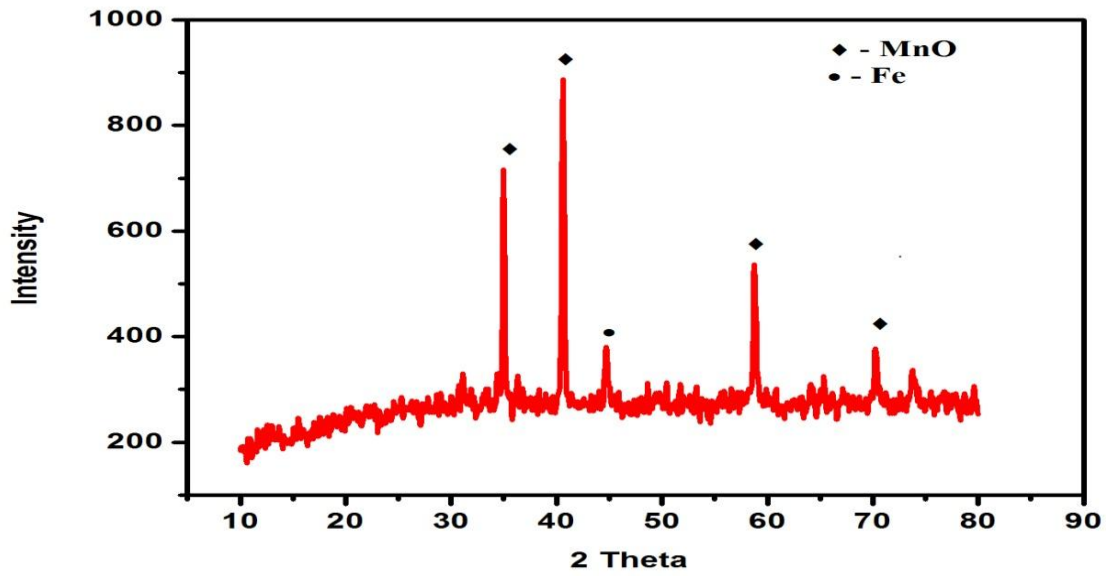
The four samples with highest E.O.R were selected for XRD Analysis to identify the major phases. The XRD results are mentioned below:

**Sample 1 :**For the Jigging sample, whose reduction was carried out at 1100°C for 60 mins with E.O.R of 69.30, the major phases were Fe and MnO. Peak of jacobsite,  $\text{MnFe}_2\text{O}_4$  was also found.. The result is given in figure 6.4 :



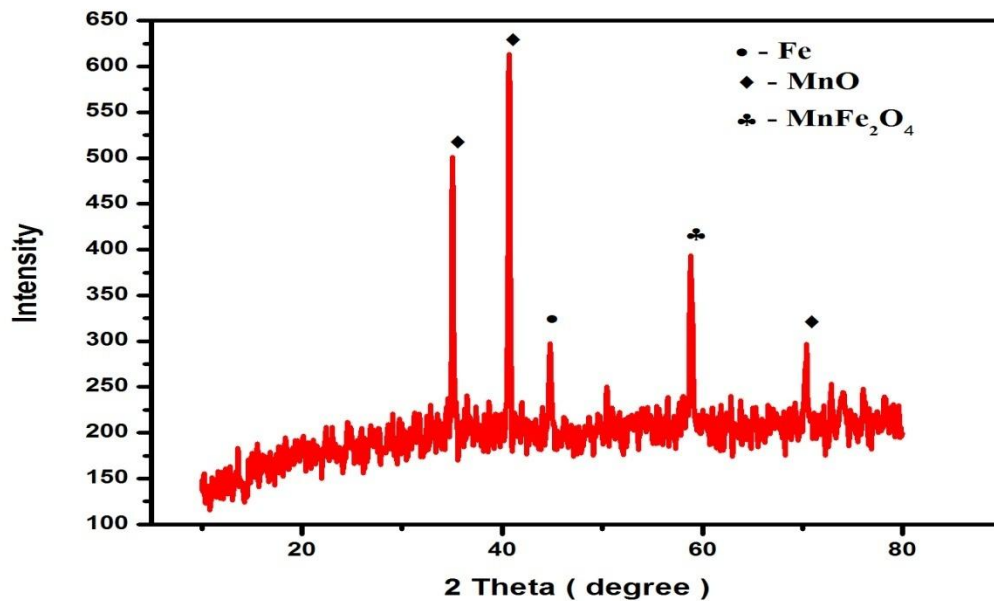
**Fig 6.4: Sample 1 :Jigging sample after reduction at 1100°C for 60 mins.**

**Sample 2:** For the Jigging sample, whose reduction was carried out at 1100°C for 90 mins with E.O.R of 68.49, the major phases were Fe and Mn. The result is given in figure 6.5 :



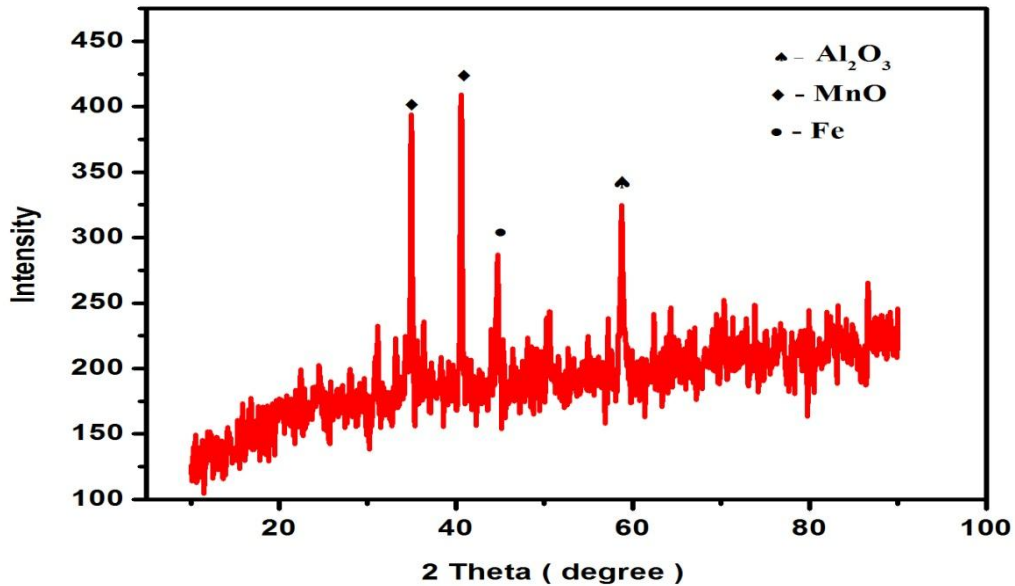
**Fig 6.5: Sample 2: Jigging sample after reduction at 1100°C for 90 mins**

**Sample 3 :** For the Tabling sample, whose reduction was carried out at 1100°C for 60 mins with E.O.R of 73.12 , the major phases were Fe , MnO and  $MnFe_2O_4$  . The result is given in figure 6.6 :



**Fig 6.6: Sample 3: Tabling Sample after reduction at 1100°C for 60 mins.**

**Sample 4:** For the Tabling sample, whose reduction was carried out at 1200°C for 60 mins with E.O.R of 73.37, the major phases were Fe, MnO and Al<sub>2</sub>O<sub>3</sub>. The result is given in figure 6.7:



**Fig 6.7: Sample 4: Tabling samples after reduction at 1200°C for 60 mins**

## 6.7 MAGNETIC SEPARATION

Magnetic separation was done for each of the four samples with the help of a hand magnet of 20 mT strength. The magnetic phases got attracted by the magnet and the non magnetic phases were separated. The weights and weight percentage of the magnetic and non magnetic parts were calculated and mentioned in table 6.13:

	TIME ( mins )	TEMP ( °C )	E.O.R ( % )	WEIGHT ( gm )		Wt. PERCENTAGE ( % )	
				MAGNETIC	NON MAGNETIC	MAGNETIC	NON MAGNETIC
<b>JIGGING</b>	60	1100	69.30	3.80	2.70	58.46	41.54
	90	1100	68.49	4.64	2.47	65.26	34.74
<b>TABLING</b>	60	1100	73.12	4.53	2.42	65.15	34.85
	60	1200	73.37	4.73	0.93	83.49	16.51

**Table 6.13: Weights and weight percentages of magnetic and non magnetic parts.**

## 6.8 SMELTING

After magnetic separation and characterization of the jigging and tabling samples, the non magnetic parts of the samples were further taken for smelting. Since both

thetablingandjigging samples after the reduction contained primarily MnO phase and some Fe phase so all the samples were mixed together and subjected to smelting with calculated amount of aluminium powder ( Appendix III) , 2% BaO<sub>2</sub> and 3% CaO so as complete the reduction of MnO and produce Mn-Fe alloy as product .

## 6.9 CONCLUSION

The gravity separation method Jigging and Tabling were effective to beneficiate the low grade manganese ore containing pyrolusite to improve its manganese content and increase its Mn/Fe ratio by separation the Mn content from its impurities like SiO<sub>2</sub>, K<sub>2</sub>O, S-blend etc. there are substantial increase in the manganese content from 38.06% in the raw ore to 41.793% and 42.843% in the jigging and tabling samples respectively. The Mn/Fe ratio also increased from 2.51 in the raw ore to 2.647 and 2.624 in the final samples after Jigging and Tabling.

After reduction ,in the XRD data it was found that MnO was the major manganese phase and Fe was the major iron phase.MnO<sub>2</sub> generally follows the reduction sequence MnO<sub>2</sub> to Mn<sub>2</sub>O<sub>3</sub> to Mn<sub>3</sub>O<sub>4</sub> toMnO to Mn. Since reduction was carried out at temperatures of 1100 and 1200°C , reduction could only extend to the formation of MnO phase and metallic Mn was not formed .But Fe being the major phase, it was clear that Fe<sub>2</sub>O<sub>3</sub> had undergone complete reduction at that temperature range but MnO phase being more stable requires higher temperature for reduction to metallic phase. After magnetic separation was carried out from the reduced samples of higher E.O.R , higher weight percentage was found for the magnetic parts compared to the non magnetic parts. This was due to complete reduction of Fe<sub>2</sub>O<sub>3</sub> to Fe phase and some other magnetic Fe phases which were magnetic.

The Non magnetic parts of Jigging and Tabling having higher Mn/Fe ratio due to removal of Fe were subjected toAluminothermic smeltingto produce Manganese Iron alloy as the final product.



*Chapter 7*

*Interpretation*

- The low grade Pyrolusite ore samples collected from TATA Steel after being crushed and sieved to required sizes , the lumps were subjected to beneficiation by Jigging and the fines were subjected to beneficiation by Tabling.
- For Jigging , 3 sizes : +5/16, +4 and -5 to +7 range were selected and 3 Motor Speeds :1100 RPM , 1450 RPM and 1800 RPM were selected and the percentage weight loss was calculated for each of the samples. With increasing the motor speed from 1100 RPM to 1450 RPM, the percentage weight loss increased for the +4 and -5 to +7 sizes but with further increase to 1800 RPM weight loss decreased. For the highest size +5/16 the percentage weight loss continued to rise with increasing the motor speed. The maximum percentage weight loss was found for the -5 to +7 size range at 1450RPM.
- For Tabling, 2 size ranges : +25 to +150 and -150 to +200 , 2 angles : 4 degree and 8 degree , 2 volume flow rates : 4 lit/min and 6 lit/min were selected. The weights of concentrate and middling/tailing were measured and the weight ratio between the two was calculated for all the samples. The highest weight ratio was found for the sample of +25 to +150 size range at 4 lit/min and 4 degree angle. The general trend in Tabling was:
  - A) On increasing the volume flow rates , the weight ratio increased .
  - B) On increasing the Angle, the weight ratio decreased.
  - C) There was no effect of the size range as such.
- Samples with highest separation from Jigging and Tabling were selected for Reduction under 2 temperatures: 1100°C and 1200°C and 2 time intervals: 60 mins and 90 mins. The Extent of Reduction was found out for all the samples and 2 samples with highest EOR from Jigging and Tabling were selected for further analysis. The 2 samples from Jigging were the one at 1100°C for 60 mins with EOR of 69.30 % and the one at 1100 °C for 90 mins with 68.49% . The 2 samples from Tabling were the one at 1200°C for 60 mins with EOR of 73.37% and the one at 1100°C for 60 mins with EOR of 73.12%.
- After Reduction and Magnetic separation, the non magnetic parts were mixed and subjected to aluminothermic smelting to produce Manganese Ironalloy.

*Chapter 8*  
*Appendix*

## APPENDIX 1

### CALCULATION OF E.O.R OF TABLING SAMPLES

#### 1.1 XRF data of highest Mn Recovery Sample after Tabling

Elements	Mass percentage
Total Manganese	41.793
Total Iron	15.785
SiO <sub>2</sub>	2.685
MgO	0.172
P	0.047
Na <sub>2</sub> O	0.079
K <sub>2</sub> O	1.3
CaO	0.333
TiO <sub>2</sub>	0.064

#### 1.2 Finding out the MnO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> percentage

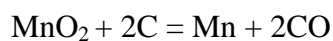
55 gmMn is present in 87 gm MnO<sub>2</sub>

41.793 gmMn is present in ( 87 / 55 \* 41.793 ) gm = **66.108 gm MnO<sub>2</sub>**

112 gm Fe is present in 160 gm Fe<sub>2</sub>O<sub>3</sub>

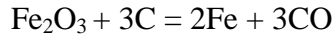
15.785 gm Fe is present in ( 160 / 112 \* 15.785 ) gm = **22.55 gm Fe<sub>2</sub>O<sub>3</sub>**

#### 1.3 Coal Required for reduction



87 gmMnO<sub>2</sub> requires 24 gm carbon for oxygen removal.

66.107 gm MnO<sub>2</sub> requires ( 24/87 \* 66.107 )= 18.24 gm carbon for oxygen removal



160 gm Fe<sub>2</sub>O<sub>3</sub> requires 36 gm carbon for oxygen removal

22.55 gm Fe<sub>2</sub>O<sub>3</sub> requires ( 36/160 \* 22.55 )= 5.0735 gm carbon for oxygen removal

So 100 gm ore requires 18.24 +5.073.5 = 23.3135 gm carbon for oxygen removal

So 10 gm ore requires 2.33135 gm carbon for oxygen removal

Now , The Coal used has 28.08 % carbon

The amount of boiler grade coal for removal of oxygen from 10gm ore = ( 2.33135 / 0.2808 )  
= **8.3025 gm**

#### **1.4 Oxygen to be removed**

87 gm MnO<sub>2</sub> contains 32 gm oxygen

66.108 gm MnO<sub>2</sub> contains ( 32 / 87 \* 66.108 ) = 24.32 gm oxygen

160 gm Fe<sub>2</sub>O<sub>3</sub> contains 48 gm oxygen

22.55 gm Fe<sub>2</sub>O<sub>3</sub> contains ( 48 / 160 \* 22.55 ) = 6.765 gm oxygen

So 100 gm ore contains ( 24.32 + 6.765 ) = **31.085 gm oxygen to be removed**

**So, EOR = % Loss in weight due to Oxygen removal / Total oxygen in 100 gm ore**

= 22.73/31.085 \* 100 = **73.12%**

= 22.81/31.085 \* 100 = **73.37%**

= 20.18/31.085 \* 100 = **64.91 %**

= 22.54/31.085 \* 100 = **72.51 %**

## APPENDIX II

### CALCULATION OF E.O.R OF JIGGING SAMPLES

#### 2.1 XRF data of highest Mn Recovery Sample after Jigging

Elements	Mass percentage
Total Manganese	42.843
Total Iron	16.324
SiO <sub>2</sub>	4.562
MgO	0.317
P	0.04
Na <sub>2</sub> O	0.11
K <sub>2</sub> O	1.136
CaO	0.692
TiO <sub>2</sub>	0.131

#### 2.2 Finding out the MnO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> percentage

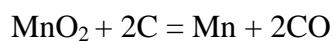
55 gmMn is present in 87 gm MnO<sub>2</sub>

42.843 gmMn is present in ( 87 / 55 \* 42.843 ) gm = **67.77 gm MnO<sub>2</sub>**

112 gm Fe is present in 160 gm Fe<sub>2</sub>O<sub>3</sub>

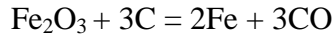
16.324 gm Fe is present in ( 160 / 112 \* 16.324 ) gm = **23.32 gm Fe<sub>2</sub>O<sub>3</sub>**

#### 2.3 Coal Required for reduction



87 gmMnO<sub>2</sub> requires 24 gm carbon for oxygen removal

67.77 gm MnO<sub>2</sub> requires ( 24/87 \* 67.77 )= 18.695 gm carbon for oxygen removal



160 gm Fe<sub>2</sub>O<sub>3</sub> requires 36 gm carbon for oxygen removal

23.32 gm Fe<sub>2</sub>O<sub>3</sub> requires ( 36/160 \* 23.32 )= 5.247gm carbon for oxygen removal

So 100 gm ore requires 18.695 + 5.247 = 23.942 gm carbon for oxygen removal

So 10 gm ore requires 2.3942 gm carbon for oxygen removal

Now , The Coal used has 28.08 % carbon

The amount of boiler grade coal for removal of oxygen from 10gm ore = ( 2.3942 / 0.2808 )  
= **8.526 gm**

#### **2.4 OXYGEN TO BE REMOVED**

87 gm MnO<sub>2</sub> contains 32 gm oxygen

67.77 gm MnO<sub>2</sub> contains ( 32 / 87 \* 67.77 ) = 24.92 gm oxygen

160 gm Fe<sub>2</sub>O<sub>3</sub> contains 48 gm oxygen

23.32 gm Fe<sub>2</sub>O<sub>3</sub> contains ( 48 / 160 \*23.32) = 6.996 gm oxygen

So 100 gm ore contains ( 24.92 + 6.996 ) = **31. 916gm oxygen to be removed**

E.O.R= 22.12 / 31.916 \*100 = **69.3 %**

= 20.58 / 31.916 \* 100 = **64.48 %**

= 21.86 / 31.916 \*100 = **68.49 %**

= 18.15 / 31.916 \*100 = **56.86 %**

### APPENDIX III

#### CALCULATION OF AMOUNT OF REDUCTANT REQUIRED FOR ALUMINOTHERMIC SMELTING

**For Jigging Sample of EOR = 69.30%**

160gms  $\text{Fe}_2\text{O}_3$  contains 48gms oxygen

So, 2.332gms  $\text{Fe}_2\text{O}_3$  contains  $(48/160 * 2.332)$ gm oxygen = 0.6996 gms oxygen

87gms  $\text{MnO}_2$  contains 32gms oxygen

So, 6.78 gms  $\text{MnO}_2$  contains  $(32/87 * 6.78)$ gm oxygen = 2.5 gms oxygen

Total oxygen in 10gms ore sample =  $2.5 + 0.6996 = 3.2$  gms

For 100% EOR, 3.2 gms oxygen has to be removed.

So for 69.3% EOR,  $(3.2/100 * 69.3)$  gm oxygen was removed = 2.21gm oxygen was removed

Now assuming the entire oxygen from hematite was removed and the remaining oxygen was present in  $\text{MnO}$

The amount of oxygen removed from  $\text{MnO}_2 = 2.15 - 0.6765 = 1.51$ gm

Amount of oxygen left to be reduced from  $\text{MnO} = 2.5 - 1.51 = 0.99$  gms

Now,  $3\text{MnO} + 2\text{Al} = \text{Al}_2\text{O}_3 + 3\text{Mn}$

So, 48gm oxygen requires 54gm aluminium for reduction.

So, 0.99 gm oxygen requires  $(54/48 * 0.99)$  gm aluminium = **1.11 gm aluminium**

**For Jigging Sample of EOR = 68.49%**

160gms  $\text{Fe}_2\text{O}_3$  contains 48gms oxygen



So, 2.332gms  $\text{Fe}_2\text{O}_3$  contains  $(48/160 * 2.332)$ gm oxygen = 0.6996 gms oxygen

87gms  $\text{MnO}_2$  contains 32gms oxygen

So, 6.78 gms  $\text{MnO}_2$  contains  $(32/87 * 6.78)$ gm oxygen = 2.5 gms oxygen

Total oxygen in 10gms ore sample =  $2.5 + 0.6996 = 3.2$  gms

For 100% EOR, 3.2 gms oxygen has to be removed.

So for 68.49 % EOR,  $(3.2/100 * 68.49)$  gm oxygen was removed = 2.19 gm oxygen was removed

Now assuming the entire oxygen from hematite was removed and the remaining oxygen was present in  $\text{MnO}$

The amount of oxygen removed from  $\text{MnO}_2 = 2.19 - 0.6996 = 1.5$ gm

Amount of oxygen left to be reduced from  $\text{MnO} = 2.5 - 1.5 = 1$  gms

Now,  $3\text{MnO} + 2\text{Al} = \text{Al}_2\text{O}_3 + 3\text{Mn}$

So, 48gm oxygen requires 54gm aluminium for reduction.

So, 0.99 gm oxygen requires  $(54/48 * 1)$  gm aluminium = **1.125 gm aluminium**

**For Tabling Sample of EOR = 73.12 %**

160gms  $\text{Fe}_2\text{O}_3$  contains 48gms oxygen

So, 2.255gms  $\text{Fe}_2\text{O}_3$  contains  $(48/160 * 2.255)$ gm oxygen = 0.6765gms oxygen

87gms  $\text{MnO}_2$  contains 32gms oxygen

So, 6.610gms  $\text{MnO}_2$  contains  $(32/87 * 6.610)$ gm oxygen = 2.431gms oxygen

Total oxygen in 10gms ore sample =  $2.431 + 0.6765 = 3.1075$ gms

For 100% EOR, 3.1075gms oxygen has to be removed.

So for 73.12 % EOR,  $(3.1075/100 * 73.12)$  gm oxygen was removed = 2.27 gm oxygen was removed

Now assuming the entire oxygen from hematite was removed and the remaining oxygen was present in MnO

The amount of oxygen removed from  $MnO_2 = 2.27 - 0.6765 = 1.59$  gm

Amount of oxygen left to be reduced from MnO =  $2.431 - 1.59 = 0.841$  gm

Now,  $3MnO + 2Al = Al_2O_3 + 3Mn$

So, 48gm oxygen requires 54gm aluminium for reduction.

So, 0.841 gm oxygen requires  $(54/48 * 0.841)$  gm aluminium = **0.946 gm aluminium**

**For Tabling Sample of EOR = 73.37 %**

160gms  $Fe_2O_3$  contains 48gms oxygen

So, 2.255gms  $Fe_2O_3$  contains  $(48/160 * 2.255)$  gm oxygen = 0.6765gms oxygen

87gms  $MnO_2$  contains 32gms oxygen

So, 6.610gms  $MnO_2$  contains  $(32/87 * 6.610)$  gm oxygen = 2.431gms oxygen

Total oxygen in 10gms ore sample =  $2.431 + 0.6765 = 3.1075$ gms

For 100% EOR, 3.1075gms oxygen has to be removed.

So for 73.37 % EOR,  $(3.1075/100 * 73.37)$  gm oxygen was removed = 2.27 gm oxygen was removed

Now assuming the entire oxygen from hematite was removed and the remaining oxygen was present in MnO

The amount of oxygen removed from  $MnO_2 = 2.27 - 0.6765 = 1.59$  gm

Amount of oxygen left to be reduced from MnO =  $2.431 - 1.59 = 0.841$  gm

Now,  $3\text{MnO} + 2\text{Al} = \text{Al}_2\text{O}_3 + 3\text{Mn}$

So, 48gm oxygen requires 54gm aluminium for reduction.

So, 0.841 gm oxygen requires  $(54/48 * 0.841)$ gmaluminium = **0.946 gmaluminium**

**So Total Alumunium needed as reductant = 0.946 + 1.125 + 1.11 + 0.946**  
**= 4.127 gms Al as Reductant.**

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