EXTRACTION OF IRON FROM USED BLASTING SAND

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DECLARATION

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ABSTRACT

Blasting sand is used as an abrasive material for sand blasting process. In Sri Lanka blasting sand made of copper slag is used for treating the ship surfaces before painting. In a year about 6000 tons of used blasting sand is rejected as a waste. This waste is a huge problem because this sand cannot be directly dumped to the environment due to environmental pollution.

Sieve analysis, Magnetic separation, Energy dispersive X-ray analysis (EDAX) tests were done in the characterization step. According to the EDAX results average iron (Fe) amount of raw material was 36% and it was increased up to 48% after the magnetic separation. Magnetic separation results showed that there were 88% percent of magnetic materials available in the raw material. Magnetic separation test for retaining materials of sieves were showed that increase of magnetic amount with the decrease of particle size giving average 82% of magnetic percentage for particles larger than 500 microns and 98% of magnetic percentage for particles smaller than 500 microns.

Considering characterization data direct reduction method was selected as the extraction method. Best raw material mixing proportion and sintering temperature was found from the optimization process done for different pellet combinations. Pellets prepared with 100 parts of used blasting sand, 7 parts of Coke and 6 parts of Arawakkalu lime and fired in 1200^0C temperature was shown excellent results for Apparent porosity test and Crushing strength tests. The subsequent melting of these pellets in the lab scale cupola recovered iron as “metallic pigs”, which confirmed to alpha iron.

Keywords: *Blasting sand, Copper slag, Iron Extraction, Direct reduction.*

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ABBREVIATIONS

Fe_2SiO_4 – Fayalite

Fe_2O_3 – Hematite

Fe_3O_4 - Magnetite

EDAX – Energy-dispersive x - ray analysis

XRD – X-ray diffraction analysis

1. INTRODUCTION

1.1 Introduction of blasting sand

Sand blasting is a surface treating method that uses stream of pressurized abrasive materials against the surface to be treated [1]. There are different kind of abrasive materials used in various field. Among them aluminium oxide, silicon carbide, crushed glass, glass beads, steel grids, steel shots and copper slag [2] are widely used in industries like metallurgical, metal forming, moulding, automotive, aviation and shipyards.[3] As copper slag is widely used as blasting material due to low cost, low dust level, lower crystalline consumption.[4] Sand blasting prepared from copper slag is widely used in Dockyard, Sri Lanka for surface cleaning before applying paint on ships.

When blasting sand is used for multiple times it becomes unusable. Dockyard produces about 6000tons of waste from blasting sand per year. This waste cannot be directly dumped due to environmental pollution and strict environmental regulations, limited availability of disposal sites in Dockyard area. [5]

This waste contains valuable metals like iron, copper, titanium and zinc. Extraction of economically valuable materials can add value to used blasting sand and removal of natural hazardous metals helps to overcome environmental issues.

Most available metal in this blasting sand is iron. So the main objective of this research is to minimize environmental issues and add value to this waste by extracting iron.

1.2 Objectives

- Characterize used blasting sand to gain a thorough understanding of its properties and select an appropriate value addition process.
- Identify the most suitable method for adding value to the material.
- Optimize the value addition process for efficiency and effectiveness.
- Execute the value addition process to assess practicality and evaluate the outcomes.

- Analyze the results obtained from the value addition process for further insights and improvements by extraction of iron

2. LITERATURE SURVEY

Iron is that the fourth most abundant metal within the earth’s crust and therefore the most abundant transition metal. Iron can form complexes with oxygen because it can easily change valence. So iron is mostly available as oxide forms. Iron ores are rocks and minerals from which metallic iron are often economically extracted. They are usually rich with iron oxides and vary in color from dark grey, bright yellow, or deep purple to rusty red. Major iron ore minerals with their iron percentages are in table 2.1,

Table 2.1: Major iron ore minerals with their iron percentages

Mineral	Formula	Average iron percentage (%)	Colour
Magnetite	Fe_3O_4	72.4	Black
Hematite	Fe_2O_3	69.9	Black to silver gray, in earthy forms is red
Goethite	$Fe_2O_3.H_2O$	62.9	Yellow-brown to red
Limonite	$FeO(OH)\cdot n(H_2O)$	55.0	Yellowish brown
Siderite	$FeCO_3$	48.2	Pale yellow to tannish, grey, brown

Iron ore can be classified according to the commercially, geologically and beneficiation requirements. Size, grade, secondary minerals and treatments were considered for commercial classification. Main genetic types, structure and mineral composition is important for geological classification of iron ore. Classification based of beneficiation is logically based on the iron-bearing mineral containing in the ore.

Iron mining methods vary depending on the type of ore being mined. Currently there are four major types of ore deposits in operation, depending on the geology and mineralogy of the ore deposits which are titanomagnetite, magnetite, pisolitic ironstone massive and hematite deposits.

2.1 Impurities of iron ore

There are impurities which lower the worth of iron ores or make them valueless. Sulphur in the ore makes iron and steel brittle by going partially in them. Although it is possible to remove sulphur by burning ore and other methods, the presence of a significant amount can be objectionable. The phosphorus in steel makes it brittle. The limit for phosphorus for steel production is one hundredth of one per cent for ten per cent of average iron in ore which is called Bessemer limit. Bessemer ores carrying 52% of iron should not exceed 0.052% of phosphorus and not more than 0.04% of sulphur. But the presence of phosphorus in iron increases the liquidity and tends to form sound castings, phosphorus rich ores are used. Titanium is opposed by users of iron ore, as it is said to form a pasty slag and in other ways interfere with the smooth running of the blast furnace.

Average iron percentage in the ore have to be not less than 50% as mined to use it without beneficiation by washing, cobbing, calciting and magnetic concentration. Fine ores are found in very large quantities; And under favorable conditions, it will be able to be used by a certain concentration process. [6]

2.2 Iron availability and production in 2019

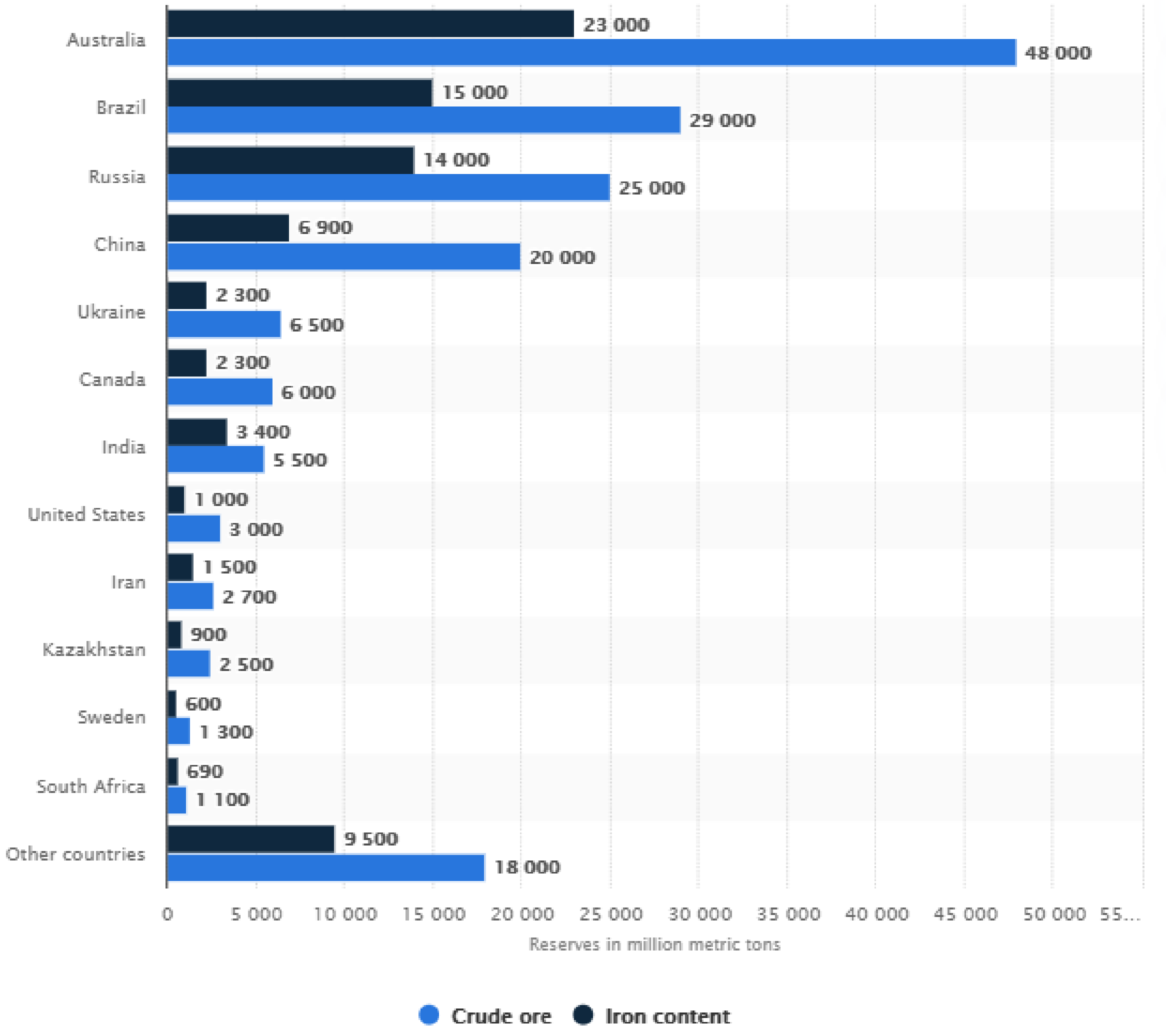


Figure 2.1: World reserves of iron ore in 2019, by country [7]

According to the figure 2.1 Top ten usable iron ore production countries in 2019 were Australia, Brazil, China, India, Russia, South Africa, Ukraine, Canada, United stated and Kazakhstan. Their iron ore production is mentioned in table 2.2.

Table 2.2: Usable iron ore production in the world 2019

Country	**Usable iron ore production (Thousand tonnes)**
Australia	930,000
Brazil	480,000
China	350,000
India	210,000
Russia	99,000
South Africa	77,000
Ukraine	62,000
Canada	54,000
United states	48,000
Kazakhastan	43,000

2.3 Iron availability in Sri Lanka

Iron-ores in Sri Lanka can be summarized under three major categories like primary deposit (magnetite), secondary deposit (hydrated iron oxide) and copper-magnetite type. Magnetite, hematite and iron oxides are the most commonly available ferrous and ferroalloy minerals in Sri Lanka. According to the estimations iron ore reserves in Sri Lanka between 10-12 million tons. Bore hole tests has been conducted for most of deposits found in Sri Lanka except few bore holes for copper – Magnetite deposit in Seruvila.

2.3.1 Primary iron deposits

Wellawaya Magnetite-Hematite deposit is one of the quality ferrous and ferroalloy deposits we have in Sri Lanka. In 1999, the Geological Survey and Mines Bureau discovered a large magnetite deposit in the Buttala area in the Uva Province. [8]

Several small magnetite reserves are found in Wilagedara and Panirendawa around Sandalanka and Chilaw respectively. These are mostly surface deposits. The

Wilagedara magnetite deposite is the first bendded magnetite deposit discovered in Sri Lanka in 1959.

2.3.2 Secondary iron deposits

Hydrated iron oxide deposits are called secondary deposits. Hydrated iron oxides such as hematite, limonite and gothite and magnetite are the high grade ore deposits in Sri Lanka. These deposits are mainly concentrated in south west sector of the island, particularly in Rathnapura district. Dela, Naragolla, Opata and Poronuwa are the iron ore available areas in Rathnapura district. Less amount of secondary iron ore deposits are available in Galle and Matara. [9]

2.3.3 Copper magnetite deposit

Copper magnetite deposit in Seruwila was discovered in 1971 by the Geological Survey department. According to laboratory XRF characterization for the systematically added field ore sample confirmed that on average 0.5-0.7% Cu and 20-50% Fe were available in the sample. Estimations of ore resources are showing in the table 2.3

Table 2.3: Estimations of Ore resource [10]

Prospect	Reserve estimates(Mt)	Cu%	Fe%
Kollankulam	0.26	0.32	48.40
Arippu	0.55	1.10	42.40

2.3.4 Dispersion of Sri Lankan iron ore deposits

The highest grade of magnetite deposits occur at Vilagedara near Thambakanda in Negombo in the Puttalam district and at Panirenda near Bingiriya and Chilaw in the Kurunegala district. According to Herath (1980) and Cooray (1967), hydrated iron ores are distributed as:

- Southwest of the island; At Ambalangoda near Godakelle and Mount Lavinia on the coast of quartz veins

- In the Ratnapura district; Kalawana, Balangoda and Rakwana, in the Sabaragamuwa province. The most important deposits are in Dela, Noragolla, Opatha and Poranuwa in the Ratnapura district and Wilpita in the Galle district.
- To some extent in the Galle and Matara districts of the Southern province. Copper / magnetite deposits also occur at Seruwila in the Trincomalee district. These deposits were most recently rediscovered in 1962, 1959 and 1971, respectively.

A layer of iron rich in nodular ironstone or ferricrete is common in many parts of the island but is particularly distributed within the dry zone. It is also referred to as nodular gravel, pisolitic gravel or lateritic gravel. On crystalline rock tops, nodular ironstone are found in the dry zone and in the northern part of the island, especially in the area between Puttalam, Kurunegala and Galgamuwa.

The magnetite-hematite deposit recently discovered at Wellawaya is one of the finest ferrous and ferroalloy deposits in Sri Lanka. The iron is on several straps and is sandwiched between ordinary rocks on the surface and can be used without much difficulty by the open quarry method. Hematite, limonite, and goitite are iron ores that are scattered in irregular masses on the surface or ridges and a few feet below or near the surface.

2.4 History of iron extraction in Sri lanka

Archaeological excavations have uncovered a wealth of information on the history of metallurgical processes in Sri Lanka. Sri Lankan metal processing industry dates back to around 3000 BC. Ancient chronicles and stone inscriptions such as Mahavamsa, Thupavamsa, Poojavaliya, Culavamsa, Dhatuvamsa, Deepavamsa mention the history of the metal industry in Sri Lanka. [11]

Evidence of the smelting of iron in Sri Lanka was found at Sigiriya and Anuradhapura in the 10th-9th BC centuries. Discoveries at Sigiriya confirm the technology behind the iron smelting process. External surfaces of specimen artifacts discovered by R.Hadildet. [12] Had a high carbon content. This shows the richness of the technology of iron production in Sri Lanka. Dehigaha-Elakana, Kirioya, Balangoda

Samanalawewa near Sigiriya were among the various places that provided evidence of iron production in Sri Lanka. Both bellow driven and natural draught driven technologies were used in those furnaces. [13]

In the study of ancient iron extraction technologies in Sri Lanka. It is important to study about Hatharabage, Sigiriya and Samanalawela sites and their technologies.

2.4.1 Ancient iron extraction technology in Hatharabage

Dr.Anandacoomaraswamy has inspected an iron making furnace at Hatharabage, in Balangoda. According to him the furnace is sheltered beneath all sides opened thatched roof of a shed. As figure 2.2 furnace contains two similar units which is called double furnace and only one unit is used at a time. It contains a 'well' where the molten metal and slag is collected. 'Well' contains two holes in front and back sides. One in front through which the molten metal and slag runs out. Little opening in the posterior where an impact of air is constrained from the howls into the well. Pole is embedded from this opening chance to time to jab and test the iron to see whether it is prepared to tap out. There was a wall made from sticks and mud which protects bellows from the heat of the furnace which showing "A" in figure 2.2. Limonite is broken up into pieces a little larger than walnut and roasted previously before charging into the furnace. By roasting, a large part of moisture is driven off and some porosity would have thus been created facilitating an ingress of reducing gas required for reduction.

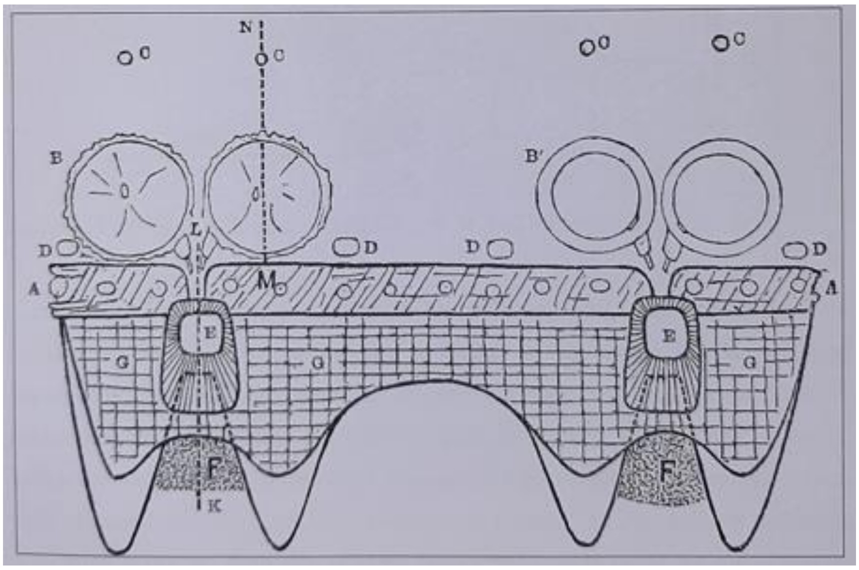


Figure 2.2: Combines plan of double furnace at Hatarabage [14]

Above A, B, C, D, E, F and G symbols are described as follows,

A-Wall of mud and wattle protecting bellows blower from heat of fire

B and B' – Bellows

C-Sticks acting as a springs to pull bellows

D-Stakes to watch the strap supporting bellows blower back in attached

E-Furnaces

F-Slope of sand, over which the slag runs out when a stick in inserted

G-Flat surface of furnace about 2 ft. 9 inches above ground at K

K, L, M, N, and N – Line of section of figure 2.2

As figure 2.3 front opening of the well bottom of the furnace in covered thickly with sand. At the lower part of the well charcoal fire is begun and in this way covered with layers of metal and charcoal till the well is filled. Additional ore and charcoal is added while the work is continuing. Occasional letting out the slag kept one person fully occupied with the work. The bellows are opened by two persons and more than three hours are normally required before the iron is ready for the recovery. When iron is found ready, the sand is cleared away, and the bloom is pushed pot through the opening, and then follows the process 'cutting the iron'

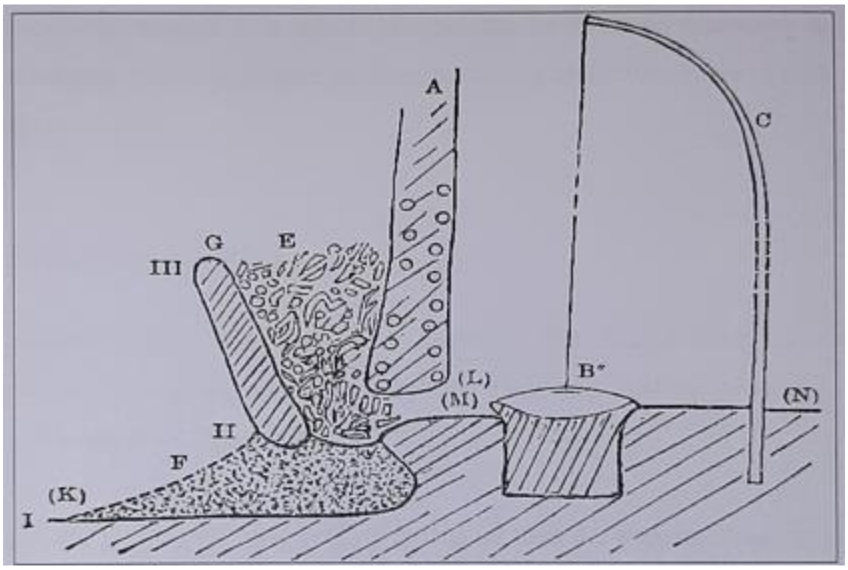


Figure 2.3: Section of furnace along KL and MN of figure 2.2 [14]

2.4.2 Ancient iron extraction technology in Sigiriya

Dehigala-alakanda near Alokawewa village and Aligala in Sigiriya were identified ancient iron ore smelting sites by the excavations done in these areas during the period 1990-1991.[15] Archaeological excavations and excavations at Sigiriya and its environs have estimated the earliest date for the smelting of iron based on the C14 carbon dating of the 10th century BC.[16] The Department of Archeology led examinations and observed proof of antiquated metal industry including iron smelting furnaces and iron scrap mounds at different spots in the Sigiriya region, Dehigaha-Ala Kanda, Alakolawawa town. The site was recognized in 1988 and uncovered in 1990 and 1991. Alakolawa is found 8.5 km southeast of Sigiriya Rock. Large furnaces were used to smelt and separate from the ores using advances bloomer process, and molten iron was used for various purposes, such as the cultivation of people around Sigiriya, irrigation, construction, and the manufacture of war weapons. Subsequently, the ascent of the iron industry added to the development of Sigiriya as an appropriate spot for urbanization. [17] The volume of iron production was assessed around from the measure of slag staying at the site. Notwithstanding, the majority of the slag was covered under the soil layers. The Department of Archeology estimates that Alokawewa has produced more than 10,000 tons of iron ore. Around 35 iron production sites have been found around the Sigiriya area, including about 5 of the same size.

Excavations at the Alokawewa site have uncovered several furnaces used in the production of iron. The furnace was made via cutting the first stone into an oval-formed pit which drawing of the structure is in figure 2.4. The front dividers are made of soil, which permits the mineral to hold heat in the heater to smelt iron. The draft was utilized to raise the temperature of the liquefying system brought about by a high smokestack and to accommodate the blast of air passed up ringers through a tuyere.

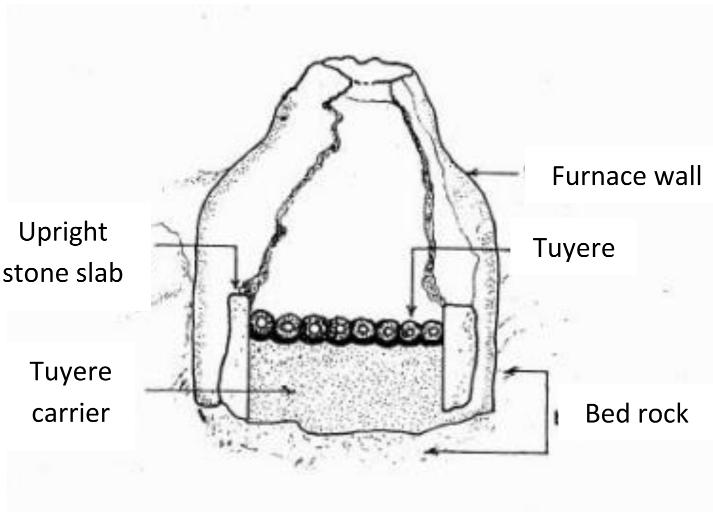


Figure 2.4: Drawing of a structure of a well-preserved furnace founded from Alokaweava site [18]

2.4.3 Wind powered iron smelting technology of Samanalawewa

Smelters in Smanalavava utilized the storm winds that lashed across the area at a speed of almost 70 Km each hour from April to August as a characteristic draught for the furnaces. Archaeologist Dr. Gil Juleff has discovered some kind of furnace. The furnace includes two basic design elements, including a semi-long-lasting back divider and a transitory straight divider. Across the front wall which was constructed on a foundation of reused tapering tuyers which are telescoped one into another and lay horizontally,to form a line as shown in figure 2.5.Another row of tuyers mounted on the top of the wall allows air to flow into the furnace at the conventional manner. The length of the furnace was 1.2 to 2 m from north to south; Depth as in the plan.The east west dimention is circa 0.3m and the high is less than 0.5m. [21] According to the and early the radiocarbon dating site belonging to the 7^{th} and early 11^{th} centuries AD.

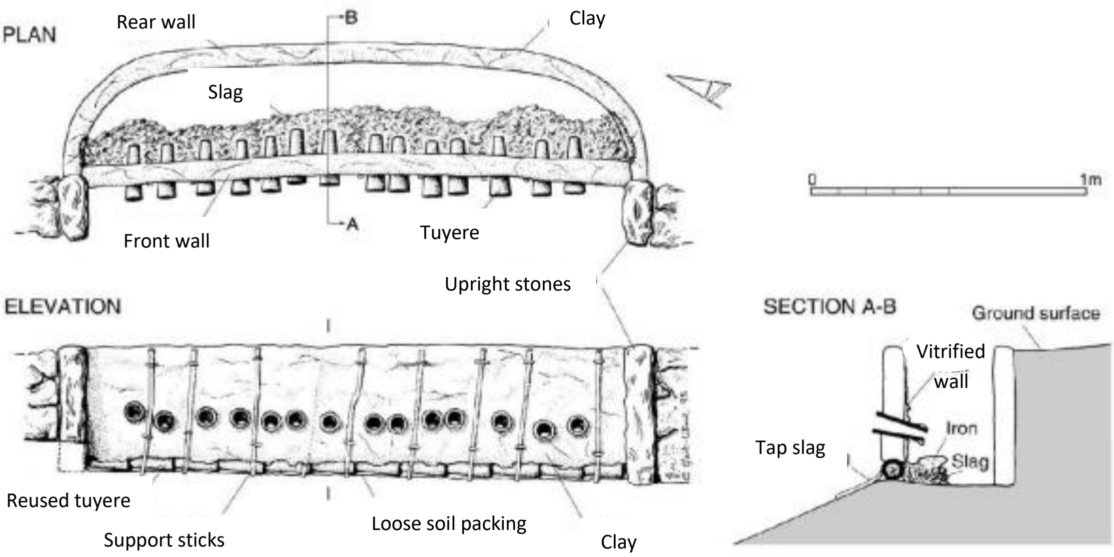


Figure 2.5: Drawing of a structure of a well-preserved furnace founded from Samanalaweva site [18]

2.5 Global copper slag generation

Generally every ton of metal production generates 2.2 tons of slag. In case of copper it produces more than 24.6 million tons of slag per year around the world. Because of that attention on cost effective waste management in this regard is an essential. [6] Characterization on copper slag has been conducted with X-ray diffraction (XRD) [5,7,8] , Scanning electron microscope (SEM)[9], X-ray fluorescent spectrometer analysis (XRF) , Atomic absorption spectroscopy (AAS)[10], X-ray conducted tomography (CT)[11] , Sieving , laser diffraction [6,8] tests. Those tests were given data about morphology, mineralogy and chemical composition. XRD test gives mineral composition data while XRF test gives chemical composition data. So XRD and XRF test data on copper slag were collected from the literature and compared with own laboratory test results for better conclusions. But consideration of literature data on copper slag was done carefully because composition, mineralogy and the morphology varies with the variation of ore, previous extraction process and cooling procedures. [6] According to the literature average 52% wt. Fayalite ($2FeO.SiO_2$) is available as major crystalline mineral with crystallization in orthorhombic system and high hardness properties [12]. Glassy phase with average 35%wt. is available as second large quantity which is an amorphous material generated due to rich silicon slag undergoes fast cooling process. Magnetite (Fe_3O_4) is available as third large quantity in copper slag with 13% which produced by over oxidation of iron oxide [6, 13]. XRF has given chemical composition analysis data in terms of weight percentage. Iron (Fe), elemental oxygen (O) and Silicon (Si) with the average weight percentage of 41.7%, 39.3% and 13.6% are available as main elements in copper slag. Except Aluminum other elements are available less than 1%. Aluminum (Al) is available 1.45% wt. and other important metals are available as follows. Copper(Cu) - 0.9% wt. , Molybdenum (Mo)- 0.121%, Cobalt (Co)- 159ppm , Tungsten (W)-92.5ppm and Nickel(Ni)-44.7ppm .[6]

2.6. The chemistry of iron extraction process

Material reduction reagents that have a very fine particle size distribution cannot be used directly and must be assembled by various processes like sintering, flaking and granulation. Direct reduction reactors and blast furnaces are the main reduction reactors used. Most common product from blast furnace is molten iron because reduction and melting happens inside blast furnace. Reduction reduces iron as a solid, and therefore the product is the so-called reduced iron (DRI). The list of physical, chemical and qualitative specifications of the tablets to be used in the iron making process must have the required characteristics. Metallurgical properties. The pellets manufactured for use in the iron making process must have properties that conform to the list of qualitative specifications for chemical, physical and metallic properties.

Pellets and additives are utilized in the pelletizing system to accomplish those determinations. Additives like limestone, hydrated lime and dolomite are utilized to modify the synthetic synthesis of lime and are regularly used to address the essential condition. Hydrated lime-like substances go about as additives and binders. Fines for anthracite or coke are added during the boxing system to decrease the fuel utilization needed to heat the ball inside. The pellets are obtained by adding enough water to the iron storage focus; this mineral is often a critical component in the formulation and development of pellets that allow surface pressure to be controlled and held together.

The total stress on the fine particles caused by water is called the neutral stress. However, moderate stress is not enough to sustain a solid grain such as iron ore. Also, when the pellets are heated, the water tends to evaporate and dissolve.

Binders are added to the adhesive to prevent such effects and to:

- Increment the strength of the pellets prior to heating which is called green strength
- At the point when a lot of gas created during water dissipation will in general break the pieces, keep the pellets from imploding during the underlying phase of heating.

2.6.1 Direct reduction method for iron extraction

Direct reduction method is classified as the processes that produce iron by reduction of iron ore below the melting point, and the product is referred to as direct reduced iron. The process that produce liquid metal like blast heater fluid metal straightforwardly from metal are alluded to as immediate smelting process. The purpose of some processes is to produce liquid steel directly from ores, and these processes are classified as direct steel making processes. Direct reduced iron (DRI) is a high quality metal product made from iron and is used as the raw material. In electric arc furnaces, blast furnaces and other more recent steel making processes.

Direct reduction is characterized as a process of creating strong iron out of small grain iron ore, pellets or lump ore, applying flammable gas or coal-based direct reduction specialists at somewhat low temperatures. Since impacting sand is dusty material pellets were made for direct reduction process. There are many benefits on agglomeration as follows, Raw material benefits:

- prevention of dust loss
- Increase porosity, density and melting abilities
- Simplified transportation

End product benefits:

- Improvement of product characteristics
- Dust free product handling
- Segregation prevention
- Enhanced appearance

Process benefits:

- Increased process flow effectiveness
- Increased process efficiency

General benefits:

- Significant dust reduction
- Improved handling and transportation
- More compatible utilization of raw material
- Improved product characteristics

2.6.2 Iron extraction from direct reduction process

In conventional iron reduction method iron ore is reduced by stepwise reaction in the blast furnace by removing oxygen from iron oxides. But there is growing tendency to produce iron by "direct reduction" method. In direct reduction oxides are reduced to metal below the melting point of the metal. This method is environmentally friendlier when compared to blast furnace method because this method use less coke. Furthermore, direct reduced iron has a well-defined chemical composition. Selection of suitable reducing agent and porosity of lump iron ore or agglomerates is very important.

2.6.3 Selection of reducing agent

Reduction is the removal of oxygen from metal oxide to extract metal. This can be achieved by adding another element or compound which is more affinity to oxygen than the metal oxide. This element or compound is known as the reducing agent. Reducing agent should have following properties,

1. Reducing agent should have a compound which is more stable than the feed compound. In other words Gibbs free energy change for that compound should more negative than the feed compound.
2. A non-metallic element or a compound

An Ellingham diagram which is a plot of Gibbs free energy (▲G) vs. temperature of metal oxides can be used to select proper reducing agent. **(Appendix B)** Carbon (C) has selected as reducing agent because it produce carbon monoxide (CO) which has more stability compared to iron oxides. To reduce the metal oxide all necessary is to heat the reactants to temperature at where carbon monoxide is more stable than iron oxide. [20]

Carbon monoxide (CO) which is the oxide of selected reducing agent carbon (C) is more stable than oxides of iron above 700^0C. So iron can be extracted by heating iron oxides with carbon above 700^0C.

2.6.4 Steps of Iron ore reduction

Fe_2O_3 is the highest oxide of the iron according to the principle of consecutive transformation of oxides. The sequence of reduction is depends on the strengths of the bonds between oxygen and iron. The affinity of the oxides for oxygen increases as Fe_2O_3 (hematite), Fe_3O_4 (Magnetite), FeO (wustite), Fe

Those stable oxides can be occurred temperature above 570^0C. Wusatile is a non-stoichiometric compound which oxygen content varies with temperature and degree of reduction ($FeO_{(1+X)}$, X varies from 0.05-0.12 depending the conditions used). Hematite has closely packed hexagonal structure while magnetite and wustite having face centered cubic (FCC) structure.

2.6.5 Iron oxygen system

This system possibly the most extensively studied system which express depth knowledge about the stability of iron bearing compounds and the thermodynamics of the system are well understood and there is much information on the kinetics of gaseous reduction related to iron oxide. (Figure 2.6)

Hematite (Fe_2O_3) is bimorphic at room temperature and the higher temperature phase remains stable at low temperature too. Higher temperature phase is alpha-hematite (α-Fe_2O_3) and lower temperature phase is gamma-hematite (γ- Fe_2O_3) where both are ferromagnetic. Hematite has rhombohedral crystal structure while, maghemite has an inverse spinal structure as magnetite with vacancies in the cation lattice.

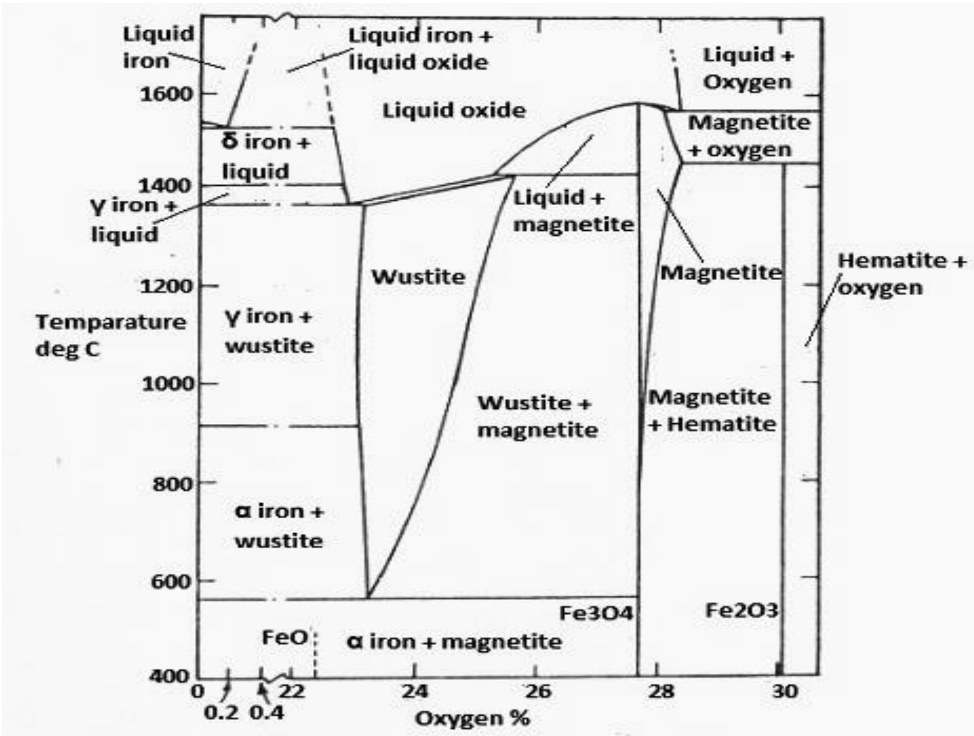


Figure 2.6: Iron – Oxygen phase diagram [21]

There is a controversy about the transformation temperature of α-Fe_2O_3 to γ- Fe_2O_3. But γ- Fe_2O_3 is stable in 773K and even in 898K. The existence of β–Fe_2O_3 was suggested by Finch and Sinha which is stable 773K-973K. The reversibility of transformation has also been observed.

In the presence of carbon monoxide (CO); Hematite reduced to Magnetite,

$$3Fe_2O_{3(s)} + CO_{(g)} \longrightarrow Fe_3O_{4(s)} + CO_{2\,(g)}$$

Fe_3O_4 has the same atomic structure as maghemite (γ - Fe_2O_3) and it is known to exist with porous as lath shaped microstructure depending on the temperature of formation and oxygen activity of the reducing gases. [22]

Magnetite has inverse spinel crystal structure with a unit cell of 32 oxygen atoms as shown in figure 2.7. It has a face-centered cubic structure and the edge length of unit cell is 0.839 nm [1]. Half of the Fe II ions and Fe III ions in this crystal structure occupy eight octahedrals and the other half of the Fe III ions occupy square places. [23]

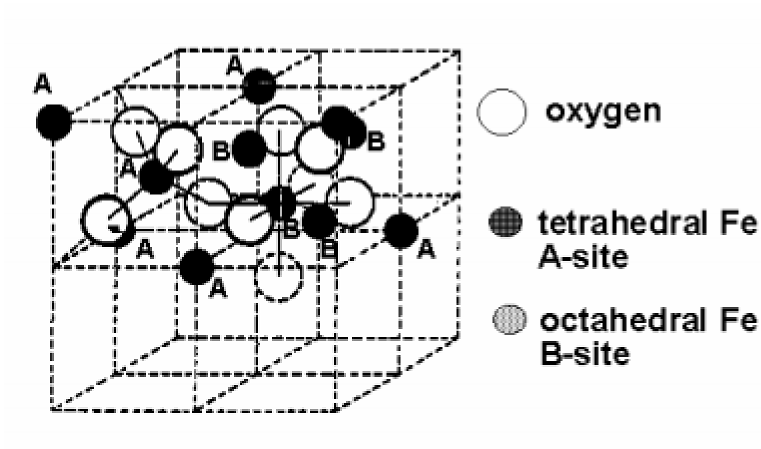


Figure 2.7: Crystal structure of Magnetite

The reduction of iron oxide to iron happens as a result of solid state diffusion of ferrous iron. Wustile is unstable at the temperature less than 843K. So magnetite is converted to sponge iron as follows.

$$Fe_3O_{4(s)} + CO_{(g)} \longrightarrow 3Fe_{(s)} + 4CO_{2(g)}$$

2.6.6 Diagram of Partial pressure of CO vs. temperature

Equilibrium of Fe and Fe Oxide with a mixture of CO and CO_2 (carbon dioxide) gases with solid carbon (C) are showing in the diagram in figure 2.7

This graph explains that reduction reactions only attained at the given temperature at the defined ratio of partial pressure (P_{CO}/P_{CO2}) or molar concentration in gaseous phase. These conditions are common with blast furnaces and cupolas and hence its importance.

In the following diagram upper field shows conditions for the stable existence for metallic iron. The mole ratio (CO/CO_2) is higher than the required value for an equilibrium at given temperature. So iron oxides reduced to metallic iron. By increasing the CO amount take equilibrium far away and promotes reduction reaction.

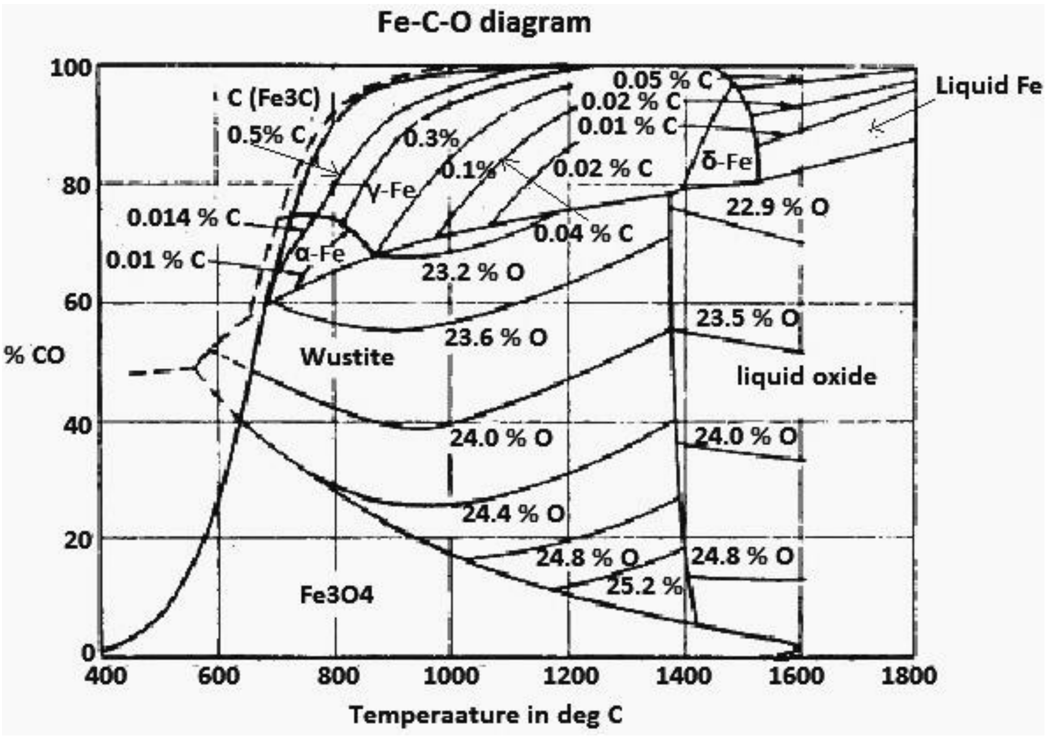


Figure 2.8: Equilibrium of iron and iron oxide with a mixture of CO and CO_2 (carbon dioxide) gases with solid carbon (C) [21]

3. MATERIALS AND METHODS

This research was conducted using waste materials as major component, such as used blasting sand and Arawakkalu lime, and followed the direct reduction method for iron extraction. This method was selected after the characterizing and the analyzing the raw material. The process was optimized for better results.

3.1 Materials

The following materials were mainly used for the iron extraction from used blasting sand. Additionally, moisture was used for pelletizing, and some other materials were utilized during the testing procedures.

(a) Used blasting sand – Iron resource *

(b) Metallurgical coke

(c) Aruwakkalu lime **

* Used blasting sand was collected from Dockyard, Sri Lanka

** Arawakkalu lime is calcite which was collected from Aruwakkalu mine at Puttalam.

3.2 Apparatus

The extraction process and the testing were conducted according to the standard procedures using following apparatus available in the laboratory.

i.	Laboratory Jaw Crusher	– Laboratory Type H crusher Denver Co.Ltd, USA
ii.	Laboratory Tema Mill	– Laboratory Disk Mill TemaBumburyMachiney Ltd Type T 300
iii.	Ball mill	- W.M. Boulton Ltd Engineers Type B.R. Jar Mill, RPM: 40 Inner diameter: 20cm, Inner height: 22cm
iv.	Sieve Analyzer	- Laboratory (BS) Test Sieve

Endecotts Ltd, London, England

Frame material: Brass

Mesh material: Stainless steel

Mesh: 106μm to 1mm

v.	Oven	- Cube Laboratory Coke Baking Oven Thk- 23 Maximum Temperature: 200^0C 6KS – 1021
vi.	Cupola	- Laboratory cupola fabricated by Mr(Eng.) S.P.Guluwita
vii.	Universal Testing Machine	- Hte Hounsfield Max Load: 50kN Max Speed: 500mm/min
viii.	Furnace	- Laboratory Furnace Nabertherm Co. Ltd German Maximum Temperature: 3000^0C
ix.	SEM	- Scanning Electron Microscope

3.3 Iron extraction experimental Procedure

Research was conducted mainly in three phases such as,

1. Characterization of blasting sand

2. Selection and optimization of iron extraction process

3. Extraction of iron from blasting sand

Characterization was conducted using different tests such as EDAX analysis, sieve analysis and magnetic separation. Three different sampling methods were used during sample collection to ensure the uniform representation of the material. Direct reduction method was used as the extraction process and process optimizations were done by enhancing the quality of the pellets through experiments. Flow chart of the iron extraction process is shown in figure 3.1.

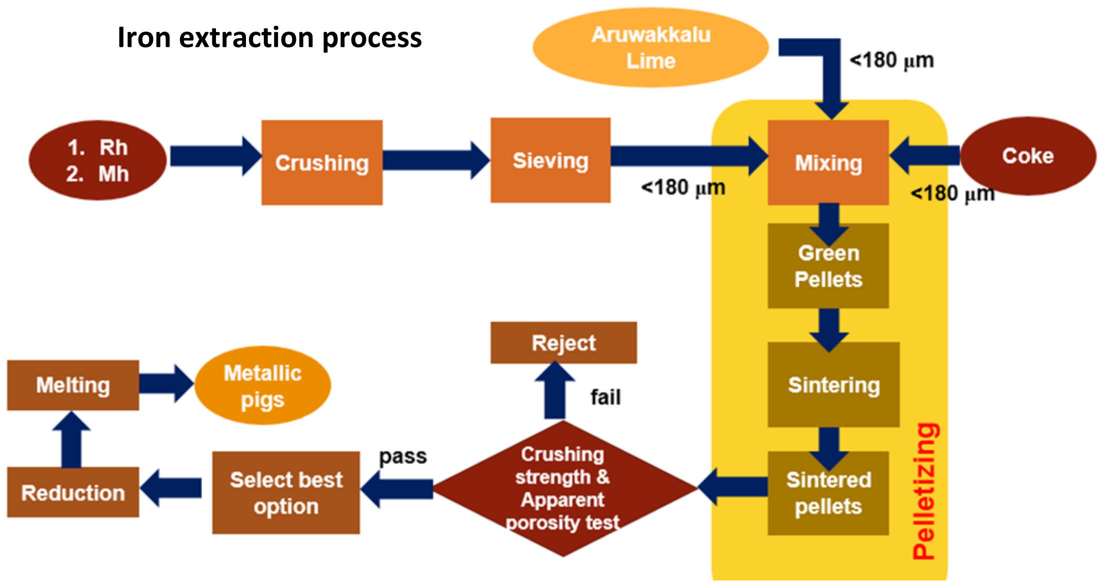


Figure 3.1 – Iron extraction process flow chart from blasting sand

3.3.1. Characterization of blasting sand

Material used for characterization was ship blasting sand collected from Dockyard, Sri Lanka. Test samples were taken using different sampling methods to get better representation of the lot been analyzed. Random sampling was used as first sampling method. This method avoids unconscious bias of researchers and opens equal chances to represent the lot from different locations [14]. But this method is suitable for large sample preparations than small laboratory experiments. As second method cone and quartering sampling was used. Sample had shoveled well before making the conical pile. The conical pile should be symmetrical and the cone had flattered into the shape of disk. Then the disk was divided into two quarters using perpendicular boards. Finally one pair from the opposite pair was removed and the remaining pair has used as a test sample. [15] This was done 3 times as the required test sample was small. Cone and quartering method is better for the large lots [16]. Grab sampling was the third method used for sampling. Material was put on a glazed paper and mixed well and homogenized by rolling glazed paper in repetitive sequence of A to C,D to B,C to A and B to D as showing in the figure 3.2[15].

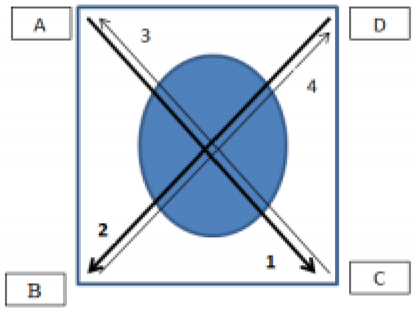


Figure 3.2 – Sequence of rolling

The sample was divided into sub samples as incremental division and the test sample was made of those sub samples. Grab sampling method has mainly been used for sampling and this method is the most suitable one as laboratory sampling method with advantages of flexible, quick and required sample size. The characterization of blasting sand was done mainly under three tests as energy dispersive x-ray analysis (EDX/ EDAX), magnetic separation and sieve analysis. EDAX was done by scanning electron microscope (SEM). EDX/EDAX analysis provides spectra with peaks corresponding to the elements with providing qualitative and quantitative analysis [17]. EDAX analysis was done for the raw samples of blasting sand taken by using random sampling and grab sampling to get quantitative analysis of elements in blasting sand. Then EDAX analysis was done with magnetic samples which were taken after the magnetic separation of raw material. Magnetic properties help to retrieve target minerals from the gang [18]. So magnetic separation was done with the test samples taken from random sampling, grab sampling and cone and quartering sampling. It was done with six samples as two from each sampling method in laboratory magnetic separator. Particle size distribution affects the physical properties of the material and it is important for further processing. In the extraction processes particle size distribution emphasize crushing and grinding requirements of the material before processing [19, 20]. Sieve analysis is an analytical technique used for determination of particle size distribution of different materials. Sieve analysis was done as the third characterization technique by using British standard (BS) sieves according to dry sieve analysis method [21]. Furthermore retaining materials of each sieve were subjected to magnetic separation independently to study the magnetic and nonmagnetic particle distribution along with the particle size.

3.3.2. Selection and optimization of iron extraction process

Direct reduction process was selected as iron extraction method after considering the physical and chemical properties of raw material.

3.3.2.1 Iron pelletizing and preparation

The raw materials used for pellet making were blasting sand, coke, Aruwakkalu lime and moisture. Coke was used as the fuel and reducing agent. Aruwakkalu lime was the binder, fluxing agent and slag producer. Appropriate amount of water was added to ore concentrate to obtain pellets, this was a fundamental factor for formation and growth of pellets because it creates surface tension to holds mineral grains cohesive.

Blasting sand was crushed in the Tema mill and took 180 micron passing material for further processing. Coke and Aruwakkalu lime was also crushed, sieved and 180 micron was taken to mix with blasting sand. They were mixed according to required proportions and mixed for 10 minutes in laboratory ball mill for homogeneous mix. Then moisture was added to agglomeration. Size of the pellets were maintained between 8-14mm diameters according to the standard. Pellets were air dried for 2 days and oven dried for 2 hours at 110^{0}C sintering them in the furnace. Pellets were sintered in different temperatures which are above 1200^{0}C and let for oven cooling.

3.3.2.2 Optimization and quality testing of pellets

Two type of blasting sand were taken. They were, raw blasting sand as it is and magnetic part of the blasting sand after the magnetic separation. Different types of pellet combinations were made by changing proportion between blasting sand, coke and Aruwakkalu lime as shown in table 3.1.

Table 3.1 – Pellet combinations and their composition ratio

Raw Material	Blasting sand	Coke	Aruwakkalu Lime	Name	Sintering temperature (⁰C)	Name
Blasting Sand	100	7	6	Rh76	1200	Rh761
					1250	Rh762
					1300	Rh763
					1350	Rh764
	100	7	7	Rh77	1200	Rh771
					1250	Rh772
					1300	Rh773
					1350	Rh774
	100	7	8	Rh78	1200	Rh781
					1250	Rh782
					1300	Rh783
					1350	Rh783
Magnetic Blasting Sand	100	7	6	Mh76	1200	Mh761
					1250	Mh762
					1300	Mh763
					1350	Mh764
	100	7	7	Mh77	1200	Mh771
					1250	Mh772
					1300	Mh773
					1350	Mh774
	100	7	8	Mh78	1200	Mh781
					1250	Mh782
					1300	Mh783
					1350	Mh783

Accordingly there were 24 different combinations of pellets 12 from each type of blasting sand. Quality tests were done for each type of pellet and best quality pellet was selected for further processing.

3.3.2.3 Quality testing on pellets

Crushing strength test and apparent porosity test were done as quality testing for sintered pellets EDAX tests were done for both green pellets and sintered pellets to get proper comparison.

3.3.2.4 Crushing strength test

Iron pellets used in iron production require specific mechanical properties to withstand various stresses during transportation, handling, and processing. To ensure their suitability, a crushing strength test was conducted to verify that the pellets can endure the mechanical forces encountered, such as impacts from falling or stacking in reactors. This test was crucial for confirming that the pellets meet the necessary strength requirements for their intended use.

Figure 3.3 – Universal tensile testing machine

Crushing strength tests was done in universal testing machine shown in figure 3.4 according to the standard ASTM – E 382 – 07. The test was done in slow rate as 15mm min^{-1}. Crushing strength should higher than 200KgF according to the quality requirements of the pellets.

3.3.2.5 Apparent porosity test of iron pelletizing

Apparent porosity was measured by the water absorption method according to the standard ASTM – C20– 00. Apparent porosity values has to be in 25%-30% to fulfill the quality requirements.

Fired pellets have oven dried at 110^0C untill get constant weight. Then the dry weight has measured(m_0). Then pellets were bollied for 2 hours while they are fully immered in the water. After keep them fully immersed in the water for additional 12 hours samples were weighted while suspended the water(m_2). Finally the saturated weight (m_1) was measured by blotted each sample by smooth tissue to remove adhering water droplets.

Apparent porosity = $\frac{Volume\ of\ open\ pores\ in\ pellet\ (m1-m0)}{Exterior\ volume\ of\ pellet\ (m1-m2)}$

Where,

m_0-dry weight

m_1-Weight of pellets after saturated with water impregnation

m_2- Weight of pellets while suspended in water

3.3.2.6 Determination of moisture content of green pellets

Moisture content of green pellets are required to be in between 10-12%. Following equation has been used to calculate moisture content of green pellets,

Moisture content = $\frac{(Wg - Wd)}{Wg}$ x 100%

Where,

Wg – Weight of green pellet,

Wd – Weight of green pellet after drying at 110^0C for 2 hours

3.3.2.7 Optimization of iron pellets

Pellet combinations which fulfilled both of above quality requirements were selected for further extraction process. As a result of pellets which pass both test are more than one EDAX comparison between green pellets and sintered pellets and physical characteristics had to be considered. Pellet quality enhancement and process optimization has happened with this activity. That will be further discussed in the results and discussion session.

3.4 Extraction of iron from used blasting sand

After the quality testing and optimization process selected pellet combination was produced in high amount for the further work. Reduction and melting was done in the laboratory cupola.

Coke bed was used as fuel and the temperature achieved in the test was 1400^0C. Molten iron was tapped finally. Extracted iron was tested in EDAX analysis and XRD.

4. RESULTS AND DISCUSSION

In this section presents the findings obtained from the experiments and analyses conducted during the research. Data, observations, and measurements related to the iron extraction process, as well as characterization tests and optimization experiments, are presented and analyzed. The implications of the findings are discussed within the broader context of the research objectives, with comparisons to existing literature and theoretical frameworks.

4.1 Experimental work

The experimental work in this dissertation focuses on the extraction of iron from used blasting sand utilizing the direct reduction method. The research involved a comprehensive series of experiments designed to optimize the extraction process. ASTM standards were followed through the experimental work.

4.1.1 Green pellets

Green pellets were prepared following ASTM E382-20 standard. Diameter of the pellets were maintained between 8mm to 14 mm according to standards. Some images of green pellets are shown in figure 4.1.

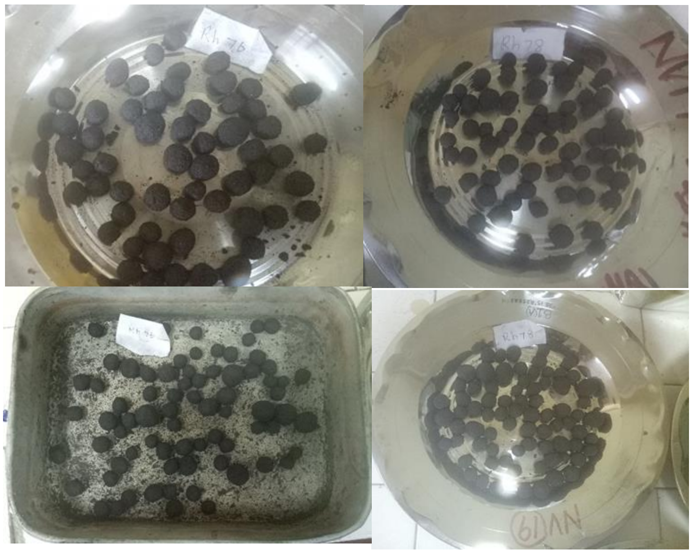


Figure 4.1:Green pellets

4.1.2 Sintered Pellets

Pellets were sintered at four different temperatures. Green pellets were air dried and owen dried before sintering according to the ASTM E382-20 test procedures. Figure 4.2 is shown some images of sintered pellets during the experiment work.

Figure 4.2: Sintered pellets

4.1.3 Reduction and Melting

Reduction and melting was done in the laboratory cupola furnace and the coke bed was heated up to 1400^{0}C temperature. Figure 4.3 is shown the side view of the furnace when coke bed is heated and figure 4.4 show the top view of it.

Figure 4.3: Heated coke bed

Figure 4.4 : Mirror image of inside

4Kg of pellets were prepared from the selected pellet combination for reduction and melting. Pellets were charged to the cupola furnace as figure 4.5. metallic pigs were received after the melting as shown in figure 4.6(a) and figure 4.6(b)

Figure 4.5: Charging of pellets

Figure 4.6(a): metallic pigs

Figure 4.6(b): metallic pigs

4.2 Results and Discussion of chemical analysis

This section presents and analyzes the findings of the study, discussing their implications within the context of the research objectives.

4.2.1 Chemical composition of coke and lime

Chemical analysis was done for the coke used in the extraction process and the observed results are shown in the table 4.1

Table 4.1: Chemical analysis of coke

Overall analysis	
Constituent Determined	**Content (%)**
Moisture	0.52
Volatiles and carbon	91.91
Ash	7.57
Ash analysis	
Constituent Determined	**Content (%)**
SiO_2	42.05
Al_2O_3	18.11
Fe_2O_3	29.91
CaO	5.19
MgO	0.70
MnO	0.05
TiO_2	0.76
Na_2O	1.34
K_2O	1.81

Chemical analysis test results of the Aruwakkalu lime is shown in table 4.2

Table 4.2: Chemical analysis of Aruwakkalu lime

Constituent Determined	Content (%)
SiO_2	4.00
Al_2O_3	0.31
Fe_2O_3	0.31
CaO	52.80
P_2O_5	0.32
MgO	0.02
MnO	0.01
TiO_2	0.06

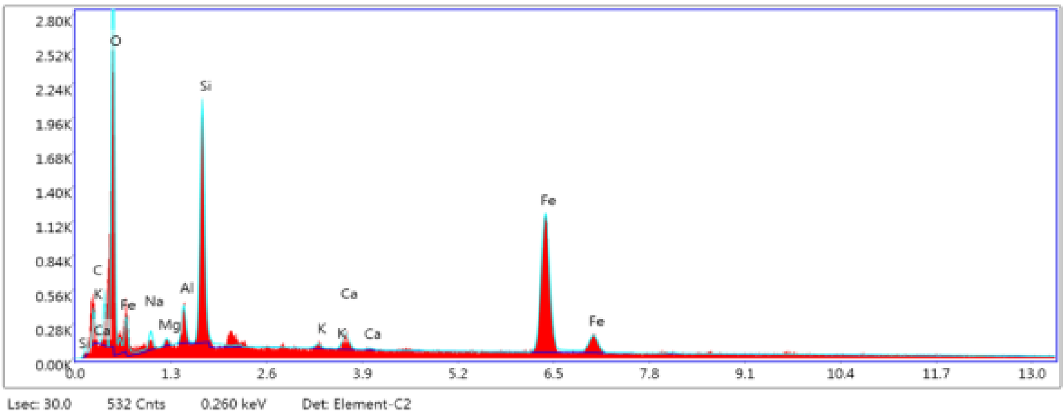
Na_2O	0.05
K_2O	0.03
Loss of ignition	39.87
Insoluble residences	2.14

4.2.2 EDAX analysis of blasting sand

Figure 4.1, figure 4.2, figure 4.3, and figure 4.4 show the EDAX analysis results of blasting sand samples collected using different sampling methods. The figures clearly display the peaks and weight percentage values for specific elements.

4.2.2.1 EDAX results of blasting sand taken by random sampling technique

EDAX results of figure 4.1 showed that most available element is Iron (Fe) with average percentage of 36.71%. Second and third places were taken by Oxygen (O) and Silicon (Si) with the average percentages of 31.81% and 13.90%.

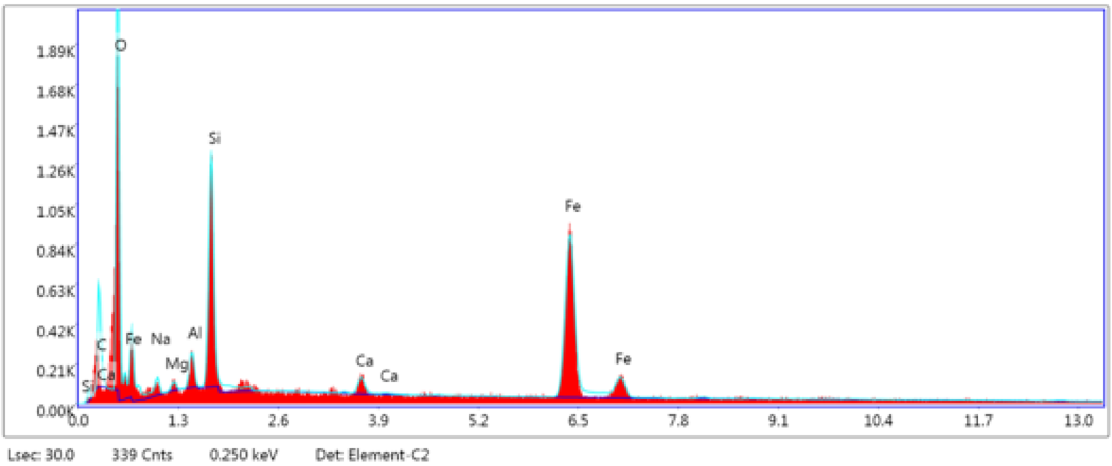


ELEMENT	C	O	Na	Mg	Al	Si	K	Ca	Fe
WEIGHT %	8.61	31.81	3.00	0.97	2.76	13.90	0.43	1.89	36.71

Figure 4.1 : EDAX results of blasting sand taken by random sampling technique

4.2.2.2 EDAX results of blasting sand taken by grab sampling technique

EDAX results of figure 4.2 give same pattern of figure 4.1 with showing most available element as Iron (Fe) with average percentage of 36.59%. Second and third places were taken by Oxygen (O) and Silicon (Si) with the average percentages of 31.83% and 11.40%.

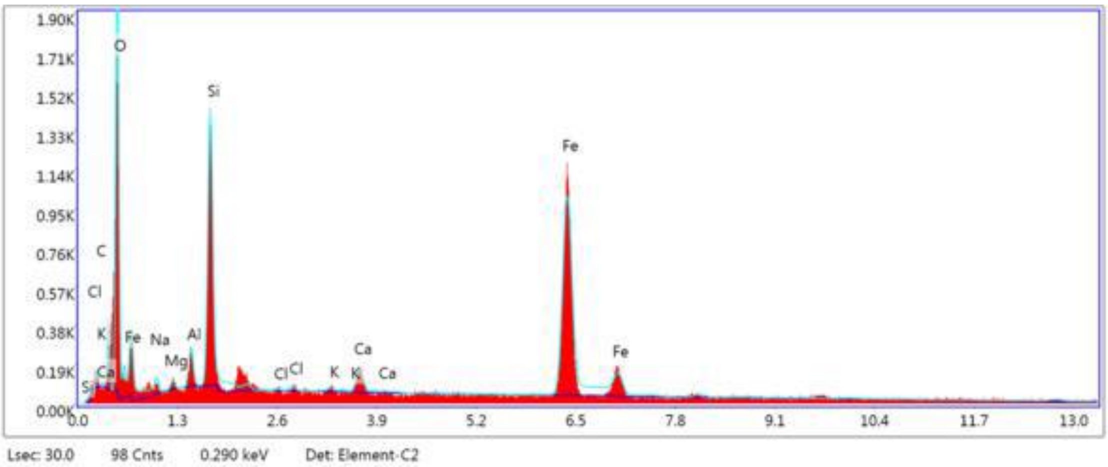


ELEMENT	C	O	Na	Mg	Al	Si	Ca	Fe
WEIGHT %	13.40	31.83	2.14	0.87	2.22	11.40	1.55	36.59

Figure 4.2: EDAX results of blasting sand taken by grab sampling technique

4.2.2.3 EDAX results of magnetic separated Blasting sand taken by random sampling technique

EDAX results done with magnetic sample of blasting sand taken by random sampling technique as shown in figure 4.3 that average Iron percentage has increased by approximately 11.62%, Silicon by 1.33%. Oxygen has reduced approximately by 4.89% while carbon reduced approximately by 8.6% which means carbon percentage reduced nearly zero after magnetic separation of the sample.

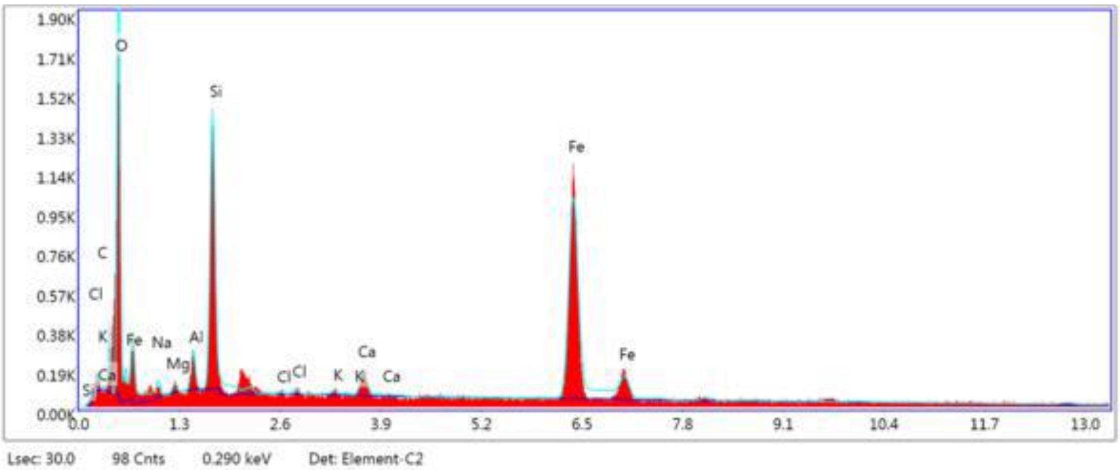


ELEMENT	C	O	Mg	Al	Si	Ca	Na	Fe
WEIGHT %	0.01	26.92	1.33	2.81	15.23	2.39	2.54	48.33

Figure 4.3 : EDAX results of magnetic separated Blasting sand taken by random sampling technique (Magnetic part)

4.2.2.3 EDAX results of magnetic separated blasting sand taken by grab sampling technique

EDAX results done with magnetic sample of blasting sand taken by grab sampling technique as showed in figure 4.4 that average iron percentage has increased by approximately 8.92%, Silicon by 3.1%. Oxygen has reduced approximately by 2.11 % while Carbon reduced approximately by 9.93 % .



ELEMENT	C	O	Mg	Al	Si	Ca	Na	Fe
WEIGHT %	3.47	29.72	0.60	2.25	14.50	1.77	1.67	45.51

Figure 4.4 : EDAX results of magnetic separated blasting sand taken by grab sampling technique (Magnetic part)

4.2.3 Magnetic and non-magnetic percentages of blasting sand used in different sampling methods

Magnetic separation for blasting sand was done with six samples and they gave 88% approximate magnetic percentage and 12% of approximate nonmagnetic percentage.It is shown in table 4.3. Objective of this test was to collect data about the Iron availability in the magnetic form and the results confirmed the high Iron availability in magnetic form.

Table 4.3 : Magnetic and nonmagnetic percentages of blasting sand taken from different sampling methods

Sample	**Magnetic percentage (%)**	**Nonmagnetic percentage (%)**
Random sampling A	86.3	13.7
Random sampling B	84.1	15.9
Grab sampling A	89.8	10.2
Granb sampling B	87.7	12.2
Cone and quartering A	91.7	8.3
Cone and quarterinb B	91.3	8.7
Average	**88.4**	**11.6**

4.2.4 Analysis of particle size distribution of blasting sand

Sieve analysis test results showed that particle size distribution of particles larger than 500 microns is high. It is clearly shown in the rapid increasing slope of cumulative distribution graph in figure 4.5

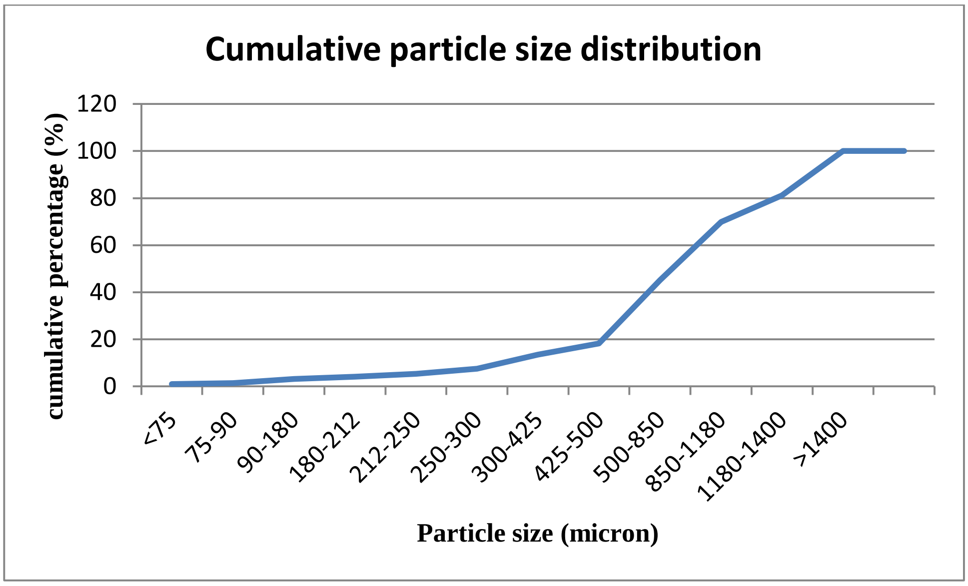


Figure 4.5 : Cumulative particle size distribution of blasting sand

4.2.5 Magnetic and nonmagnetic particle distribution with particle size

Magnetic separation was done for sieved materials to study about the magnetic and nonmagnetic distribution with the particle size. It shows that magnetic percentage increases with the reduction of the particle size. Materials with less particle size than 500 microns give high magnetic percentage than 95% while larger particles give 82% of magnetic percentage as shown in figure 4.6

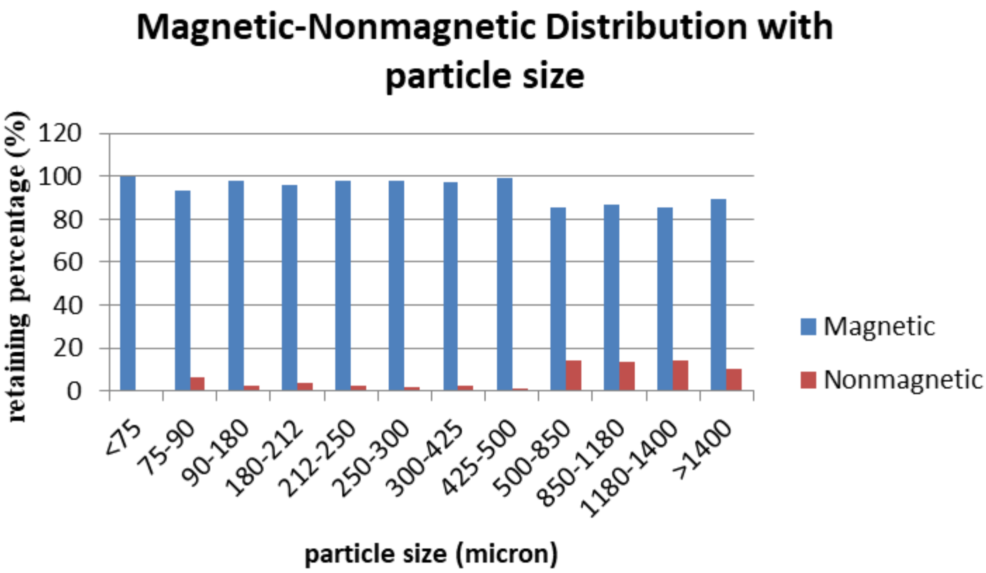


Figure 4.6: Magnetic and nonmagnetic particle distribution with particle size

Because smaller particles show high magnetic percentages, pellets were made using two different blasting sand types. Raw blasting sand and magnetic part of the magnetic seperated blasting sand. So there were two type of pellets were made. Aruwakkalu lime and coke were used as other raw materials for pellatizing.

Chemical tests were done for the Arawakkalu lime and coke to confirm that they have required compositions.

4.2.6 Comparison of moisture content of green pellets

Green pellets were subjected to moisture content test and only pellets which staisfy moisture content requirement has proceed to further process. Required moisture content value range is 10 – 12% . Moisture content of green pellets are very important for the agglomeration and bonding characteristics.

4.2.7 Crushing strength behavior of sintered pellets

Pellets were sinthered in different temperatures and the different combinations of pellets were subjected to crushing stength test and apparent porosity test. Results are showing in figure 4.7. According to the standard crushing strength should be higher than 200Kgf and apparent porosity should be between 25%-30%.

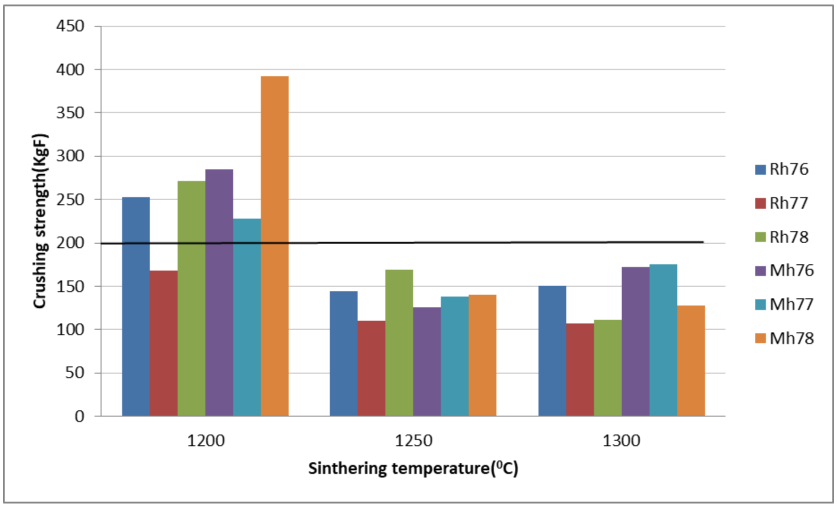


Figure 4.7: Crushing strength values graphical illustration

4.2.8 Porosity behavior of sintered pellets

Apparent porosity test results of different pellet combinations are shown in figure 4.8. One pellet has exceeded the apparent porosity quality requirement limits, while three pellets have satisfied the required quality requirements.

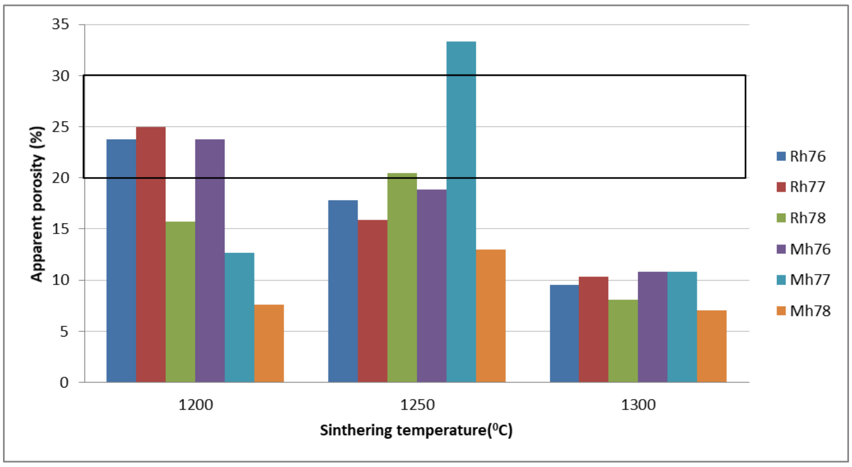


Figure 4.8 -Apparent porosity values graphical illustration

Pellet combinations which satisfy the quality requirements are shown in table 4.4.

Table 4.4 : Quality requirements satisfied pellets

Crushing strength test passed (> 200Kgf)	**Apparent porosity test passed (25 - 30)%**
Rh761	Rh761
Rh781	Rh771
Mh761	Mh761
Mh771	
Mh781	

Only Rh761, Mh761 have passed from the qulality checking test. So both pellet combination has qulified to process extraction step. Because iron percentage increase by magnetic speration is only about 10% and extra processsing steps required for magnetic seperation. Rh761 sample has selected for the iron extraction.

4.2.9 EDAX analysis of metallic pigs

Extracted iron metallic pigs were tested in EDAX analysis and results are shown in figure 4.9 and table 4.5. According to EDAX results iron percentage has increased up to 78% which was 36% before. Silicon percentage has decreased to 2% while Carbon decrease to zero.

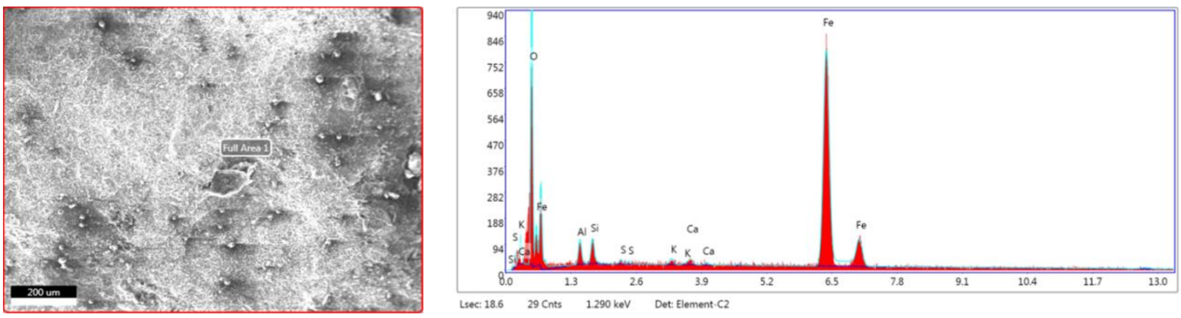


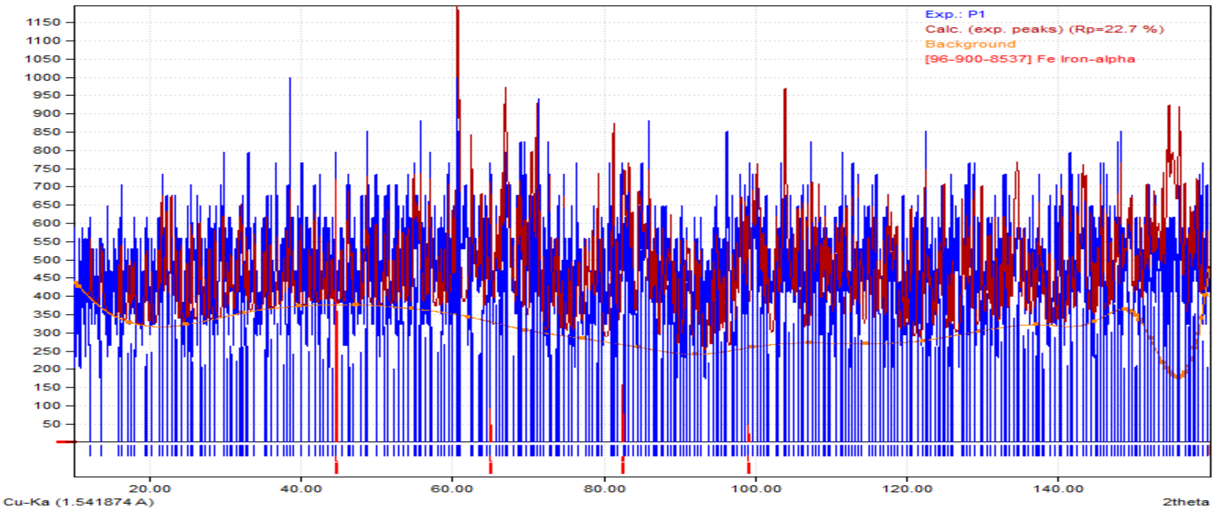
Figure 4.9: EDAX results of metallic pigs

Table 4.5- EDAX results of metallic pigs

Sample Name	Fe	O	C	Si	Cu
Metallic pigs 1	73.92	19.30	-	2.15	-

4.2.10 XRD analysis of metallic pigs

XRD test and results of metallic pigs are shown in figure 4.10. There is a formation of alpha iron according to the test results.



C	96-900-8537	Fe	Iron-alpha	0.5115	0.9963	0.7195	13.29	0.7853

Figure 4.10: XRD results of metallic pigs

5. CONCLUSIONS

As the used blasting sand contains around 37% iron, it is necessary to extract iron as the primary step in value addition to the used blasting sand. Since the material is in powder form and the iron percentage is less than 50%, the direct reduction method is optimal for the extraction process. Pellets prepared using a 100:7:6 mixing ratio between blasting sand, lime, and coke and sintered at 1200°C have yielded optimal values in quality requirement tests. As copper slag contains Fayalite around 52% by weight, and the melting point of Fayalite is less than 1300°C, pellets sintered above 1350°C have been melted away, and pellets sintered at 1300°C also showed partially melted behavior. This factor also affected the selection of the optimum sintering temperature, which was determined to be 1200°C.

Although the iron percentage increased by around 10% after magnetic separation, the pellets made from the primary used blasting sand and the pellets made from the magnetic part of the magnetic-separated blasting sand didn’t show much difference in terms of iron percentage after sintering. Considering the additional step required for magnetic separation and the low amount of iron percentage increment associated with it, magnetic separation is not necessary.

In this research, the direct reduction process was conducted in the cupola furnace. Even within the cupola furnace, the iron percentage increased up to 74%. If this process were conducted in the Corex reactor that percentage would be increased up to 90%. The main reason for the decrease in iron percentage in the cupola furnace was the contamination of iron pigs with the slag.

The average iron percentage in natural iron ore in Sri Lanka is around 50%. Mining and beneficiation processes are required to extract iron from these ores. Used blasting sand contains around 37% of iron and is available as waste in Dockyard Sri Lanka. Therefore, this material can be bought at a low price without investing money in mining. Thus, extracting iron from used blasting sand is economically advantageous compared to iron extraction from conventional iron ore.

After the extraction of iron, valuable metals can be further extracted as an extension of the value addition to the used blasting sand. Copper slag was concentrated at the bottom of the pellet when it was heated at 1200°C because its melting point was lower than 1200°C. This observation was additionally noted and could be helpful for the development of a copper extraction method from used blasting sand.

6. RECOMMENDATIONS

The direct reduction was conducted in the laboratory cupola furnace. It is advisable to utilize the Corex reactor for extracting iron from used blasting sand to minimize contamination of slag with metallic pigs. The use of the Corex reactor will enhance the purity of the extracted iron in the above process.

As used blasting sand contains a high amount of Fayalite, the sintering temperature should be maintained below the melting point of Fayalite, which is around 1200°C

Used blasting sand typically contains around 36% Iron. Implementing a beneficiation process such as leaching can significantly enhance the effectiveness of the extraction process.

Accidentally sintered pellets at 1150°C have unexpectedly met quality requirements better than those sintered at other temperatures. If 1150°C is consistently used as the sintering temperature, further experiments could potentially enhance pellet quality while reducing costs.

After extracting iron from the blasting sand, the remaining material can be repurposed for further extraction of valuable materials such as copper or utilized for other purposes like construction. This approach maximizes resource utilization and minimizes waste, contributing to both environmental sustainability and economic efficiency.

7. REFERENCES

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