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## **Direct reduction of iron at Algerian Qatari Steel (AQS)**

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# *Dedication*

*With a deep affection, I am grateful to my family, my source of success and happiness...*

*Boulaiche Fouad*

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## List of Abbreviations

<b>Abbreviations</b>	<b>Meaning</b>
AI	% content of powders
AQS	Algerian Qatari Steel
ASU	Air Separation Unit
BOF	Basic Oxygen Furnace
CCS	Compression Crush Strong
CDRI	Cold Direct Reduction of Iron
DRI	Direct Reduction of Iron
DRP	Direct Reduction Plant
EAF	Electric Arc Furnace
HBI	Hot Briquetted Iron
HDRI	Hot Direct Reduction of Iron
M	Degree of metallization
Nm <sup>3</sup>	normal cubic meters
QSI	Qatar steel international
TI	% content of pellets.
WSA	World Steel Association

# *Introduction*

# Introduction

The production of iron is a crucial and foundational component of the global economy and industrial development. Iron, in its various forms, is one of the most widely used metals worldwide due to its versatility and strength. Iron production has a rich and ancient history that dates back thousands of years. Early civilizations, such as the Hittites and Egyptians, were among the first to smelt iron and use it for tools, weapons, and construction. The Iron Age, a pivotal period in human history, marked the transition from bronze to iron as the primary material for making tools and weaponry, leading to significant advancements in agriculture, warfare, and infrastructure.

Iron and steel are indispensable materials in modern society, with a wide range of applications across various industries. They are essential for constructing buildings, bridges, and infrastructure, manufacturing vehicles, machinery, and appliances, and producing packaging materials, among many other uses. The global demand for iron and steel remains consistently high, making iron production a critical component of the world's industrial base and economic development.

The production of iron involves several key methods, with two primary approaches: smelting iron ore in a blast furnace to produce pig iron and further refining it to obtain various grades of steel. Here are the main steps in the iron production process [1]:

- **Iron Ore Mining:** The process begins with the extraction of iron ore from mines. Iron ore can be found in various forms, such as hematite, magnetite, and taconite.
- **Ore Processing:** Extracted iron ore is processed to remove impurities, typically in the form of gangue minerals. The result is a concentrated iron ore, often in the form of pellets or sinter.
- **Iron Smelting:** The concentrated iron ore is then smelted in a blast furnace. This process involves heating the ore with coke (a carbon-rich material) and limestone, which acts as a flux to remove impurities. The result is molten pig iron, a crude form of iron with high carbon content.

- **Steel Production:** To obtain steel, pig iron is further processed through various methods such as the basic oxygen furnace (BOF) or electric arc furnace (EAF). These processes remove excess carbon and other impurities to produce different grades of steel with specific properties.

The Midrex process used at **Algerian Qatari Steel (AQS)** company is a highly efficient and environmentally friendly method for the production of iron and direct reduced iron (DRI). In this process, iron ore pellets or lump ore are heated in a shaft furnace with a mixture of natural gas and hydrogen, reducing the iron oxide in the ore to metallic iron. The distinguishing feature of the Midrex process is the use of a gas reforming system that converts natural gas into hydrogen-rich gas, which facilitates the reduction of iron ore without the need for coke, a carbon-intensive material used in traditional blast furnace methods [2]. The Midrex process offers several advantages, including lower greenhouse gas emissions, reduced energy consumption, and the ability to use a wider range of iron ores, making it a sustainable and cost-effective approach to iron production.

The main objective of this work is to describe this DRI process of the AQS complex for the production of iron with properties demanded by the international market.

## References

1. Gordon, Y., Kumar, S., Freislich, M., & Yaroshenko, Y. (2015). The modern technology of iron and steel production and possible ways of their development. *Steel in Translation*, 45, 627-634.
2. Bonelli, G. (2021). North Africa: Focus on Direct Reduction Ironmaking. *Steel Times International*, 45(1), 36-42.

# Chapter I

**Description of the Bellara Steel Complex &  
Direct Reduction Process**

## I.1. Introduction

The Algerian Qatari Steel company (AQS) has been created in December 2013 to execute the new steel complex in Bellara, Jijel, Algeria. It is the outcome of a joint investment between Algeria and the Qatari counterpart. Qatar steel international (QSI), owned 49% of shares. Whereas, 46% is owned by sider industrial group and 05% by National investment funding FNI. AQS operates in the industrial zone of Bellara, in the municipality of El-Milia, Jijel province, located 400 km from the capital, Algiers. The project has two areas of implementation:

- The Bellara site (El Milia), AQS steel complex is implemented along a surface of 216 hectares.
- The Djendjen port (Taher municipality), a 10-hectare plot operated by AQS under a concession regime, equipped with facilities for receiving and transiting imports of iron oxide pellets (raw material).



**Figure I.1.** Aerial view of AQS steel complex.

## I.2. Objective of the project

The Algerian Qatari Steel (AQS) steel complex is currently operating at its full production capacity, close to 100 %. A portion of the production is intended for export to the international market, with the signing of several contracts with various foreign clients, particularly with Canada, China, Italy, Tunisia, and other markets are being finalized. As for the domestic market, it currently represents 30 % of the production, a percentage that may fluctuate depending on national demand. The project was carried out in two phases to achieve a capacity of over 4

million tons per year of steel products. AQS started production and commercialization of iron products at the end of 2017, with an annual production of two (02) million tons of rebar and wire rods of various diameters. The second phase involves the commissioning of the Direct Reduced Iron (DRI) unit, dedicated to increasing the production capacity to over 4 million tons per year. After the Gara Djebilet iron ore deposit in Tindouf Province development, Algeria will be one of the lowest cost DRI producers in the world (iron oxide pellets typically make up 80 -90 % of DRI OPEX).

### **I.3. Principal production units**

The Bellara Steel Complex is equipped with the most advanced technology in the world and includes three main environmentally friendly production units, which guarantee maximum production efficiency and product quality that meets international standards.

#### **I.3.1. DRI production unit**

A Direct Reduction unit with a production capacity of 2.5 million tons per year of reduced iron from iron oxide pellets from which oxygen is removed in a gas reduction furnace using reducing gases such as CO and H<sub>2</sub> (**Figure I.2**). Midrex Technologies, Inc. and Paul Wurth were contracted in 2016 to supply a 2.5 million tons/year direct reduction plant (DRP) capable of producing both CDRI and HDRI. First product was produced by the DRP in February 2021. During 2022, 74% of total production (1.7 million tons) was HDRI.



**Figure I.2.** DRI production unit.



### I.3.2 Melting shop unit

The two melting shops of Bellara Steel Complex are composed of electric arc furnaces with a total production capacity of 2.2 million tons per year to transform raw steel into liquid and billets with well-defined characteristics.



**Figure I.3.** Melting shop.

### I.3.3. Rolling mill's unit

Three Rolling Mills are used in Bellara Steel Complex to obtain steel products designated for shipment either in smooth or screened form. Total production capacity of 2 million tons per year for both rebar and wire rod.

## I.4. Secondary Installations

### I.4.1. Lime production unit

Lime is produced by calcining calcium carbonate or magnesium at temperatures above 800 °C, causing the decarbonation of the raw material to produce calcium oxide or magnesium oxide (quicklime). The lime is then maintained at temperatures between 1200 and 1300 °C to adjust its reactivity. Slightly calcined lime is the most reactive and commonly used in the steel industry. The calcination equipment consists of banks of straight kilns equipped with filters as shown in **Figure I.4.**



**Figure I.4.** Lime production unit.

#### **I.4.2. Air Separation Unit (ASU)**

The ASU (Air Separation Unit) refines air through a low-temperature distillation process ( $-196\text{ }^{\circ}\text{C}$ ), which enables the production of oxygen, nitrogen, and argon on a large scale. In this unit, ambient air is captured to separate oxygen, nitrogen, and argon using cryogenic distillation technology, followed by selective distillation that separates the components based on their boiling temperatures (**Figure I.5**).



**Figure I.5.** Air Separation Unit.

### I.4.3. Electrical station

The power transformation and distribution station is the installation where the voltage is lowered to be delivered in the appropriate quantity and voltage to meet the needs of different consumption points. These electrical installations consist of three types of devices: transformers that lower the voltage, circuit breakers capable of automatically interrupting the circuit when necessary, and disconnectors that establish or interrupt a circuit as needed, allowing the routing of electrical energy. It is a closed and remotely controlled installation. At



the Bellara Complex, there are four outdoor transformers.

**Figure I.6.** Electrical station.

### I.4.4. Water treatment mill

The industrial water treatment system of the complex consists of the following treatment stations: a specific conditioning and treatment station for cooling water for each of the steelmaking process units (direct reduction, steelmaking, and rolling), connected to a centralized final treatment station for the final adjustment of the composition. The stations are designed and equipped with the necessary purification technologies to ensure that the composition of the final effluent strictly complies with the applicable limit values. The complex has a full and self-sufficient capacity to treat all types of generated wastewater and



achieve the required level of final water quality in accordance with applicable environmental regulations.



**Figure I.7.** Water treatment mill.

#### **I.4.5. Station Reception and transport of Raw Materials**

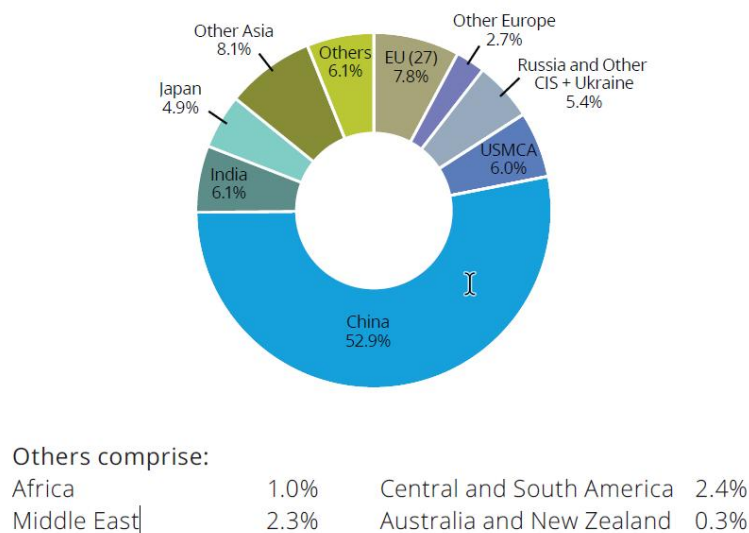
This station plays a vital role in loading, unloading and storing raw materials in a safe and economical way. Accordingly, the importance of this station lies in the ease of moving iron ore to the top of shaft furnace and moving cold sponge iron or DRI to storage placement. It then helps productivity and increases yield. AQS plant based on midrex process is designed to operate successfully using a wide variety of Iron oxide pellets and lump ores, imported from several countries around the world. It was transported by the railway line directly from the port to the plant storage yard.



**Figure I.8.** A view of the Station.

## I.5. World crude steel production

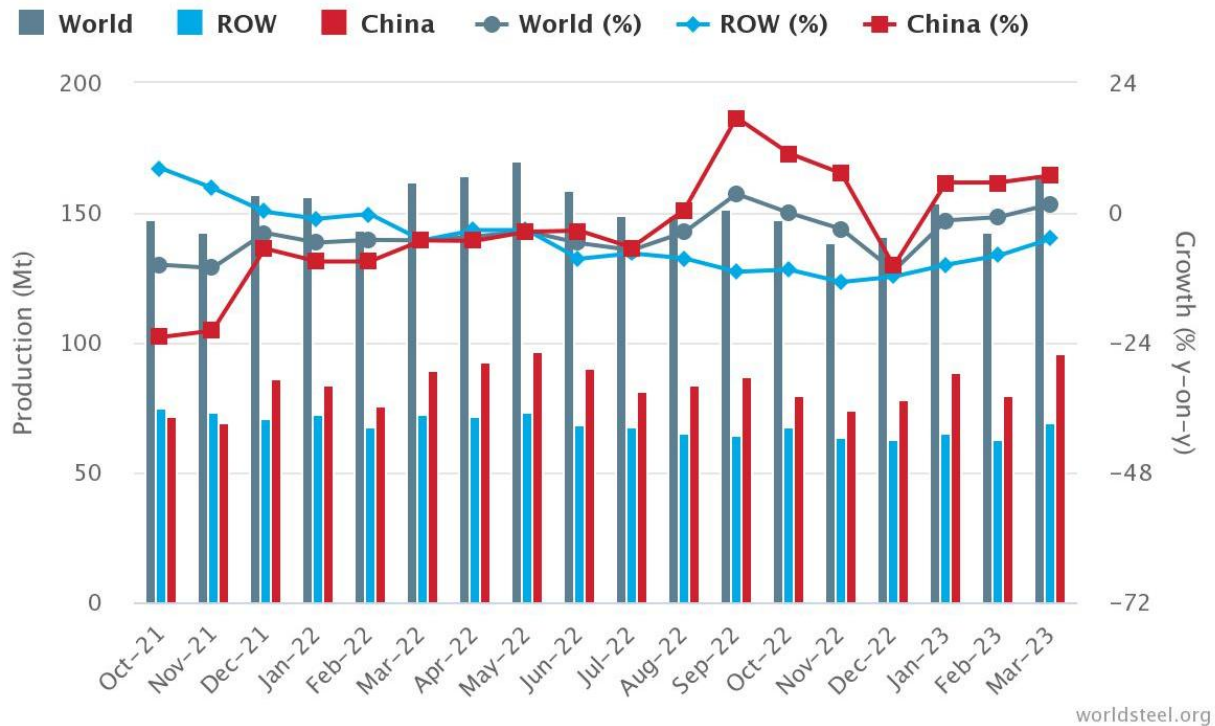
The magnitude of the impact of the conflict taking place in Ukraine vary across regions, depending on their direct trade and financial exposure to Russia and Ukraine. However, the steel demand will see further growth by 2.2% in 2023 to reach 1 881.4 billion tons. But it is primarily China that has largely driven this development and remains by far the world's largest producer with 1.033 billion tons, as stated by the World Steel Association (WSA) in a press release [1, 2]. Its share of global production has further increased to reach 52.9 % of the total. Algeria occupies the 39rd place in this ranking with a production of 3,5 Mt in 2021 against 3,0 Mt in 2020 [1, 2].



**Figure I.9.** Steel production and use: geographical distribution 2021[2].

World crude steel production for the 63 countries reporting to the World Steel Association (worldsteel) was 165.1 million tons (Mt) in March 2023, a 1.7% increase compared to March 2022. Africa produced 1.4 Mt in March 2023, up 8.4% on March 2022. Asia and Oceania produced 124.8 Mt, up 4.1%. The EU (27) produced 11.9 Mt, down 5.6%. Europe, Other produced 3.5 Mt, down 14.1%. The Middle East produced 3.1 Mt, down 17.5%. North America produced 9.3 Mt, down 2.6%. Russia & other CIS + Ukraine produced 7.6 Mt, up 3.0%. South America produced 3.5 Mt, down 7.6%.

In the top 10 steel-producing countries, China produced 95.7 Mt in March 2023, up 6.9% on March 2022. India produced 11.4 Mt, up 2.7%. Japan produced 7.5 Mt, down 5.9%. The United States produced 6.7 Mt, down 2.1%. Russia is estimated to have produced 6.6 Mt, up 0.4%. South Korea produced 5.8 Mt, up 1.9%. Germany is estimated to have produced 3.3 Mt, down 0.5%. Brazil produced 2.7 Mt, down 8.7%. Türkiye produced 2.7 Mt, down 18.6%. Italy produced 2.2 Mt, up 2.7%.



**Figure I.10.** Crude steel production by region between October 2021 and Mars 2023 [1].

## I.6. Manufacturing industrial technological process

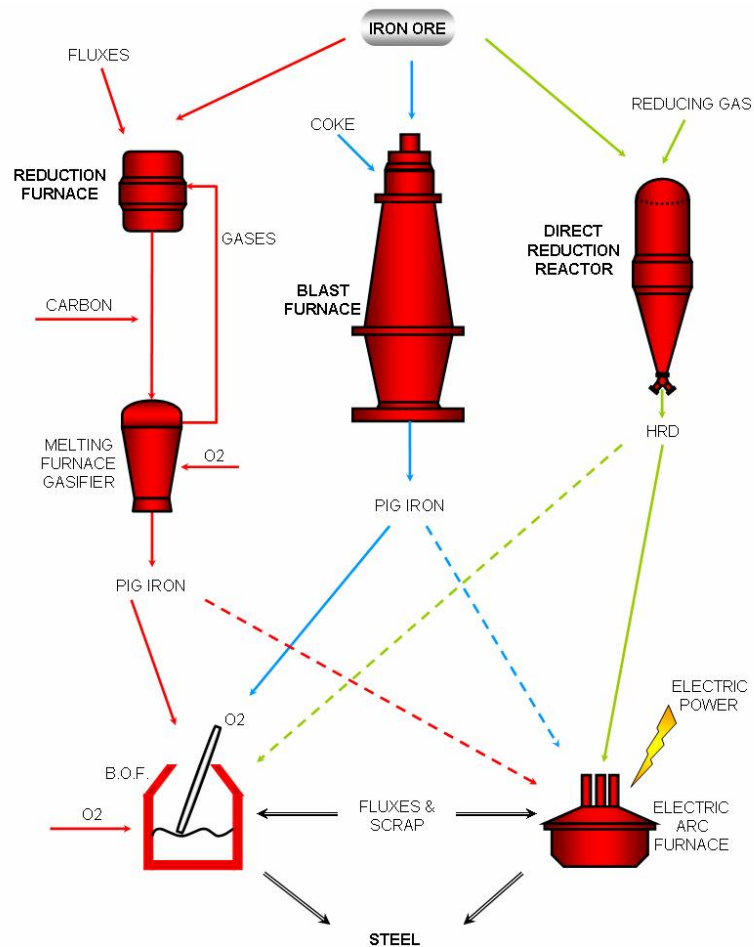
Steel is the basic material for industrial growth. Consequently, it is of foremost relevance for any country seeking its autonomy to produce the steel needed to be self-sufficient. Nowadays, there are three routes to obtain steel from iron ore.

- Blast furnace producing Pig Iron
- Direct Reduction Process producing Direct Reduced Iron (DRI)
- Smelter producing Pig Iron

Products from these processes are refining in:

- Electric Arc Furnace
- Basic Oxygen Furnace (BOF)

In which finally liquid steel is produced. **Figure I.11** shows a general view of these steel fabrication routes.



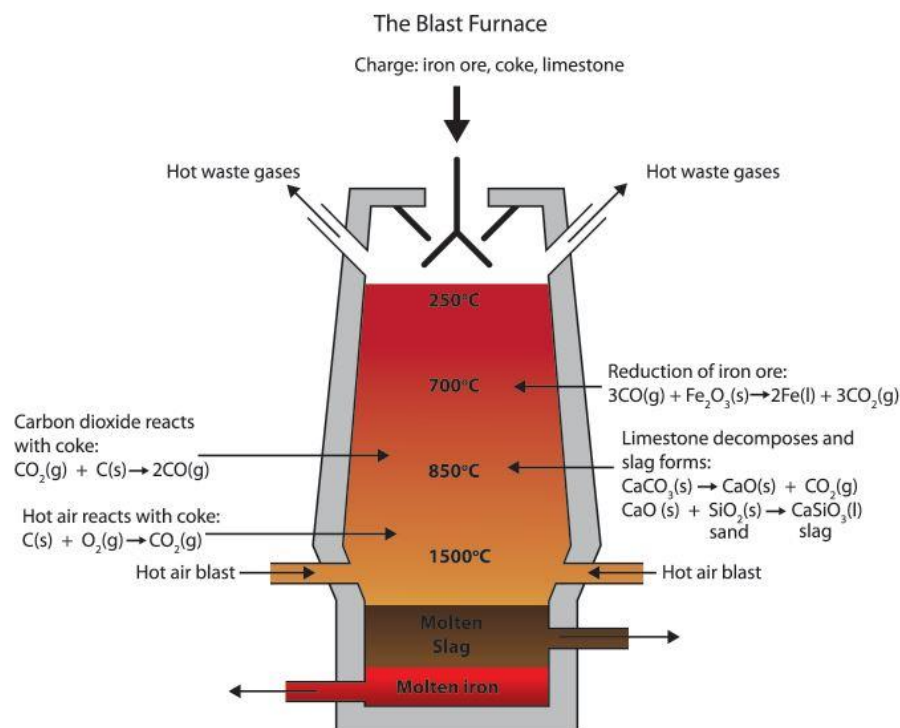
**Figure I.11.** Steel production processes [3].

### I.6.1. Blast furnace

Iron ore mixed with coke and fluxes is fed into the blast furnace where the iron reduction and melting take place. The necessary energy to carry out this process is released by the combustion of coke with preheated air, usually enriched with Oxygen, which is injected into the blast furnace through tuyeres. The product melt obtained is named ‘Pig Iron’. It contains a high percentage of Carbon, sulfur, and silicon, which are adjusted to the required level in the refining process.

Key steps of the process are as follows:

- upper part of the furnace - free moisture is driven off from the burden materials and hydrates and carbonates are disassociated.
- lower part of the blast furnace shaft - indirect reduction of the iron oxides by carbon monoxide and hydrogen occurs at 700-1,000°C.
- Bosh area of the furnace where the burden starts to soften and melt - direct reduction of the iron [and other] oxides and carbonization by the coke occurs at 1,000-1,600°C. Molten iron and slag start to drip through to the bottom of the furnace.



**Figure I.12.** Blast Furnace Process [4].

### I.6.2. Smelter (melting furnace for pig iron production)

This Technology consists in preheating and carries out a partial reduction of iron ore in a furnace similar to Direct Reduction Reactors using as reducing agent the gases generated during the process second step, which is the melter furnace (Smelter).

Final reduction is completed into the melter furnace (Smelter). Smelter-Gasifier is fed with DRI, Carbon and Oxygen to obtain pig iron, slag, and gases that are used for the reduction of





The reducing gas can be generated externally to the reduction furnace, or can be generated from hydrocarbons introduced into the reduction zone of the furnace. In the former case, the reducing gas is produced from a mixture of natural gas (usually methane) and recycled gas from the reducing furnace.

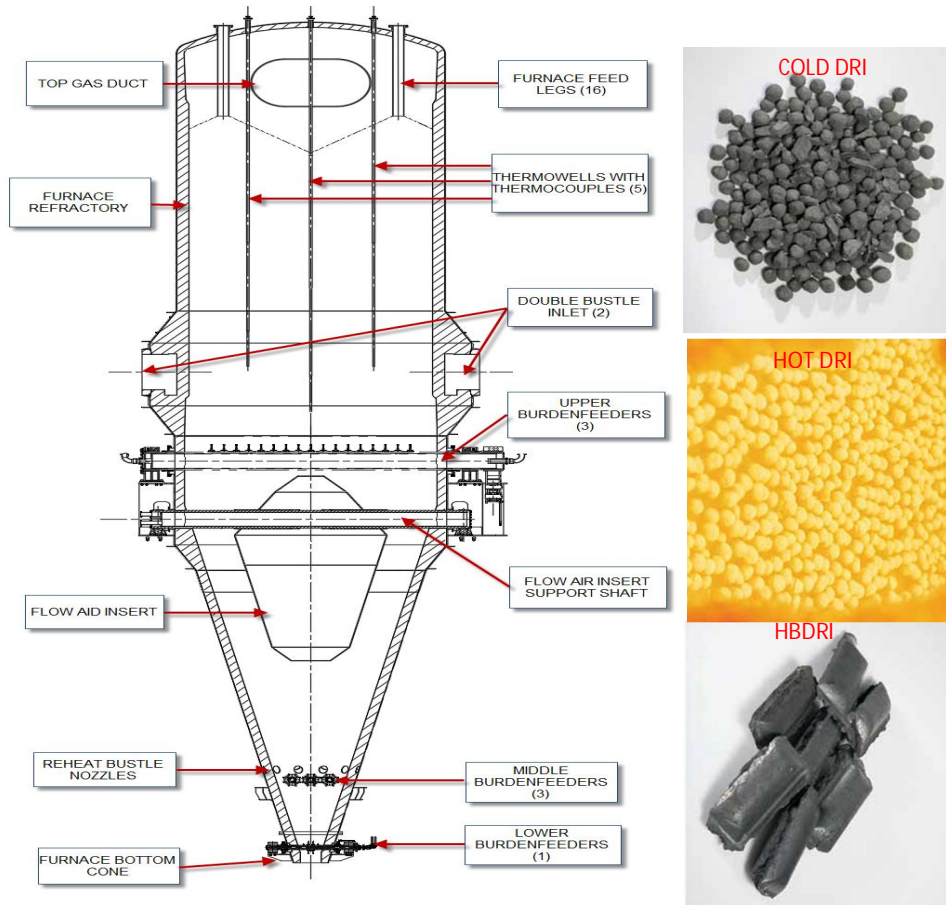
Reducing gases and the temperature at which the reducing reactions take place depend on the Direct Reduction Process, but the temperature never reaches the melting point of the solid. Direct Reduction Processes commercially more active are HYL-ENRGIRON and MIDREX (**Figure I.13**).

### **I.7. Direct reduction basic concepts**

Main components of Iron ore are: Hematite ( $\text{Fe}_2\text{O}_3$ ), Magnetite ( $\text{Fe}_3\text{O}_4$ ), Wüstite ( $\text{FeO}$ ) and gangue. Lump ores and pellets cannot be charged directly into the electric furnace because the associated Oxygen with iron must be firstly removed, because steel production is based in metallic iron and Carbon, and not in Hematite and Magnetite. If it were removed in the electric furnace, the necessary energy to accomplish the removal of Oxygen would be electric energy and the chemical agent to remove it, would be the Carbon from the electrodes. This process would be, besides impractical, quite expensive.

In a direct reduction process, Oxygen is removed from the iron oxides contained in the iron ore by reducing gases action. Reduction is accomplished until some degree around 96 % but not completely. The removal of residual Oxygen, consequently the reduction, is completed in the electric arc furnace.

In the Direct Reduction Process, iron ore is reduced directly, without melting it and the product obtained is a porous metallized solid known as Direct Reduced Iron (DRI), if this product is briquetted at high temperature, greater  $650\text{ }^\circ\text{C}$  the product is known as Hot Briquetted Iron (HBI). The Direct Reduced Iron (DRI) or Hot Briquetted Iron (HBI) is melted to obtain liquid steel in a process subsequent to direct reduction. The furnace currently in use to carry out the melting process is the electric arc furnace.



**Figure I.14.** Midrex reduction furnace [5].

### I.7.1. Metallization

Degree of metallization is the extent of conversion of iron oxide into metallic iron during reduction. It is defined in percentage of the mass of metallic iron divided by the mass of total iron in the DRI or HBI:

$$M (\%) = \frac{Fe^{\circ}}{Fe_T} \times 100$$

Where M is the degree of metallization,  $Fe^{\circ}$  is the metallic iron content which includes the iron combined with Carbon ( $Fe_3C$ ) and  $Fe_T$  is the Total iron in the DRI which is the sum of metallic iron; iron as iron carbide ( $Fe_3C$ ) and the remaining iron as oxide.

### I.7.2. Reduction

The degree of direct reduction products is the ratio of the Oxygen removed from the iron ore to the original amount contained in the iron ore:

$$R (\%) = \frac{O_2\text{removed}}{O_2\text{initial}}$$

Where:

R (%) = Percent of reduction.

O removed is the Oxygen removed from the iron ore.

O initial = Initial is the Oxygen in the iron ore.

Reducing gases have great affinity for the Oxygen contained within iron oxides of iron ores. The main reducing substances commonly used in direct reduction processes are: Hydrogen and Carbon Monoxide.

Due to the thermodynamics of the direct reduction reactions, their equilibrium reactions and due to the fact that almost all these reactions are endothermic, reducing gases are heated up at temperatures between 750 °C and 1100 °C, therefore, the iron ore should be preheated from room temperature up to the reaction temperature. As a rule of thumb, the higher the operation temperature, the higher the reaction velocity will be, therefore, a higher process production capacity will be obtained.

The highest operation temperature will be set by the metallurgical properties of the processed iron ore, such as sticking and swelling tendencies.

### **I.7.3. Reducing gases**

Reducing gases are generated by the reaction between an organic fuel (energetic substance) and an oxidizer, producing gaseous mixtures made of Hydrogen and Carbon Monoxide, among others.

### **I.7.4. DRI stability**

Due to the high metallic iron content in the DRI it is bound to reoxidize if proper conditions are achieved. DRI is likely to react with atmospheric Oxygen or with water to revert the reducing reactions.

The main problem is that DRI reoxidation reactions are exothermic; therefore, they generate heat as well as toxic and explosive gases such as Hydrogen and Carbon Monoxide. Reoxidation

reactions are carried out when DRI, is at high temperature environment with an oxidizing atmosphere i.e., air or humidity. Accordingly, to obtain a stable product, it is required as a prerequisite, that the DRI obtained as product from the direct reduction process be cold and dry. With this, it is easier to store and transporting. It is usual to measure DRI reactivity in Nm<sup>3</sup> (normal cubic meters) of Oxygen consumed by reaction per ton of DRI at a predetermined temperature.

### **I.7.5. Iron ores**

Iron ores have different chemicals, physical and metallurgical properties that affect their behavior in the Direct Reduction Processes.

#### **I.7.5.1. Iron Ores Chemical Analysis**

Main iron oxides components in iron ores are: Fe<sub>3</sub>O<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub> or FeO which are present in different percentages, due to this, they are not reduced in the same way. Others constituents different to iron oxides, such as CaO, MgO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaCO<sub>3</sub> etc. are gangue. Even though the gangue does not directly enter in the reduction reactions, it is very important because it determines some metallurgical properties of the solid. The effect of the gangue on these properties is measured through the binary or quaternary basicity ratios.

$$B_2 = \frac{\text{CaO}}{\text{SiO}_2} \quad \text{and} \quad B_4 = \frac{\text{CaO} + \text{MgO}}{\text{SiO}_2 + \text{Al}_2\text{O}_3}$$

Pellets with a basicity ratio between 0 and 1 are considered acids, and materials with a basicity ratio more than 1 are considered basic pellets. Since the refractory lining in the Electric Arc Furnace is based on basic elements (MgO), it is well accepted that DRI from basic pellets optimizes the steelmaking costs, because there is not any chemical attack on the refractory lining.

#### **a) Physical Properties**

**Iron Ore Mechanical Strength:** Iron ores must have mechanical strength enough to withstand the handling mainly during the transport and charge to the Direct Reduction Process.

Iron Ore Particle Size Distribution Particles of almost the same homogenous size are preferred to avoid flow channeling which causes problems in pressure drop and a non-uniform product quality.

## b) Metallurgical Properties

### · Reducibility

Iron ore reducibility is a measure of the rate of reaction between the reducing agents and the Oxygen from the iron oxides. The reduction behavior or “reducibility” of an iron ore depends on several parameters such as: Crystallographic structure, Particle ore porosity, particle size, process temperature, operation pressure, among others.

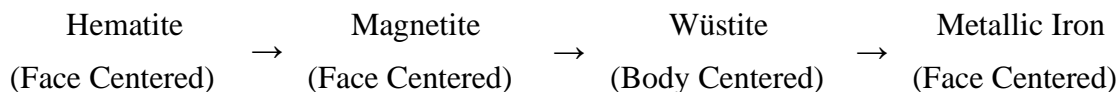
### · Sticking

This phenomenon can be defined as the tendency of pieces of iron ore or pellets to stick together to form large blocks or clusters or, even, to stick to the walls of the reduction furnaces. Practically, temperature of reduction is one of the most important parameters for this phenomenon which is usually more and more critical when temperature increases. Sticking provoke serious solid flow problems, causing gases channeling and a non-uniform product quality.

In the Direct Reduction Process the clustering phenomena has been overcome by the use of iron ore coating process. Coating process consists in cover the pellets with a thin layer of cement or calcium before they go into the Direct Reduction Reactor. Coating process diminishes the probability to have incipient fusion between the metallic irons of pellets which provokes the clustering phenomenon.

### · Swelling

All iron ores develop a volume change while being reduced, because crystallographic changes occur in the molecular arrangement, depending on the different states of the reduction process.



The volume change is identified as “Swelling Index” and represents a percentage of bed volume change. When the swelling is significant, it induces similar problems to those caused by sticking.

### I.7.5.2. Direct reduction of iron oxide

#### I.7.5.2.1. Reduction chemistry

**Table I.1** shows the sequence of reactions that occurs when producing one mole of iron, starting with hematite ( $\text{Fe}_2\text{O}_3$ ) feed in the reduction process. Each reaction is reversible; therefore, for any reaction to proceed in the direction of reducing iron, the ratio of reductant ( $\text{H}_2$  or  $\text{CO}$ ) to oxidant ( $\text{H}_2\text{O}$  or  $\text{CO}_2$ ) must be greater than the equilibrium value governed by the reduction chemistry. For example, **Table I.1** shows also the minimum reduction equilibrium ratios for each reaction at 760 °C.

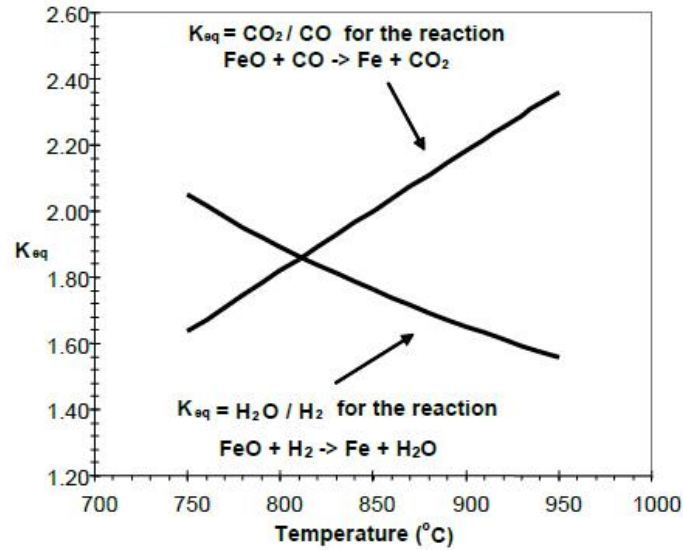
**Table I.1.** Reduction reactions and minimum reduction equilibrium ratios at 760 °C.

Reduction Reaction	Reaction Name	Equilibrium Ratio
$\frac{1}{2} \text{Fe}_2\text{O}_3 + \frac{1}{6} \text{H}_2 \rightarrow \frac{1}{3} \text{Fe}_3\text{O}_4 + \frac{1}{6} \text{H}_2\text{O}$	Hematite to Magnetite	$\text{H}_2/\text{H}_2\text{O} = 1.3 \times 10^{-5}$
$\frac{1}{2} \text{Fe}_2\text{O}_3 + \frac{1}{6} \text{CO} \rightarrow \frac{1}{3} \text{Fe}_3\text{O}_4 + \frac{1}{6} \text{CO}_2$	Hematite to Magnetite	$\text{CO}/\text{CO}_2 = 1.5 \times 10^{-5}$
$\frac{1}{3} \text{Fe}_3\text{O}_4 + \frac{1}{3} \text{H}_2 \rightarrow \text{FeO} + \frac{1}{3} \text{H}_2\text{O}$	Magnetite to Wustite	$\text{H}_2/\text{H}_2\text{O} = 0.6$
$\frac{1}{3} \text{Fe}_3\text{O}_4 + \frac{1}{3} \text{CO} \rightarrow \text{FeO} + \frac{1}{3} \text{CO}_2$	Magnetite to Wustite	$\text{CO}/\text{CO}_2 = 0.7$
$\text{FeO} + \text{H}_2 \rightarrow \text{Fe} + \text{H}_2\text{O}$	Wustite to Iron	$\text{H}_2/\text{H}_2\text{O} = 2.0$
$\text{FeO} + \text{CO} \rightarrow \text{Fe} + \text{CO}_2$	Wustite to Iron	$\text{CO}/\text{CO}_2 = 1.7$

A plot of the equilibrium ratio versus temperature, shown in **Figure I.15**, represents the final stage of reduction. In the MIDREX Furnace, the reducing gas contacts the iron oxide in a counter flow manner so the final stage of reduction is accomplished by the reducing gas with the highest reducing potential; i.e., the highest ratio of reductants ( $\text{H}_2$  and  $\text{CO}$ ) to oxidants ( $\text{H}_2\text{O}$  and  $\text{CO}_2$ ). In the MIDREX Process, this ratio is 11-12.

A sufficient flow of reducing gas (relative to flow of iron oxide) must be maintained constantly to ensure the  $\text{H}_2/\text{H}_2\text{O}$  and  $\text{CO}/\text{CO}_2$  ratios equal or exceed the ratios in **Figure I.15** after the final stage of reduction of wustite to metallic iron.





**Figure I.15.** Equilibrium ratio versus temperature for reduction of wustite to iron.

In MIDREX Plants, the term “utilization” is used to express the ratio of the calculated minimum flow requirement to the actual flow supplied. “One hundred percent gas utilization flow” refers to the case of supplying just enough reducing gas to maintain the reductant-to-oxidant ratios at the equilibrium values after the final stage of reduction of wustite to metallic iron and the consequent oxidation of the reducing gas (Figure I.15). In actual operation, about 10.0-20.0 % higher gas flow than this is supplied, and the corresponding gas utilization is 80.0-90.0 %. This permits a reducing potential higher than equilibrium at the location in the furnace where the final stage of reduction begins. The higher gas flow also ensures sufficient gas flow in sections of the furnace where the bed permeability may be lower. After reduction of wustite to metallic iron (FeO to Fe), the reducing gas still has enough reducing potential to accomplish the reduction steps of magnetite to wustite ( $\text{Fe}_3\text{O}_4$  to FeO) and hematite to magnetite ( $\text{Fe}_2\text{O}_3$  to  $\text{Fe}_3\text{O}_4$ ). These steps proceed in the direction of reducing iron at much lower reductant-to-oxidant ratio compared to the reduction of wustite to metallic iron, as shown in Table I.1.

#### I.7.5.2.2. Reduction Furnace Energy Requirements

Table I.2 shows the heat of reaction for the different reduction steps, depending on whether  $\text{H}_2$  or CO is used as a reductant. The overall reduction reaction is endothermic. The reaction energy and the heat required to bring the iron oxide up to reaction temperature are supplied by the sensible heat of the reducing gas. Whether the reduction of FeO to Fe is endothermic or



exothermic is not of practical consequence because most of the reduction occurs high in the burden where the burden temperature is not critical

**Table I.2.** Heat of reaction for reduction.

Reaction	With H <sub>2</sub>	With CO
Fe <sub>2</sub> O <sub>3</sub> --> Fe <sub>3</sub> O <sub>4</sub>	Exothermic	Exothermic
Fe <sub>3</sub> O <sub>4</sub> --> FeO	Endothermic	Endothermic
FeO --> Fe	Endothermic	Exothermic

The volume of hot reducing gas required to satisfy the reduction reactions is sufficient to heat the entering iron oxide to the required reaction temperatures and still exit the furnace as top gas with a temperature of about 350-450 °C. This very high temperature difference between the top gas and the iron oxide at the top of the furnace heats the material rapidly to reaction temperature and makes the furnace insensitive to wet iron oxide feeds.

It is desirable to maintain the final reduction zone (FeO --> Fe) at a temperature high enough to permit rapid reaction but low enough to keep the material from clustering. At 825 °C, the heat of H<sub>2</sub> reduction is +70,700 kcal/t Fe (endothermic), while the heat of CO reduction is -82,200 kcal/t Fe (exothermic). The conventional MIDREX Plant operates with an excess of hydrogen over carbon monoxide, tending to make the final reduction region isothermal.

#### I.7.5.2.3. Reduction Mechanism and Kinetics

The rate of reaction in the reduction process is a function of several factors, some of which are controllable. The reduction mechanism consists of the following series of steps:

1. Diffusion of the reducing gas (H<sub>2</sub> or CO) through the boundary layer of gas surrounding the iron oxide lump or pellet.
2. Diffusion of the reducing gas into the lump or pellet through the pores in the material.
3. Chemical reaction at the iron oxide interface.
4. Diffusion of the oxidized gas (H<sub>2</sub>O or CO<sub>2</sub>) out of the lump or pellet.
5. Diffusion of the oxidized gas back through the boundary layer into the main gas stream.

The rate of each of these steps has an effect on the total time required for the production of metallic iron. Steps 1 and 5 are influenced by the temperature, the concentration of the diffusing

gases, and the velocity of the gas stream through the burden. In the MIDREX Furnace, turbulent flow conditions ensure an adequate supply of reductant at the surface of the iron oxide particle.

The diffusion rate of gases into and out of the material in steps 2 and 4 also is a function of temperature and concentrations and is influenced by the physical structure of the material. In step 3, temperature and concentration are important, as are the relative quantities of hydrogen and carbon monoxide in the reducing gas.

It is neither necessary nor practical to determine the rates of each of the individual reduction steps. The total residence time available in the reduction zone of the MIDREX Furnace is approximately 4-6 hours, which is more than adequate for most iron oxide materials.

### **I.7.6. Reforming**

#### **I.7.6.1. Introduction**

Reforming is the reaction of a hydrocarbon (such as methane, propane, etc.) with  $H_2O$  and/or  $CO_2$  to produce the reductants  $CO$  and  $H_2$ . Natural gas is the usual source of hydrocarbons in the MIDREX Process. The reforming reactions are endothermic; therefore, they require input of heat for the reaction to proceed in the direction of producing  $CO$  and  $H_2$ . Because of this, a catalyst is required to increase the reaction rate. The reductant-rich reformed gas leaving the reformer is used to reduce iron oxide to metallic iron in the MIDREX Furnace. In the reduction furnace reactions, the reductants  $CO$  and  $H_2$  are converted to the oxidants  $CO_2$  and  $H_2O$ , which are mixed with natural gas and recycled as feed gas to the reformer tubes. The reforming of recycled gas to produce reducing gas is unique to the MIDREX Process.

#### **I.7.6.2. Reactions in the Reformer**

The important reactions that occur (or can occur) in the MIDREX® Reformer are shown in **Table I.3**. Reforming reactions (reforming with  $CO_2$  and with  $H_2O$ ) produce the desired reductants,  $CO$  and  $H_2$ , by reacting the oxidants,  $CO_2$  and  $H_2O$ , with a hydrocarbon such as methane. When these reactions proceed in the reverse direction, the process is called “methanation”.

**Table I.3.** Reactions in the reformer.

Reaction number	Reaction Formula	Reaction Name
1	$\text{CH}_4 + \text{CO}_2 \leftrightarrow 2\text{CO} + 2\text{H}_2$	Reforming with $\text{CO}_2$
2	$\text{CH}_4 + \text{H}_2\text{O} \leftrightarrow \text{CO} + 3\text{H}_2$	Reforming with $\text{H}_2\text{O}$
3	$\text{CO} + \text{H}_2\text{O} \leftrightarrow \text{CO}_2 + \text{H}_2$	Water – Gas Shift Reaction
4	$\text{CO} + \text{H}_2 \leftrightarrow \text{C} + \text{H}_2\text{O}$	Carbon-depositing (Water – Carbon)
5	$2\text{CO} \leftrightarrow \text{C} + \text{CO}_2$	Carbon-depositing (Boudouard)
6	$\text{CH}_4 \leftrightarrow \text{C} + 2\text{H}_2$	Carbon-depositing (cracking)
7	$\text{C}_n\text{H}_{2n+2} \leftrightarrow n\text{C} + (n+1)\text{H}_2$	Carbon-depositing (cracking)

The water-gas shift reaction (reaction 3) is important because of its close approach to equilibrium in the reformer. Because it is difficult to measure the water content of the reformed gas, the water content is calculated from the reformed gas analysis and the reformed gas temperature using the water-gas shift equilibrium curve.

Reactions 4-7 are carbon-depositing reactions. Reaction 7 is a generalization of reaction 6 and applies to any hydrocarbon, including heavier hydrocarbons such as propane and butane. Carbon deposition must be avoided in the reformer to prevent catalyst degradation or temporary capacity loss.

In conventional steam reforming, there is 200.0-300.0 % more steam than the amount required to react the hydrocarbon to completion. Use of such excess oxidant cannot be tolerated in the MIDREX Process because the excess oxidant in the reformed gas would impede the reducing reaction in the reduction furnace. For this reason, the MIDREX Reformer is operated at near stoichiometric conditions. In true stoichiometric reforming, there would be just enough oxidant in the reformer gas feed to react the entire hydrocarbon with no oxidant left over. In practice, MIDREX reforming is carried out with about 40.0% more oxidant than required for true stoichiometric reforming but is still termed stoichiometric reforming. Operating this close to stoichiometric requires proper operation of the MIDREX Reformer at all times. Improper operation can result in carbon depositing reactions fouling the catalyst, degradation of the catalyst, or failure of the high temperature alloy reformer tubes.

### I.7.6.3. Reforming Reactions

The reforming reactions take place in tubes filled with REFORMEX® Catalyst in the MIDREX Reformer. Reforming is highly endothermic or heat consuming. **Table I.4** shows the heats of

reaction for the two reforming reactions in the range 700-1000 °C. As seen in **Table I.4**, reforming with CO<sub>2</sub> is approximately 15.0 % more endothermic than reforming with H<sub>2</sub>O.

The great majority of reformed gas required for reduction in the furnace is produced in the reformer and introduced to the furnace with the bustle gas. However, some reforming also occurs in the furnace. This is called “in-situ” reforming. In-situ reforming occurs because methane and oxidants are present in the furnace. Natural gas is added to the furnace to increase product carbon.

Furnace methane is increased by adding enrichment natural gas to the reformed gas mixer and by introducing natural gas directly to the furnace at the transition zone. After reducing iron oxide to metallic iron, CO and H<sub>2</sub> are converted to the oxidants CO<sub>2</sub> and H<sub>2</sub>O. The catalytic surface of the DRI allows these oxidants to react with some of the methane or heavier hydrocarbons from the natural gas to form more CO and H<sub>2</sub> inside the furnace, which increases DRI production.

Reforming with CO<sub>2</sub> produces equal quantities of H<sub>2</sub> and CO, while reforming with H<sub>2</sub>O produces three times more H<sub>2</sub> than CO. Therefore, the H<sub>2</sub>/CO ratio of the reformed gas is determined by the relative amounts of CO<sub>2</sub> and H<sub>2</sub>O in the reformer feed gas. The CO<sub>2</sub> reforming reaction is slower than the H<sub>2</sub>O reforming reaction. Because the MIDREX Reformer reforms CO<sub>2</sub> and H<sub>2</sub>O, it must operate at a higher temperature than a conventional steam reformer to achieve a high reaction rate.

As seen in equations 1 and 2, there is a volume expansion from the reforming reactions. However, there is no volume change in the iron oxide reduction reactions. Because the volume of water condensed in the top gas scrubber is less than the reforming volume expansion, more gas leaves the top gas scrubber than is required for the reformer feed gas. This excess quantity of top gas is utilized in the reformer main burners as top gas fuel.

**Table I.4.** Heat of reaction for reforming.

Temperature (°C)	Endothermic Heat of Reaction (kcal/Nm <sup>3</sup> CH <sub>4</sub> )	
	Reforming with CO <sub>2</sub>	Reforming with H <sub>2</sub> O
700	2785	2407
800	2787	2418
900	2784	2425
1000	2776	2428

#### **I.7.6.4. Reformer Catalyst**

A catalyst is a material that accelerates a reaction without being consumed or materially changed by the reaction. Reforming is a heterogeneous catalytic reaction because the reactants are in the gaseous phase and the catalyst is a solid. Reforming catalysts are composite catalysts because they contain more than one chemical constituent; i.e., the active constituent and its support. Catalyst performance depends on the intrinsic chemical and physical properties of the catalyst and on the extent of its degradation through use in the reformer.

Catalyst activity, the most important factor in successful stoichiometric reforming, depends on the original chemical composition of its active constituent and its support material. The active constituent in most reforming catalysts is nickel; although some catalysts use cobalt (nickel is more active than cobalt at lower temperatures). At higher temperatures, the activity of nickel and cobalt are about the same. The higher the percentage of active constituent, the greater the catalyst activity. The support material for most reforming catalysts is a high purity alumina.

The required chemical composition of the catalyst primarily depends on the natural gas feedstock. Carbon deposition on the catalyst will degrade catalyst performance. Therefore, if the natural gas has a high concentration of higher hydrocarbons, the use of a lower-activity catalyst may be necessary to prevent carbon deposition on the catalyst from hydrocarbon cracking.

Since the feed gas is entering the reformer at 580 °C and the reformer floor temperature is above 1000 °C, the feed gas will heat rapidly above 700 °C when entering the reformer tube and contacting the active catalyst.

Due to a high preheat, the reformer feed gas is heated above 700 °C at the bottom of the tube. Therefore, the reformer tube is completely full with a high performance, active catalyst like REFORMEX R7GG (unless a split catalyst loading is used for higher hydrocarbons). The activity of this catalyst must be as high as possible to maximize the throughput of a tube consistent while avoiding carbon deposition.

Catalyst activity also is influenced by carrier properties such as size and shape, porosity, pore size distribution, and surface area. Greater surface area and smaller pore size make the catalyst more active.

The size and shape of the catalyst affect its pressure drop characteristics. The pressure drop decreases with larger size, uniform sizing, and ring-shape instead of a solid-shape.

The size and shape of the catalyst also affect its heat transfer properties. At the high temperatures used in stoichiometric reforming, radiation is believed to be the primary mode of heat transfer. Heat is transferred radially from the tube wall to the center of the tube in the following manner:

- Radiation from the tube wall to adjacent ring of catalyst particles.
- Heat conduction through the first ring of catalyst particles.
- Radiation from the first ring of catalyst particles to next ring of catalyst particles.
- The last two steps are repeated until the centerline of the tube is reached.

Based on this heat transfer mechanism, the heat transfer rate increases with solid, larger particle size, rather than rings. Heat transfer to the center of the tube must be adequate to ensure the center does not cool and cause carbon deposition. Increasing particle size also decreases activity because of the decrease in outside surface area.

Considering all the above factors, a solid, uniformly-sized particle with an average diameter about one-eighth to one-tenth of the tube diameter is best for the active catalyst.

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# Chapter II

**Quality control of raw materials and DRI**



## II.1. Plant design criteria:

### II.1.1. Core plant capacity:

The following are the key design parameters for the Algerian Qatari steel direct reduction plant:

- Production rate of 312,5 t/h. assuming 8,000 hours of annual operation, this equates
- To annual production of 2,500,000 tons of cdri/hdri.
- Min, Average 94 % metallization
- Min, Average 84,2 % metallic iron, based on an oxide with 67,7 % total Fe
- Min 2,5% carbon cdri /hdri
- Oxide teed material, 100% DR grade oxide pellets.
- Feed gas preheat of 600 °C, combustion air preheats of 595 °C, top gas fuel preheats of 290 °C, and preheated enrichment and transition zone natural gas of 310 °C.

### II.1.2. Iron Oxide Feed:

The midrex process is capable of reducing a wide variety and various mixtures of iron oxide pellets and lump iron ore. More than 50 different types of oxide material have been used commercially in midrex plants around the world.

**Table II.1.** physical & chemical characteristics of oxides.

Physical characteristics	Pellets
Average cold compression strength	>250Kg/pellet (min)
Tumble index	>94%(min) +6.73mm <5%(max) -0.5mm
Abrasion index	4%(main) -0.5mm
Bulk density	2.0-2.3t/m <sup>3</sup>
Size Distribution	<4% 18 to 50 mm >94% 8 to 18 mm <4% (max) -6mm
Porosity	>28%(min.)
Chemical Characteristics	Pellets
Fe(total)	66.7%
FeO	<1%
SiO <sub>2</sub> & Al <sub>2</sub> O <sub>3</sub>	<4.5%

CaO & MgO	0.4%-1.8%
P	<0.04%
S*	<0.010%
H <sub>2</sub> O	<3.0%

S\* Sulfur release of iron must not cause H<sub>2</sub>S in reformer feed gas exceed 5 ppm.

**Table II.2.** Reduction characteristics.

Reduction Characteristics	Pellets
Midrex Linder Test (760 C)	>93% MET
Midrex Linder Degradation	<2% -3.36 mm
Compression strength	>90% Kg/pellet
Fines Formation (Tumbling) of +6.3 mm Metallized Material	>85% +6.73 mm
Midrex Hot load Test (816 C)	>94% MET
Midrex Hot load Degradation	<2% -3.36 mm
Clustering (hot load test) (after 10 revs. In tumble drum)	0% +25 mm

### II.1.3. Natural Gas:

The plant receives natural gas at incoming pressure 29 – 31 bar and temperature of -10 to 50 °C with the following composition (dry basis):

**Table II.3.** composition of received natural gas.

Component	Volume % (nominal)
CH <sub>4</sub>	82.72
C <sub>2</sub> H <sub>6</sub>	8.26
C <sub>3</sub> H <sub>8</sub>	2.23
i-C <sub>4</sub> H <sub>10</sub>	0.37
n-C <sub>4</sub> H <sub>10</sub>	0.59
i-C <sub>5</sub> H <sub>12</sub>	0.13
n-C <sub>5</sub> H <sub>12</sub>	0.15
C <sub>6</sub> <sup>+</sup>	0.09
N <sub>2</sub>	3.23
CO <sub>2</sub>	2.01
He	0.22
S as H <sub>2</sub> S	<5 ppm
Total Sulfur	<10 ppm
Moisture	<80 ppmv

The core plant design is based on a continuous supply of natural gas. However, in case of a gas failure, the plant is designed for an emergency fail safe shutdown from full operating conditions.

#### II.1.4. Electricity:

Electricity power required for plant operation is supplied at 33 KV ,50 Hz (cycles per phase). The plant electricity consumers are supplied as follows:

**Table II.4.** Electricity power required for plant operation.

Medium voltage:	6.6 KV, +/- 5% 50 Hz, +/- 3%, 3-phase Motors > 200 KW
Low voltage:	400 V, +/-5% 50 Hz, +/- 3% 3-phase Motors 200 KW or less
Emergency power:	6.6 KV, / 400 V +/-5% 50Hz, +/-3% 3-phase
Light & Utilities:	230 VAC, +/-5%, 50Hz, +/-3%, 1-phase
Control voltage:	230 VAC 24 VDC for I/O signals
Uninterrupted power supply	230 VAC +/-0.5%, 50Hz +/- 0.1%, 1-phase

#### II.1.5. Makeup water:

Based on the water analysis provided by AQS, it is necessary to utilize a Reverse Osmosis water Treatment System to obtain a suitable water quality for the DR plant. The following table shows the required Makeup water quality.

**Table II.5.** required Makeup water quality of Midrex process.

Midrex Requirements	
Turbidity	<2.0 NTU
Total hardness as CaCO <sub>3</sub>	<50 ppm
Ca hardness CaCO <sub>3</sub>	<35 ppm
Mg hardness CaCO <sub>3</sub>	N/A
Total alkalinity as CaCO <sub>3</sub>	<50 ppm
Chlorides as NaCl	<15 ppm
Silica as SiO <sub>2</sub>	<10 ppm
Total dissolved solids	<100 ppm
Total suspended solids	<10 ppm
Suspended solids size	<425 um
Iron	<0.3 ppm
pH	7-8
Service	uninterrupted

### II.1.6. Nitrogen:

The nitrogen has a purity of 99.95% N<sub>2</sub>. AQS supplies Nitrogen gas to the DR facility as the purge Gas system. The primary purpose of the nitrogen backup system is for maintaining inert Atmosphere around DRI material (i.e. the hot transport conveyor, PDC, passivation silos ...etc)

Nitrogen is also used for furnace sealing during plant idle conditions and/or loss Seal Gas oxygen control.

Gaseous nitrogen usage of up to 13000 Nm<sup>3</sup>/h is required at a supply pressure of 8.0 bar. The anticipated gaseous nitrogen system flow rate during plant idle conditions is approximately 700-4,000 Nm<sup>3</sup>/h.

In addition, a liquid nitrogen system (supplied by AQS) serves as a back-up to the gaseous nitrogen system. This system consists of a liquid nitrogen storage tank and vaporizer unit. This liquid nitrogen system assures that sufficient inert gas is always available to purge the plant of hazardous gases during times when the gaseous nitrogen supply is interrupted.

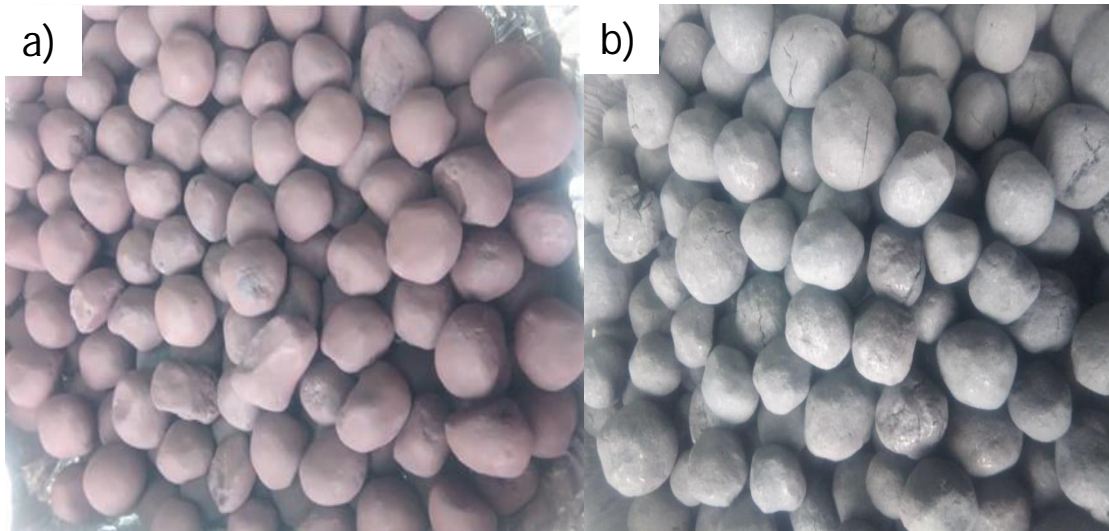
### II.1.7. Oxygen:

The plant is designed for a maximum rate gaseous oxygen of approximately 6,250 Nm<sup>3</sup>/h and a nominal rate of approximately 3,100 Nm<sup>3</sup>/h, 24hours/day, 365days/year at 9.0 bar minimum pressure, which is approximately 10 Nm<sup>3</sup> of oxygen per ton of product. The oxygen composition is 95% oxygen (minimum) the balance being nitrogen.

## II.2. Quality control of raw materials

### II.2.1. Raw Material

The essential raw materials in the reduced iron industry are iron oxides in the form of geometric pellets (also referred to as pellets) after undergoing an agglomeration process to eliminate impurities such as phosphorus, sulfur, and alumina. The pellets take the form of spheres with diameters ranging from 8 to 18 mm. Their production involves a combination of chemical and thermal processes to enhance the iron ore content to 67-72% iron and impart the desired chemical and mechanical characteristics. The iron oxide must undergo qualitative control to ensure the proper progression of the direct reduction stage in the furnace.



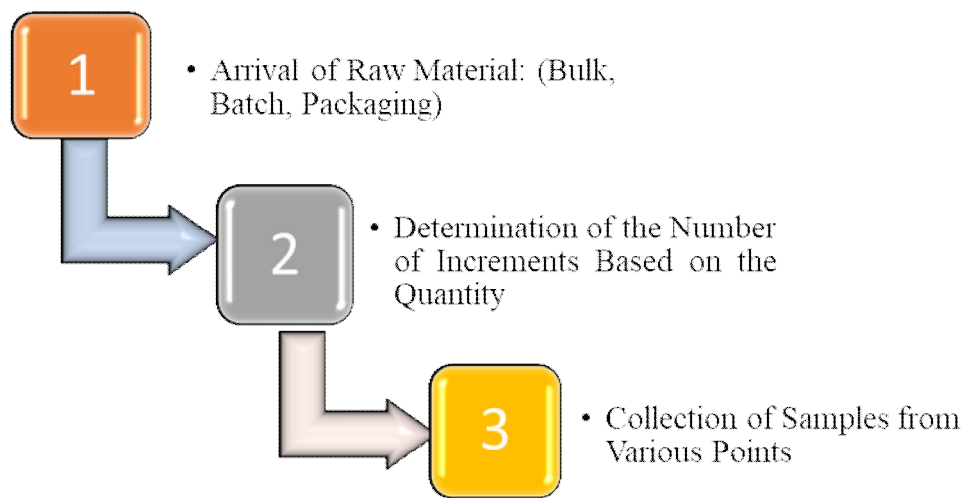
**Figure II.1.** Iron oxide pellets (a) before reduction and (b) after reduction (DRI).

The raw material has reached the final storage site at Bellara from the Djen-Djen port in Jijel, following the unloading of maritime transport vessels. Currently, this raw material is being transported by train, pending the commencement of the railway line in the near future. During

the unloading process, quality control operators initiate their work with the sampling operation, adhering to well-defined methods.

### II.2.1.1. Sampling:

This operation is conducted in the storage area after each arrival of the raw material to verify its properties and composition against the specifications established beforehand during the procurement of this material from international markets. The diagram below illustrates an organizational chart of the sampling method:



**Figure II.2.** Flowchart of the Sampling Method.

### II.2.1.2. Physical Tests:

Physical tests were conducted on a composite sample to determine the specifications of the iron oxide that will be used in the reduction process:

#### II.2.1.2.1. Moisture Content:

This test is performed to determine the water content in the raw material. A drying method is applied to evaporate the water. To do this, an initial quantity of the composite sample with a mass ( $M_1$ ) of 2 kg is taken and placed in an oven at  $105 \pm 2$  °C for 4 hours to ensure the removal of water absorbed by the raw material. After cooling, the mass of the final sample is weighed again, noted as  $M_2$ . The moisture content is then calculated using the following formula:

$$\text{Moisture Content (\%)} = ((M1 - M2) / M1) * 100$$

The average moisture content of the tested raw material in our study is 2.2%.

#### II.2.1.2.2. Particle Size Distribution:

To prevent furnace blockage, the particle size distribution of the iron oxide must be known and determined in percentage according to the ISO 4701:2019 standard (Iron ores and direct reduced iron - Determination of size distribution by sieving). According to this standard, the particle size distribution of the raw material must meet the values presented in the table below:

**Table II.6:** physical specification required on the raw material.

Granulometry (mm)	CCS Kg/ Pellet	Porosity %	density T/m <sup>3</sup>	Moisture %	T.I. %	A.I. %	
+16.0	0.32	298	28.0	2.0	1.09	96.7	2.93
+12.5	58.12						
+9.5	39.66						
+6.35	0.93						
+3.35	0.56						
-3.35	0.42						
<16.0 & >9.5	97.78						

TI: % content of pellets.

AI: % content of powders.

These two measures represent the percentage content of compression strength. This test is conducted on a Tumbler machine, where iron oxide pellets are subjected to 200 compression cycles. The granules that remain unbroken after this process are labeled as TI, while the pellets that have turned into powder are labeled as AI.

Consequently, the raw material needs to pass through an automatic vibrating sieve (Figure II.3) comprised of multiple sieves with varying diameters ranging from 19.5 mm down to at least 3.3 mm. Larger grains over 19.5 mm will be retained on the first sieve and so forth.



**Figure II.3.** Automatic Vibrating Sieve.

In order to calculate the particle size distribution of the granules, 2 kg of granules were placed on a 19.5 mm sieve. The obtained sieving results for the raw material granules are presented in the following table:

**Table II.7:** oxide screen result.

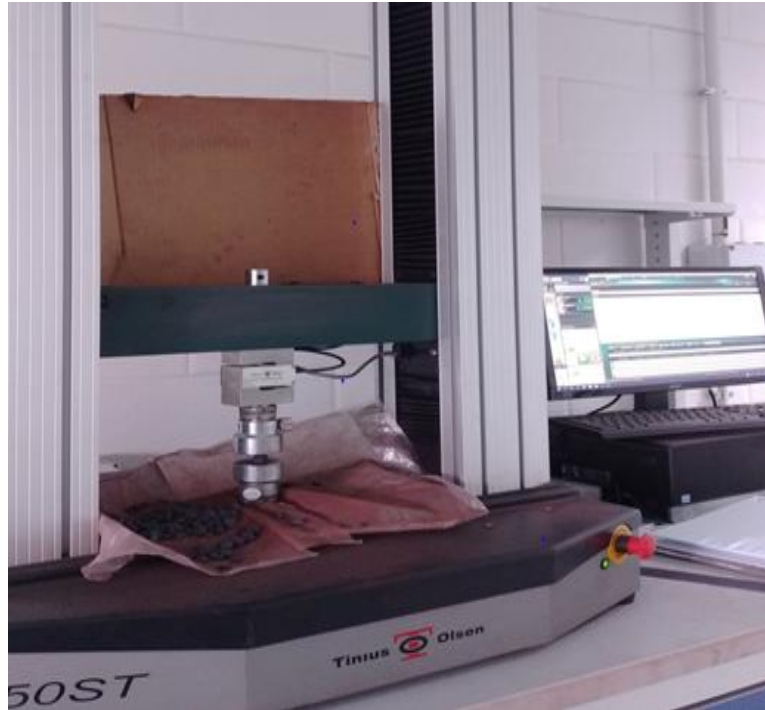
Oxide screen results (mm)	+19,0	+16,0	+12,5	+9,50	+6,30	+3,30	-3,30	<16.0 & >9.5
%	0	0.32	58.12	39.66	0.93	0.56	0.42	97.78

The particle size distribution of the tested raw material complies with the ISO 4701:2019 standard, with 97.98% of particles having a particle size between 16 and 9.5 mm, in accordance with the standard (90.04%).

#### II.2.1.2.3. Crushing Strength (CCS - Compression Crush Strong)

To ensure good crushing strength in accordance with the ISO 4700 standard, at least 60 pellets per composite sample of the raw material, ranging in size from 9.5 to 16 mm, are tested. The procedure involves applying progressive force using a hydraulic press and measuring the resistance in kg/pellet.





**Figure II.4.** Compression Testing Machine.

**Table II.8:** Result of crushing strengths.  $+9.5 < \text{diameter} < 12.5$  mm.

N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	Average Kg/ Pellet
<b>1</b>	261	<b>13</b>	234	<b>25</b>	154	<b>37</b>	344	<b>49</b>	227	308.22
<b>2</b>	260	<b>14</b>	133	<b>26</b>	340	<b>38</b>	298	<b>50</b>	371	
<b>3</b>	299	<b>15</b>	250	<b>27</b>	345	<b>39</b>	219	<b>51</b>	338	
<b>4</b>	327	<b>16</b>	289	<b>28</b>	388	<b>40</b>	282	<b>52</b>	253	
<b>5</b>	426	<b>17</b>	367	<b>29</b>	388	<b>41</b>	451	<b>53</b>	150	
<b>6</b>	298	<b>18</b>	372	<b>30</b>	312	<b>42</b>	288	<b>54</b>	387	
<b>7</b>	293	<b>19</b>	432	<b>31</b>	363	<b>43</b>	295	<b>55</b>	293	
<b>8</b>	447	<b>20</b>	351	<b>32</b>	301	<b>44</b>	407	<b>56</b>	291	
<b>9</b>	179	<b>21</b>	263	<b>33</b>	314	<b>45</b>	200	<b>57</b>	280	
<b>10</b>	151	<b>22</b>	511	<b>34</b>	322	<b>46</b>	295	<b>58</b>	372	
<b>11</b>	161	<b>23</b>	240	<b>35</b>	294	<b>47</b>	342	<b>59</b>	188	
<b>12</b>	337	<b>24</b>	216	<b>36</b>	554	<b>48</b>	243	<b>60</b>	237	

**Table II.9:** Result of crushing strengths. 12.5 < diameter <16 mm.

N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	N°	CCS Kg/ Pellet	Average Kg/ Pellet
<b>1</b>	514	<b>13</b>	354	<b>25</b>	272	<b>37</b>	343	<b>49</b>	257	<b>398.58</b>
<b>2</b>	474	<b>14</b>	214	<b>26</b>	380	<b>38</b>	395	<b>50</b>	171	
<b>3</b>	293	<b>15</b>	461	<b>27</b>	288	<b>39</b>	339	<b>51</b>	404	
<b>4</b>	594	<b>16</b>	416	<b>28</b>	332	<b>40</b>	447	<b>52</b>	416	
<b>5</b>	758	<b>17</b>	393	<b>29</b>	556	<b>41</b>	247	<b>53</b>	393	
<b>6</b>	328	<b>18</b>	345	<b>30</b>	425	<b>42</b>	134	<b>54</b>	345	
<b>7</b>	446	<b>19</b>	888	<b>31</b>	342	<b>43</b>	373	<b>55</b>	888	
<b>8</b>	418	<b>20</b>	372	<b>32</b>	421	<b>44</b>	470	<b>56</b>	372	
<b>9</b>	607	<b>21</b>	344	<b>33</b>	385	<b>45</b>	410	<b>57</b>	344	
<b>10</b>	356	<b>22</b>	484	<b>34</b>	430	<b>46</b>	429	<b>58</b>	484	
<b>11</b>	387	<b>23</b>	354	<b>35</b>	290	<b>47</b>	234	<b>59</b>	354	
<b>12</b>	282	<b>24</b>	511	<b>36</b>	390	<b>48</b>	241	<b>60</b>	511	

The obtained results from these tests, as presented in the two tables above, provide insight into the compression resistance during screening and loading of the raw material into the furnace. This helps prevent the phenomenon of furnace blockage and the formation of blockage (casting). Iron oxide pellets with a resistance exceeding 250 kg/pellet comply with the reduction process.

It should be noted that the reduction process of iron oxide requires high-quality raw material. These results are crucial for adjusting the parameters set in the DCS (Distributed Control System), which will be altered based on the chemical composition provided by the quality engineer.

These reduction process parameters include:

- Ø Reduction temperature
- Ø Reducing gas flow
- Ø Cooling gas flow
- Ø Material loading and unloading speed

### II.2.1.3. Chemical Analyses:

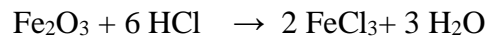
Chemical analyses of both the raw material and the reduced iron are carried out in the direct reduction laboratory using qualitative methods such as X-ray fluorescence (XRF) and

chemical methods including titration (oxidation-reduction reaction) and gravimetric methods based on complex precipitation reactions of undesirable elements.

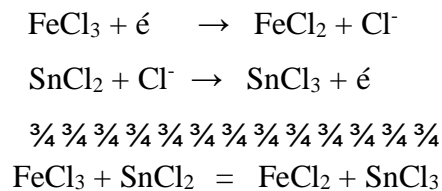
### II.2.1.3.1. Titration:

For the qualitative analysis, the sample needs to be prepared according to international standards. This involves three necessary steps: drying, crushing, grinding, and sieving to obtain fine particles with a size below 180  $\mu\text{m}$ , ensuring accurate results in line with ISO 2957-1/ISO 2957-2 standards. As per the standard requirements, the sample mass needs to be accurately determined based on the desired element.

To determine the iron content in the raw material sample, the sample is dissolved in an acid (hydrochloric acid or sulfuric acid) in a beaker on a hot plate at a temperature between 80 and 100°C to prevent water evaporation.

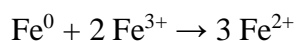


The resulting solution is mixed with a tin chloride solution. A redox reaction occurs, and a color change is observed, resulting in a colorless solution that indicates the complete reduction of Fe(III) to Fe(II) according to the reaction:



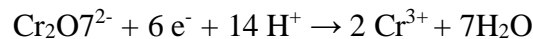
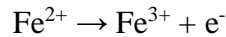
This is an exothermic reaction, and the solution is rapidly cooled to avoid reversible reactions.

To determine the degree of metallization (M), a reduced iron (DRI) sample is added to an Erlenmeyer flask with a solution of known iron chloride concentration. The solution is agitated for 45 minutes to dissolve the metallic iron.

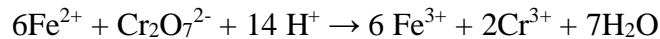


The iron content is determined using titration with a solution of potassium dichromate ( $K_2Cr_2O_7$ ) of known normality. The equivalence point is reached when the color changes from green to violet.

The volume used is employed to calculate the iron concentration in the solution and subsequently the iron content in the sample. The electrochemical reactions involved in this titration are:



The overall balanced equation for the titration reaction is:



For metallic iron ( $Fe(0)$ ), the same titration procedure is applied to the solution obtained after dissolution with  $FeCl_3$ .

### II.2.1.3.2. Impurity Determination:

Impurities ( $SiO_2$ ,  $Al_2O_3$ ,  $MgO$ ,  $CaO$ , and  $P$ ) are undesirable chemical elements present in the raw material. The protocol followed to determine the mass percentage of these elements is as follows:

The first step is to dissolve the raw material in acids to precipitate impurities such as  $SiO_2$ . The recovered solution is then analyzed to determine the composition of other impurities.

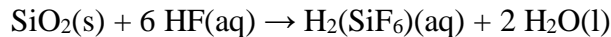
To do this, we dissolve the sample in 37% hydrochloric acid. The sample is placed in a beaker, and after rinsing the interior walls of the beaker with distilled water, a sufficient amount of acid is added. The dissolution is carried out on a hot plate at a temperature between 70-90 °C. Nitric acid is added to facilitate the release of nitrous acid ( $HNO_2$ ) for a few minutes. To ensure the removal of all free acids in the solution, perchloric acid ( $HClO_4$ ) is added. The release of gases is complete when a white smoke is observed. The resulting solution is filtered through a membrane.

#### a) $SiO_2$ Determination:

The precipitate obtained after membrane filtration contains silica ( $SiO_2$ ) as well as other oxides such as  $Cr_2O_3$  and  $TiO_2$ . To determine the silica content, this precipitate is transferred to a

platinum crucible and placed in a muffle furnace at 900 °C for 7 minutes. The crucible is then transferred to a desiccator to preserve it and prevent moisture absorption.

The total mass of the crucible with the precipitate is noted as (M1). The crucible is transferred to a hot plate, and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and hydrofluoric acid (HF) are added. Hydrofluoric acid is one of the few known liquids capable of dissolving silica (SiO<sub>2</sub>), according to the following chemical equation:



The end product is transferred back to the muffle furnace to complete the elimination of silicon. The total mass of the crucible with the precipitate after removal from the furnace is noted as (M2). The SiO<sub>2</sub> content in our sample is calculated using the following formula:  $\text{SiO}_2 (\%) = (\text{M1} - \text{M2}) / \text{M0}$  Where M0 is the initial mass of the sample.

This gravimetric method is part of ISO 2598-1-1992, which specifies two gravimetric methods for determining the SiO<sub>2</sub> content of iron ores.

#### **b) Determination of Other Impurities:**

The first step was to dissolve the raw material in acids, and the recovered solution after filtration is used to determine the content of other impurities. Aliquots of 25 mL of this mother solution are taken to determine the content of each chemical element. Aliquots are the result of distributing an initial volume into several equal parts. In short, it's a representative sample of the remaining characteristics. For example, in a solution, you take a 10 mL aliquot that represents 100 mL of the prepared solution in terms of characteristics like concentration.

- **Total Hardness (CaO and MgO):** Total hardness is due solely to calcium and magnesium ions. Hardness is determined using a complexometric titration with EDTA. Ammonium and ammonium chloride are added to two aliquots, and the hot plate temperature is raised to 70-90°C for 20 minutes to form a precipitate. After precipitation, the two solutions are filtered and then heated to 100°C to evaporate the ammonia. After cooling, the two solutions are titrated with an EDTA solution using specific indicators, and the color changes from red to blue.

The content of CaO and MgO is determined using the following equations:  $\text{CaO} (\%) = (\text{VEDTA} \times \text{Normality} \times \text{Mequi} / \text{Sample Mass}) \times \text{factor}$   $\text{MgO} (\%) = (\text{Total Hardness} -$

VEDTA - N x Mequi / Sample Mass) x factor Where: Total Hardness = CaO% + MgO%  
 Mequi1: equivalent weight of CaO Mequi2: equivalent weight of MgO Sample Mass:  
 mass of the sample used

- Alumina (Al<sub>2</sub>O<sub>3</sub>):** Similarly, a 25 ml solution is placed in a beaker on a hot plate, and a sodium hydroxide solution (NaOH, 50%) is added to form a precipitate. The final solution is filtered through a membrane to recover the precipitate. The solution's pH needs to be 5 to 6.  $\text{Al}_2\text{O}_3 + \text{H}_2\text{O} + \text{NaOH} \rightarrow \text{NaAl}(\text{OH})_4$   $\text{NaAl}(\text{OH})_4 \rightarrow \text{NaOH} + \text{Al}(\text{OH})_3 \downarrow$  The solution is heated until evaporation, then a colored indicator (xylene orange) is added, and the solution is titrated with a zinc sulfate solution (0.01 N). To calculate the percentage of Al<sub>2</sub>O<sub>3</sub>, the following formula is used:  $\text{Al}_2\text{O}_3 (\%) = (\text{Blank} - \text{VZnSO}_4) \times \text{N} \times \text{Mequi} / \text{M} \times \text{factor}$  Where: Blank: Standard solution Mequi: equivalent weight of Al<sub>2</sub>O<sub>3</sub> Sample Mass: mass of the sample used
- Phosphorus:** To determine the phosphorus content, a solution prepared with standard reagents is used to complete the experiment. This solution oxidizes phosphorus to PO<sub>4</sub>.

Two solutions, A and B, are prepared. Solution A is made from a diluted ammonium molybdate solution in ammonia and distilled water. Solution B is prepared by diluting nitric acid in distilled water with the proportion of 1/2.3. The solutions are mixed, and the mixture is allowed to precipitate for 24 hours.

A filter paper from the titrated solution during total hardness determination is taken and dissolved in nitric acid and the mixture of solutions A and B. The solution is heated for 2 hours and then passed through a membrane filter. The membrane needs to be rinsed with nitric acid (1/100) and a solution of ammonium nitrate (20%) until the pH is around 7. The filter residue is recovered using a sodium hydroxide (0.1 N) solution, and then the solution is titrated with hydrochloric acid (0.1 N). The P content is calculated using the following formulas:  $\text{PO}_4 (\%) = (\text{VNaOH} - \text{VHCl}) \times 1.34 / \text{M} \times 10$   $\text{P} \% = \text{Result} \times 0.321$

The results of impurity analysis in the raw material using the chemical titration method and gravimetric method are presented in the table below:

**Table II.10:** Pellet impurity analysis results.

Elements	T. Fe	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	S	P	FeO	SiO <sub>2</sub>
Content %	64.75	1.13	0.85	0.07	0.02	0.05	1.00	4.8

### II.3. Natural gas analysis

Gas chromatography (GC) is typically the analytical technique used to determine the composition of the natural gas and calculate its energy content.

**Figure II.5.** chromatography analysis.

**Table II.11:** chromatography analysis of natural gas.

Natural gas			
Date:	23/8/2023		Average
Sampling Time	8:20		
Element			
CH <sub>4</sub> (Methane)	83.80		83.80
C <sub>2</sub> H <sub>6</sub> (Ethane)	8.14		8.14
C <sub>3</sub> H <sub>8</sub> (Propane)	1.81		1.81
iC <sub>4</sub> H <sub>10</sub> (Iso Butane)	0.24		0.24
nC <sub>4</sub> H <sub>10</sub> (Normal Butane)	0.47		0.47
iC <sub>5</sub> H <sub>12</sub> (Iso I P)	0.08		0.08
nC <sub>5</sub> H <sub>12</sub> Normal I P)	0.15		0.15
C <sub>6</sub> +	0.006		0.006
N <sub>2</sub> ( Nitrogen )	5.07		5.07
CO <sub>2</sub> (Carbon dioxide)	0.23		0.23
G.K.CAL	9570.89		9570.89
N.K.CAL	8661.67		8661.67
MOL.WT	18.75		18.75
SP.GR	0.68		0.68
C <sub>3</sub> +	2.756		2.756
C <sub>5</sub> +	0.236		0.236
H <sub>2</sub> S (PPM)	<0.5		<0.5

#### II.4. Quality Control of Direct Reduced Iron (DRI) Production

Using the raw material with the previously mentioned physico-chemical properties, a reduced iron product (DRI) was obtained with a degree of metallization of 94.71% (Table II.12), confirming the product's quality according to international standards.

In this table, it can be observed that the values of the degree of metallization for the initial tests are lower than those of the subsequent tests. This confirms the intervention of the production team in adjusting production parameters during the early stages to improve the degree of metallization, which is evident from the fourth test conducted on the produced reduced iron. These tests are carried out over a 24-hour production period, and a degree of metallization between 94% and 95% is achieved.



**Table II.12:** Analysis Results of Produced Iron (DRI, production from **08/08/2023** to **09/08/2023**).

N° of Test	Carbone %	Sulfure %	Total Fe %	Metallc Fe %	Metallization %
1	2.58	0.001	91.41	87.21	95.41
2	1.88	0.001	91.75	87.66	95.54
3	1.99	0.001	91.14	86.70	95.13
4	2.54	0.001	91.58	87.39	95.42
5	2.34	0.001	90.59	84.03	92.76
6	2.31	0.001	91.57	87.27	95.30
7	2.13	0.001	91.52	86.54	94.56
8	2.21	0.001	91.47	86.74	94.83
9	2.26	0.001	90.79	86.77	95.57
10	2.12	0.001	91.18	84.58	92.76
11	2.31	0.001	90.85	84.80	93.34
12	1.94	0.001	91.26	87.53	95.91
<b>Average</b>	2.22	0.001	91.26	86.44	94.71

The results of impurity analysis conducted on the iron obtained after reduction using the chemical titration method and the gravimetric method are presented in the table below. The impurity content remains within the required quality standards:

**Table II.13:** Impurity Analysis Results of the Reduced Material (DRI).

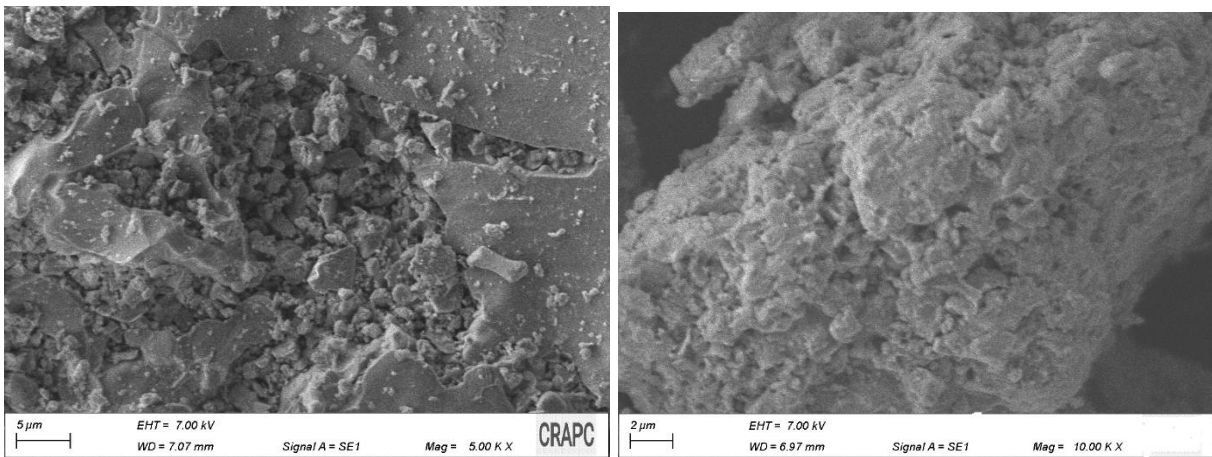
Chemistry						
Element	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	P	S
<b>Target (%)</b>	<b>4.8</b>	<b>1.13</b>	<b>0.85</b>	<b>0.07</b>	<b>0.05</b>	<b>0.02</b>

## II.5. Scanning Electron Microscope (SEM) analysis

Scanning Electron Microscopy (SEM) allows for the description of all topographical aspects of the solid. The material being analyzed must be conductive to avoid electron charging phenomena. When coupled with an EDX sensor, it is possible to determine the chemical composition.

In our case, SEM analysis was carried out on two samples, the first concerns pellets of the raw material and the second concerns iron produced after direct reduction (DRI). Both samples were crushed and analyzed in powder form.

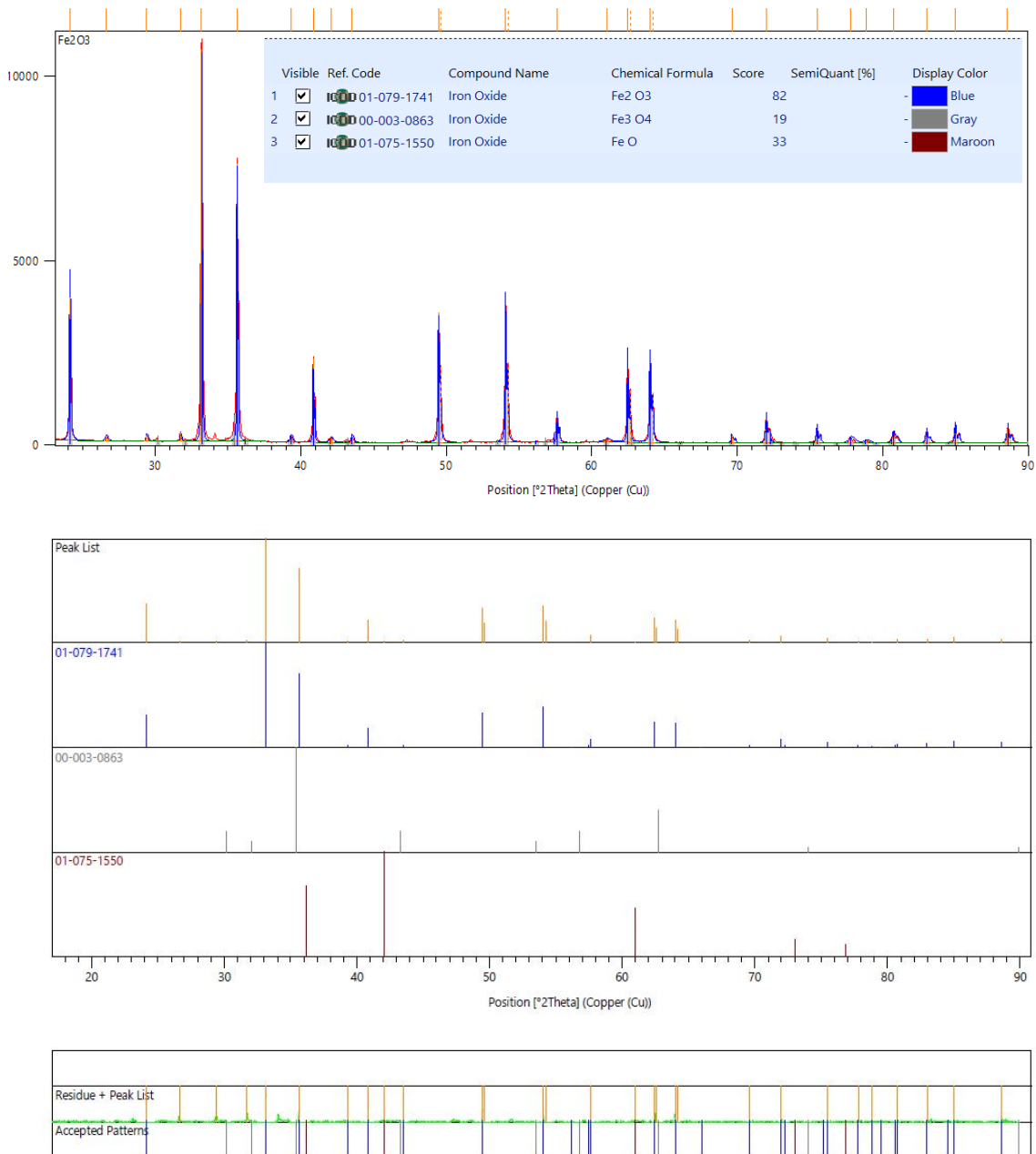
**Figure II.6** shows the SEM images of the two powders. Both powders are compact and agglomerated with a fine particle size. A difference is visible between the raw material composed essentially of iron oxide and the reduced material where metallic iron is produced. The grain morphology is less uniform in the case of the raw material with the presence of large and small particles. Conversely, after reduction, we obtain a more or less homogeneous material of metallic iron.



**Figure II.6.** Micrograph of the powder of a) raw material and b) direct reduced iron (DRI).

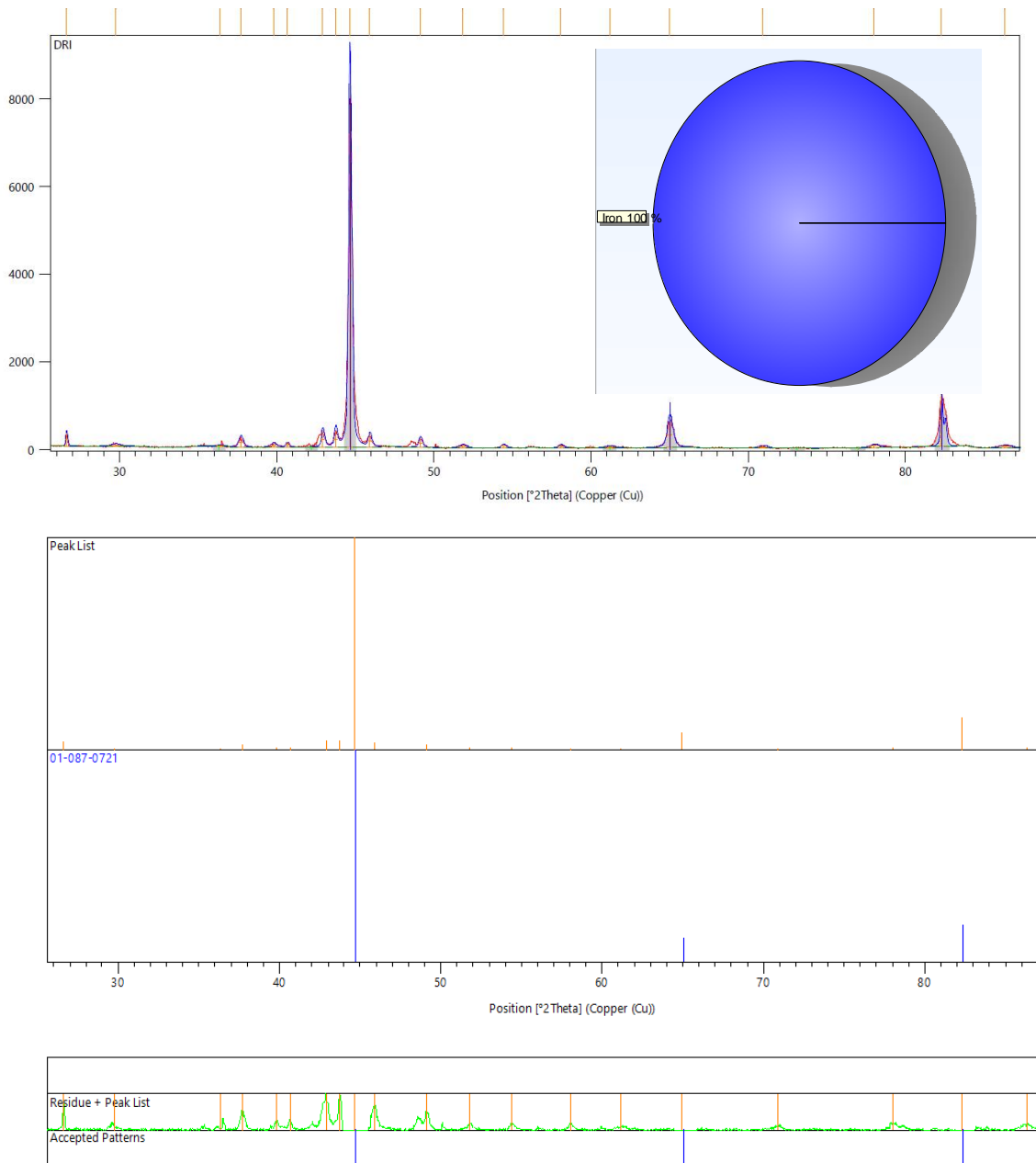
## II.6. X-ray diffraction (XRD) analysis

The X-ray diffraction (XRD) technique is used to characterize the structure of powders and crystalline materials (minerals, metals, ceramics, crystalline organic products). The diffractograms were obtained using a Malvern Panalytical powder diffractometer with  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.54056 \text{ \AA}$ ) and  $2\theta$  ranging from  $4$  to  $90^\circ$ . This technique allows the recognition of products with the same chemical composition but different crystalline forms and also identifies all phases of crystalline compounds in the analyzed sample.



**Figure II.7.** X-ray diffractogram of a sample of iron ore raw material.

Figure II.7 shows the X-ray Diffractogram of the starting iron ore used for the production of DRI. The XRD shows that the sample contains several crystalline compounds and in particular hematite (Fe<sub>2</sub>O<sub>3</sub>), Fe<sub>3</sub>O<sub>4</sub> and FeO. Data processing was carried out using the HighScore plus software with the 2019 version of the International Center for Diffraction Data (ICDD) database. The XRD shows the maximum positions of the main minerals. Significant peak overlap is apparent. This analysis confirms that hematite is the majority composition of iron ore.



**Figure II.8.** X-ray diffractogram of a powder of direct reduced iron (DRI).

The XRD of the obtained iron powder (**Figure II.8**) after direct reduced iron (DRI) confirms the reduction of iron oxide to iron and attests the results obtained in **Table II.12**.

*General conclusion*

# General conclusion

This work aimed to describe the direct reduction process of iron at Algerian Qatari Steel company (AQS) as well as the various chemical, physical, mechanical and thermal characterizations in order to produce a steel with the properties demanded by the national and international market.

The complex uses a large direct reduction unit using the MIDREX process and natural gas as the reducing gas source and energy source. Iron oxide pellets are reduced (oxygen is removed) to solid-state metallic iron by a reducing gas composed of hydrogen and carbon monoxide which itself is manufactured by reforming natural gas. The result is a solid product called sponge iron or reduced iron (DRI). The various physicochemical characterizations carried out on the raw material and the DRI product confirm the obtaining of a reduced iron product (DRI) with a degree of metallization of 94.71% attesting the high quality of the product in accordance with international standards.

The MIDREX process is an efficient and environmentally friendly method for direct iron reduction, widely used in the steel industry. It utilizes natural gas to reduce iron ore pellets, producing high-quality direct-reduced iron (DRI) that can be used as a feedstock in steelmaking. This process offers several advantages, including lower greenhouse gas emissions, energy efficiency, and reduced reliance on traditional blast furnaces. Overall, the MIDREX process plays a crucial role in modern steel production by enhancing sustainability and resource utilization.

## **Abstract**

The Bellara Steel Complex of Algerian Qatari Steel company (AQS) uses one of the best advanced technologies for the production of reinforcing steel bars and rods of different diameters. The raw material, in the form of pellets primarily composed of iron oxide (hematite,  $Fe_2O_3$ ), is reduced in the direct reduction unit using MIDREX technology with the aid of gas.

In this study, we focused on the direct iron reduction process using MIDREX technology by describing the process and conducting various tests to ensure the quality of the reduced iron after this crucial first step in obtaining a finished product that meets international market requirements.

**Keywords:** AQS, Steel, DRI, MIDREX, XRD.

## **Résumé**

Le Complexe sidérurgique de Bellara de la compagnie Algerian Qatari Steel (AQS) utilise l'une des technologies les plus avancées pour la fabrication du rond à béton et de file machine de différents diamètres. La matière première sous forme de pellets constitués essentiellement de l'oxyde de fer (hématite,  $Fe_2O_3$ ) est réduite dans l'unité de la réduction directe en utilisant la technologie MIDREX à l'aide du gaz.

Dans ce travail, nous nous sommes intéressés à l'étude du procédé de réduction directe du fer en utilisant la technologie MIDREX à travers la description de ce procédé et la réalisation des différents tests pour assurer la qualité du fer réduit après cette première étape crucial pour l'obtention d'un produit fini conforme aux exigences du marché international.

**Mots clés :** AQS, Acier, DRI, MIDREX, DRX.

## **المخلص:**

يستخدم مجمع بلارة التابع للشركة الجزائرية القطرية للصلب (AQS) واحدة من أحدث التقنيات لتصنيع قضبان تسليح الخرسانة وقضبان الأسلاك بأقطار مختلفة. يتم اختزال المادة الخام على شكل كريات تتكون أساساً من أكسيد الحديد (الهيماتيت،  $Fe_2O_3$ ) في وحدة الاختزال المباشر باستخدام تقنية MIDREX و الغاز.

نهتم في هذا العمل بدراسة عملية اختزال الحديد المباشر باستخدام تقنية MIDREX من خلال وصف هذه العملية وإجراء اختبارات مختلفة للتأكد من جودة الحديد المختزل بعد هذه الخطوة الحاسمة الأولى للحصول على منتج نهائي يلبي متطلبات السوق الدولية.

**الكلمات الرئيسية :** AQS ، الصلب، DRI ، MIDREX ، الأشعة السينية.