CARBONIZATION STUDY OF NON COKING COALS AND CHARACTERISATION OF THEIR PROPERTIES FOR APPLICATION IN SPONGE IRON MAKING

A

Thesis

Submitted by

RAKESH KUMAR

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS

For the award of master of technology

In

Steel technology

Roll No.-212MM2339

M. Tech.

Department of Metallurgical and Materials Engineering

National Institute of Technology, Rourkela – 769008

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Under Supervision of

Prof. Dr. **M. Kumar**

Department of Metallurgical and Materials Engineering
National Institute of Technology, Rourkela – 769008

Department of Metallurgical and Materials Engineering
National Institute of Technology, Rourkela

CERTIFICATE

The research titled **"**carbonization study of non-coking coals and characterization of their properties for application in sponge iron making**",** submitted by Rakesh Kumar for the award of degree of Master of Technology in steel technology, has been carried out under my supervision at the department of Metallurgy and material science of National Institute of Technology, Rourkela. The work is comprehensive, complete and fit for evaluation.

Prof. M. Kumar

Metallurgical & Materials Engineering
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Abstract

In view of unavailability of costly coking coal (used for blast furnace route) in several countries including India, DRI route of iron making is getting momentum. At present India produces around 20MT sponge iron per annum, in which coal-based process have major contribution.

Studies on the effect of different carbonization temperatures (400, 600,800, 900°C), chemical and physical properties (proximate analysis, reactivity, ash fusion temperatures and caking characteristics), and calorific or heating value of different coal samples, procured from different mines in India, were undertaken in the present project work for their judicial selection in Indian sponge iron plants. In the present project work, studies on chars prepared from Western coalfield limited (Madhya Pradesh), Southern east coalfield limited (Chhattisgarh), Mahanadi coalfield limited basundhara (Orissa) and Jharsuguda (belpahar), Kalinga and Lingaraja non coking coal at four different carbonization temperature (400, 600, 800,900°C) were characterized for their chemical (proximate and ultimate analysis), physical (apparent density and apparent porosity) properties, ash fusion temperature, calorific value, XRD, and reactivity. It is found that the fixed carbon content, ash content increases while volatile matter and calorific value reduced with increase in carbonization temperature. Reactivity of the coal chars towards CO_2 was measured at these carbonization temperatures and it was investigated that with increase of carbonization temperature, the reactivity of chars towards CO_2 was reduced. It was also investigated that Mahanadi coalfield limited (MCL) coal has maximum fixed carbon content and Jharsuguda mines coal has higher calorific value while SECL, korba char has highest fixed carbon content. Idea behind this is to found good coal and match its property according to its application in sponge iron plant. Studies on the effect of different soaking time and different heating rate also carried out and it was investigated that different soaking time does not give any significant effect on its chemical and physical properties but heating rate gives certain effect on its properties.

Keywords: Non-coking coal, proximate analysis, ultimate analysis, calorific value, true density, fixed carbon content, reactivity, ash fusion temperature, porosity, density, carbonization, soaking time, heating rate.

Chapter 01

Introduction

1.1 Theory and Objective:

The sponge iron route of iron making based direct reduction process has of late come up as one of the major alternative route of iron making in India. Presently total steel production in India is around 56 million tons per annum and this has to be increased up to the level of 120 million tons by 2020. In order to achieve this target, direct reduction (DR) route was develop in the country at 1980. There was only one sponge iron plant in India i.e.in Andhra Pradesh but there are approximate 300 sponge iron plants in the country and they are producing around 20 million tons per annum of direct reduced iron. In view of the difficulties involve in blast furnace route of iron making (unavailability of coking coal, emission of a lot of polluted gases and capital investment) leads to promote the massive utilization of sponge iron route.

India's anticipation for the demand of steel as well as its mineral resources, liberalization of economic policy and the continuous globalization of market have attracted many foreign and national steel magnates to invest in the steel sector in India. In result of the abundant and easy availability of low-graded (D, E, F, and G) non-coking coals in India, more attention is being paid in recent years toward their utilization in sponge iron plant (DRI). Over the last few years, coal-based (rotary kiln) direct reduction processes have gained importance as a major potential alternative route of iron making in India. The DRI routes of iron making technology has more economic viability due to its ability to produce a significant amount of electricity through the use of hot waste gases and char. The quality of coal char used in this route is one of the most important problems to be solved to use them more effectively in these processes. For successful utilization of coal in sponge iron making, the properties that need to be well understood are fixed carbon, volatile matter, ash and sulphur contents, caking and swelling indices, ash fusion temperatures, char reactivity and its strength, bulk density, porosity, density and their reduction ability of iron ore. [1] coal and char with low ash content high fixed carbon content, low sulphur content, high ash fusion temperature (150-200°C more than operating temperature of the rotary kiln), high reduction potential (reactivity towards CO_2) and high calorific value or heating value. The chemical composition of coal ash must be known for this process of iron making because Presence of TIO_2, Al_2O_3, SIO_2, and Fe_2O_3 tends to increase the ash fusion temperature but presence of alkali oxide like Fe_2O_3, CaO, MgO, Na_2O, and SiO_2 commonly decrease the ash fusion temperature.

India has a huge reserve of coal, of which only 14% is of the coking variety and the remaining86% of poorly coking or non-coking type. Decreased reserves of coking coal in India [2] and increased import restrictions prompted us to look for alternatives to BF route. Therefore, it was thought beneficial to use this coal for iron production [3, 4] by blending it with other suitable materials [5, 6]. Non coking coal has no sticking tendency so it can be frequently used in sponge iron plant. The parameters required for the coking of coal primarily concern proximate analysis, ultimate analysis.

Presently, non-coking coal is one of the major raw materials required for production of sponge iron. Our major concern is about production of sponge iron in the rotary kiln for that non coking coal is used as source of reducing agent and heat source. [7]

1.2 Different routes of iron making:

1**. Blast furnace route**: - blast furnace route of iron making needs coke i.e. obtained from carbonization of coking coal.

Disadvantage of blast furnace route

- This route of iron making is very costly because 86% of the total coal reserves of India are non-coking coal and rest 14% is coking coal. So India is importing coking coal from Australia. Therefore it is very costly and scarce.
- gestation period is higher
- Capital investment involved in blast furnace route is high.
- Secondly the blast furnace route of iron making emits a lot of polluting gases in atmosphere which are very harmful.
- Less flexibility in production capacity

2. Sponge iron route or DRI route: - in view of difficulties faced in blast furnace route and in order to promote the massive utilization of non-coking coal in iron making, the DRI (direct reduced iron) or sponge iron route was developed. Direct-reduced iron (DRI), also known as sponge iron, [8] is produced from direct reduction of iron ore (in the form of lumps, pellets or fines) by a reducing gas produced from natural gas or coal. The reducing gas is a mixture majority of hydrogen (H_2) and carbon monoxide (CO) which acts as reducing agent. This process of directly reducing the iron ore in solid form by reducing gases is called direct reduction.

Advantage of DRI route over blast furnace route: followings are the advantages of DRI routes over BF route:

- Environment friendly compared to blast furnace route
- Huge amount of availability of non-coking coal
- Simple plant operation
- Good flexibility in production
- Attractive investment cost
- The energy requirements of a sponge iron plant are much less than blast furnace plant.

1.3 Factors to be Considered for Selection of Coal in Sponge Iron making [9]:-

1. Chemical composition (proximate and ultimate analysis): The coal to be used in sponge iron making should have fixed carbon content more than 40%. Fixed carbon content not only acts as reducing agent but also provides some of the heat energy for the process to occur. Volatile matter content should be in the range of 27-30% in order to utilize it respectively in sponge iron making. If the volatile matter content more than 32% it will be difficult to control such a huge volume of gas in the reactor. As the ash content in the coal increases, the amount of valuable fixed carbon content decreases and the amount of slag formed in the reactor also increases. High slag formation in the reactor increases the energy consumption and flux consumption in the kiln. Not only this, high ash formation in the kiln unnecessarily occupies more effective volume of the kiln. Hence ash content in the coal should be as low as possible.

2. Total hydrogen and carbon contents: Total hydrogen and carbon content in the coal should be as high as possible to increase the heating or calorific value of the coal.

3. Sulphur and phosphorous content: Generally Sulphur and phosphorous content in the coal should be in the range of 1to 1.5%.

4. Reactivity of coal towards carbon dioxide gas (CO_2): Reactivity of coal for its utilization in sponge iron making should be more than 2cc/gm./°C. Higher the reactivity more is the cogeneration and better is the reduction of iron ore.

5. Energy or calorific value: coal used in the sponge iron making partially meets the thermal requirement of the process. Higher the calorific value, more will be heat generation and hence lesser will be the amount of coal required for iron making.

6. Ash fusion temperature: Coal used in sponge iron making should be high. Softening temperature of coal ash should be at least 150-200°C more than the operation temperature of the rotary kiln.

7. Coal char strength: Non-coking coal used in sponge iron making gets converted into coal char. The coal char should have higher strength in order to allow free passage of gas.

8. Bulk density: bulk density of the material effect

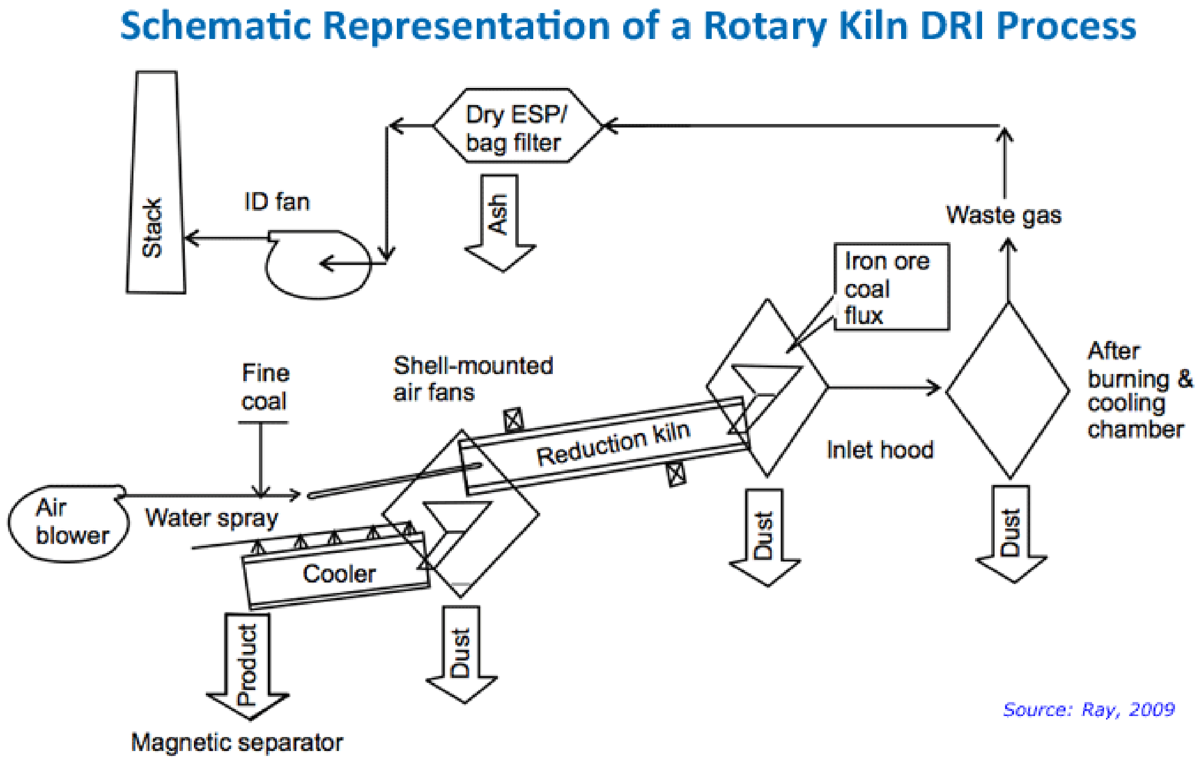
a) The production rate

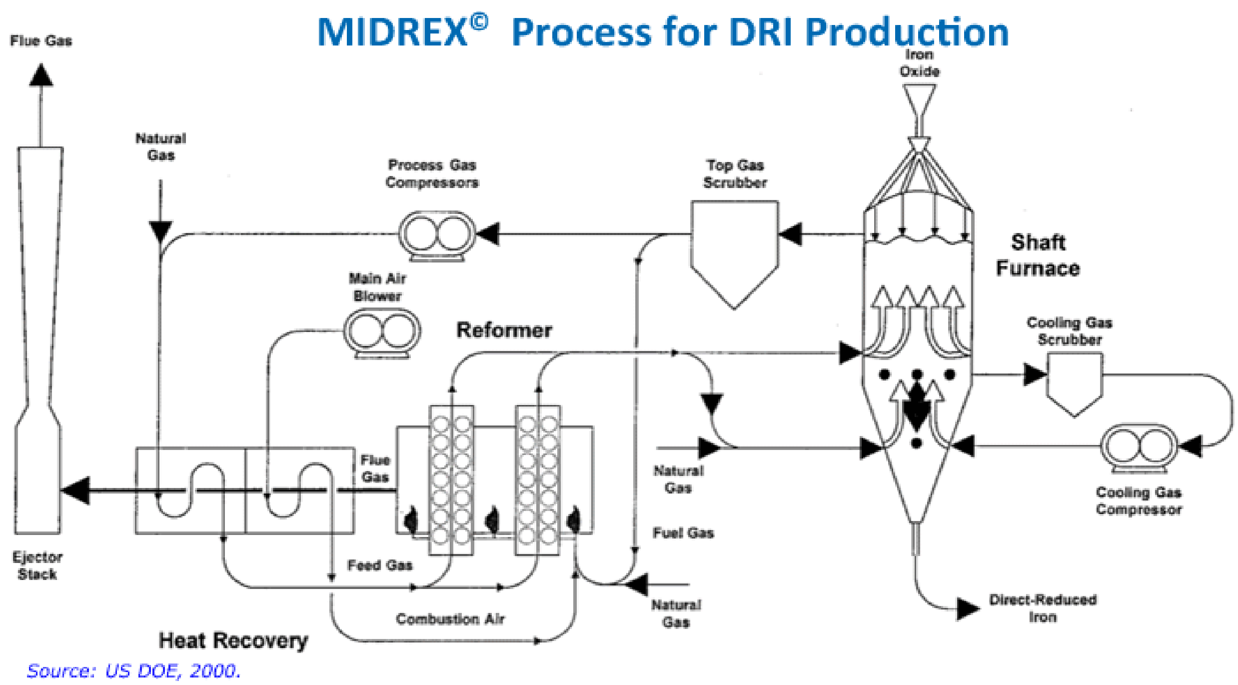
b) The transportation cost

Higher the bulk density more the production rate and lower the transport cost. For use of coal in sponge iron making its bulk density should be more than 800kg/m³.

9. Size of coal: for the use in sponge iron making, the size of the coal should be in the range of 4-8mm. If size is large means less surface area and less reactivity. If size is small, less porosity then gases cannot escape.

1.4 schematic representation of rotary kiln process

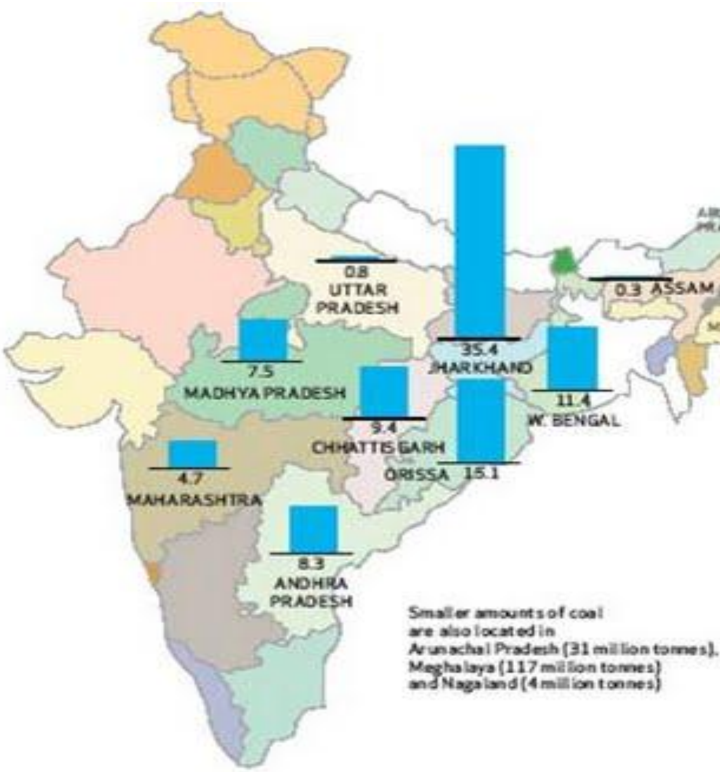




1.5 State-wise Coal reserves in India :(All figure in billions tones). [10]

Table 1.1: state wise coal reserves in India

State	Proved	Indicated	Inferred	Total
Jharkhand	40.2	34	6.61	80.81
Odisha	25.5	36.57	9.44	71.51
Chhattisgarh	14	33.51	3.43	50.94
West Bengal	12.5	13.41	4.77	30.68
Madhya Pradesh	9.30	12.30	2.80	24.40
Andhra Pradesh	9.61	9.60	3.04	22.25
Maharashtra	5.71	3.12	2.10	10.93
Uttar Pradesh	0.89	0.19	0.00	1.08
Meghalaya	0.10	0.02	0.48	0.60
Assam	0.48	0.05	0.01	0.54
Nagaland	0.01	0.00	0.32	0.33
Bihar	0.01	0.00	0.15	0.16
Sikkim	0.00	0.07	0.05	0.12
Arunachal Pradesh	0.03	0.05	0.02	0.10



State wise Coal Reserves in India

Note- 86% of the total coal reserves of India is non-coking coal and rest 14% is coking coal.

1.6 Grading of Indian coal:

Dried Coals of India have been graded into various categories starting from A to G, as shown in Table 1.2. This grading is based on the ash content and energy value of coal. As proceed from grade A to G quality of coal is decrease.

Table 1.2: Grading of Indian coals

Grading of coals	Calorific values range (kcal/kg)
A	5700-6300
B	4800-5700
C	4200-4800
D	3400-4200
E	2400-3400
F	1300-2400
G	>1300

Normally D, E and F coal grades are available to Indian Industry [11]

1.7 Production and export of iron ore during last decade

India is one of the major producer of iron ore after China, Brazil and Australia. India is contributing more than 7% of total producton of iron ore in the world. As shown in Table1.3, total production of iron ore increases reguraly because of demand of iron ore for steel industry. Steel production in the country is griwing at faster rate therefore requirement of iron ore by steel industry increases continously. Consumption of iron ore by domestic industries is increases but the production is still much higher then consumption so leads to increase in export of iron ore. So, production and export of iron ore increases.

Table1.3: production and export of iron ore in India[12]

Year	Production	Export
2001-02	87.13	42
2002-03	99.08	48
2003-04	123.16	63.11
2004-05	147.22	78
2005-06	164	90
2006-07	181.20	94.36
2007-08	213	104.22
2008-09	213.87	106.55
2009-10	218	117
2010-11	207	98.18
2011-12	168	62
2012-13	158	45.22

1.8 Year wise sponge iron production in India:

Presently total amount of steel production in India is around 56 million tons per year and this has to be reached up to the significant level of 110 million tons by 2016. To achieve this target, direct reduction (DR) route was develop in the country at 1980. There was only one sponge iron plant in India in 1980 i.e.in Andhra Pradesh but there are approximately 300 sponge iron plants in the country at present that leads to increase in production of sponge iron and now total production of sponge iron is around 20 million tons per annum [13].

Table1.4: year wise data for sponge iron (DRI) production in India

Year	Production	Growth
1993-94	2.42	66.40
1994-95	3.40	41.51
1995-96	4.42	29.82
1996-97	5.02	13.70
1997-98	5.30	6.11
1998-99	5.20	-1.5
1999-00	5.35	23
2000-01	5.48	26.21
2001-02	5.45	-9.12
2002-03	6.92	27.1
2003-04	8.11	17.10
2004-05	10.30	27.48
2005-06	11.5	11.35
2006-07	16.31	-
2007-08	20	-

Chapter 02

2. Literature review:

Characteristics of non-coking coals by M. Kumar and S.K. Patel (2008) [14]

Prof. M. Kumar and Prof S.K. Patel research work carried out on characterization of non-coking coals procured from different coal mines were undertaken for the selection in sponge iron plant. They found that sulphur content (range .40-.66) is not a problem. They also found that majority of coals have no caking characteristics. Majority of coal ash were found to have high ash fusion temperatures (IDT>1100, ST>1349, HT>1500, FT>1500). The result shows that fixed carbon content increases in the chars, reactivity of coal towards carbon dioxide (CO_2) decreases but majority of char have significantly higher reactivity (more than 4cc/gm./°C). Further studied were carried out on the reduction potential of coal sample and founded that chars exhibited higher degree of reduction potential led to reduce the time of reduction. Their result show that the all the coal sample are efficiently applicable in industry for production of sponge iron.

India Prof. Kalyan sen of emeritus (2008) [15]

Prof. Kalyan sen studies on the effect of chemical and physical properties (proximate analysis, reactivity towards carbon dioxide and ash fusion temperatures, Sulphur content, chlorine content), and calorific or heating value of coal sample were procured from coal mines in India. He found that coal have maximum 30% ash content and calorific/heating value in the range of 4940-6200kcal/kg. Majority of coal ash were found to have high ash fusion temperatures (IDT>1280) and efficient fixed carbon content. All the characteristics of coal are frequently useful for sponge iron plant.

Characterization of properties and reduction behavior of iron ores for application in sponge iron making Prof. M. Kumar and S.K. Patel Mineral Processing and Extractive Metallurgy Review, [16]

Prof. M Kumar and S.K. Patel studied on characterization of non-coking coal and iron ore used for sponge iron plant. He found that all the coal sample have initially good reduction potential there after it reduced. The result obtained show that slow heating provided higher reduction ability.

Byong Chul Kim [17]

Prof. Byong chul kim studies were carried out on the effect of carbonization temperature on coal characteristics. He found that as the carbonization temperature of coal sample increased the reactivity of coal decreased. During his research work he found that higher temperature lead to decrease in active carbon site and it lead to reduction in gasification rate. This effect was mostly found in low rank coal with compare to high rank coal. It was found that carbonization temperature have a great effect on coal sample with high volatile matter content [18] .Heating rate employed during carbonization of coal sample affects the product yield and distribution. Rapid carbonization leads to higher reactivity of char [19].higher heating rate depressing the development of carbon structure that leads to increase the active sites there by decreases the gasification rate of char [20]

pushpa singh nandita choudhury A. Sarkar, P. Sarkar [21]

Investigation was carried out on reactivity assessment on two high ashes, non coking coal (bituminous) of India. It has been found that inertinite rich low rank coal (2.88-22.93) has more reactivity than vitrinite rich high rank coal (2.24-9.40) at different temperature (420, 455, 490, 525, 560°C). Their result shows that vitrinite rich high rank coal, the combustion sustain for larger period while inertinite rich low rank coal has lower combustion period comparatively higher rate of combustion.

Carbonization analysis by Prof. M. Kumar and Prof. R.C. Gupta [22]

Prof. M. Kumar and Prof S.K. Patel investigated the effect of different carbonization temperature on the properties of char obtained during their research work on carbonization of non-coking coals procured from Dhanbad coal mines. The result shows that increasing of carbonization temperature from 400°C to 1000°C increases the expulsion of gases from the coal sample therefore volatile matter content and hydrogen content decreases continuously. With decreases of hydrogen content the calorific value decreases. The result of reactivity of coal chars at four different carbonization temperatures 400, 600, 800, 1000°C shows that increased of carbonization temperature decreased the reactivity of char towards carbon dioxide (CO_2). Apparent density initially decreased with increasing carbonization temperature up to 400°C there after increases while the true density increases continuously.

Reymond C. Everson et al:

His work was regarding the characterization and combustion of coal. The result obtained by him investigate that the char has low porosities which indicate the presence of high inertinite in that coal (obtained by South African mines). [23]

Cypress Rene, Soudan Clere, Moinet [24]

Results were carried out on the pyrolysis of blends of bituminous coal and iron ore (hematite or magnetite) using thermo-gravimetric and analysis of gases. The primary reduction of iron ore by CO and H_2 has been observed at temperature between 400°C and 500°Cat the heating rate of 3.2°K/min. At 600°C magnetite is progressively reduced to wustite there after iron. Reduction of iron ore by CO becomes more important at higher temperature, CO being produced from boudouard reaction. Production of CO depends on reactivity of char toward $CO_{2.}$ Chars should have high reactivity for proper reduction of iron ore.

Narcin N., Aydin S., Sesen K., Dikec F. [25] : Reduction potential of coal sample depends on the fixed carbon content and total carbon content that is determined by proximate analysis and ultimate analysis respectively. Iron ore reduction with domestic lignite coal in semi rotary tube furnace depends on coal consumption ratio ($C_{FIX}/C_{TOT.}$) Narchin et al established that at temperature 1000°C and coal consumption ratio 0.40, reduction of iron ore was completed in about 90 min. this result may helpful in industrial process.

Dutta, S. Wen, C.Y. and Belt, R.J. (1977) [26]

The results were carried out on Gasification of char towards CO_2 atmosphere at temperature range of 840-1100°C that can be divided into two stages, the first stage due to pyrolysis and the second stage due to char-CO_2 reaction. Reactivity in the first stage is mainly a function of the volatile matter content of the char/coal and the rate of heating. The reactivity of a char in the second stage char-CO reaction is found to depend more on its coal seam than on the gasification scheme used for its production. Each char/coal sample is found to have its own characteristic rate curve. The rate of reactivity characteristics of char and coal are due to their pore characteristics, which again change with conversion and temperature. The rate of char-CO_2 reaction is found to have little relation with the total nitrogen surface area of the pores. The study shows a tendency that only a fraction of the total nitrogen surface area which is occupied by pores above a certain size is available for reactions.

D.D. Haldar: [27]

Beneficiation of non-coking coal was carried out to improving its properties like heating/calorific value, volatile matter content, char characteristics and strength and reducing the inert content. Beneficiation of non-coking coal has been done to provide all the suitable properties to the coal i.e. most suitable for iron production. The studies of up gradation or beneficiation of coal is that coal particles recovered as cleans should have the coking property in case of coking coal and combustible behavior in case of non-coking coals i.e. suitable for iron production.

Binayak mohapatra and Dharanidhar patra [28]

The results were carried out on reduction behavior of iron ore lumps by non-coking coal in the temperature range of 850-1000°C and it was found that reduction time and temperature influenced by the degree of reduction i.e. increases with reduction temperature. In reduction time of 15-120minutes the reduction rate was improved up to 50-70% thereafter decreases. He investigated that there was no effect of type of coal on the degree of reduction of iron ore lumps. The reduction behavior of iron ore lumps was identical in all the studied coals.

Romeo M. Flores et al [29]:

According to Mr. Romeo M. Flores the different composition of coal effects porosity, permeability and volatile gases and these characteristics affects the performance of coal in combustion. Shrinkage and swelling characteristics are depending on the gas content in the coal. % of fixed carbon measures energy value of coal.

Petrographic study of coal by Claudio Avila et al [30]:

Claudio Avila et al research work studies were carried out on 25 different coal sample procured worldwide. Coals characterization shows important change in vitrinite (it leads to porosity) and intrinsic potential. Proximate analysis of all 25 coals sample were carried out in two different condition 1) as received and 2) after dried (ash free) its results show that fixed carbon and volatile matter obtained in 2nd condition is higher with compare to obtained in 1st condition but the ratio of carbon content and volatile matter increases. Petrographic study of coal sample reveals its texture property of coal, it was done by microscope attached with photomultiplier at 10x320 magnifications to measure reflected intensity of light beam.

Mohsen S. Masoudian et al [31]:

Search works were carried out the various effect of carbon dioxide on coal. Results reveal that with the adsorption of CO_2 the strength and elastic modulus were changed. This result was reversible. CO_2 adsorption may changes the micro structure as observed in SEM.

Chapter 03

3. Aims and objective of present project work:

The present project work was under-taken for study with the following objectives:

1. To study the proximate and ultimate analyses of coals obtained from different mines of India.

2. To study the calorific values of all these selected coals.

3. To study the ash fusion temperature (IDT, ST, HT, FT) of the coal ashes.

4. To study the reactivity of coal chars towards carbon dioxide gas.

5. To study the apparent porosity and true density of these coal samples.

6. To study the effect of carbonization temperature on the characteristics of coal chars obtained.

7. To study the effect of heating rate on the properties of coal chars obtained.

8. Analysis of the results obtained to assess their suitability in sponge iron making.

Chapter 04

4. <u>Experimental procedure:</u>

4. 1. Selection of Material: -

The non-coking coal of was collected from Western coalfield limited (MP), Southern east coalfield limited (CG); Mahanadi coalfield limited basundhara (Orissa) and jharsuguda (belpahar) for present project work.

4. 2. Proximate analysis: - [(Indian standard 1350 Part I -1984)]

It determines the percentage of moisture, volatile matter, percentage of ash, percentage of fixed carbon content in the coal. These constituents were determined as per Indian standard method [32]. The short descriptions of the procedure followed are as follows:

Moisture content:

1 gm. of dried sample of -72 mesh size coal powder was taken in a borosil glass crucible and placed in air oven and maintained up to temperature of 105-110^{o}C for one hour. The loss in weight expressed as amount of moisture content in the sample.

% loss in weight = % moisture content

Volatile matter content:

1 gm of dried sample of-72 mesh size coal powder was taken in a cylindrical crucible of silica covered with lid then kept the crucible in the furnace maintained at a temperature of 925-950^{0}cand kept for 7 minute only the crucible was taken out and loss in weight was determined.

The % of volatile matter calculated as-

% volatile matter= %loss in weight - %moisture content

Ash content:

1 gm. of dried sample of -72 mesh size coal powder was taken in a shallow disc of silica. The disc along with coal sample was placed in a furnace maintained at temperature of 770-800ºc and kept there till complete burning usually it takes one hour.

The % of ash content calculated as-

%ash content = %weight left

Fixed carbon content:

The fixed carbon content calculated as

Fixed carbon % = 100 – (%moisture+%ash+%volatile matter)

4. 3. Ultimate analysis:-

The objective of ultimate analysis of coal sample is to find the total carbon content and hydrogen content. Ultimate analysis has been performed in Punjab technical university with the help of one of my friend.

4.4. Calorific/heating value :

The amount of heat generated by combustion of unit weight of the coal sample is described as calorific value. The calorific values of coals and their chars were determined as per Indian standard method [33]. The procedure followed is as follows:

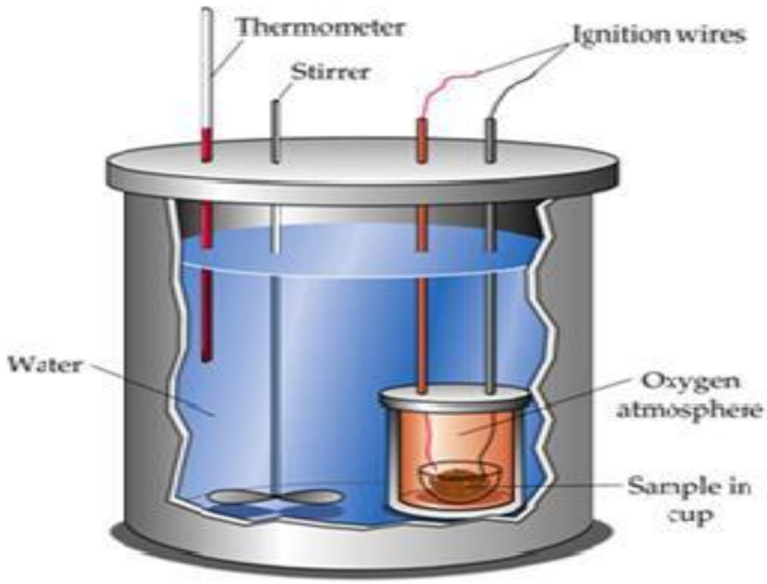
Instrument used: Bomb calorimeter

Process:

Calorific value or heating value of coal sample was determined in bomb calorimeter. 1gm. of prepared briquetted coal sample was placed with cotton thread, chromyl/platinum wire in a bomb. Oxygen gas was supplied in the bomb at pressure of 25kg/cm². Now this bomb was placed in water filled vessel (as shown below) that was connected with power source for spark.

[34]

The briquetted sample was combusted in presence of O_2. The rise of temperature was noted down every one minute till the attainment of maximum temperature. There after cooling start.



Arrangement of bomb calorimeter as describe in the above diagram

Formula:

GCV = [{WE*(ΔT+.04)}/W]

GCV: Gross calorific value

WE: Water equivalent = 2400kcal/^{o}c

ΔT: Temperature difference between maximum and minimum temperature

W: weight of the sample

4.5. Reactivity of coal:

It is the ease by virtue of which the coal reacts with oxidizing gases like carbon dioxide, air, water vapour. Higher the reactivity towards carbon dioxide gas more will be the amount of carbon mono oxide (CO) formed and better will be the reduction of iron ore in the rotary kiln. [34]

Process:

Before measurement of reactivity, coal is required to be converted into coal char by heating the coal at a temperature of 950°C for 2 hours in absence of air. The coal char obtained is then processed for determination of its proximate analysis. 5gm. sample of -16 and +500 micron size of prepared char sample is taken in the quartz tube, sealing the ends of sample. The sample is kept in the uniform heating zone of the furnace then nitrogen gas is passed at the rate of 50c.c./min and the sample is maintained at temperature of 1000±5°C. When the temperature is stabilized CO_2 gas is passed at the rate of 100c.c. /min. for 25 minute after that CO2 gas flow is stopped and again nitrogen is passed at the earlier rate of 50c.c. /min. until the temperature of reacted sample is bought down to 150°C. While passing nitrogen no ash should blow off from the quartz tube. The reacted sample is taken out from the furnace then weighted.

Formula used to calculate the reactivity of coal sample:

Reactivity= $11.61*W/ (5*C_{FIX}-W/2)$ c.c. /gm. Sec.

W= weight loss or reacted part of sample

C_{FIX}= fixed carbon content of the char.

4.6. Ash fusion temperature:

Ash fusion temperature is very important parameter in sponge iron plant. It gives an idea about the melting characteristics of coal ash. This ash fusion temperature test consists of four different characteristics i.e.:

1) Initial deformation temperature (IDT), initial shrinkage in shape.

2) Softening temperature (ST), rounding of corners of prepared sample was observed.

3) Hemispherical temperature (HT), change of cube into hemisphere.

4) Flow fluid temperature (FT), hemispherical form of sample flow as fluid.

Method of determination: [35]

A cube of 3 to 4 milligram of ash was prepared and prepared sample was heated in a special kind of furnace (i.e. known as heating microscope) at the heating rate was 10ºC per minute and maximum temperature up to 1600ºC. Initial shrinkage in the shape was observed at a particular temperature that temperature was noted down i.e. known as initial deformation temperature (IDT) heating was continued then rounding of corners of prepared sample was observed that temperature was noted down i.e. known as Softening temperature (ST) there after cubic sample was changed into hemisphere at a temperature, that was also noted down this temperature is known as hemispherical temperature (HT) finally hemispherical form of sample flow as fluid this temperature was also noted down this temperature is known as flow fluid temperature (FT)

4.7. Determination of Apparent density and apparent porosity: [36]

Apparent density and apparent porosity of coal sample were determined by using hot test boiling water method. In this method a coal sample of 15-20mm size was dried in an air oven at temperature of 105-110^{O}C and the weight of this dried sample was taken. This dried sample was suspended in a beaker containing hot boiling water with the help of thread. The sample was kept in the hot for around 15 to 20 minute. Now the suspended weight of sample and thread recovered in a chemical weighting balance. Sample was then removed from the thread and the weight of thread only while immersed in water was recorded. Finally the weight of water saturated sample was taken in air and the below mentioned formula was used to determine the apparent density and apparent porosity of coal sample.

FORMULA:

Apparent porosity= (W-D)/ {D-(S-s)}

Apparent density= D/ {D-(S-s)}

D= dried weight of coal sample;

W= weight of water saturated sample in air;

S= suspended weight of sample + thread while immersed in water;

s= suspended weight of thread only while immersed in water;

4.8. Caking index:[37]

Powder coal sample of -72 mesh size is usually mixed sand powder of same size in different ratio. The total weight of the sample was 25gm. Mixture was taken in a crucible and the crucible was placed in furnace maintained at a temperature range of 925-950°C. The sample was kept at this temperature for 7 min and then taken out from the furnace and cooled in air. The crucible kept inverted and 500gm. weight was kept on each of the cake form. Weight of the powder generated from each cake form was measured. The cake from which the weight of powder form was less than 1.25gm was usually consideration for caking index.

4.9. Carbonization of non-coking coal:

Carbonization is a heat treatment process of carbon containing material like coal. Carbonization can be used to increase the fixed carbon content, true density, mechanical strength, wear resistance and c-c bond strength. Improvement in these properties also depends on the different carbonization conditions.

Process:

Carbonization of weighted amount of separate air dried sample was carried out in furnace from room temperature to required carbonization temperature of 400^{O}C, 600^{O}C, 800^{O}C, 900^{O}C at the different heating rate about 10^{o}C and 20^{O}c per minute for different soaking time (1hr. and 2hr.) respectively. Coal sample was taken in a stainless steel box closed with a lid having an out let for the expulsion of volatile matter. Closed box was placed in a furnace and heated slowly from room temperature to predefine carbonization temperature of 400, 600, 800 and 900°C at the different heating rate of 20°C/min. and 10°C/min. Now the samples were removed from the furnace and their proximate analyses were calculated. Soaking time of the sample at these temperatures was 1 hour. The char samples were allowed to cool in the furnace itself. Obtained chars were weighted to determine the char yield and further determine their physical and chemical properties.

Chapter 05

5. <u>Result and Discussion</u>:

5.1 Characterization of coal sample:

Chemical and physical properties (proximate analysis, ultimate analysis, reactivity, caking index, ash fusion temperatures, calorific values, apparent porosity and apparent density) of different coal samples and their chars procured from different mines in India were carried out and data have been listed in Tables…. Proximate analysis results provide knowledge about fixed carbon content; volatile matter content, ash content and moisture content and the results obtained are listed in Tables... Fixed carbon content show the indication of availability of carbon required for iron ore reduction in sponge iron plant. Ash fusion temperature of procured coal is high which is good for sponge iron plant. Proximate analysis result of the different sample as shown in the Table 5 indicates fixed carbon content, volatile matter content, and ash content in the range of 33-54%, 23-36%, and 11-38% respectively. It was found that basundhara coal sample has highest fixed carbon content while Jharsuguda (OCP) belpahar mines coal has gross calorific value 8222.22kcal/kg, which is highest among all sample. The coal to be used in sponge iron making should have fixed carbon content more than 40% and volatile matter contents in the coal should be in the range of 27-30%. Basundhara coal sample and western coal field limited, Madhya Pradesh coal sample best suited for sponge iron plant because their carbon contents is 54% and 46% respectively and volatile matter contents 29% and 23% respectively in the range required for sponge iron plant.

Table 5.1: Experimental data of proximate ultimate analysis:

MINES NAME	PROXIMATE ANALYSIS				Ultimate analysis	
	Moisture content (%)	Volatile matter (%)	Ash content (%)	Fixed carbon content (%)	Total carbon content (%)	Hydrogen content (%)
SECL,KORBA(CG)	8	27	27	38	58.60	3.65
WCL,MP	2	29	23	46	61.50	4.12
MCL,BASUNDHARA ORISSA	6	29	11	54	67.40	4.65
JHARSUGUDA(BELPAHAR)	14	29	22	35	54.20	4.85
KALINGA	10	23	34	33	50.40	3.20
LINGARAJA	11	25	23	41	57.60	3.80

Above data shows that carbon content of Basundhara (MCL) mines coal has relatively high carbon content, low ash content among all four mines whereas Sarni (WCL) mines coal has low moisture content. So I can say that Basundhara coal is more suitable for industrial application.

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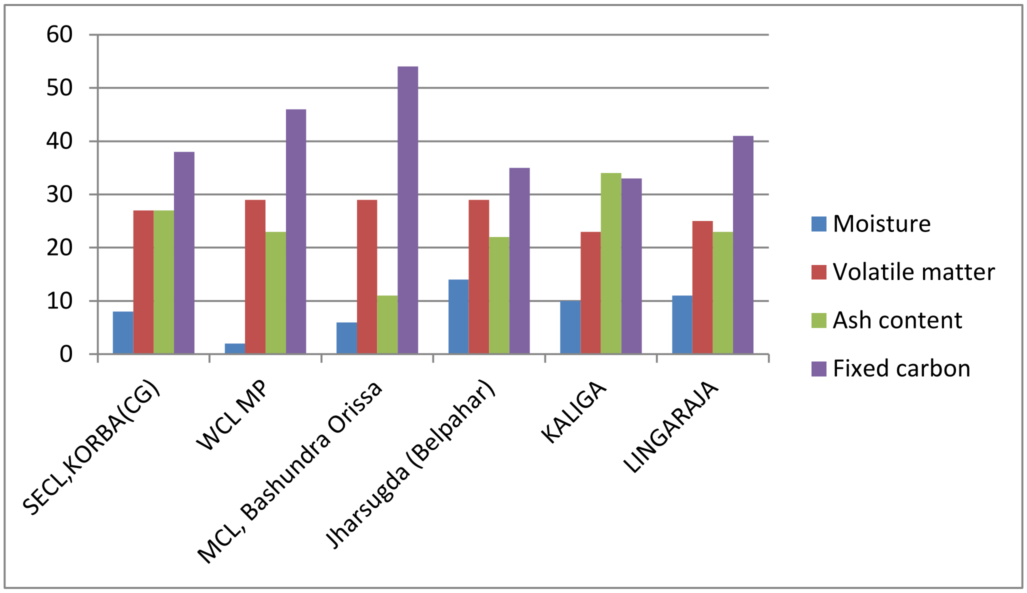


Fig5.1: Graphical Representation of Proximate Analysis of coal

5.2 Analysis of calorific value:

Calorific value of all coal samples were calculated and it was found that all the sample have calorific values in the range of 5610-8122.22 kcal/kg as shown in Table 5.2. Result shows that the calorific value of Jharsuguda (belpahar) mines coal has gross calorific value 8222.222 kcal/kg is highest among all sample, means it generate 8222.222 kcal amount of heat per kilogram mass of coal.

Table 5.2: Calorific value of several coal samples:

Mines name	Gross calorific values (kcal/kg)
SECL,KORBA (CG)	5760
WCL,MP	6318.987
MCL, BASUMDHARA	7776
BELPAHAR, JHARSUGUDA	8222.222
KALINGA	5610
LINGARAJA	6124

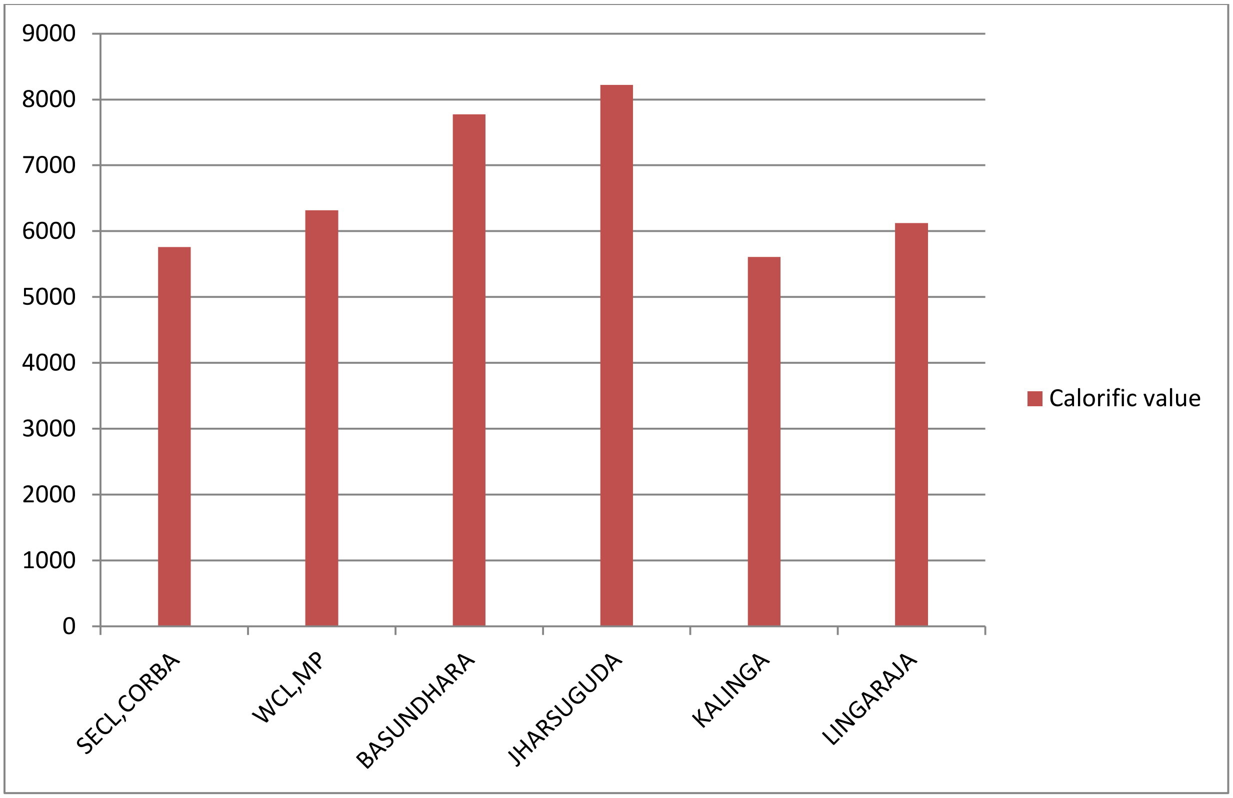


Fig 5.2: The graphical representation of above listed calorific values

5.3 ANALYSIS OF REACTIVITY OF DIFFERENT COAL SAMPLE:

$$C + O_2 = 2CO \text{ (endothermic reaction)}$$

It is actually the reactivity of coal char. During sponge iron making the non-coking coal carbonizes and form coal char. The reactivity of the non-coking coal for its utilization in sponge iron plant should be more than 2cc/gm./^{0}C. Higher the reactivity more is the cogeneration and better is the reduction of iron ore. As the reactivity of coal increases, the reduction temperature of the kiln decreases. It indicates that by the use of higher reactivate coal, one can reduce the operation temperature of the kiln. This may result in higher saving of energy and power. It was found that all the coal sample have reactivity more than 2cc/gm./^{0}C as shown in the Table 5.3 i.e. suitable for sponge iron plant.

Table 5.3: reactivity of several coal samples:

Sample source	Reactivity (c.c./gm./ Sec)
SECL,KORBA (CG)	3.65
WCL,MP	3.14
MCL, BASUMDHARA	3.85
BELPAHAR, JHARSUGUDA	2.75
KALINGA	5.65
LINGARAJA	3.25

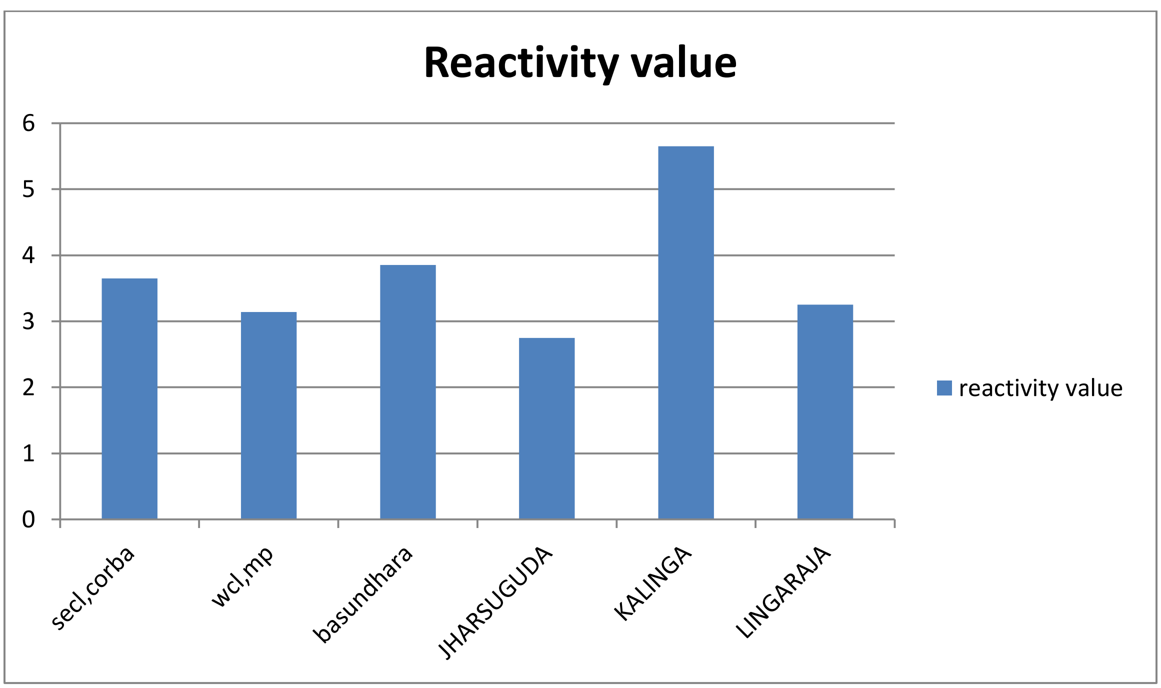


Fig.5.3: graphical representation of above listed reactivity

5.4 ANALYSES OF ASH FUSION TEMPERATURES RESULTS

Ash fusion temperatures (IDT, ST, HT, and FT) are used to measure coal ash fusibility and its agglomeration characteristics and provide information about ring formation in the sponge iron plant. Lower ash fusion temperature of coal char gives rise to ring formation in the sponge iron plant and jam formation in the boiler of the thermal power plant, reducing reactivity of char and forming agglomerated mass and reduces the effective kiln diameter. The decrease in kiln diameter reduces the rate of transport of material in sight kiln. The coal to be used in sponge iron making the initial deformation temperature (IDT) of the coal ash should be at least 150^{0}C more than the operative temperature of the kiln since the softening temperature of coal ash must exceed 1300°C. The most important factor affecting the ash fusion temperature is the chemical composition of ash (TIO_2, Al_2O_3, SIO_2, and Fe_2O_3 tend to increase the ash fusion temperature).

The ash fusion temperatures (IDT, ST, HT, and FT) results of coal ashes obtained from different coal mines are presented in Table 5.4. It is clear from this table that the IDT, ST, HT and FT values of basundhara coal ash are the highest. Other coal ashes {WCL (MP), belpahar, SECL (korba), kalinga and lingaraja} showed approximate the same values of IDT, ST, HT, and FT. The higher ash fusion temperatures are expected to be due to oxides like AL_2 O_3, SiO_2 and TiO_2. For the industrial uses, coals with higher ash fusion temperatures are preferred. Results shows that selected coal samples have sufficient high ash fusion temperature that is appears to be suitable in sponge iron plant.

Table 5.4: Experimental data of ash fusion temperature:

MINES NAME	ASH FUSION TEMPERATURE(^{O}C)			
	Initial deformation temperature(IDT)	Softening temperature (ST)	Hemispherical temperature (HT)	Flow temperature (FT)
SECL,KORBA(CG)	1085	1176	1432	1476
WCL,MP	1063	1192	1419	1492
MCL,BASUNDHARA ORISSA	1108	1206.6	1481.2	1510
JHARSUGUDA (BELPAHAR)	1098	1188	1430	1460
KALINGA	1020	1165	1449	1475
LINGARAJA	1035	1187	1410	1468

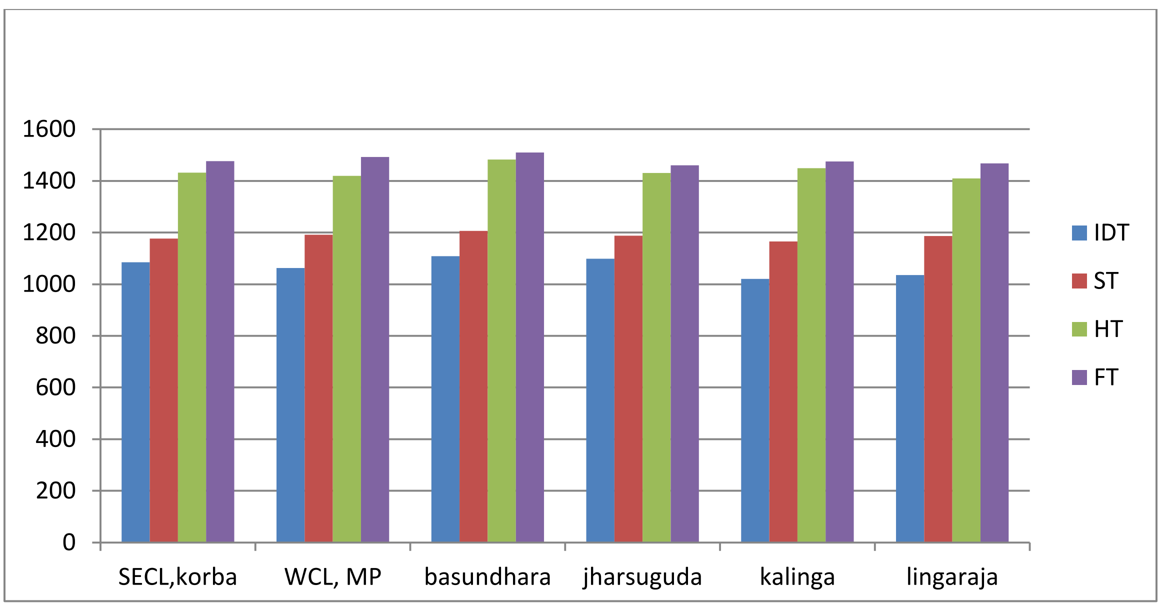


Fig. 5.4: Graphical representation of above calculated ash fusion temperature

5.5 APPARENT POROSITY AND APPARENT DENSITY:

Higher the porosity more will be the surface area of coal exposed towards oxidizing gases and higher will be the reactivity. It was found that apparent porosity of all coal samples in the range of 14.94-40.88% as shown in Table 5.5

Table 5.5: Experimental data of apparent porosity and apparent density:

Mines name	Apparent porosity (%)	Apparent density
SECL,KORBA(CG)	22.39	1.5
WCL,MP	14.94	1.46
MCL,BASUNDHARA ORISSA	40.88	1.484
JHARSUGUDA(BELPAHAR)	19.66	1.56
KALINGA	24.66	1.44
LINGARAJA	29.56	1.51

5.6 Result analysis caking characteristics:

Caking index gives the idea about sticking tendency of coal particles. Higher the caking index, more the sticking of coal sample between its particles.

- As result of caking we get lumpy mass
- The caking index of the coal is because of its vitrinite content.

Non coking coal have generally low caking index usually less than 3. [38] The result obtained established that maximum sample have nil caking characteristics except WCL, MP and MCL Basundhara coals as shown in Table5.6.

Table 5.6: caking indices of different coal samples:

Coal sample	Caking index
SECL,KORBA(CG)	NIL
WCL,MP	1.8
MCL,BASUNDHARA ORISSA	2.2
JHARSUGUDA(BELPAHAR)	NIL
KALINGA	NIL
LINGARAJA	NIL

5.7 Effect of carbonization temperature on chemical composition of different coal sample:

Carbonization means heating of carbonaceous (enriched in carbon) material in absence of air. Degree of carbonization increases with the increase of carbonization temperature. With increases of the carbonization temperature from 400°C to 900°C the chemical properties like fixed carbon content, ash contents increases while volatile matter decreases. The result shows that increasing of carbonization temperature from 400°C to 900°C increases the expulsion of gases from the coal sample therefore volatile matter content and hydrogen content decreases continuously. With decreases of hydrogen content the calorific value decreases. [39] The result of reactivity of coal chars at four different carbonization temperatures 400, 600, 800, 900°C shows that increased of carbonization temperature decreased the reactivity of char towards carbon dioxide (CO_2).

Results show that with increases in the carbonization temperature from 400-900°C expulsion of gases increases that leads to increases in fixed carbon content whereas decreases in volatile matter content as shown in the Table 5.7.

Table 5.7: proximate and ultimate analysis of chars obtained from different coal source listed below:

Coal source	Carbonization temperature (°C)	Carbonization time	Proximate analysis (wt. %)				Reactivity (cc/gm./sec)	Ultimate analysis (wt. %)	
			Moisture content	Volatile matter content	Ash content	Fixed carbon content		Total carbon content	Hydrogen content
SECL, Korba	400	1 hour	7	30	24	39	4.16	49.22	3.87
	600		8	8	27	57	4.08	59.22	1.45
	800		3	5	31	61	3.92	62.35	.20
	900		1	3	32	64	3.72	64.66	Nil
MCL Basundhara	400	1 hour	4	26	19	51	4.22	59.20	4.12
	600		3	13	26	58	4.12	60.22	1.62
	800		2	6	30	63	3.98	64.56	.36
	900		1	3	31	65	3.87	65.33	Nil
WCL, MP	400	1 hour	3	27	24	46	3.53	53.34	3.66
	600		2	21	27	50	3.41	54.67	1.22
	800		2	14	30	54	3.26	55.25	.20
	900		2	7	32	59	3.14	59.65	Nil
Belpahar	400	1 hour	12	26	24	38	3.12	45.46	2.88
	600		10	16	28	46	2.96	46.69	1.02
	800		7	11	32	50	2.85	51.25	.18
	900		2	5	34	59	2.77	61.20	Nil

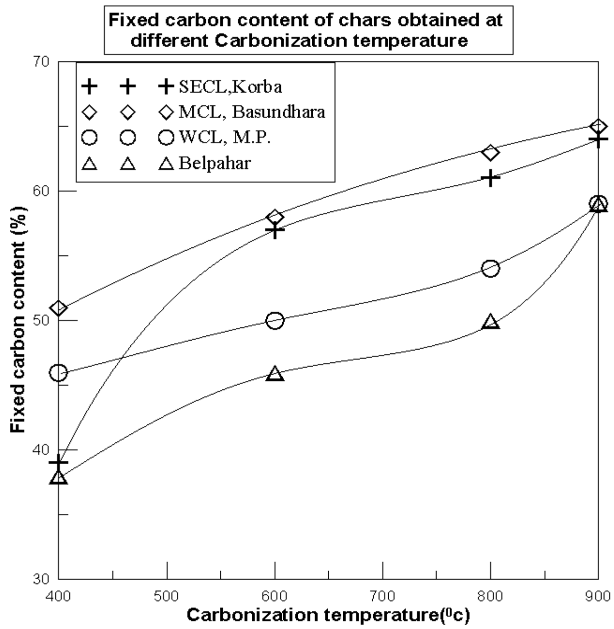


Fig. 5.8: Variation of fixed carbon content with carbonization temperature

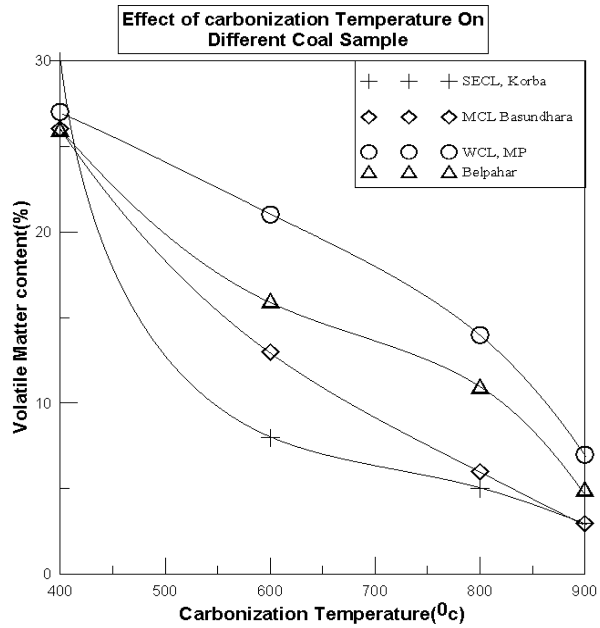


Fig. 5.9: Variation of volatile matter content with carbonization temperature

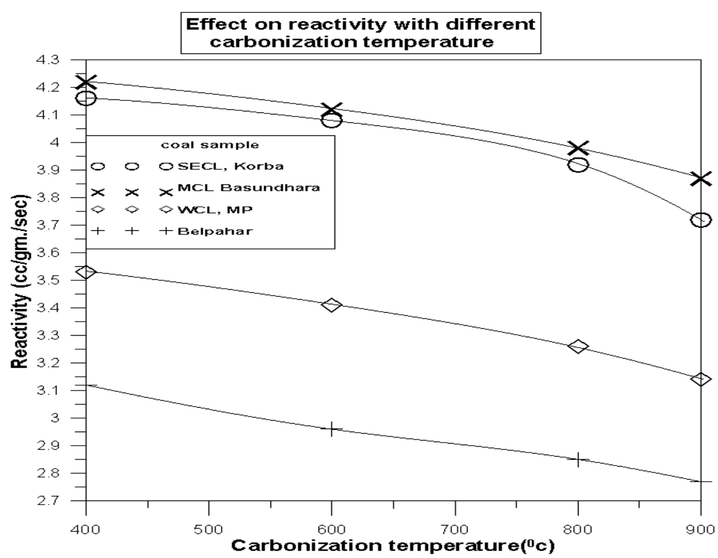


Fig 5.10: Variation of reactivity of different chars towards

CO at different carbonization temperature

5.8 Effect of carbonization temperature on physical properties of chars:

Result shows the variation of apparent density and porosity of coal char with increasing of carbonization temperature (400, 600,800and 900°C). Apparent density initially decreased with increasing carbonization temperature up to 400°C there after increases whereas apparent porosity initial increases with increases in carbonization temperature up to 400°C there after decreases slowly as shown in the following Tables. Increase in porosity up to 400°C shows increase in new pores through the evolution of volatile matter there after shrinkage of pores structure through structural rearrangement of the carbon matrix. [40]

Table 5.8.1: Effect of carbonization temperature on physical properties of SECL Korba coal sample

Carbonization temperature (°C)	Apparent density	Apparent Porosity (%)
-	1.5	22.39
400	1.12	31.65
600	1.36	30.5
800	1.48	29.7
900	1.6	27.56

Table 5.8.2: Effect of carbonization temperature on physical properties of WCL MP Coal sample

Carbonization temperature (°C)	Apparent density	Apparent Porosity (%)
-	1.46	14.94
400	1.35	24.6
600	1.4	23.5
800	1.45	22.5
900	1.50	21.7

Table 5.8.3: Effect of carbonization temperature on physical properties of Basundhara Coal sample

Carbonization temperature (°C)	Apparent density	Apparent Porosity (%)
-	1.484	40.88
400	1.32	52.6
600	1.39	51.75
800	1.46	50.15
900	1.5	48.20

Table 5.8.4: Effect of carbonization temperature on physical properties of Belpahar Coal sample

Carbonization temperature (°C)	Apparent density	Apparent Porosity (%)
-	1.56	19.66
400	1.40	29.7
600	1.48	28.54
800	1.54	27.65
900	1.62	26.42

Table 5.8.5: Effect of carbonization temperature on physical properties of kalinga Coal sample

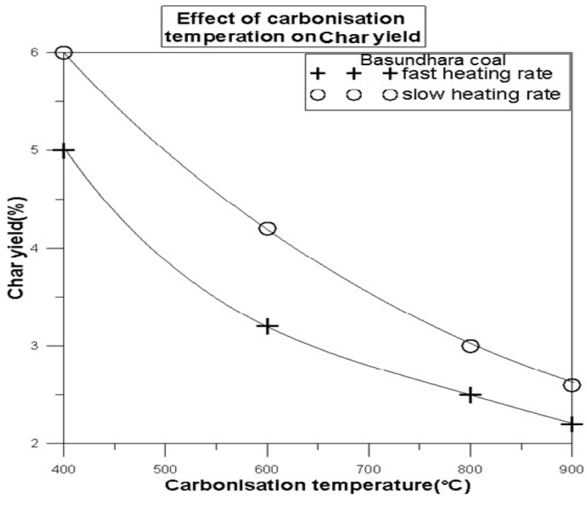
Carbonization temperature (°C)	Apparent density	Apparent Porosity (%)
-	1.44	24.66
400	1.29	31.5
600	1.32	30.54
800	1.39	29.7
900	1.45	28.56

Table 5.8.6: Effect of carbonization temperature on physical properties of Lingaraja Coal sample

Carbonization temperature (°C)	Apparent density	Apparent Porosity (%)
-	1.51	29.56
400	1.36	36.55
600	1.43	35.46
800	1.55	34.9
900	1.45	33.6

5.9 Effect of heating rate on coal sample:

Analysis of different heating rate during carbonization were carried out and it was investigated that Slow heating rate gives higher carbon yield than fast heating rate because of more paralytic carbon deposition during slow heating rate. Slow heating rate allow the volatile matter to remain in the coal matrix for longer period of time i.e. slow heating rate gives more opportunity to the volatile matter to undergo the process of cracking and deposition of more carbon. Fast heating rate expel the volatile matter quickly from the coal sample and thus deposit less carbon in the coal sample. Fast heating rate gives higher porosity because lower amount of paralytic carbon deposition in pores and some void and cracks are formed. Fast heating rate gives higher reactivity because higher porosity and more defects.



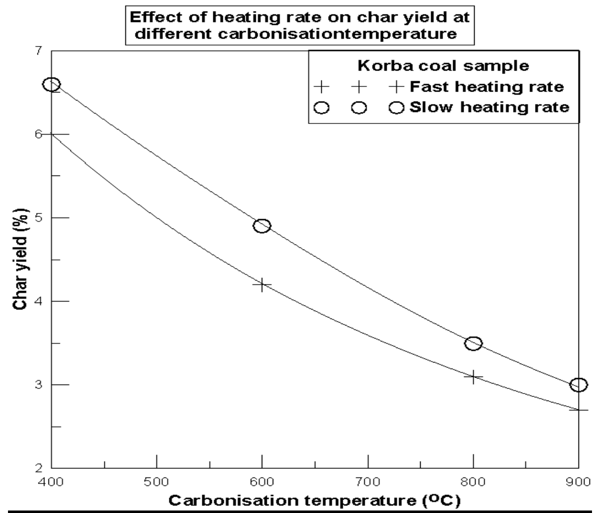


Fig.5.11: Variation of Chars yield of Basundhara coal sample Sample with carbonization temperature at different rate

Fig.5.12: Variation of Chars yield of Korba coal with carbonization temperature at different heating rate

Above figures show that with increases in the carbonization temperature from 400-900°C at different heating rate the char yield is decreases continuously. With increase in heating rate the chars yield is decreases because of different amount of gas expulsion

5.10 Effect of different soaking time:

Effects of different soaking time (1hr, 2hr) work were carried out on physical and chemical properties of coal. It was investigated that reactivity decreases marginally whereas gross calorific value, volatile matter content, fixed carbon content and ash content increases marginally with carbonization temperature at different soaking time as shown in the **Table** 18.

Table 5.10: Effect on the variation of proximate analysis, reactivity and GCV of southern coal field limited (SCL) Korba with carbonization temperature at different soaking time.

Carbonization temperature (°C)	Soaking time	Fixed carbon content (%)	Ash content (%)	Volatile matter (%)	reactivity (c.c./gm./ Sec)	Gross calorific value (kcal/kg)
400	1 hr.	39	24	30	3.54	5445
600		57	27	8	3.26	5585
800		61	31	5	2.87	5624
900		64	32	3	2.75	5667
400	2hr.	42	26	26	3.44	5452.22
600		59	29	8	3.1	5592.6
800		62	31	3	2.54	5638.66
900		64	33	2	2.3	5677.5

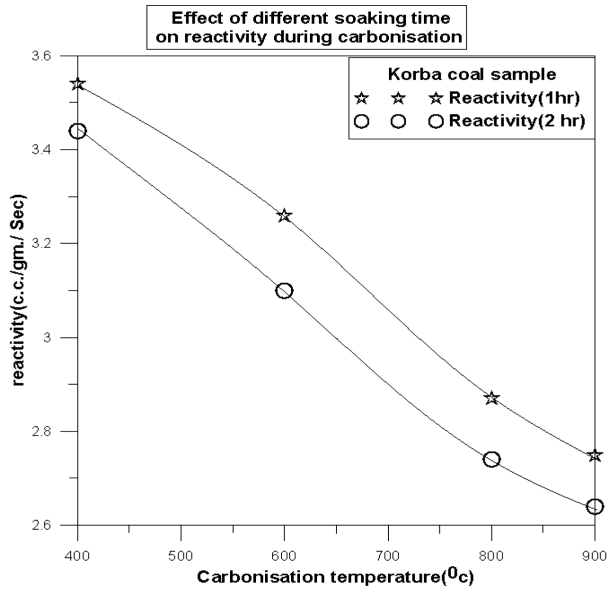


Fig 5.13: The variation of reactivity with carbonization temperature at different soaking time

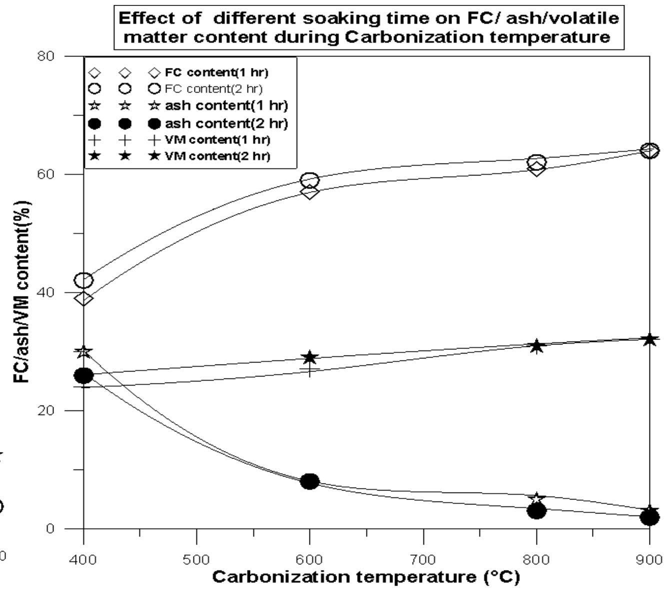


Fig 5.14: The variation of fixed carbon content, ash content and volatile matter content with carbonization temperature at different soaking time.

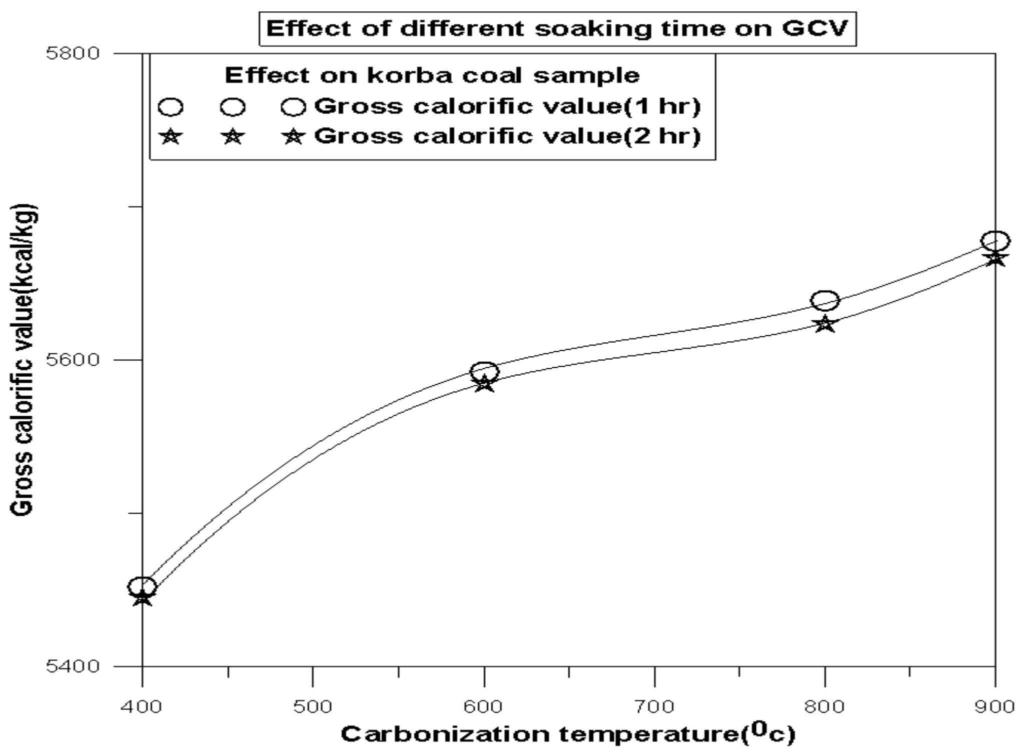


Fig 5.14: variation of gross calorific values with carbonization temperature at different Soaking time

5.11 XRD Analysis:

The X-ray diffraction (XRD) studies on different coal ashes (mesh size -200) were carried out on Xpert 3040Y00, Holland in the 2θ range of 20-80°. It was investigated that main compounds were found to be SiO_2, TiO_2, Al_2O_3 and MnO_2. These are the refractories oxide have high temperature resistivity properties that effect the ash fusion temperature in the coal ashes.

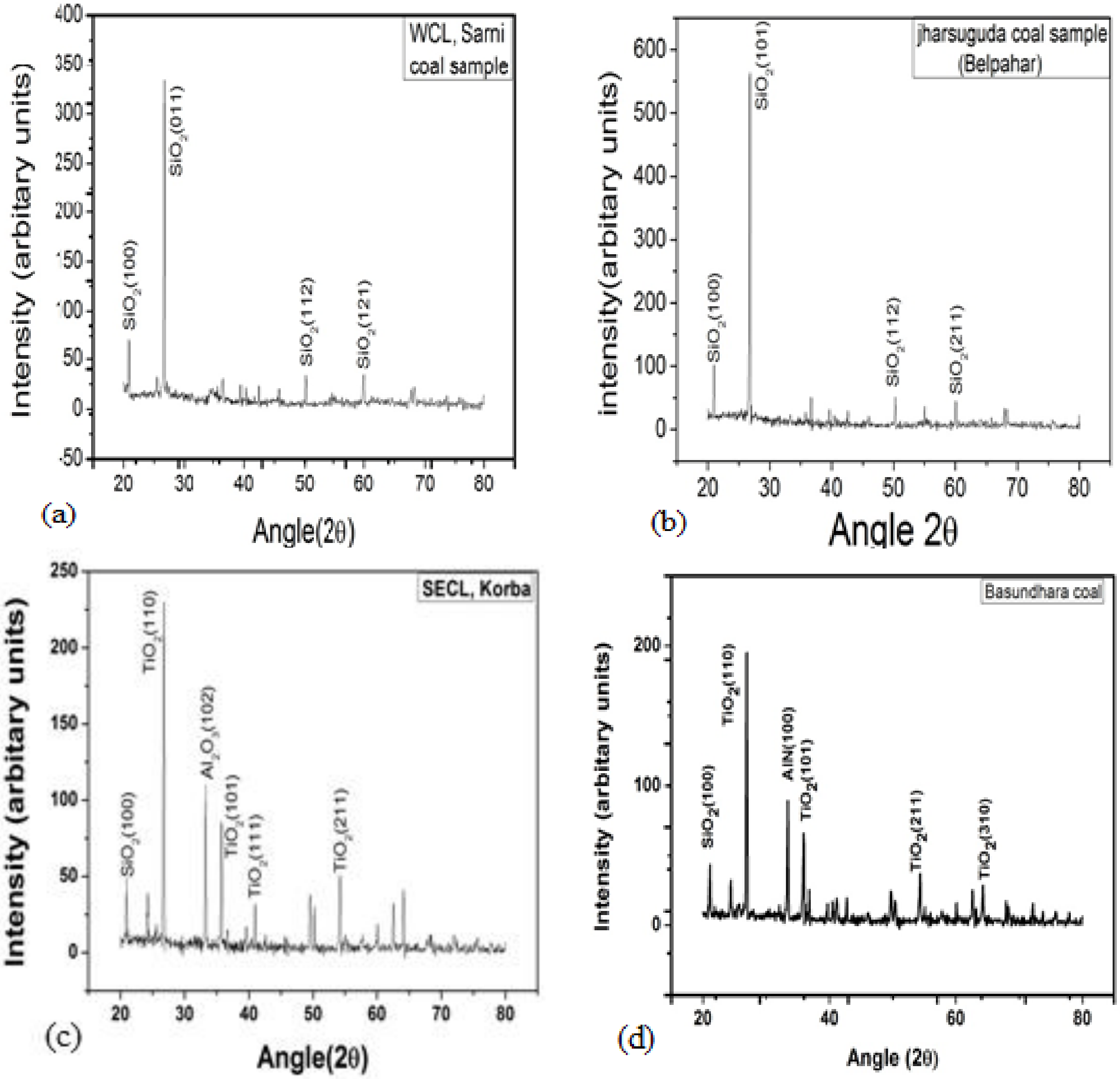


Fig.5.15 XRD analysis of coals (a) wcl sarni, (b) Jharsuguda, (c)SECL korba (d) Basundhara

Chapter 06

[Conclusion and scope for future work]

Conclusion:

Studies on the chemical and physical properties and their variation with different carbonization temperatures, heating rate and soaking time of different coal samples were carried out and following investigation were concluded.

1. Proximate analysis of different coal samples were carried out and it was concluded that western coal field limited Sarni (MP) and Basundhara coals were most suitable for sponge iron plant.

2. It was found that almost all the coals have no caking characteristics.

3. Almost all the coals were found to have high ash fusion temperatures that ensure there is no jam formation in the rotary kiln.

4. Reactivity of chars towards CO_2 was studied and exhibited suitable value for sponge iron plant.

5. With increase in carbonization temperature (400 to 900°C), the fixed carbon content, ash content and porosity increases.

6. With increase in carbonization temperature (400 to 900°C), the reactivity of coal char decreases.

7. The calorific value or heating value and hydrogen content decreases with increase in carbonization temperature (400 to 900°C).

8. Apparent density initially decreased with increasing carbonization temperature up to 400°C there after increases.

9. Effect of different heating rate during carbonization has been performed and result shows that char yield decreases with heating rate.

10. Effect of different heating rate has been carried out and it was investigated that there was no significant effect on the coal properties.

Scope for the future work

1) The studies carried out at present may be extended for other coals also.
2) Studies on other physical as well as chemical properties of coal are also suggested to be carried out in future.
3) Studies on reduction of iron ore by using these coals also need to be carried out in future.

Chapter 07

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