

A DISSERTATION

submitted in partial fulfilment of the requirements for the award of the degree

of

MASTER OF ENGINEERING

in

METALLURGICAL ENGINEERING (EXTRACTIVE METALLURGY)

By

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DEPARTMENT OF METALLURGICAL ENGINEERING UNIVERSITY OF ROORKEE ROORKEE August 1972

CERTIFICATE

Certified that the dissortation entitled "Kinetics of Pre-roduction of Iron Ore Briquettes Using Wood Charcoal Under Nitrogen Atmosphere", which is being submitted by Mr. Anil Kanta Tripathy in partial fulfilment for the award of Degree of Master of Engineering in Metallurgical Engineering (Extractive Metallurgy) of University of Roorkee, Roorkee is a record of his own work carried out by him under my supervision and guidance. The matter embodied in this dissertation has not been submitted for award of any degree or diploma.

This is further to certify that he has worked for a period of eight months from January 1972 to August 1972 for preparing this dissertation at this University.

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Roorkee .1972 Dated: August

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PREFACE

Since, blast furnace is the only versatile unit for production of pig iron, every effort has been made in recent years to increase its productivity and decrease the coke rate. It has been realised that by charging the prepared burden of proper size, particularly the pre-reduced pellets or briquettes, the coke rate can be decreased and the capacity of the furnace can be increased considerably. Many countries e.g. Japan, Canada, U.S.A. etc. have reported excellent results on increase in production and decrease in coke rate by charging pre-reduced materials in their blast furnaces. On average, it can be said that every 1% metallization of the burden decreases coke rate by 1/25. However, no such work has been reported under Indian conditions.

Therefore, the present investigation has been under taken with a view to study the influence of time, temperature, particle size of raw materials and Fe_2O_3/C ratios under Nitrogen atmosphere on rate of reduction of iron ore and to elucidate the exact mechanism of reduction of iron oxide.

This dissertation is divided in four Chapters:

In Chapter I, mechanism of pre-reduction of iron ore and factors affecting the rate of pre-reduction are described.

The Second Chapter deals with the methods of analysis of the raw materials, calibration of thermocouple and flowmeter, the method of briquette preparation and the experimental procedure used for carrying out the reduction with varying Fe_20_3/C ratios at different mesh size of rion ore and wood charcoal at $900^{\circ}C$, $1000^{\circ}C$, $1050^{\circ}C$ and $1100^{\circ}C$ in nitrogen atmosphere.

The results of the experiments conducted on reduction of -60 to + 85, -85 to + 100 and -100 to + 150 mesh size iron ore wood charcoal briquettes in nitrogen atmosphere at 1/2, 1/3 and $1/4 \text{ Fe}_2 0_3/C$ ratios for $900^{\circ}C$, $1000^{\circ}C$, $1050^{\circ}C$ and 1100°C during the time interval (15 minutes to 180 minutes) are elicited in Chapter-3. It has been found that reduction upto 90% proceeds through two stages, stage one 1.e. upto 30% reduction is controlled by chemical reaction and the activation energy for this stage is found to be 23-25 Kcal/gm. mole at all temperatures, particle sizes and Fe₂03/C ratios. Stage two 1.e. upto 90% reduction is found to proceed in accordance with semi logarithmic rate law i.e. $r = c \log t + x$. The activation energy for this stage has also been calculated and on average found to be 35-38 Kcal/gm. mole for all temperatures, particle sizes and Fe_2O_2/C ratios. Based on this, it is suggested that, diffusion of iron through FeO is a most probable rate determining step.

In Chapter Four, a short description of applications of pre-reduced materials in iron and steel making furnace is given. In the end suggestions for further work are also included.

ACKNOULEDGEMENTS

It is with doep sense of gratitude that the author wishes to thank Sri S.C. Koria, Locturer in Metallurgical Engineering Department, University of Roorkee, Roorkee, the guide for the project, who took keen interest in every style of the project.

The author expresses his profound gratitude and sincere thanks to Dr. M.N. Saxena, Professor & Head, Department of Metallurgical Engineering, University of Roorkee, Roorkee, for providing the necessary facilities in the department.

Finally, the author extends his thanks to the members of staff of the Department of Metallurgical Engineering, University of Roorkee, Roorkee, for their timely suggestions.

(A.K.TRIPATHY)

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CHAPTER-1

1.1. INTRODUCTION:

In recent years, a large number of significant developments⁽¹⁻⁵⁾ have taken place in the technology of iron making in the blast furnace, including oxygen enrichment and humidification of blast, higher blast temperature, self and superfluxed sintor and pellets, fuel injection through tuyeres, charging of prepared burden and employment of high top pressure. The technology of these developments have been fairly will established and the reported results⁽⁶⁺⁸⁾ on increase in production and decrease in coke rate have exceeded beyond prediction. However, the quest for increasing the productivity and decreasing coke rate has never lasted and this has given brith to a new and significant development " Pre-reduction ".

Pre-reduction is defined as beneficiation ⁽⁸⁻¹²⁾step in which oxygen is an impurity. Pre-reduction can be achieved by using low grade solid fuels either externally or internally to iron ore. The exploitation of this technology on commercial scale has taken place throughout the world in recent years and highly encouraging results on decrease in coke rate and increase in production have been reported. The Steel Company of Canada⁽¹⁾ has achieved 23% increase in productivity and 20% decrease in coke rate by charging pre-reduced pellets (88.7% Fe and 90.6% metallization) which comprises of 30% of the total charge. Hegachida plant, Yawatva⁽¹⁾ (Japan) reported 18.4% increase in production and 18% decrease in coke rate by charging sponge iron (69% Fe and 58% metallization) comprising of 40% of the total charge. Republic Steel Company of U.S.A.⁽⁸⁾ has reported 9% increase in production and 11.3% decrease in coke rate by charging metallized briquettes (71% Fe and 84% metallization) comprising of the 14% of the total charge. The theoretical calculations made by Jacques, Astier⁽²⁾, on the use of pre-reduced material to make liquid hot metal, reveal that 97% decrease in coke rate can be attained for each 10% metallization of the whole burden.

In view of the scarcity of high grade coking coal and limited capacities of Indian blast furnace use of pre-reduced burden is highly desirable.

1.2. MECHANISM OF THE PRE-REDUCTION OF IRON ORE:

In general two hypothesis^(13,15) have been put forward to account for the reduction of iron oxide which occurs when (13) a mixture of oxide and carbon is heated. In the first hypothesis the reduction reaction can be represented as follows :-

> $Fe_x O_y (S) + (OS) = Fe_x O_{y=1} (S) + CO(g)$ (1) Where x = 1,2,3 and y = 1,3 or 4

The reduction reaction is supposed to begin at the points of contact between carbon and iron oxide particles. Once the oxide particle is completely covered by a shell of reaction product, further reduction is possible only by the diffusion of reacting species to the oxide-metal interface coupled with a simultaneous diffusion of reaction products away from the interface. This hypothesis is vaild, provided carbonmonoxide

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produced during the reduction reaction is completely removed from the site of reaction to prevent it from taking part in reduction of iron oxide. It has been also beliefved (13,14) that due to the extremely low rate of diffusion of solid carbon compared to that of reaction between carbon and oxygen in the oxide, carbon diffusion is the governing factor (13,15) of all true direct reduction. This concept (14) however, has been changed recently by consideration of the diffusion of iron ion (15)into the oxide lattice at high temperature followed by interaction with carbon atoms. Thus while carbon diffusion occurs, the governing factor is to be iron diffusion.

The second hypothesis⁽¹³⁾ that has been considered may be represented as follows :-

> $F_{e_{\mathbf{x}}} \circ_{\mathbf{y}} (S) + Co (g) = F_{e_{\mathbf{x}}} \circ_{\mathbf{y-1}} (S) + Co_{2} (g)$ $Co_{2} (g) + C (S) = 2 CO (g) \qquad \dots (2)$

Where x and y take the same values as in (1)

According to this hypothesis, carbon monoxide is supposed to reduce iron oxide particles producing iron and carbon dioxide, one molecule of the latter gas reacts with one atom of carbon particles producing two molecules of carbon monoxide which in turn reduces more iron oxide. It is worthwhile that the total reaction is identical i.e.

 $Fe_x \circ_y (S) + C (S) = Fe_x \circ_{y+1} (S) + Co(g)$ in both hypothesis; therefore thermodynamic considerations can not assist in deciding which is taking place, as the total reaction is identical in both cases.

The view most widely held at present is that the two stage hypothesis (2) is the more probable (e.g. Esin and Gel'd) (13-15).

1.2.1. APPLICATION OF THE FIRST HYPOTHESIS:

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Tammann and Zroruikin⁽¹⁷⁾ and Baukloh^(16,18) et.al. supported the first hypothesis. Baukloh et.al, tried to establish a parallelism between the first products of solid-solid reduction and the dissociation pressure of the oxides. Although these authors claimed to have overcome the great difficulties associatel with thorough degassing of the reacting substances and the interaction of the products of reduction, some of their experimental results (13-16) do not substantiate this. The preliminary dissociation of the oxide to give oxygen, which then combined with the carbon, was proposed by Tammann and Zrorouikin⁽¹⁶⁾. The calculation of Gelid and Egin (13-15) of the dissociation pressure of FeO showed that the amount of dissociated Og available was insignificant compared with the actual amount of reduction occuring. So Baukloh and Durrer considered the diffusion of carbon and iron to be the rate governing step for the entire reaction.

It has been substantiated by several authors^(13,15) that endothermic reactions in the solid state follow the general diffusion laws that are applicable to crystalline materials. Hence the temperature at which a reaction begins is closely related to the thermal vibration of the atoms or molecules in the crystal lattices. A theoritical equation for the dependence of inter solid reactions on time, particle size, and temperature

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was developed by Jander⁽¹⁴⁻¹⁹⁾ by applying the diffusion laws -

$$\left[1-\left(1-\frac{R}{100}\right)^{1/3}\right]^{2} = 2 DC_{0} t/r^{2} = R t$$

Where

 $R_1 = Reduction degree (percentage)$

t = time

D = Diffusion constant

 C_0 = Initial concentration of diffusion element

r = Mean radius of element to be diffused.

K = Constant

If Jander's equation is valid, a plot of $\left[1-\left(1-\frac{1}{13}\right)^{1/3}\right]^2$ versus t must have a constant slope and so also if the rate of carbon diffusion controls the overall reaction velocity. According to Jander, an endothermic reaction must follow the above equation.

On the contrary, if the exothermic reaction between CO and Fe_2O_3 occurs, the linear relation between the two terms will not be established. Sambong: (13-15) conducted the reduction experiment in Nitrogen with the mixtures of 1 gm of ferric oxide with 0.9 gm of graphite and showed that reduction was comparatively fast in the initial period and then slowed down, indicating CO participation in the reduction.

Recently, the diffusion of iron $atoms^{(15)}$ instead of carbon has received considerable attention in connection with the reduction of iron oxide. It has been stated that diffusion of iron ions within the oxide crystal is the rate determining process in the reduction of certain oxides. According to Lu, et.al^(13,15), the rate of iron oxide reduction is not diffusion controlled, but probably controlled by a surface reaction at the oxide iron interface. This statement was based on the fact that the rate of reduction is nearly constant at constant composition of the gaseous phase in the course of the reduction of Wustite to metallic iron.

Mckewan⁽¹⁵⁾ also presented a rate equation on the assumption that iron ore in the process of reduction is a simple two phase system, oxide-metal and that rate of reaction is controlled at the metal oxide interface.

1.2.2. APPLICATION OF SECOND HYPOTHESIS:

The mechanism of the 2nd hypohtesis⁽¹³⁾ is a type of chain reaction. A molecule of CO is formed in the bed, this reduces the iron oxide to give CO_2 , which then react with coal to produce two molecules of carbon monoxide, which are available for further reduction. In the reduction of hematite(Fe₂O₃) to iron by Carbon monoxide, the slowest stop is the reduction of Wustite (FeO) to Fe by carbon monoxide.

As postulated by Whalafalla and Co-Workers (13,17), it is supposed that carbon monoxide is first adsorbed on the oxide lattice according to reaction (3).

 $CO(g) \xrightarrow{K_1} CO (adsorbed on oxide)(3)$ This reaction is followed by the reaction (4) through the reaction (5)

> $CO(ad) + CO(g) \xrightarrow{K_2} CO_2(ad) + C$ (4) $CO_2(ad) \xrightarrow{K_3} CO_2(g)$ (5)

Reaction (4) involves the interaction of a gaseous carbon monooxide molecule, CO(g), with an adsorbed molecule, CO(ad), and was shown to be the slowest and rate determining steps for the mechanism of reduction of Wustite by carbon monoxide. The products of this reaction are an adsorbed CO_2 molecule and a free carbon atom. Reaction (6) involving the interaction of a free carbon atom with a ferrous oxide molecule producing adsorbed carbon monoxide.

> C + FeO (S) $\xrightarrow{K_{1}}$ CO (ad) + Fe (6) CO (ad) $\xrightarrow{K_{2}}$ CO(g)(7) CO₂ (ad) + Fe(S) $\xrightarrow{K_{6}}$ FeO (S) + CO(ad)(8)

In the second stage of the reduction process, it is postulated that the carbon dioxide is first adsorbed on carbon particle according to reaction (9) and is followed by reactions (10) and (11).

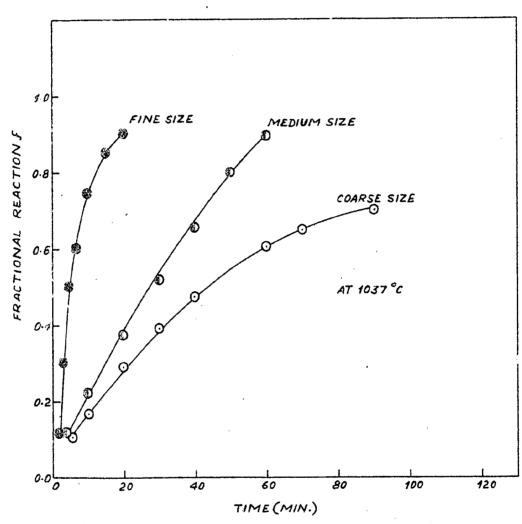
If gas-solid reactions were the only ones occurring in the heated bed of ore and coke, a sufficiently rapid flow of inert gas would diminish⁽¹⁶⁾ their rate almost to zero, as the gases would be swept out of the bed before having time to react with solid material.

The solid material may contain appreciable amounts of adsorbed oxygen, which will tend to desorb at high temperatures

and could then provide the necessary carbon monoxide to initiate the reaction. The products of desorption were removed in the very rapid nitrogen stream (30 c.c./sec.).

Out of two types of reactions, the solution loss reaction is almost stopped by a sufficiently rapid flow of gas through the bed. The reaction between one and coal can proceed only at the points of contact, so that once iron is produced at these positions the reaction will slow down considerably. The change in the rate of reaction with amount of reduction for an infinite flow is very much less than that with a very small flow of inert gas through the bed.

The rapid rate of reduction with a low flow of gas through the bed can be explained in the following way (14). The reduction of Fe₂03 to Fe₃04 is practically irreversible at high temperatures and the values of CO/CO2 ratio at equilibrium are about zero. Hence when CO is passed slowly over heated FegO3, practically every CO molecule can react with the oxide to give CO2. A large fraction of this CO2 can react with coal to give CO, since the concentration of CO will be low, and hence the reduction of the iron oxide will be very rapid. As soon as FegQ, is produed on the surface and reduction proceeds to FeO, the equilibrium is less favourable to gaseous reduction and CO generation. So the amount of reduction occuring as a result of the direct interaction of oxide and coal can be ignored. From this it follows that the amount of reduction occuring depends on two factors (14,16). the ease of gaseous reduction of the oxide and (11) the (i) ease of solution of the coal by CO2.





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1.3. FACTORS AFFECTING PRE-REDUCTION OF IRON ORE BRIQUETTES:

The following factors are affecting the pre-reduction of iron ore briquettes:

1.3.1. Effect of the type of reductant used (16)

If the low grade coke is used as reductant, then it has got less effectiveness in bringing about the reduction of iron oxide, than lignite coke and mood charcoal. This is because of the difference in reactivity of carbon of wood charcoal, lignitic low grade coke.

1.3.2. Effect of Temperatures:

Yun⁽¹⁵⁾ studied on " direct reduction of ferric oxide by solid carbon in vacuum" and he found that at higher temperatures the degree of reduction will be higher and at lower temperatures such as 700°C and 800°C, the degree of reduction was found to be extremely small. The investigation on kinetics of iron ore reduction by carbon was carried out above 947°C in Nitrogen atmosphere, it was found that the degree of reduction was higher.

1.3.3. Effect of Particle Size :

It has been reported (13,16) that for a constant hematite particle size and a Fe₂O₃/C ratio e.g. 1/3, the observed time for half complete reduction with coarse, medium and fine size of carbon particles at 1037°C are illustrated in fig. 1.3.3.

1.3.4. Effect of Hemattite/carbon ratio:

The reduction rates of iron oxide are also affected by the amount of carbon used for carrying out the reduction.

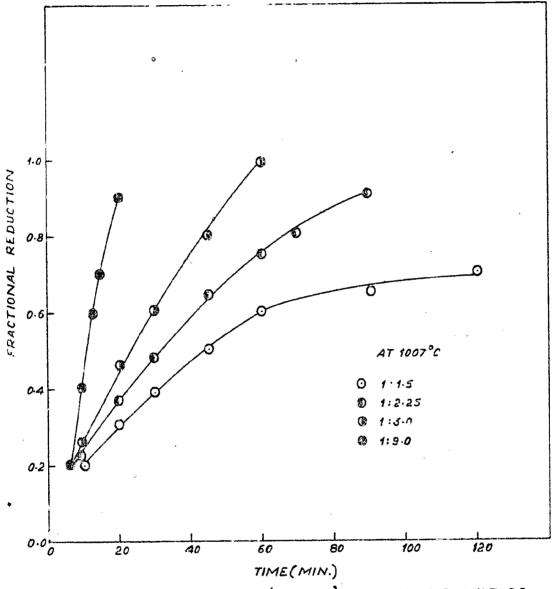


FIG.1.3.4 THE EFFECT OF THE 'FOR 03/C' RATIO OF THE MIXTURE ON THE REDUCTION OF HEMATITE.

By using carbon more than stoichiometric requirement the (20)reduction rate increases, Studied carried out Ghosh and Tiwari with different percentages of lignite coke as reductant in the range from 15 to 35% was illustrated in fig. 1.3.4. and suggested that the higher amount of carbon increases the reduction rate. Similar results have also been reported by Rao^(13,16) where the Fe₂O₃/C ratio was varied between 1/1.5 and 1/9.

1.3.5. Effect of Mitrogen gas flow:

Baldwin⁽¹⁴⁾ carried out his experiments on reduction of iron oxide with solid coke under different nitrogen gas flows. With increasing gas flow rate, the degree of reduction will be higher and also given that with further increasing the gas flow rate, it will not affect the rate of the reduction.

CHAPTER-2

EXPERIMENTAL WORK

This Chapter describes the methods of analysis of the raw materials, calibration of thermocouple and flowmeter, the method of briquette preparation and the experimental procedure used for carrying out reduction with varying Fe_2O_3/C ratio at different mesh sizes of iron ore and wood charceal.

2.1. <u>RAW MATERIALS</u>: The raw materials used for the present study were iron ore, obtained from Gua Mines and wood charcoal, used as reductant procured from local markets of Roorkee.

2.1.1. Iron Ore: Iron ore fines were subjected to sieve analysis, chemical analysis and ignition hoss determination. 2.1.1.1. <u>Sieve Analysis</u>: The crushed iron ore fines were sieved in Denvar sieve set (sieves arranged according to Taylor's series) and the fines in the size range -60 to +85, -85 to + 100 and - 100 to + 150 mesh were collected and stored in bottles to study the effect of particle size on the rate of reduction reaction.

2.1.1.2. <u>Chemical Analysis</u>: The mhemical analysis of iron ore fines was done by titrating a reduced solution of the iron ore in hydrachloric acid againsta standard solution N/10 of potassium dichymate.

The chemical analysis of the iron ore fines used in this investigation was 56% Fe and 20% gaunge. 2.1.1.3. Loss on Ignition : Loss on ignition of iron ore was determined at 900°C, 1000°C, 1050°C and 1100°C by heating one gram sample at the respective temperatures. From the weight loss measurements, percentage loss on ignition was calculated and shown in table 1.

2.1.2. <u>HOOD CHARCOAL</u>: The wood charcoal fines were analysed for sieve analysis, proximate analysis (before and after volatile matter removal).

2.1.2.1. <u>Sieve Analysis</u> : The sieve analysis of wood charcoal fines were performed in a similar way as described in 2.1.1.1. 2.1.2.2. <u>Proximate Analysis</u> : The proximate analysis of wood charcoal as received from market was done to determine the amounts of moisture, volatile matter, ash and fixed carbon and the results are reported in table 2.

From the present study, the volatile matter of wood charcoal was removed. It was done by heating wood charcoal to 900[°]C for a length of an hour in an inert atmosphere. The proximate analysis of this treated wood charcoal is shown in table 2.

2.3. <u>Preparation of briquettes</u>: The iron ore and wood charcoal powders of same particle size were seperately weighed and throughly mixed in a mixer for about 8 hours, to ensure proper mixing. The proportion of Fe_20_3 of iron ore to carbon of wood charcoal was varied from mixture to mixture at constant particle size. In this way, the mixtures $Fe_20_3 + 2$ C, $Fe_20_3 + 3$ C and $Fe_20_3 + 4$ C were prepared by taking one gm. mole of Fe_20_3 and 2.3 and 4 gm atom of carbon respectively at -60 to + 85, - 85 to + 1000 and

-100 to + 150 mesh sizes. To all these mixtures, 5% water was added to promote effective binding during briquetting.

From the above mixture, a sample of 10 gm was taken and compacted on universal testing machine using a die of 1.9 cm dia with the help of a plunger at 420 kg/cm² pressure. The length of the briquettes usually varied in between 1.5 cm to 1.9 cm.

The briquettes thus prepared were first air dried and then dried in an oven for two to three days at 100-150⁰C till the constant weight attained.

2.3. <u>CALIBRATION</u>: The thermocouple and the flowmeter were calibrated before theywere used in the experiment.

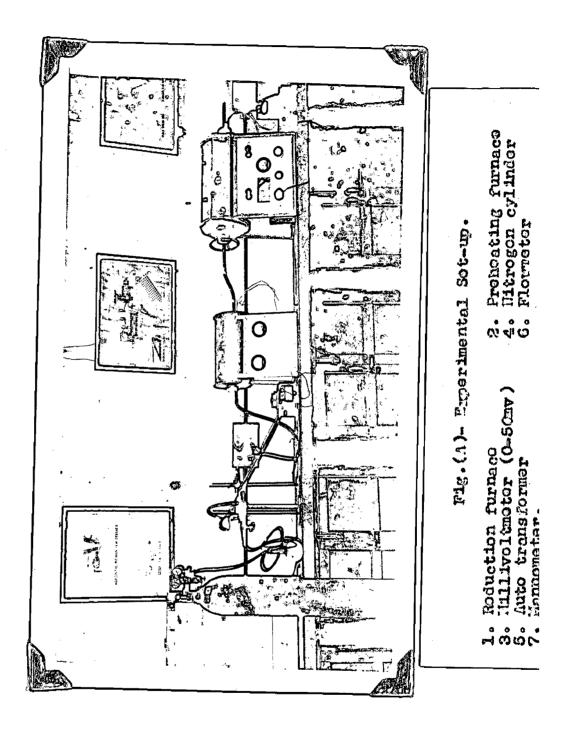
2.3.1. <u>Thermocouple</u>: Chromel Alumel (chromel about 90% Ni and 10% Cr and Alumel about 94% Ni, 2% Al, 3% Nn and 1% Si) thermocouple was calibrated against a standard thermocouple using temperature recorder and milli woltmeter. The calibration graph is shown in the figure no.1.

2.3.2. Flowmeter: The flow meter was calibrated against the standard device (orificemeter). The calibration graph is shown in the figure no.2.

2.4. <u>EXPERIMENTAL PROCEDURE</u>: Reduction experiments were performed in both ends open Kanthal wire wound allumina tube furnace of 7.5 cms diameter, 75 cms long.

One end of the furnace was closed by a steel flange that had in let for preheated oxygen free nitrogen gas. A constant flow of 1.8 liters/min. was maintained throughout the experiment. This high rate of flow is maintained to avoid the effect of

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flow rate on the percentage reduction of iron ore⁽¹⁴⁾. The other end of the furnace was closed by a flange with the provision for insertion of thermocouple and outlet for gases. Using a calibrated chromel and Alumel thermocouple the temperature profile along the length of the furnace was dotermined. It was found to be uniform ($\pm 5^{\circ}$ C) over a length of 10 cms as shown in the experimental set up figure (A).

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The briquette made out of compacting the desired proportion of iron ore and wood charcoal fines followed by removal of moisturewas weighed in a chemical balance. Three briquettes at a time of different mesh sizes were kept in a stainless steel boat and the boat was slowly introduced into the uniform temperature zone of the furnace through the opening end after flushing with the preheated (500-600°C) oxygen free nitrogen gas. The opening end of the furnace was closed by a steel flange. Usually 3 to 5 minutes elapsed to attain the uniform temperature of the briquette. After the reduction was carried out under isothermal condition for a predetermined length of time. the boat was slowly withdrawn from the temperature zone to the cooling end of the furnace and the briquettes were cooled to room temperature in a stream of nitrogen gas. At the end of cooling, briquettes were taken out and weighed to ascortain weight loss occurred during experiment. Briquettes were crushed and analysed for residual carbon content by combustion +absorption method.

In this way, the experiments were conducted at 900°C, 1000°C, 1050°C and 1100°C for different intervals of time

(ranging from 15 to 180 minutes) by varying Fe_2O_3/C ratios, 1:2, 1:3 and 1:4 for particle sizes, - 60 to + 85, - 85 to 100 and - 100 to + 150 mesh to study the influence of time, temperature, Fe_2O_3/C ratio and particle size on the rate of reduction.

CHAPTER-3

RESULTS AND DISCUSSIONS

3.1. EFFECT OF TEMPERATURES :

The percent iron oxide reacted in the iron ore wood charcoal briquettes of -60 to + 85, -85 to + 100 and - 100 to + 150 mesh sizes at 1/2, 1/3 and 1/4 Fe₂O₃/C ratio in the temperatures 900°C, 1000°C, 1050°C and 1100°C for different intervals of time (15 minutes to 180 minutes) under mitrogen atmosphere are given in tables 3.1 to 3.3., 4.1 to 4.3, 5.1 to 5.3 and plotted in figures 3.1.1. to 3.1.3., 3.2.1. to 3.2.3. and 3.3.1. to 3.3.3. From the figures the pronounced effect of temperature particularly in the initial stages (upto 30% reduction) is quite discernible. For example, percent reduction of iron oxide varies from 20% at 1000°C to 50% at 1100°C in 15 minutes according to the reaction $Fe_2O_3 + 3C$ at -60 to + 85 mesh size. Similar observations could also be made from other figures (3.1.1. to 3.1.3, and 3.3.1. to 3.3.3.).

After 30% reduction the percent iron oxide reacted was found to obey the following linear relationship.

 $r = C \log t + \pi$ (1)

where C and x are constant, t is time of reduction and r is the per cent reduction. This relationship has been obtained from the figures 4.1.1. to 4.1.3, 4.2.1. to 4.2.3. and 4.3.1. to 4.3.3. In plotting per cent reduction vs long (time), initial log (time) values upto 30% reduction has not been taken into account since they are found to be highly temperature sensitive. The values of constant c at all temperatures, mesh sizes and Fe_2O_3/C ratios were found to be nearly constant and reported in table-6. Equation (1) was found to valid at all temperatures studied. In this range, the activation energy values for reduction process have been calculated in a similar way as calculated by Jena and et al. cited⁽²¹⁾ by Koria et.al. in his dissertation in their experiments on sintering of uranium dioxide. By differentiating equation (1) with respect to time t we get,

$$dr/dt = C/t$$
.
or $dr/dt \propto 1/t$

.....(2)

Now according to Arrehenius relationship $K = A e^{-Q/RT}$ and equation (2), the reciprocal of time for attaining a definite reduction should be proportional to $e^{-Q/RT}$ at all temperatures, where Q is activation energy, R is gas constant and T is absolute temperature. The log (1/t) is plotted against 1/T $x10^{40}K$ for the reduction reactions $Fe_2O_3 + 2C$, $Fe_2O_3 + 3C$ and $Fe_2O_3^{44}C$ at different particle sizes =60 to +85, -85 to + 100 and =100 to + 150 mesh sizes and are shown in figures 5.1.1. to 5.1.3. 5.2.1. to 5.2.3 and 5.3.1. to 5.3.3. From the elopes of these plots, activation energy values have been calculated and are reported in table 7.

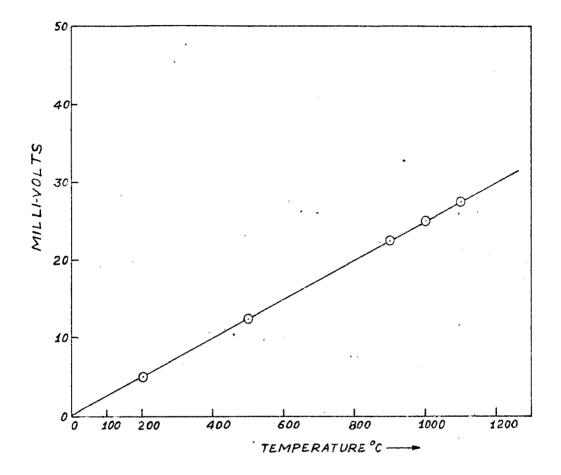
From the results reported above, it is found that upto 30% reduction the rates of reactions $Fe_2O_3 + 2C$, Fe_2O_3+3C and $Fe_2O_3 + 4C$ are controlled by chemical reaction

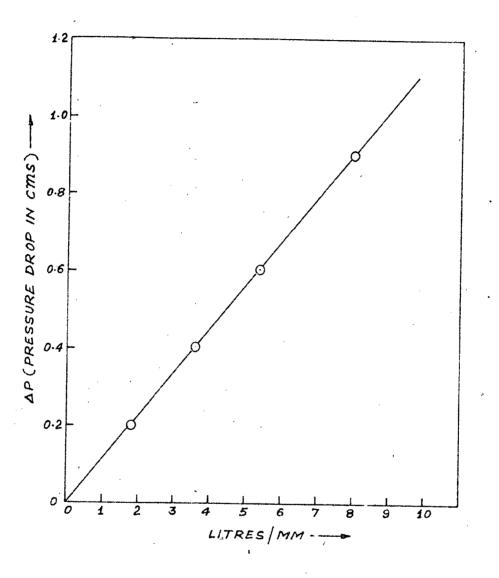
and the activation energy values are found to be 23 to 25 Kcal/gm. mole and a shown in table 7. After 30% and upto 90% reduction the reaction was found to be diffusion controlled. From the activation energy values obtained (35 to 38 Kcal/gm. mole) in the present investigation, it is considered that diffusion of Fe through FeO is the most probable rate determining step since this value is in closed agreement with the reported ⁽¹⁸⁾ value (35 Kcal/gm. mole) for diffusion of Fe through FeO. Further it is also found that particle size of reactants have no effect on activation energy which suggests that in all the cases diffusion of Fe through FeO is the most probable rate determining step.

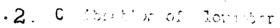
3.2. EFFECT OF Fe202/C RATIO

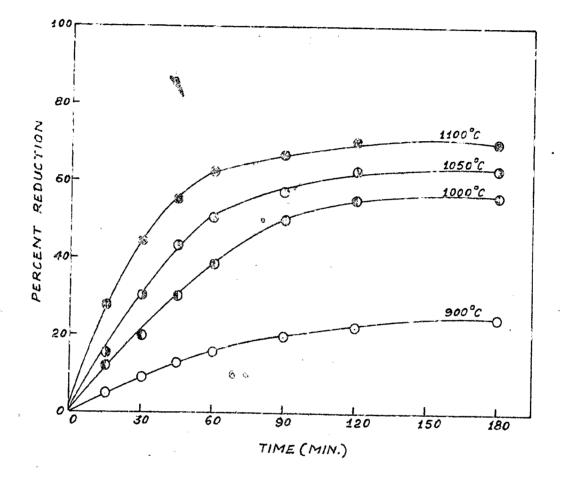
The values of percent reduction of -60 to + 85 mesh size iron ore wood charcoal briquettes for different interval of time at 1000° C are plotted at different Fe₂O₃/C ratio i.e. 1/2, 1/3 and 1/4 and are shown in figure 6. From the plot, it is quite conspicuous that as the ratio of Fe₂O₃/C decreases from 1/2 to 1/4 the rate of reduction considerably increases. In other words it can also be said that reduction proceed at much higher rate in the mixture of proportionately higher carbon. 3.3. EFFECT OF PARTICLE SIZE:

The values of percent reduction at different Fe_2O_3/C ratio i.e. 1/2, 1/3 and 1/4 for different intervals of time at $1000^{\circ}C$ of -60 to + 85, -85 to + 100 and -100 to + 150 mesh sizes iron ore, wood charcoal briquettes are shown in figure 7.1, 7.2 and 7.3. From the plot, it is observed that for -100 to + 150 mesh size particles at different Fe_2O_3/C ratio have higher rate of reduction.



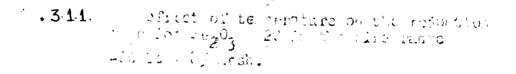


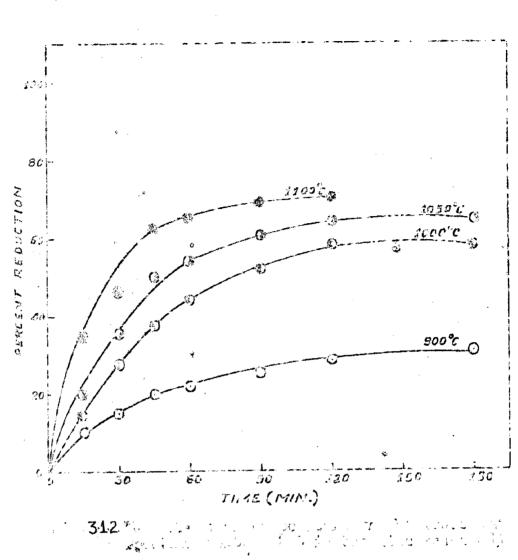




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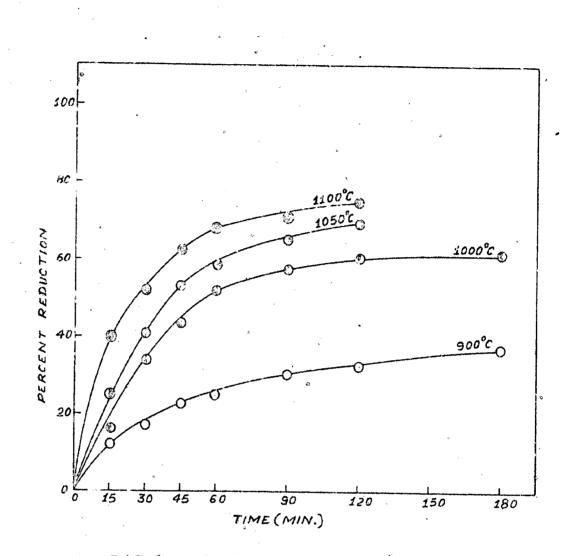


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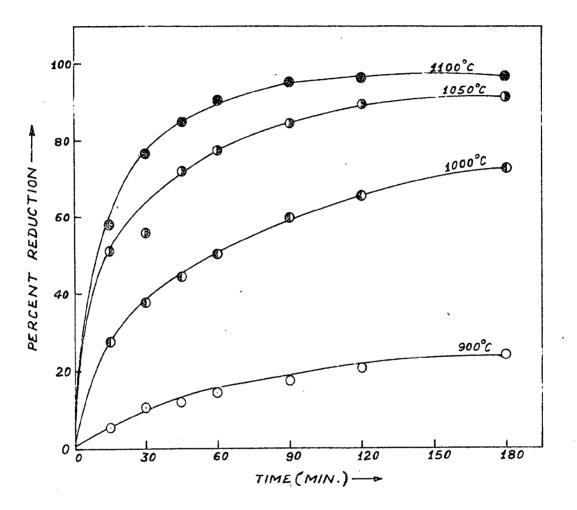
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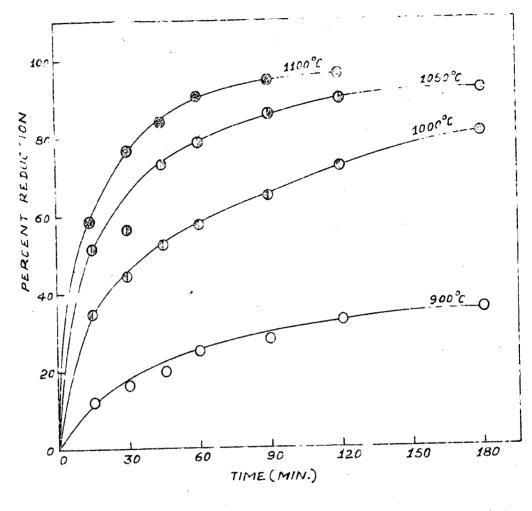
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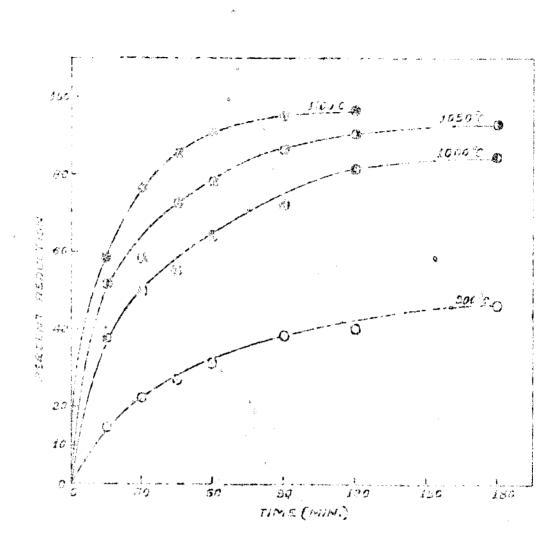


.i. 3.2.1. I c effect of telporature on the reduction reaction (e.G. - 3.6 in the size range -60 to - Efficient."



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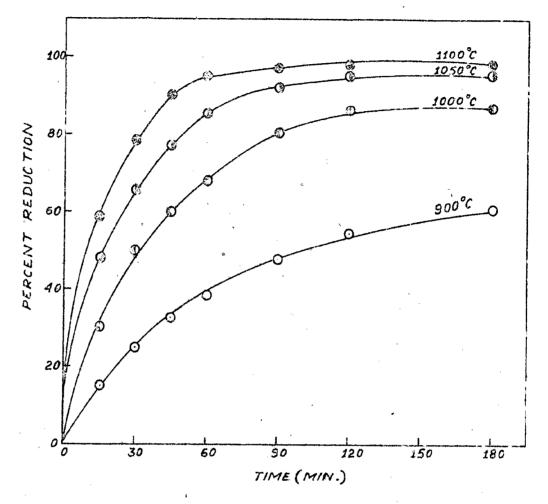


Fig 334 The effect of temperature on the relevance process is received at 40 in the function of the second of the function 100 for the function

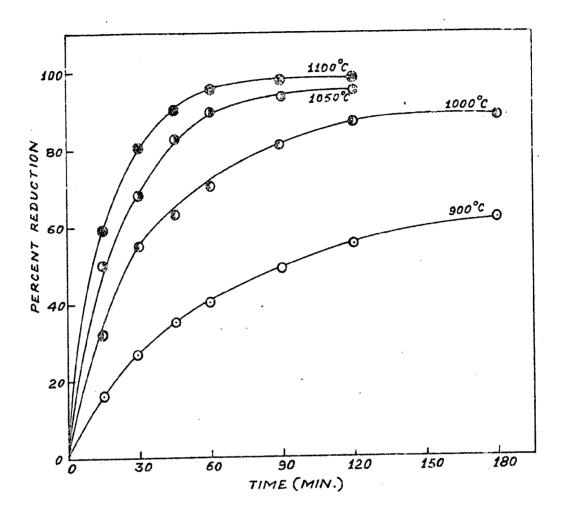
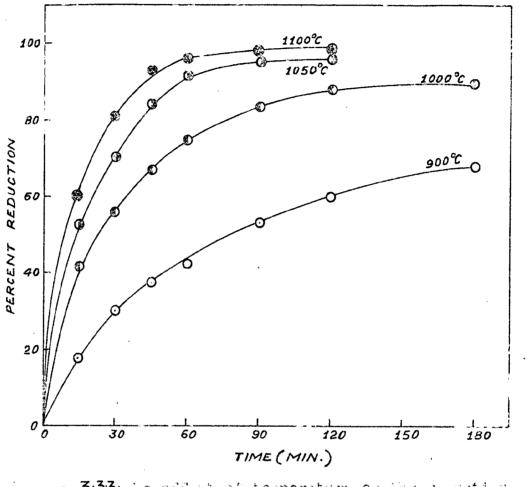
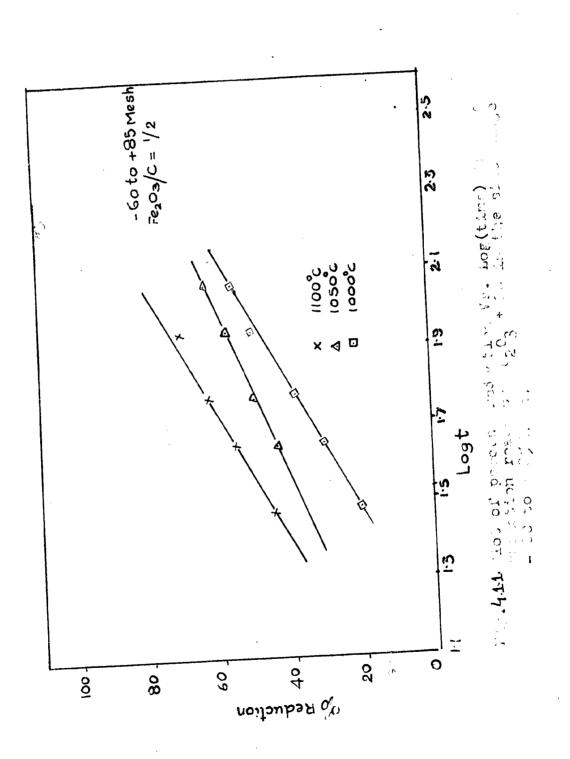


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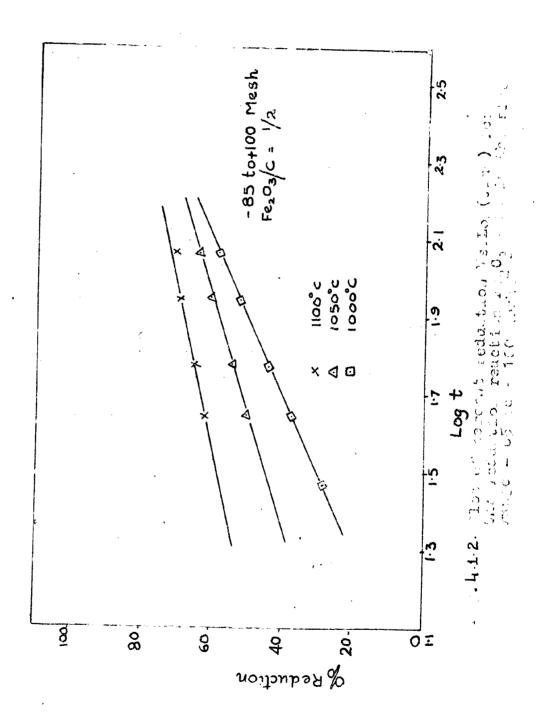
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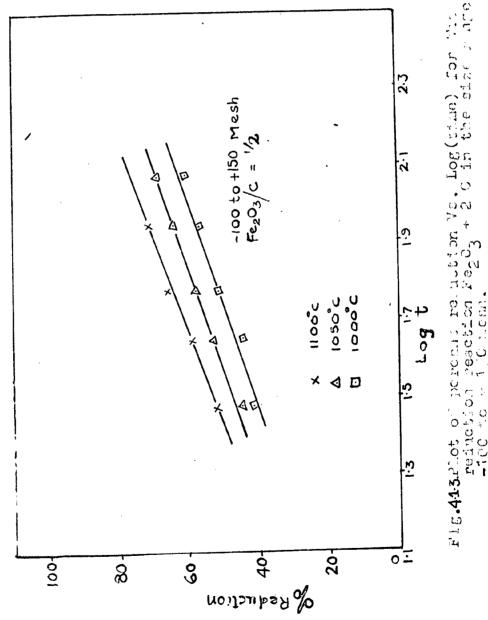
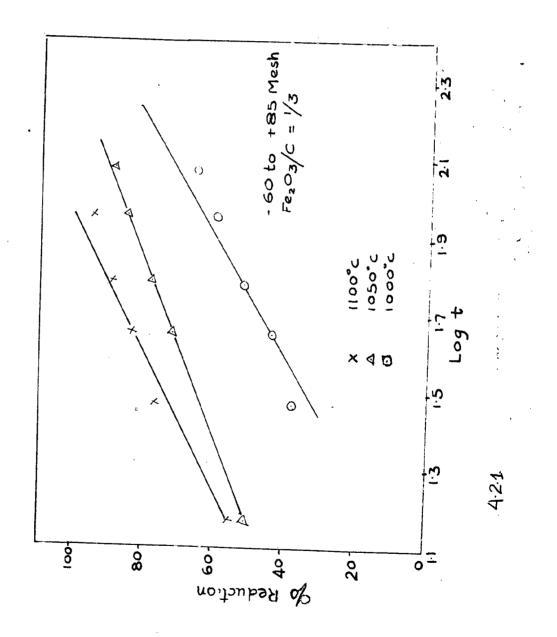
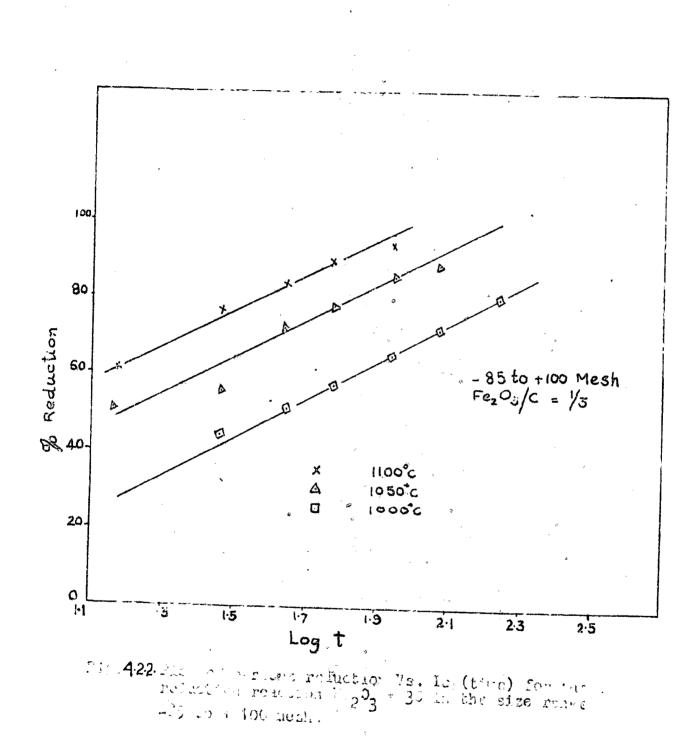
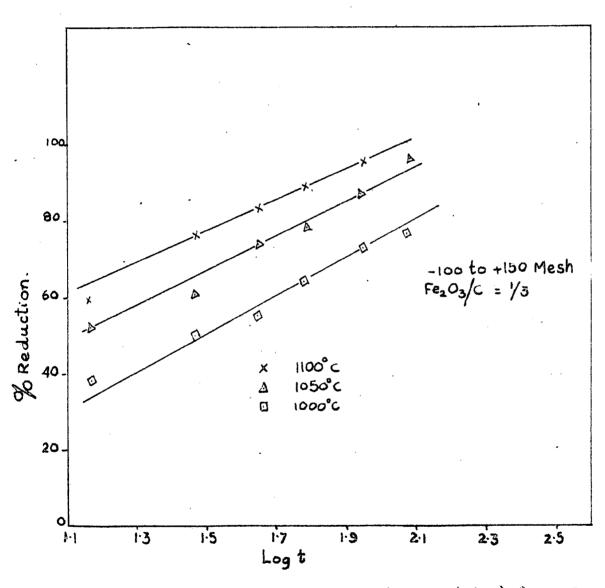


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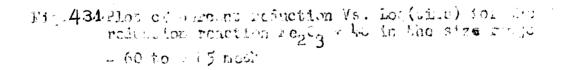


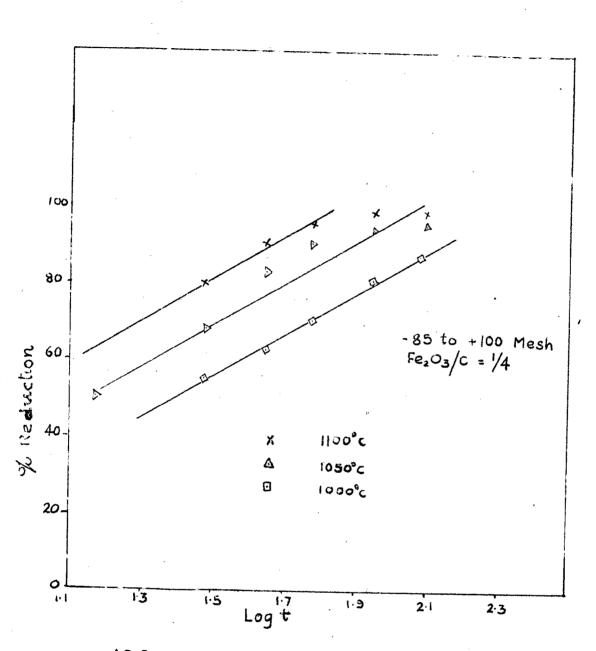


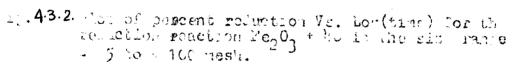
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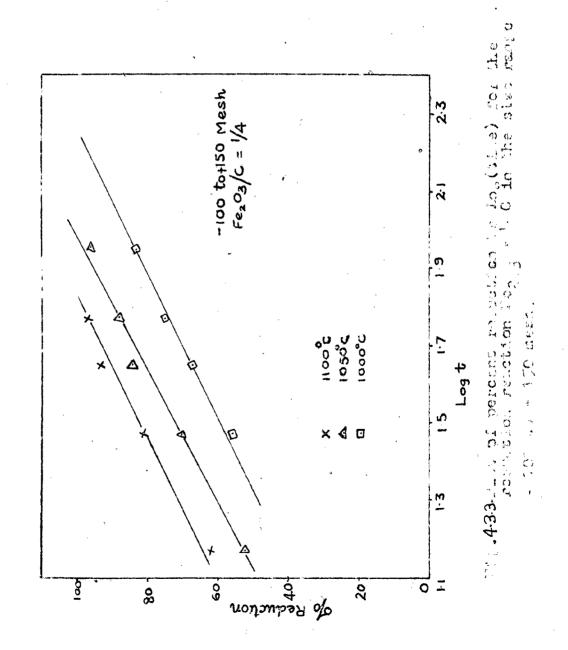
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100 Ø 80. -60 to + 85 Mesh. $Fe_2 O_5/C = 1/4$ Reduction. 100°C × ∆ 1000°C ٥ 20 0 2.3 17 L.cg.t 1.9 13 2.1 1.5 1-1

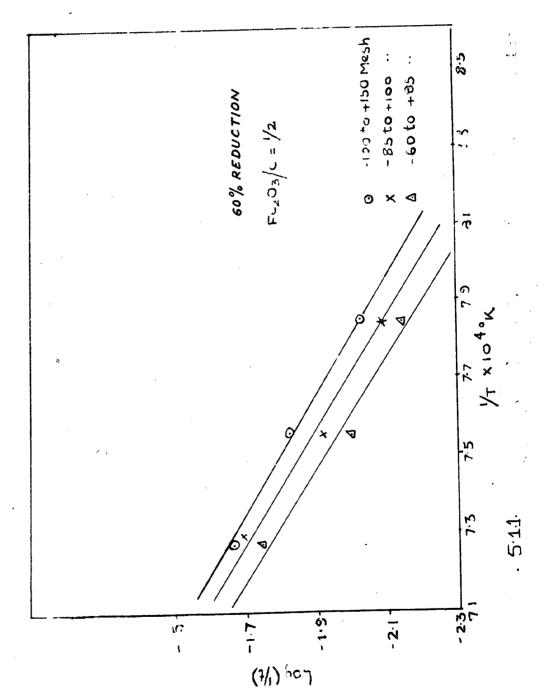




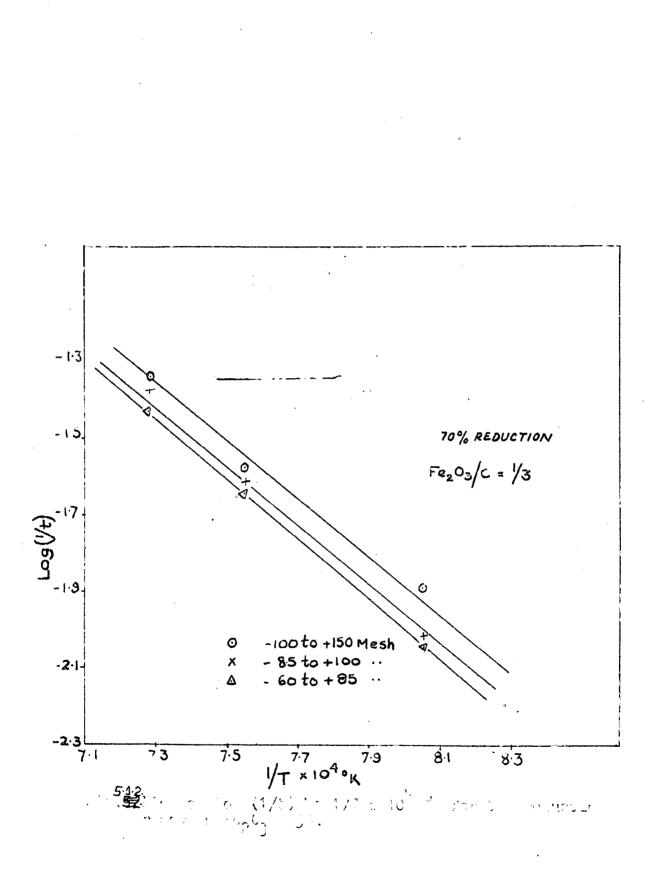


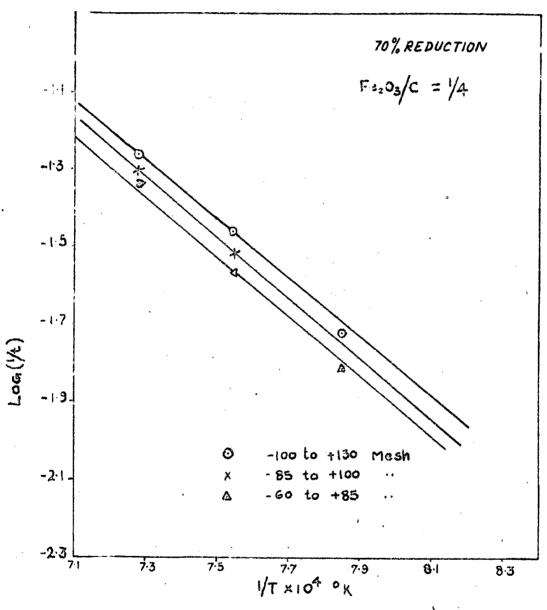


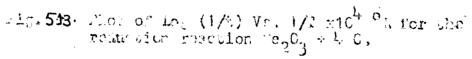
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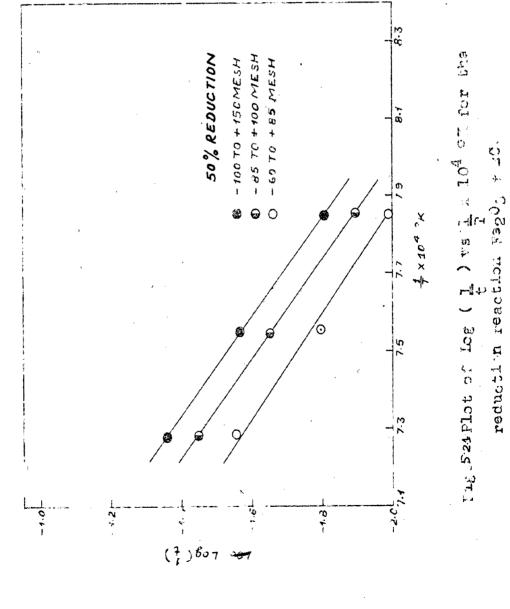


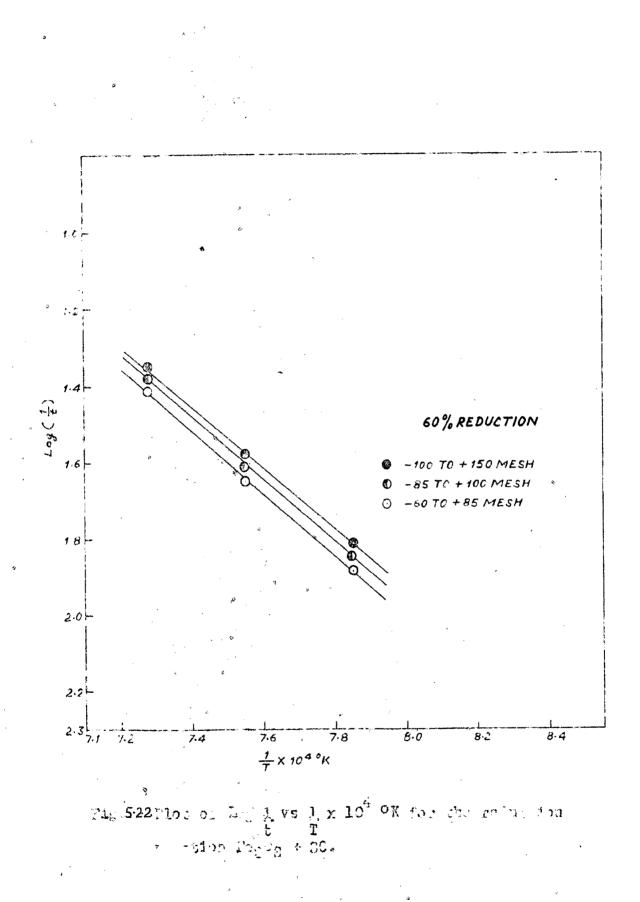
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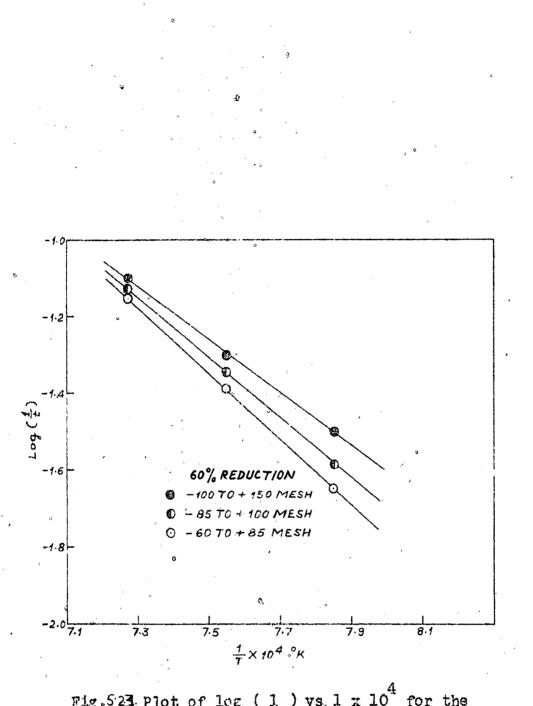


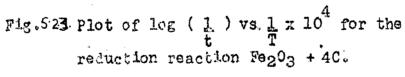


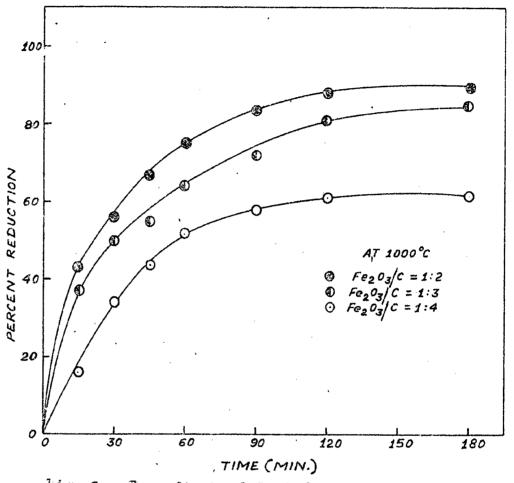






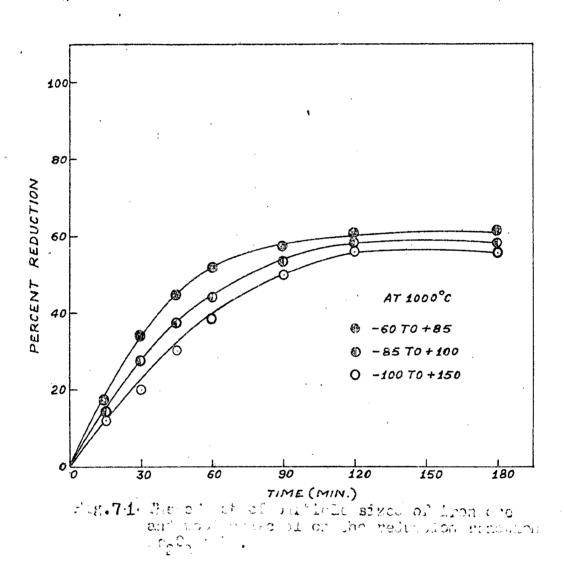


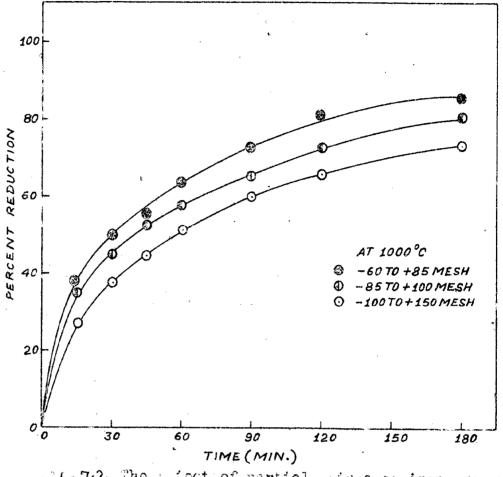




Vic. 6. In flect of Ze_{20}/C ratio of the mixture on reduction of 2^{-100} to + 150 mesh size into one priducties.

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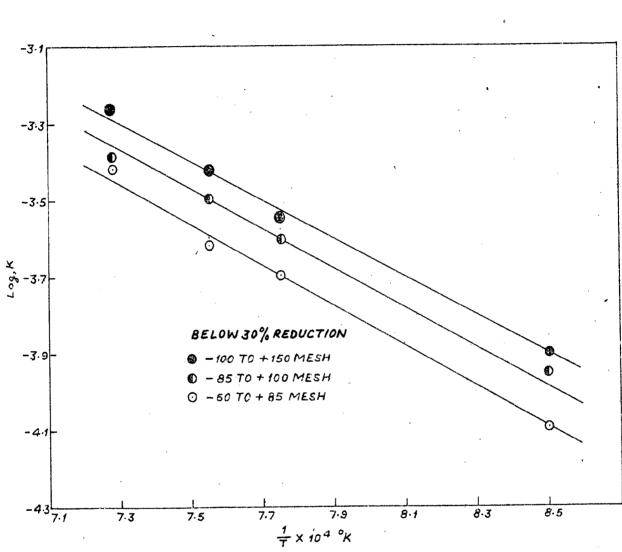
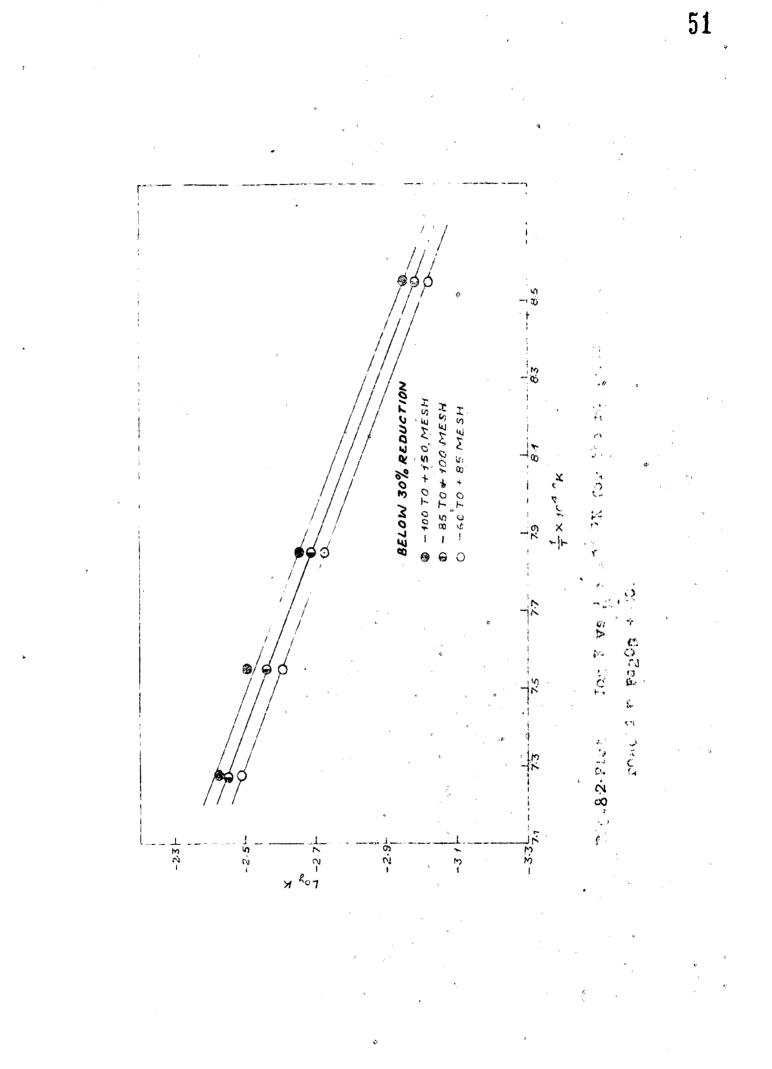
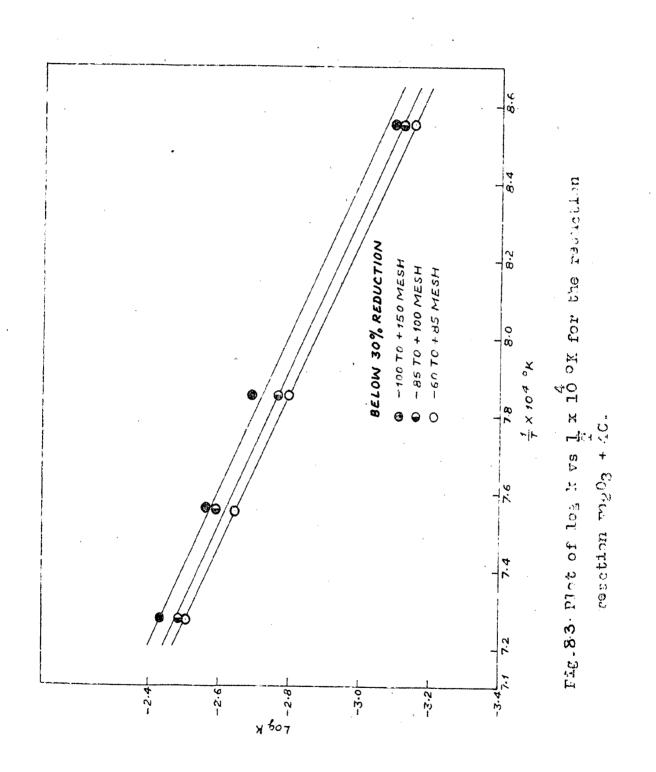


Fig. 8.1 Plot of Log K vs 1×10^4 oK for the reduction reaction Fe₂0₃ + 2C.





CHAPTER_4

APPLICATIONS OF PRE_REDUCED AGGLOMERATES IN PIG IRCH AND STREL MAKING PROCESSES

The applications can be discussed as follows :

4.1. IN PIG IRON MAKING PROCESSES:

4.1.1. <u>Blast Furnace</u>: Blast furnace by virtue of its excellent productivity and economics occupies unique position in the field of iron making. However, its supermecy has been threatened by the growing shortage and increased prices of metallurgical grade coking coal. Under such conditions, the benefits that could be gained from reducing coke consumption are of significant importance to iron makers. Pre-reduced iron ore with high degree of metallisation 60-80% offers such advantages.

The pre-reduced materials to be suitable as blast furnance feed should possess the following physical and chemical properties such as :

- Sufficient physical strength to withstand the mechanical handling and load when charged in the blast furnance.
 Bandataran be montifation accient strengtheric of wards
- Resistance to reoxidation against atmospheric oxygen and humidity.
- 3. Free from slag forming constituents (S102, Al203 etc.) as they increase slag volume and lime consumption.
- 4. Free from alkali elements such as Zn, Pb as they accelerate wear of the lining.
- 5. Amount of residual oxygen should be as low as possible or degree of reduction should be as high as 80 to 90%.

It has been realised by many countries ⁽²³⁻³⁰⁾ that charging of pre-reduced burden in the blast furnace decreases coke rate and increases productivity. On average, a decrease in coke rate of 6% may be expected from 10% increase in burden metallisation.

Against these advantages must be set the cost of making metallised pellets. Economic consideration suggest that advantages are unlikely to be sufficient unless there are special local reasons for using metallised pellets in the blast furnace.

Nevertheless, increasing use of pre-reduced materials in the blast furnace is advocated and anticipated to be around 20 million tons⁽¹¹⁾ by the end of 1980 and 300 million tons⁽¹⁰⁾ in the year 2000.

4.1.2. Electric Reduction Process:

Processing of pre-reduced iron ores in electric pig gram furnaces may offer greater advantages (22) as compared to blast furnace. This is due to the direct reduction between ore and solid carbon which is predominent in electric pig iron furnaces. The direct reduction is highly endothermic and electrical energy has to be supplied to meet this. When prereduced ore is used in place of the normal burden, the electrical energy requirements can be greatly reduced. The decrease in electrical energy requirements coupled with building of large transformers lead to possible building of electric smelting units in the range of 1200 to 2000 metric ton⁽²⁾ of hot metal per day. The availability of electrical energy at cheaper rates will decide the future erection of these furnaces. However, it has been anticipated that around 400 million tons of pre-reduced iron ore will be processed in electric pig iron furnaces in the year 2000.

4.2. IN STEEL MAKING PROCESSES :

4.2.1. L.D. Converter :

Metallized agglomerates can be used as a coolant material or as a substitute for scrap in L.D. converter. Very little work has been reported in the literature concerning the use of metallised materials in L.D. converter. However, it has been established that the semi continuous feeding of metallised ore of high iron content of L.D. converter during the fourth and eighth minutes of the blowing period produces very satisfactory results.

In those countries where there is shortage of scrap and high cost of importing it, metallised materials could reduce the scrap scarcity and provide economic source for steel production.

4.2.2. Electric Steel making :

Metallised agglomerates have been tested in electric arc furnaces with very encouraging results. A substantial reduction in power consumption and heat timinings have been reported by using metallised pellets in place of scrap.

The technique of charging from the roof of the electric furnace is a very efficient method of utilizing sponge iron. Besides, other advantages are;

- 1. Materials handling can be simplified.
- 2. Energy costs will be decreased because of the improved electrical characteristics of the arc furnace.
- 3. Economical furnace operation can be aided by such factors as improved refractory life, decreased electrode breakage and decreased labour costs.
- 4. Because of the continuous nature of operation, it appears that the process could be automated.

The development work on continuous hot charging will lead to continuous production of steel by using electrically controlled dosing equipment and electric moving fields channels. The final refining in electric moving field channels will create extremely favourable conditions for the operation of continuous casting plants. The future of sponge iron usage, in electric furnances, therefore, would, appear to be most promising. About 500 million tons of pre-reduced iron ore is anticipated to be consumed in ultra high powered electric furnaces from 1980 to the end of this century.

4.2.3. Continuous Steelmaking :

If pre-reduced agglomerates with more than 90% metallisation of constant physical and chemical quality be made available then, these can be used effectively in the continuous steelmaking processes. In BISRA " Spray refining" and IRSID processes the continuous feeding of pre-reduced material in the powder or granular form may be quite important and bettor operational control could be achieved. British Steel industry will be using metallised charges as a coolant material for "Spray refining" process.

CONCLUSION

The following conclusions have been drawn from the present study.

(1) The kinetics of reduction of mixed iron ore wood charcoal briquettes of -60 to +85, - 85 to + 100 and -100 to +150 mesh sizes at $\frac{1}{2}$, 1/3 and $\frac{1}{2}$ Fe₂0₃/C ratios for 900°C, 1000°C, 1050°C, and 1100°C in nitrogen atmosphere was investigated. The effect of temperature, particle size and ratio of Fe₂0₃/C was investigated.

(2) The effect of temperature on the degree of reduction particularly in the initial stages (upto 30% reduction) was found to be pronounced throughout the range of temperature studied.

(3) It has been found that reduction upto 90% proceeds through two stages. Initial stage of reduction i.e. upto 30% was controlled by chemical reaction while later stage by diffusion of iron through FeO.

(4) Particle size of iron ore and wood charcoal was not found to affect the activation energy values.

(5) The reduction was found to proceed at higher ratio with proportionately higher carbon content in the mixture.

SUGGESTIONS FOR FURTHER WORK:

Following aspects can be studied for further work.

- (1) Beyond 90% reduction study could be done to elucidate the mechanism of reduction.
- (2) Effect of lignite coke, low grade coke and other low grade solid fuels on rate of reduction of iron ore can be studied and their suitability for production of pre-reduced agglomerates can be assessed.
- (3) Effect of nitrogen gas flow on the rate of reduction.
- Pilot plant studies can be made to produce pre-reduce
 briquettes and their suitability for production of steel
 can also be assessed.

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APPENDIX *

The percentage reduction has been calculated on oxygen basis. Weight loss obtained after reduction is due to carbon, oxygen and removable ingradients (volatile matter in the reductant and ignition loss of iron ore fines) removed from the briquettes. Thus the oxygen removed during reduction is calculated by substracting the sum of weight of carbon removed and removable ingradients from the weight loss data obtained. Then using the following formula :

> Oxygen removed : Theoritical removable oxygen

z 100

the percentage reduction is found out.

TABLE -1

PERCENTAGE LOSS ON IGNITION OF IRON OHE

Temperatures	Mean loss in weight (gms.)	% loss on ignition
900 ⁰ C	0.070	7%
1000 ⁰ C	0.081	8.1%
1050 ⁰ 0	0.083	8.3%
1100°C	0.085	8.5%

TABLE-2

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PROXIMATE ANALYSIS OF WOOD CHARCOAL

Ingradients	As received	After Volatile matter renoval
Moisture	4.7%	1.2%
Volatile matter	22.4%	4%
Ash	9.3%	14.8%
Fixed carbon	66.4%	80%

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TABLE 3.1

PERCENT REDUCTION OF IRON OXIDE IN IRON OR BRICUETES FOR THE REACTION FE203+2C FOR SIZE 60 TO 4

Time	0006	199 - Yu	1000°C		1050°C		110000	
(ainutes)	(minutes) 2 of carbon in the reduced briquettes	1 ro- Juction	<pre>% re- 1% of carbon 1 duction. in the reduced briquettes</pre>	% reduc- tion	<pre>% reduct % of carbon tion in the reduced briquettes</pre>	12 roduct	252	of carbon % the reduct d briquette ti
ъ	10.55	R	sth*6	12%	\$20.9	15%	R	285
30	9.6%	9.2%	8.5%	20%	6.8%	30.25	9. 58	°*trt 5
45	84°6	13%	6.8%	30.3%	5.28	13. S	3.5%	55.
60	86	16%	5.8%	38.5%	¥.	50.5%	1:3%	62.
06	8.8%	20%	14%	20%	3.25	57.45	1.28	g 66.
120	8.6%	22.5%	3.25	55.8%	1.45	62.8%	42	202
180	8.4%	24.55	38	565	1.20	636	0.85	502

TABLE 3.2

PERCENT REDUCTION OF IRCH OXIDE IN MCM ORE BRIGUERTES FOR THE REACTION Fe203 +2C FOR -85 TO + 100 HEM SIZE

Tino	ю6 •	900°C	in an 1 0	10000	105	1050°C	1100°C	ç
(minustes)	C.	e -	C	4 21	c ¹	в, +	c4	R.
5	9.2%	101	8. 8%	14%	8.4%	20%	6%	35%
30	8.9%	15%	8	27.85	5.6%	35.65	4.23	146 55
45	8.8%	20%	6.8%	37.4%	Ser.	50%	SE	62.3
60	8.65	22%	. h6%	2111	3.8%	24%	1.63	65%
06	8	25.4%	NA NA	528	3.45	60.25	1.04%	69.3
120	7.8%	28.5%	34	8	5	64%	o.9%	70.8
180	2.4%	31%	2.85	58%	1.0%	64.23	0.85	725

calculation of percent reduction is shown in Appendix.

 $c_{f} = Percent$ of carbon in the reduced briguettes

R₁ = Percent reduction

TABLE 3.3

PERCENT REDUCTION OF IRCH OXDE IN IRCH ORE BRICHERTES FOR THE REACTION Feron + 2C FOR

	0006	0 ^C	1000 ⁰ C	^o	• •	1050°C	4900C	, c
(minute)	5	t H	ະ ບິ	et.	5	er F	5	a.
15	200°6	N	8.8%	16%	8.6%	25%	5.25	2
30	8.6%	17%	7.65	34%	R	10.7%	3.4%	52.
45	8.14	22.5%	6.25	43.4%	24.4	538	8	ي. ه
60	8.0%	25%	4.4%	52%	R	29°58	1.18	68%
90	7.85	30,2%	3.2%	57.52	2.6%	65.4%	0.8%	242
120	6.6%	32.5%	2.6%	60.8%	12	\$69	0.53	74.8
180	6.23	3773	2.4%	61.2%	0.6%	20%	0.15%	758

 c_{4} = Percent of carbon in the reduced brightes.

R₁ = Percent reduction

* Calculation of percent reduction is shown in Appendix.

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TABLE 4.1

PERCENT REDUCTION OF JRON OXIDE IN IRON ORE BRIQUETTES FOR THE REACTION FO + 3C FOR

Tine	800 ⁰ C		10000	-	1050°C	30C i	110000	5
(-sata)		H H		8.	C1	R1	C1	R1
ĴĮ	žer	6%	10.8%	27.2%	6.4%	53%	4.2%	08%
30	12.8%	10.2%	9.6%	37.8%	6.02%	565	3.585	76.2%
46	12.6%	12%	8.2%	44.6%	5.8%	12.2%	8.8	84.5%
60	12.25	14.3%	7.2%	51.3%	4.6%	77.45	1.65	90.15
80	35%	17+2%	6.04%	60.0%	4.15	84.55	1.65	8
120	11.6%	20.69	5.15	65.8%	2.24%	89.25	1.45	23.25
091	118	24.35	3	73%	1.845	91-5%	1.46	53. 3K

 $G_{\lambda} = Percent of carbon in the reduced briquettes.$

R₁ = Percent reduction

* Calculation of percent reduction is shown in Appendix. •

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TABLE 4.2

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UNTER FOR THE REACTION F0203+3C FOR PERCENT REDUCTION OF ROM OXIDE IN ROW ORE BILD

These stars	δa i	soo ^o c	Ю.	10000		1050 C		3100°C	
(*suju	л С	 #	υT		 æ	°.	1 14	°0 ^{r4}	æ ⁻⁷
						•		2	
	12.8%	12%	8		352	6.2%	51.3%	4.05	56.23
	12.4%	16-2X	8.65	-	44.6%	5.8%	56.45	3.65	76.85
	11.8%	19-5%	3-40		62.6%	4.45	73.15	3.25	845
	11.02%	25%	25	-4	67.5%	45	78.25	52	80.35
i.	10.58%	285	6.6%		65 _%	3.4%	86%	1-8%	94.25
	9•84%	32.45	4.2%	- ·	72.45	8	89.5%	1.2%	96.25
	R.40%	25-68	3.6%		80.65	1.65	91.8%	1.0%	85.45

C1 = Percent of carbon in the reduced briquottes.

R₁ = Percent reduction

*

· celculation of percent reduction is shown in Appendix.

TABLE 4.3

PERCENT REDUCTION OF IRON OXIDE IN IRON ORE BRIGUETTES FOR THE REACTION Re-0-+3C ROR -100 FO +150 HERE SIZE

Tico	1 800 ⁰ C		70	1000 ⁰ C	301	1050°c +	3100°C	
(aîns.)		 	ч С	• • • •	5	н Н	• • CT	R1
35	12.4%	15/2	9.48	37 .5 %	5.6%	51.8%	3.65	58.65
8	11.92%	22.38%	7.2%	50%	4.65	58 %	2.85	m
Ş	11+2%	27.3%	6.4%	555	4.2%	72.65	₩. 0	85.6%
60	10.76%	31.2%	5.2%	64.25	3.8K	78.45	3.6%	516
00	9•6%	38.55	3*8%	72.5%	2.6	88.2%	1. 48	85%
031	8.5%	40.2%	2.7%	81-75	1.85%	90.65	1.2%	80°85
180	7.85	46.6%	2.2%	85*25	1.6%	200	3.05	818

c1 = Percent of carbon in the reduced briquettes.

 $R_{\rm A}$ = Percent roduction

· calculation of percent reduction is shown in Appendix.

TABLE 5.1

PERCENT REDUCTION OF IRON OXIDE IN IRON GRE BRIGUERTES FOR THE REDUCTION Fe203+4C FOR -60 to + 85 NEXH SIZE

Time	3000 ⁰ C		1000 ⁰ C		1050°C		11000	Ţ
(atns)	C.	R ₁	c1	* *- #	c+	r.	c+ -	a
12	15.0 45	22%	13.4%	30.08	10.4%	48%	5.5	58.2
30	14.2%	25%	10.4%	20%	7.65	69.63	61.33	78.1
45	13.4%	32.5%	8.4%	60%	6.4%	77.5%	12	206
60	12:45	38.2%	7.8%	68%	ĸ	85*5%	3.8%	958
06	10.8%	148 X	65	80.5%	¥.2	\$26	3.62	\$16
120	9.8%	54.8%	5%	86.8%	3.8%	95%	N.C.	97.8
180	26	60.1%	4.8%	878	20.M	95.25	3.15	97.85

 C_{1} = Percent of carbon in the reduced briquettes.

R_f = Percent reduction

* Calculation of percent reduction is shown in Appendix.

TABLE 5.2

PERCENT NEDUCTION OF INCO OXIDE IN IRCH ONE BRICHETES FOR THE REACTION Fe203 +4C FOR

	900°C		1 10000	C	1050°C	**	110000	
(mins.)	C,	R	t c4	ħ	1 C1	Rq +	• •	R
15	15.58	16.25	138	38%	10%	50%	26	59%
30	13.8%	27%	9 °8%	55%	2.44	68.23	63	80.2
45	12.45	351	88	635	5.2%	83%	¥	106
60	- 11.9%	40.15	7.65	703	4.85	206	3.33	95.8
06	107	364	6.2%	81%	¥2.4	93.5%	3.25	98%
120	9.6%	55.8%	R	878	3.6%	95:28	M.	98%
180	8.6%	62.23	4.8	88	3.625	95.4g	N.	98 . 2

 $R_{i} = Percent reduction$

 C_{1} = Percent of carbon in the reduced briquettes

* Calculation of percent reduction is shown in Appendix.

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TABLE 5.3

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PERCENT REDUCTION OF IRON OXIDE IN IRON ORE BRIGUERTES FOR THE REACTION Fe.203+ 4C FOR -100 to + 150 MESH SIZE

Tine !	9000	.	1000°C	1	1 050°C		1100°G	
(suim)	c,		- •** C	Re • •	 C	R.	• •• ບ້	æ
5	15.25	18%	12.8%	42.83	9.6%	52.6%	8.8%	603
30	13.4%	308	9.6%	568	22	204	5.8%	80.8
45	12%	37.5%	8%	678	5.15	84%	¥	932
60	11.65	42.25	6.8%	758	1.2X	926	3.45	96x
90	10.2%	558	5.28	83.5%	3.8%	6 5%	R	98. 4
120	200°6	603	4.8%	88%	3.45	9 6 %	2.8%	4.80
180	7.8%	69.25	4.6%	89.5%	¥.	96.25	2.68	4.96

 c_{i} = Percent of carbon in the reduced briquettes

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R₁ = Percent reduction

* Calculation of percent reduction is shown in Appendix.

TABLE-6

VALUES OF CONSTANT C OBTAINED BETWEEN 30% AND 90% REDUCTION FOR INCN ORE BRIQUEITES REDUCED WITH WOOD CHARCOAL UNDER NITROLEN ATMOS HERE

Reduction Reactions	-60 to +85 mesh	values of constant -c. o +8585 to + 100 sh mesh	-100 to +150
F6203 + 20	50-67	3050	1+0450
Fe203 + 30	50-62	50-52	60-70
Fe203 +4C	1+0-60	58-62	3+0-50

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	INTROGEN ATMOS FILERE	
Reduction reactions	<u>Below 30% reduction</u> -60 to + 85 -85 to +00 -100 to +150 mesh пеsh пеsh	Between 30% and 90% reduction -60 to +85 neah -85 to + 100 -100 to + 150 nesh
Fe203+2C	25.0 Kcal/gm. 24.8 Kcal/ 25 Kcal/gm. mole gm.mole mole	35.8 Keal/gm. 35.8 Keal/gm. 35.4 Keal/gr mole mole nole
Fe203+3C	23.8 Keal/gm. 25 Keal/gm. 25 Keal/gm. mole mole	38.6 Kcal/gm. 35.6 Kcal/gm. 35.6 Kcal/gm mole mole mole
Fe2034tC	25 Kcal/gm. 25 Kcal/gm. 25 Kcal/gm. mole mole mole	35.4 Kcal/gm. 35.6 Kcal/gm. 35.4 Kcal/gm mole mole nole
3 		

TABLE 8.1

EFFECT OF Fe203/C RATIO ON TIME SPAN (t,) VALUE (-85 to + 100 mesh)

Mixture	Fe ₂ 0 ₃ /C ratio	Time for 900°C	half com 1000°C	plete reduc 1050 ⁰ C 11	tion 00 ⁰ C
$Fe_{2}0_{3} + 2C$	1/2		80 mins.	50 mins.	27 mir
Fe203 + 3 C	1/3		40 mins.	15 mins.	9 mini
Fe2 ⁰ 3 + 4 C	1/4	90 mins.	27 mins.	12 mins.	7 min:

TABLE 8.2

EFFECT OF FOO/C RATIO ON TIME SPAN (t,) VALUE (-100 TO + 150 MESH)

Mixture	Fe ₂ 0 ₃ /C ratio	Time for	half compl	ete reduc	tion
		900°C	1000 ⁰ C		
$Fe_{2}O_{3} + 20$	1/2	 **	55 min.	40 min.	25 min:
$Fe_2o_3 + 3C$	1/3	÷.	30 min.	13 min.	8 min.
Fe203 + 4C	1/4	78 mins.	24 min.	11 min.	6 min

TABLE 8.3

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HFFECT OF PARTICLE SIZE ON TIME SPAN (CA

	Particle sizes	Tine for hal	Time for half complete reduction	fuction	
		2000 t	1000°C	1050 ⁰ C	1100 ⁰ C
Fe203+2C	-60 to +85 Hesh		90 mins.	60 mins.	37 mins.
	-85 to + 100 Mesh	.	80 mins.	50 mins.	27 mins.
	-100 to + 150 mesh		55 mins.	40 mins.	25 ains.
Fe203 + 30	- 60 to + 85 Mean	ł	56 mins.	15 mins.	10 mins.
	- 85 to + 100 mean	ł	Ho mins.	th mins.	9 mins.
	- 100 to + 150 mesh	*	30 mins.	13 mins.	8 mins.
$Fe_20_3 + Wc$	- 60 to + 85 Mesh	96	30 mins.	16 mins.	to mins.
	- 85 to + 100 mesh	06	27 mins.	12 mins.	7 mins.
	-100 to + 150 mesh	78	24 mins.	11 ains.	6 mins.

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