

Kinetic Investigation of Reduction of Hematite Fines By the Volatile Matter of Coal Fines

The hematite is from the Koira mines and coal from the Talcher belt mines.

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Abstract— In the present investigation, an attempt has been done to study the simultaneous effects of the major processing variables on the extent of reduction of iron ore fines surrounded by coal fines in a crucible placed in muffle furnace. The project goal is to compare the reduction potential of two different coals in reduction of iron ore fines, by obtaining the energy of activation values required by the reduction reactions. The variables studied are time and temperature, coal/ore ratio, size of the iron ore fines and size of the coal fines. The reduction was done at three different fixed temperatures. The degree of reduction of fines was calculated at different time intervals, at fixed temperatures and variable parameters. The experiments were statistically designed such that the effect of each variable can be quantitatively assessed and compared. Then the observations were noted down and graphs were plotted between degree of reduction and time; then the appropriate rate law, was chosen and subsequently, the values of rate with time graph was plotted. From it, the slope was calculated which gives the equilibrium constant. Finally the Arrhenius equation was graphically used to determine the activation energy of the reduction reactions. Lastly, the activation energy versus parameter varied graph was plotted to find out the changes in activation energy with respect to change in the major parameters. (*Abstract*)

Keywords—iron ore fines; coal fines; coal/ore ratio; size of fines; degree of reduction; rate laws; Arrhenius equation; activation energy (*key words*)

I. INTRODUCTION

Talcher is known for its coal reserves. Talcher Coalfield is subdivided into five production/ administrative areas: Talcher, Jagannath, Kalinga, Lingaraj and Hingula. The coalfields are managed by Mahanadi Coalfields Limited (MCL), a subsidiary of Coal India Limited (CIL). The establishments of Talcher Thermal Power Station (TTPS), Heavy Water Plant and the Collieries (MCL) have enhanced the importance of the place. The coal production of Talcher Coal field has attained phenomenal growth during the last 30 years from 0.91 million tons in 1972-73 to 53.67 million tons in 2007-08. The coal is of lower grade containing only about 35 per cent of fixed carbon, 40 per cent volatile matter and 25 per cent ash content. In our experiment we have used coal from Lingaraj mines Ananta mines and Dera mines. The iron-ore from the Koira mines (obtained through Nerverem Power Steel Ltd.) used in this experiment is hematite with trace magnetite which was separated before the experiments were conducted. In this paper, the reduction potential of the local coal on the local iron-ore

was investigated and the results presented in the form of activation energy versus the parameters of the reactions.

II. THEORY

A. Coal gasification and Boudouard Reaction

Coal gasification refers to a process that breaks down coal into its components, by subjecting it to high pressure and high temperature in addition to the use of steam and oxygen. This leads to the production of synthesis gas, which is mainly a mixture containing carbon monoxide and hydrogen. Coal gasification can be utilized to produce methanol as alternative energy source. However, the production of methanol from coal is not that simple. Coal contains many other compounds and impurities that would interfere with the methanol synthesis process. Coal often contains such compounds as nitrogen, sulfur, ash, hydrogen, oxygen and water. These together form the Volatile Matter content (except Ash) of the coal. Before coal can be gasified, it must first be dried. Then afterwards the coal can be gasified to produce syngas. The reversible reaction of carbon dioxide with solid carbon to form carbon monoxide is termed Boudouard reaction which is the main reaction during coal gasification. This reaction is highly endothermic and has very large activation energy for chemical kinetics. Therefore it proceeds at an appreciable rate only at sufficiently high temperatures for a given reactivity of carbon. Below 973K, formation of carbon dioxide (exothermic) is preferred while above 1173K, formation of Carbon Monoxide (endothermic) is preferred. In our experiment, however, we have used temperatures ranging from 873K to 1073K, and have used the Volatile Matter (VM) of the coal as the reductant, instead of the usual carbon content as in Directly Reduced iron (DRI) plants.

B. Kinetics of reduction of hematite

Industrially iron is produced from iron ores, principally Hematite, and Magnetite by a carbo-thermic reaction that is reduction with carbon, in a blast furnace at temperature about 1073-1873K; Other than this classical route that is blast furnace technology of iron production, the iron also can be produced from its ore by the direct reduction of iron ore by a reducing agent which is coal based or may be a gaseous reducing agent, which is called directly reduced iron (DRI) or sponge iron. DRI is produced by reduction of the ore fines by either the hydrogen evolved in the form of VM from the low grade coal fines, at temperatures less than 1223K, while carbon of the coal is the

primary reductant above this temperature. Reaction kinetics in iron ore reduction deals with the rate at which iron oxides are converted to metallic iron by the removal of oxygen. The rate of a chemical reaction increases with increase in temperature. For this reason the reaction kinetics are not generally a matter of great importance in the blast furnace because of the high temperatures at which the furnace is operated. On the other hand, in DR processes where the iron is reduced in the solid state, the maximum temperature is below the melting point temperature and the reaction rates are slower. For direct reduction of iron ore, the mechanisms are complex because the oxide must go through a series of step wise changes before the conversion is complete. The slowest step in the process determines the overall reaction rate and is referred to as the rate controlling step. Reduction by Hydrogen (present in the VM) takes place in three steps; first, the hematite converts to magnetite, then magnetite to wustite and finally wustite to iron. The reaction is topochemical in nature which implies that formation of spongy or porous product layer over the reactant particles influence the reaction kinetics. The diffusion of reductant gas through the porous layer and the reaction at the interface of product layer and reactant, are rate controlling steps. The former is rate determining in diffusion-controlled reaction, the latter in chemically controlled ones and both in mixed controlled reactions. Please do not revise any of the current designations. The reduction of the iron oxides takes place in a series of sequential steps. The overall rate will be determined by the slowest process in the series. The possible consecutive steps are:

- Transport of gaseous reductant from the bulk gas phase to the particle surface through a boundary gas film.
- Molecular diffusion of the gaseous reductant through the product layer to the reaction interface.
- Adsorption of the gaseous reductant at the interface.
- Reaction at the interface (reaction between adsorbed reductant and oxygen of the lattice).
- Desorption of the gaseous products from the interface.
- 6. Mass transport of iron and oxygen ions and transformations in the solid phase, formation and growth of reaction products e.g. magnetite, wustite, iron.
- Molecular diffusion of gaseous products through the product layer to the particle surface.
- Transport of the gaseous products from the particle surface through the boundary gas film to the bulk gas phase.
- The rate limiting cases are chemical controlled (steps 3 to 6) and diffusion controlled (steps 1, 6, 7, 8), or mixed controlled in complex cases.

III. EXPERIMENT

Aim of the experiment was to carry out the reduction of iron-ore fines by coal-fines, under fixed temperature, at different time intervals, for: Set_01: variable ore to coal ratio, Set_02: variable coal fines size, with fixed ore to coal ratio, and Set_03: variable ore fines size, with fixed coal size and fixed ore to coal ratio, to determine the activation energy, and

observe the changes in activation energy (E_a) with respect to variation in ore to coal ratio, coal fines size, and ore fines size, respectively. Before the actual experiment, the coal samples were taken for proximate analysis to obtain the moisture content, volatile matter content, carbon content and ash content of the coals. Two of the coal samples with better volatile matter contents were selected for the experiment.

The procedure of the entire experiment, including the proximate analysis is as follows:

A. Proximate analysis of coal samples and initial sizing

A collection of three coal samples were taken from three different locations. Sample 01 was taken from Lingaraj, sample 02 was taken from Dera and sample 03 was taken from Ananta mines. Then the proximate analysis of the coal samples was done. 1 g of each of the samples of +70 mesh size was taken. In Proximate analysis first of all the moisture content was measured by heating all three samples in a drying oven at 110°C for about 1 hour. Moisture content was then obtained by calculating the percentage of weight loss. Then after removing the moisture content it was heated in a muffle furnace at 750°C for 1 hour to measure the content of volatile matter in the samples. The percentage weight loss of this moisture free coal gives the volatile matter content. Then after removing the volatile matter, the ash content of the samples was measured by heating the samples at 1123K till it is completely burnt for two hours. The percent weight of residue is the ash content. The carbon content is established by subtracting the sum of moisture content, VM content and Ash content from 100. The two coal samples (sample 02 and sample 03) with higher VM content was selected for further experiments. Then the coal samples were sized and screened in a 70, 100 and 120 mesh to obtain -70, -100, -120 mesh sizes. The iron-ore collected from the Koira mines via Nerverem Power Steel Ltd., had the following composition (according to the essay obtained from the Nerverem Power Steel Ltd.): 61.89% Fe(total), 3.86% LOI (lean ore of iron), 3.98% Laterite, 4.06% Moisture, 0.03% CaO (lime) 0.08% MgO (periclase) 2.5% silica. This ore was crushed, ground and screened into (-70, +80) mesh, (-80, +90) mesh, and (-90) mesh sizes. Any magnetic particle if present, were separated and disposed, and the ore fines were stored for further use.

B. Set_01 of the experiment: parameter varied is the ore to coal ratio by weight

- for the set_01 of our experiment, the reduction of iron ore (-90 mesh size) was carried out with -120 mesh coal fines, with iron ore: coal ratio 1:1, 1:2, 1:3 and 1:4, at fixed temperatures 873K, 973K and 1073K and at different time intervals for both the coal samples 02 and 03. Then the magnetic values after reduction were separated, weighed and the data obtained were used to calculate the degree of reduction which is given by the ratio of amount of oxygen removed by the reduction process to the total amount of oxygen available for removal. The degree of reduction is denoted by α .
- From the α values, the rate laws were obtained by calculating the $g(\alpha)$ values (the $g(\alpha)$ is the rate law function of degree of reduction. Several predetermined rate laws were used to find several sets of $g(\alpha)$ values. Thereafter, correlation coefficient and standard

deviation of the sets of values were calculated to determine the rate law that operated in our experiment. The rate law that had the highest correlation coefficient and lowest standard deviation was selected. In our case, the mixed controlled rate law was seen to be the operative law:

$$g(\alpha) = [(1+\alpha)^{2/3} - 1]^2 \quad (1)$$

- The $g(\alpha)$ values were plotted with time (in seconds) for all the ore to coal ratios. According to the equation

$$g(\alpha) = kt \quad (2)$$

where k is the equilibrium constant and t is the time, we find that the slope of the plot gives the value of equilibrium constant of the reduction reaction.

- Next, the plot of negative natural logarithm of k versus reciprocal of absolute temperature used is plotted for the three ore to coal ratios for both coal samples. The slope of this plot, according to the Arrhenius equation, gives the value of the ratio of E_a to R , where R is the universal gas constant. The obtained value of the slope, multiplied with the value of R , thus, provided the value of the activation energy of the reactions in kilojoules per mole.
- Finally, graphs showing the variation of activation energy with variation in ore to coal ratio by weight, are plotted for both the selected coal samples 02 and 03.

C. Set_02 of the experiment: parameter varied is size of coal fines

- For the set_02 of our experiment, the reduction of iron ore fines (size kept constant at -90 mesh) was carried out at a fixed ore to coal ratio (1:1), with three different sizes of coal fines (-70, -100 and -120 meshes) separately, at constant temperatures of 873K, 973K and 1073K, for various time intervals, for both the coal samples 02 & 03.
- Then the magnetic particles were removed by using a yoke, and measured. Then the different α values were calculated, and from then the $g(\alpha)$ values. In this case, the rate law was "spherical contact model"

$$g(\alpha) = 1 - (1 - \alpha)^{1/2} \quad (3)$$

instead of the mixed controlled rate law as shown in equation (1).

- Then, similar steps as in set_01 were carried out one after the other to obtain the values of equilibrium constant, and activation energy for the reactions.
- Finally, the plot of activation energy versus coal fine size for both the coal samples was plotted.

D. Set_03 of the experiment: parameter varied is size of iron-ore fines

- For the set_03 of our experiment, the reduction of three different sizes of iron ore (hematite) fines (-70+80, -80+90, -90 meshes) was carried out at a fixed ore to coal ratio (1:1), with coal fines (size kept constant at -100 mesh) separately, at constant temperatures of 873K,

973K and 1073K, for various time intervals, for both the coal samples 02 & 03.

- Then the magnetic particles were removed by using a yoke, and measured. From the data obtained, degrees of reduction values were obtained. Then the $g(\alpha)$ values were calculated and the appropriate rate law was selected. In this case, the same rate law as in equation (3) was found to operate.
- Then, similar steps as in set_01 were carried out one after the other to obtain the values of equilibrium constant, and activation energy for the reactions.
- Finally, the plot of activation energy in kilojoules per mole versus ore fine size was plotted.

IV. OBSERVATIONS AND CONCLUSIONS

The tables showing the proximate analyses of coal samples (Table I), the values of equilibrium constant obtained after extensive calculations (the actual numerical calculations; data tables of weight of magnetic particles obtained from yoke, degree of reduction value tables and $g(\alpha)$ tables; and statistical analyses and plots of α versus time and $g(\alpha)$ versus time have not been shown) and data analysis (Table II, III and IV), and the values of activation energy obtained for both samples of selected coal for all the three sets of experiments (Table V, VI and VII) are given in the following sections. The plots of negative logarithm of equilibrium constant ($-\ln(k)$) versus reciprocal of the absolute temperature ($1/T$) for all the experiments (Fig. 1, 2, 3, 4, 5 and 6) and the final plots of activation energy versus reaction parameters for both the coal samples in all the sets (Fig. 7, 8 and 9) have been shown.

A. Figures and Tables:

TABLE I. PROXIMATE ANALYSIS OF COAL SAMPLES

Table 1	Proximate analysis of coal samples				
	Sample weight taken is 1g for all samples				
sample	Moisture %	Volatile matter (VM) %	Carbon %	Ash%	Selection status
01	11.1	11.7	34.8	42.4	no
02	8	19.5	30.35	42.15	Yes
03	4.5	25.86	29.6	40.04	Yes

TABLE II. VALUES OF EQUILIBRIUM CONSTANT FOR SET 01

Sample 02 of coal	Set_01: parameter varied is ore to coal ratio		
	Equilibrium constant values in order of 10^{-5}		
Ore: coal ratio	Temp = 873K	Temp = 973K	Temp = 1073K
1:1	5.95624	6.07911	6.19657
1:2	2.95081	5.01322	4.85403
1:3	2.16673	3.14812	3.69440
1:4	8.29557	1.73668	5.76360

Sample 03 of coal	Set_01: parameter varied is ore to coal ratio		
	Equilibrium constant values in order of 10^{-5}		
Ore: coal ratio	Temp = 873K	Temp = 973K	Temp = 1073K
1:1	2.83913	2.85024	3.44645
1:2	5.08242	6.94037	6.54402
1:3	4.94520	5.83684	7.35645
1:4	2.90892	4.66470	6.88711

TABLE III. VALUES OF EQUILIBRIUM CONSTANT FOR SET 02

Sample 02 of coal	Set_02: parameter varied is size of coal fines		
	Equilibrium constant values in order of 10^{-4}		
Size of coal fines	Temp = 873K	Temp = 973K	Temp = 1073K
-70 mesh	1.870	2.153	2.967
-100 mesh	2.5396	4.209	5.867
-120 mesh	5.294	5.723	6.937
Sample 03 of coal	Set_02: parameter varied is size of coal fines		
	Equilibrium constant values in order of 10^{-4}		
Size of coal fines	Temp = 873K	Temp = 973K	Temp = 1073K
-70 mesh	3.627	4.222	5.885
-100 mesh	3.736	4.605	6.532
-120 mesh	4.1237	4.496	5.2379

TABLE IV. VALUES OF EQUILIBRIUM CONSTANT FOR SET 03

Sample 02 of coal	Set_03: parameter varied is size of ore fines		
	Equilibrium constant values in order of 10^{-4}		
Size of ore fines	Temp = 873K	Temp = 973K	Temp = 1073K
-70+80 mesh	2.6533	3.8639	5.6063
-80+90 mesh	3.0813	4.5423	5.4180
-90 mesh	2.5396	4.2094	5.8677
Sample 03 of coal	Set_03: parameter varied is size of ore fines		
	Equilibrium constant values in order of 10^{-4}		
Size of ore fines	Temp = 873K	Temp = 973K	Temp = 1073K
-70+80 mesh	3.1451	3.8762	5.2283
-80+90 mesh	3.0922	3.8449	5.0406
-90 mesh	3.7367	4.6052	6.5320

TABLE V. VALUES OF ACTIVATION ENERGY FOR SET 01

ORE: COAL RATIO	SET_01: PARAMETER VARIED IS ORE TO COAL RATIO	
	ACTIVATION ENERGY VALUES ARE IN KILOJOULES PER MOLE	
	FOR COAL SAMPLE 02	FOR COAL SAMPLE 03
1:1	1.536661041	7.46978661
1:2	20.06088164	10.30036595
1:3	20.98935791	15.32388582
1:4	74.60467446	33.55309665

TABLE VI. VALUES OF ACTIVATION ENERGY FOR SET 02

SIZE OF COAL FINES	SET_02: PARAMETER VARIED IS SIZE OF COAL FINES	
	ACTIVATION ENERGY VALUES ARE IN KILOJOULES PER MOLE	
	FOR COAL SAMPLE 02	FOR COAL SAMPLE 03
-70 MESH	18.67	17.54
-100 MESH	10.34	9.19
-120 MESH	32.73	29.48

TABLE VII. VALUES OF ACTIVATION ENERGY FOR SET 03

SIZE OF ORE FINES	Set_03: parameter varied is size of ore fines	
	activation energy values are in kilojoules per mole	
	For coal sample 02	For coal sample 03
-70+80 mesh	29.033	19.601
-80+90 mesh	22.180	18.890
-90 mesh	32.725	21.485

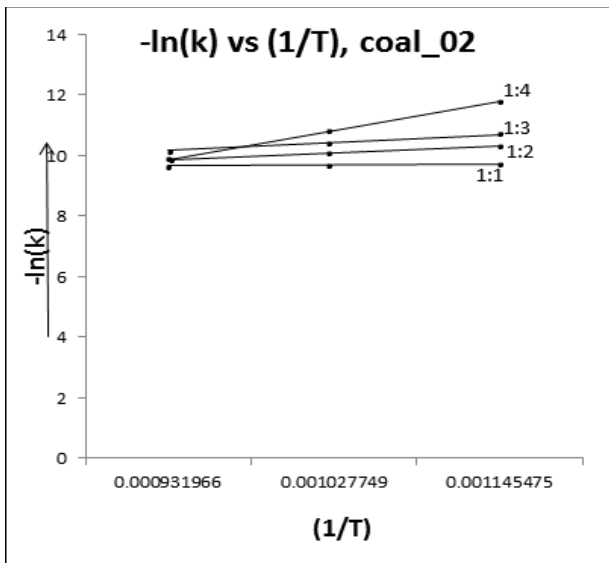


Fig. 1. $-\ln(k)$ vs $1/T$ graph for coal sample 02, set_01 of the expt.

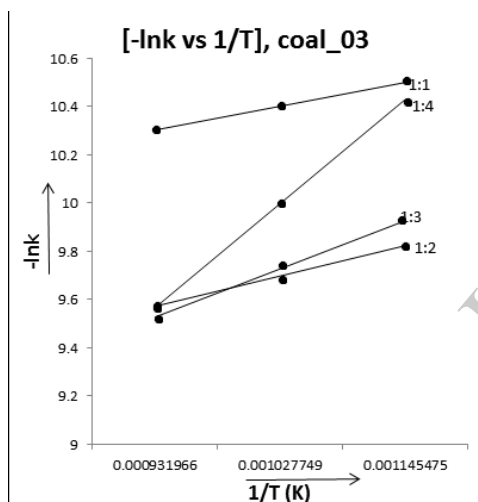


Fig. 2. $-\ln(k)$ vs $1/T$ graph for coal sample 03, set_01 of the expt.

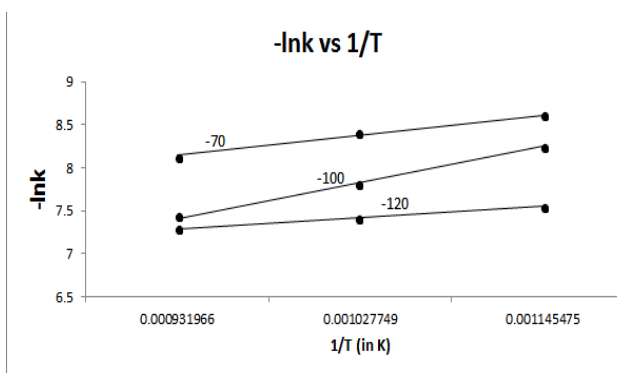


Fig. 3. $-\ln(k)$ vs $1/T$ graph for coal sample 02, set_02 of the expt.

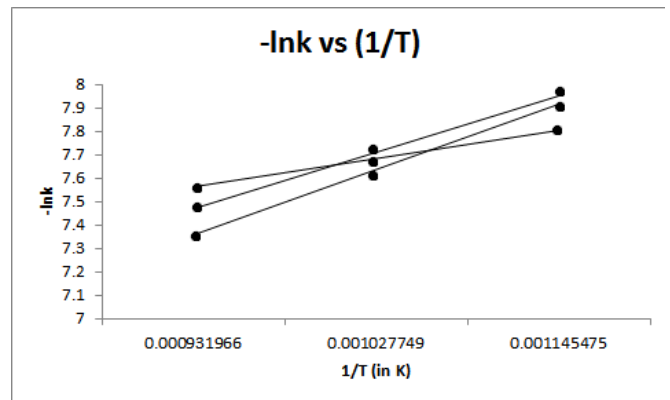


Fig. 4. $-\ln(k)$ vs $1/T$ graph for coal sample 03, set_02 of the Expt.(the lines shown are in order for increasing coal size)

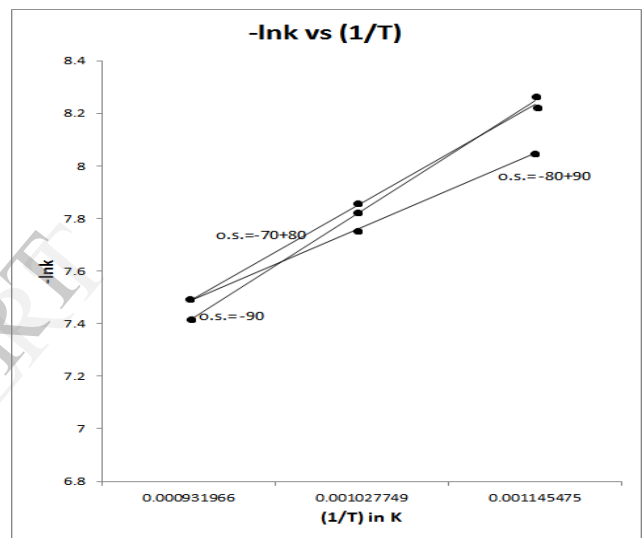


Fig. 5. $-\ln(k)$ vs $1/T$ graph for sample 02, set_03 of the expt.

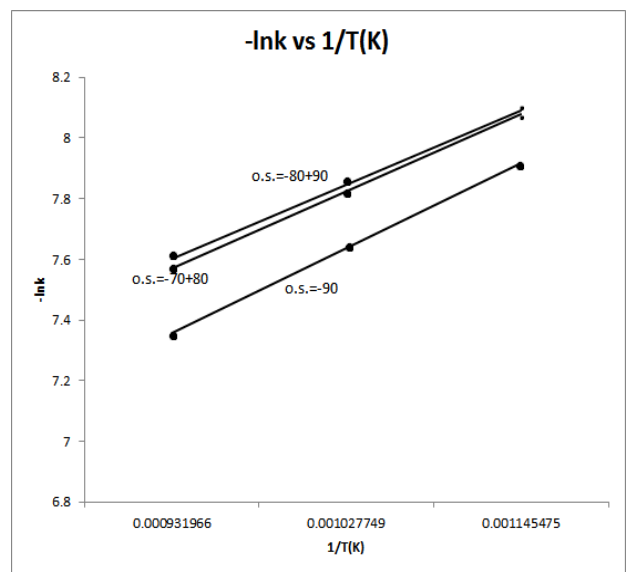


Fig. 6. $-\ln(k)$ vs $1/T$ graph for coal sample 03, set_03 of the expt.

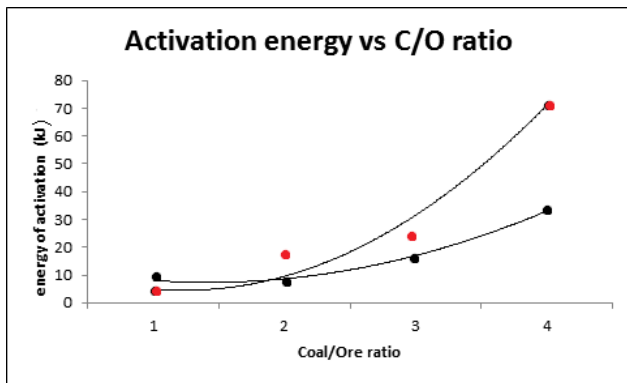


Fig. 7. Change of activation energy with respect to change in ore to coal ratio.

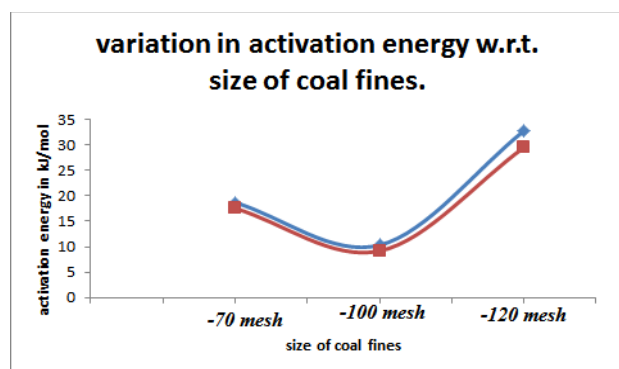


Fig. 8. Change of activation energy with respect to change in size of coal fines.

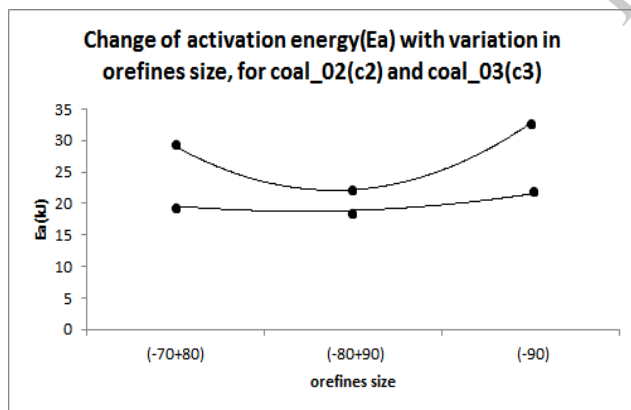


Fig. 9. Change of activation energy with respect to change in size of ore fines.

The figures (from Fig. 7 to Fig. 9) constitute the main conclusions of the paper. As we can see from the graph of the variation in activation energy of the reaction w.r.t. ore to coal ratio, the activation energy slowly rises at first and then increases suddenly with increasing amount of coal, for both the samples of coal chosen. This trend is compatible with the theory and can be explained as follows: With increasing coal to ore ratio, the amount of volatile matter that is released from the coal increases, while the exposed ore surface area decreases due to more amount of carbonaceous material. Thus, the

volatile matter released is increasingly unable to diffuse through the coal and reach the ore surface to cause reduction of the ore. At lower ratios, the mixed controlled reduction reaction proceeds somewhat easily due to more exposed surface area of ore, while in the case of higher ratios, the carbonaceous material covers the ore particles completely and creates a barrier for the evolving gases to react. The gases escape without reacting with the ore to a larger extent. This increases the activation energy of the reduction reaction. Since coal sample 02 has lower VM content and higher ash content, coupled with higher fixed carbon content than coal sample 03, the initial activation energy, for ore: coal ratio 1:1 of coal sample 02 is less than that of coal sample 03. But with increasing ratios, the higher carbon and ash content leads to higher blanketing of the ore surface, leading to greater rise in activation energy than for reduction done with coal sample 03.

The graph of the variation of activation energy with size of coal fines clearly shows that the activation energy first decreases and then rises with decreasing size of coal fines, and this trend is followed by reduction of the ore fines by both the coal samples chosen. This trend is compatible with the theory and can be explained as follows: With decreasing size of coal fines, the surface area of the coal fines increases, thus there is higher rate of evolution of volatile matter and thus higher rate of reduction of the ore, keeping the ore to coal ratio constant. The loss in permeability caused by finer coal size is more than compensated by the higher rate of gas evolution. When the coal fine size further decreases, the rate of volatile matter evolution is not able to compensate for the decrease in bed permeability, or porosity, thus, the VM gases escape without causing much reduction. Since sample 02 has lesser VM content, and higher ash content plus higher fixed carbon content, than coal sample 03, the activation energy for the reduction by sample 02 remains slightly more than that for sample 03. The lesser carbon and lesser ash content coupled with higher VM content of sample 03, makes it a better reductant than sample 02, thus leading to lower activation energy values than sample 02.

The graph of the variation of activation energy with size of iron ore fines clearly shows that the activation energy first decreases and then rises with decreasing size of ore fines, and this trend is followed by reduction of the ore fines by both the coal samples chosen. This trend is compatible with the theory and can be explained as follows: With decreasing size of ore fines, the surface area of the ore fines increases, thus there is higher rate of reduction of the iron ore by the volatile matter, keeping the ore to coal ratio constant. The loss in permeability caused by finer ore size is more than compensated by the higher rate reduction caused by higher amount of exposed surface area. When the ore fine size further decreases, the rate of reduction by the volatile matter is not able to compensate for the decrease in bed permeability, or porosity, thus, the VM gases escape without causing much reduction as they are not able to reach the ore surface in sufficient amounts due to loss in porosity. Thus active surface area is lesser, and activation energy increases. Coal sample 02 has lesser volatile content, higher ash and fixed carbon content than coal sample 03, and thus, rate of reduction is slower than that of coal 03. Hence, the activation energy values for coal sample 02 are always higher than that for coal sample 03. Moreover, the rate of change of activation energy with respect to size of coal fines is higher for sample 02 due to limited and lesser amount of volatile matter content, hence the curve shown.

Overall, it can thus be concluded that with decreasing ore amount, or increasing coal amount, i.e, increasing coal to ore ratio, the activation energy for the reduction of the iron ores from Koira mines by Talcher belt coal fines increases exponentially. Moreover, with decrease in either size of coal fines or ore fines, there is initially a dip in the activation energy values, which then rises steeply with further decrease in the size of the fines. Thus, an optimum size of the fines, and an optimum ore to coal ratio have to be assigned for lowering the activation energy, hence time taken and costs, for the reduction of the Koira ore fines by Talcher coal fines.

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