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Research Paper

REDUCTION OF IRON ORE PELLETS BY STATISTICAL DESIGN OF EXPERIMENTS

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Generation of iron ore fines needs sintering and pelletization before its being used in blast furnace /iron ore reduction. Further huge deposit of non coking coal has compelled Iron and Steel Industries to use these coals after gasification. Carbon dioxide gas coming out as flue gas is source of air pollution. In the present study, an attempt has been made to study the reduction behavior of CO/CO_2 produced after gasification of high ash non coking coal with flue gas coming out from a furnace under isothermal condition through statistical design of experiments. It was observed that degree of reduction increases with increase in temperature, time and concentration of CO in CO/CO_2 mixture. Temperature has strongest effect followed by time and concentration of CO in CO/CO2 mixture. The produced syn gas is used for smelting purpose then flux requirement to the process will be decreased because the ash of coal will not enter into the smelting furnace. In this way high ash non coking coal will be used in iron and steel industry for reduction and smelting purpose.

Keywords: Pelletization, Degree of Reduction

INTRODUCTION

Generation of iron ore fines is increasing day by day due to mechanized mining operation, sizing, beneficiation and friable nature of iron ore. Apart from these fines, there are good deposits of blue dust which is not accepted by blast furnace due to its fineness. These fines are only used in blast furnace after sintering and pelletization. There are also huge deposits of non-coking coal which can be used for reduction of iron ore fines after gasification.

It has been well established that reduction of iron ore by carbon in blast furnace and direct reduction processes proceed via gaseous media, i.e., by indirect reduction (Fruehan, 1977; Chakravorty *et al*., 1991; Haque *et al*., 1992; Lu *et al*., 2003; Kumar *et al*., 2009). Several investigations (Shalni *et al*., 2005; Kumar *et al*., 2009; Yi Ling-Yun *et al*., 2012) have demonstrated that the rate of reduction is highly sensitive to the form of reducing agent and that it increases with increasing temperature and decreasing size of

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the agglomerate. It has been pointed out (Bryk *et al*., 1986; Zohao-Caiwarg *et al.,* 2012) that the rate of heat transfer from the surface to the centre of sample played an important role in controlling the reduction kinetics of a mixed ore-coal pellet. Thorough investigation has also been carried out to study the reduction behavior of composite and double layer pellet under isothermal as well as non isothermal conditions. In the present investigation an attempt has been made to study the reduction behavior of CO/CO_2 produced after gasification of high ash non-coking coal under isothermal condition. The statistical method has been used for experimental design to know the effect of each variable and also their interactional effect.

EXPERIMENTAL

Raw Material

Iron ore was collected from Rungta mines near at Chaibasa, Jharkhand and heated to eliminate free and combined moisture. Iron ore was assayed 64.6 % Fe, 2.1% SiO_2, 2.9 % Al_2O_3 and LOI 2.5 %. Non Coking Coal was collected from Chitra Mines of Eastern Coalfields Limited, West Bengal. Non Coking Coal having 48.3% ash, 2.6% moisture, 22.0% volatile matters and 27.1% fixed carbon on air dried basis. Gross Calorific Value (GCV) of coal was found 3570 Kcal/kg. Ash analysis of the coal is shown in Table 1. It indicates ash is refractory and acidic in nature. It was pyrolised to remove volatile matter and the resultant char assayed 35.2% fixed carbon and 63.8% Ash. The same char was used for gasification purpose to produce a mixture of CO and CO_2.

Table 1: Ash Composition

Ash Compound	Percentage Share (%)
SiO_2	61.74
Al_2O_3	26.37
Fe_2O_3	6.99
TiO_2	1.44
P_2O_5	0.25
SO_3	0.12
CaO	0.35
MgO	0.09
Na_2O	0.47
K_2O	2.18

Pellet Preparation

Iron ore fines were crushed to –1 mm in roll crusher and then subjected to grinding in ball mill to 80 % passing 0.045 mm. Pellets were produced in disc pelletizer with the help of 0.5% bentonite and 10.5 % moisture. Green pellets were dried in an oven and then fired in a high temperature muffle furnace at 1320^{0}C for 20 min. These pellets were stored for reduction tests.

Gasification

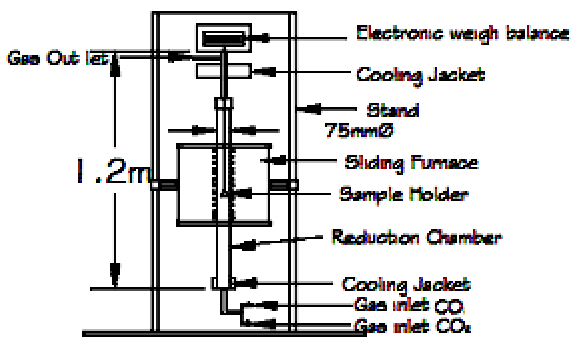
Char produced from non-coking coal was gasified in a vertical gasification chamber with the help of carbon dioxide gas .The bed temperature was maintained at 1000^{0}C .The product gas was used for reduction purpose. Desired CO/CO_2 ratio was maintained by mixing CO_2 to the product gas.

Experimental Procedure

The reduction test was carried out in a thermo gravimetric furnace with a provision for recording weight loss. Pellet was allowed to hang inside the chamber and its weight was recorded. The line diagram of the reduction test apparatus is shown in Figure 1. The furnace was switched on and nitrogen gas was allowed to flow till the desired reduction temperature was achieved. Now flow of nitrogen gas was stopped and a mixture of CO/CO_2 was allowed to flow for a desired time and weight loss was recorded. Then the pellet was cooled under Nitrogen atmosphere. The degree of reduction 'R' of pellet was calculated by using the following formula.

$$\frac{\text{Eight of Oxygen Removed from Pellet}}{\text{Weight of Removable Oxygen in the Pellet}} \times 100$$

Figure 1: Line Diagram of the Reduction Test Apparatus



Statistical Design of Experiments

Statistical design of experiments is advantageous in getting optimum condition with a limited number of experiments. The regression equation obtained is useful in highlighting the effect of individual variables and their relative importance in the process. The interactional effect of each variable is known which is not possible in classical experiments. In the present investigation, a simple fractional design of experiment has been used where two level of all the variables are taken.

RESULTS

To study the reduction behavior of iron ore pellets by CO/CO_2 mixture, a series of experiments have been conducted .The variables parameters and their range selected have been presented in Table 2 where actual (natural scale) and coded (dimensionless scale) values have been tabulated. The design matrix and results showing the degree of reduction are shown in Table 3.

Here x_1 is temperature (^{0}C), x_2 is time (in minute), x_3 is concentration of CO in CO/CO_2 mixture (partial fraction), Y the degree of reduction (%) and X_1, X_2 and X_3 are in coded form. The regression equation for the matrix is represented as

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{123}X_1X_2X_3 \qquad ...(1)$$

where a_0 is the degree of reduction when all the parameters are at the base level and a_1, a_2 and a_3 show the effect of corresponding parameters.

Table 2: Actual Value and Coded Value of Variables

	Temperature (^{0}C)	Coded value	Time (min)	Coded Value of CO	Concentration in CO/CO_2 Mixture, Partial Value	Coded Value
	x_1	X_1	x_2	X_2	x_3	X_3
Upper Level	1050	+1	40	+1	0.90	+1
Lower Level	950	-1	20	-1	0.50	-1
Base Level	1000	0	30	0	0.70	0

Table 3: Design Matrix and Result of Experiments for Determination Of Percentage (%) Degree Of Reduction (R)

Sample: Iron Ore From Rungta Mines Plus Syn Gas Produced From Coal Of Chitra Mines

Expt.No	X_1	X_2	X_3	R	X_1*R	X_2*R	X_3*R	X_1*X_2*R	X_1*X_3*R	X_2*X_3*R	X_1*X_2*X_3*R
1	+1	+1	+1	98.60	98.6	98.6	98.6	98.60	98.60	98.60	98.6
2	+1	+1	-1	90.56	90.56	90.56	-90.56	90.56	-90.56	-90.56	-90.56
3	+1	-1	+1	84.36	84.36	-84.36	84.36	-84.36	84.36	-84.36	-84.36
4	+1	-1	-1	72.73	72.73	-72.73	-72.73	-72.73	-72.73	72.73	72.73
5	-1	+1	+1	66.49	-66.49	66.49	66.49	-66.49	-66.49	66.49	-66.49
6	-1	-1	+1	42.83	-42.83	-42.83	42.83	42.83	-42.83	-42.83	42.83
7	-1	+1	-1	57.95	-57.95	57.95	-57.95	-57.95	57.95	-57.95	57.95
8	-1	-1	-1	29.38	-29.38	-29.38	-29.38	29.38	29.38	29.38	-29.38
9	0	0	0	68.01	0	0	0	0.00	0.00	0.00	0
10	0	0	0	67.52	0	0	0	0.00	0.00	0.00	0
11	0	0	0	68.64	0	0	0	0.00	0.00	0.00	0
	0	-1	0	542.90	149.60	84.30	41.66	-20.16	-2.32	-8.50	1.32

The regression coefficients of regression equation (1) are calculated by using following equations:

The regression coefficients were estimated by

$a_0 = \Sigma Y_i/N$ ...(i)

$a_j = \Sigma X_{ij} Y_i/N$ ...(ii)

$a_{nj} = \Sigma(X_{ni}X_{ij})\ Y_i/N$ and so on ...(iii)

where

Y_i = Value of degree of reduction (%) in the i^{th} trial shown in ith row

N = Total no. of trials

X_{ij} = Coded value in ith row and j^{th} column

The value of different coefficients calculated by above relations has been used to get the final regression equation.

The relationship between coded and actual value is given by

$X_1 = (x_1 – 1000)/50$; $X_2 = (x_2 – 30)/10$ and $X_3 = (x_3 – 0.70)/0.20$

Using these relationship regression Equation (1) can be converted to the uncoded form. Random experiments were carried out to test the regression equation for its validity. The conditions were fixed at the base level, i.e., $x_1 = 1000^0$C, x_2 = 30 minute and x_3 = 0.70 (concentration of CO in CO/CO_2 mixture, partial value). Three identical tests were conducted and all the three trials gave more or less identical fractional reaction, i.e., 68.01%, 67.52% and 68.64%, the calculated value of fractional reaction was 67.9%. To test the significance of each coefficients students 't' test was carried out at 95% confidence level. It was found that only the coefficients a_0, a_1, a_2, a_3, a_{12} and a_{23} are significant. Fisher's test at 95% confidence level was done for validity of the equation and it was found that the regression equation is correct and adequate.

Using Student 't' test and Fisher's test at 95 % confidence level, final regression equation developed for degree of reduction (R) for iron ore through coal gasification is

$$R = 67.9 + 18.7X_1 + 10.5X_2 + 5.2X_3 - 2.5X_1X_2 - 1.1X_2X_3$$

Table 4 shows the experimental and calculated value of degree of reduction. Since the difference is negligible, it can be said that the regression equation is adequate. From the final regression equation, it is seen that all the coefficients a_1, a_2 and a_3 have positive value which shows that with the increase in temperature, time and concentration of CO in CO/CO_2 mixture, degree of reduction increases. Interactional effect of time and temperature as well as time and concentration of CO in CO/CO_2 mixture is significant and negative. Among all these parameters temperature has strongest effect as well as interactional effect of time and concentration of CO in CO/CO_2 mixture has least effect on degree of reduction.

Table 4: Experimental and Calculated Value of Percentage Degree of Reaction (R)

Experiment no	Experimental R	Calculated R
1	98.6	98.7
2	90.6	90.4
3	84.4	84.8
4	72.7	72.3
5	66.5	66.4
6	42.8	42.4
7	58.0	58.1
8	29.4	29.8
9	68.0	67.9
10	67.5	67.9
11	68.7	67.9

CONCLUSION

On the basis of results obtained and analysis done the following conclusions have been drawn.

1. Regressions equations developed for calculating degree of reduction (%) is as follows.

   $$R = 67.9 + 18.7X_1 + 10.5X_2 + 5.2X_3 - 2.5X_1X_2 - 1.1X_2X_3$$

   The regression equation developed will be useful in predicting the degree of reduction under specified condition of temperature, time and concentration of CO in CO/CO_2 mixture.

   For achieving a particular degree of reduction the adjustment of processing parameters could be done accordingly.

2. Among all these parameters Temperature has strongest effect followed by Time and Concentration of CO in CO/CO_2 mixture.

   Temperature > Time > Concentration of CO

   The effect of time is just double of % CO in CO/CO_2 mixture where as the effect of temperature is 1.8 times higher than the effect of time. Therefore, higher temperature will be most effective parameters in controlling parameter in controlling the degree of reduction followed by reduction time and CO/CO_2 ratio will have least effect. With the syngas is used for smelting purpose then the flux requirement to the process will be decreased, because the ash of coal will not enter into the smelting furnace, which will result into decreased heat consumption and low slag volume. In this way high ash non coking coal will be used in Iron and Steel industry for reduction and smelting purposes at the same time a good percentage of carbon dioxide exposed to atmosphere will be utilized for gasification purpose.

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