

OPTIMISATION OF PROCESS VARIABLES IN SINTERING
THROUGH STATISTICAL DESIGN OF EXPERIMENTS.

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A B S T R A C I

An attempt has been made to quantify the effects of the variables on strength in sintering of iron ore/blue dust received from Bailadila, NMDC. For this, the method of design 2^n factorial experiment is used to obtain a response surface equation.

Results indicate if the coke breeze increases in the range of variation of the variable in sintering, the strength increases but strength decreases when moisture increases from lower level to higher level. Blue dust does not effect the strength.

The optimum level of variables for the maximum strength of the sinter product is determined by the method of hexagonal design of experiment. The results indicate that for a basicity of 1.9 an acceptable quality of sinter can be produced. Blue dust amounting to 40.0% of all the iron ore fines taken together can be incorporated in the raw mixture without imparting the strength of the finished sinter.

I N I R O D U C T I O N

In spite of knowing effect of all the additives but there is lack of information regarding the quantitative effect of the variables on strength properties. The number of processing variables which affect the properties are many and consequently complete interactions results due to these variables interacting with each other. In the conventional method, single factor experiments are planned when each is varied keeping other at constant level of variables. To reach an optimum level of variables, for maximum properties one has to perform a large number of tests even when the optimum combination is not always guaranteed. Under this circumstance, use of statistical design of experiments is extremely helpful. By performing fewer experiments in a planned manner, one can reach the optimum combination of variables in shorter time, thus saving considerable labour and cost.

The most important factors of the raw materials

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quality which effect the sinter strength are

- a) Coke content in the raw mix
- b) Quality of iron ore fines content in the raw mix
- c) Quality of limestone fines content in the raw mix
- d) Amount of dolomite fines, mill scale, flue dust etc.

With higher addition of coke breeze strength gets increased along with FeO content. Addition of higher amount of blue dust keeps Al_2O_3 within 7.0%. Limestone is added for fluxed sinter. It also increases permeability and zone of slag. Dolomite prevents formation of faylite. Mill scale increases permeability and strength.

Based on this information and the single factor experiments, the base level for coke breeze, blue dust and moisture was decided. Since these variables interact with each other in complex manner, it was thought to develop a regression equation connecting the input variables like coke breeze, blue dust and moisture with the strength values for the base level sinter product. For working out the optimum treatment combination from their regression equation experiments were performed by hexagonal design of experiment. The optimum treatment combination thus obtained was employed for sintering.

Work Plan

The work illustrates the statistical design and analysis of a sintering process through a systematic approach in the following lines :

- a) Factorial design
- b) Sintering tests according to the matrix of the design.
- c) Development of a mathematical model - regression equation.
- d) Test of significant co-efficients and adequacy of the equation.
- e) Plots drawn to see the effects of the variables and their interactions.
- f) Orthogonal design - hexagonal type
- g) Optimisation

a) Factorial Design

Many experimental situations require the examination of effects of varying two or more variables. It is shown that in a complete exploration of such a situation it is not sufficient to vary one factor at a time, but that all combinations of the different factor levels must be examined in order to elucidate the effect of each factor and the possible ways in which each factor may be modified by the variation of the others. In the analysis of

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the experimental results, the effect of the each factor can be determined with the same accuracy as if only one factor had been varied at a time and the interaction effects between the factors can also be evaluated. Here experiment in which each factor is tested at two levels.

A 2^3 full factorial experiment was design as shown in Table 1 and 2 with low and high levels of the variables as indicated.

Table 1: 2^3 Factorial Design

	-	+
A = Coke breeze	5.5	7.5
B = Blue dust	20.0	40.0
C = Moisture	6.5	7.5

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Table 2: Matrix of the experiments design

Test No.	% Coke breeze (A)	% Blue dust (B)	% Moisture (C)
1	5.0 (-1)	20.0 (-1)	6.5 (-1)
2	7.0 (+1)	20.0 (-1)	6.5 (-1)
3	5.0 (-1)	40.0 (+1)	6.5 (-1)
4	7.0 (+1)	40.0 (+1)	6.5 (-1)
5	5.0 (-1)	20.0 (-1)	7.5 (+1)
6	7.0 (+1)	20.0 (-1)	7.5 (+1)
7	5.0 (-1)	40.0 (+1)	7.5 (+1)
8	7.0 (+1)	40.0 (+1)	7.5 (+1)
9	6.0 (0)	30.0 (0)	7.0 (0)
10	6.0 (0)	30.0 (0)	7.0 (0)
11	6.0 (0)	30.0 (0)	7.0 (0)

Sintering experiments :

The raw mix for sintering was made of the iron ore fines, blue dust, limestone, dolomite, lime, Mn-ore, mill-scale flue dust, coke breeze, water and return fines. The amount of variables were used according to the matrix of the experiment design. A computer programming was developed to calculate the

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weight percentage of each ingredient in the raw mix. The same programming was utilised for calculation of raw mixture composition under different conditions of operating variables.

Ignition and process of sintering

The wet mix was then poured into the sinter box and filled upto the top level. Before pouring ^{Heaters} 2 kg. of -25+15mm size sinter was poured into the box and used as bedding layer. The gas burner was ignited over the raw mix surface and the suction fan switched on. The igniting flame was held for two minutes and then put out. The suction during the ignition was maintained at 500-800mm of WG and then increased to 1000mm of WG as soon as the burner was put out. The exhaust temperature and suction were noted every half a minute. The temperature of the wind box gradually noted and reached a maximum and started decreases indicating the completion of the sintering. The sinter was then allowed to cool under suction till the temperature of exhaust gas dropped to 100° C. The sinter block was then taken out and broken to -50mm size. The quality of -50+10mm size material was weighed and taken as the finished sinter. The quantity of -10mm size material was also weighed and taken as return fines produced to be used for the next tests.

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Tumbling test :

11.3 kg. of -50+10mm size sinter was tumbled for 200 revolutions in a 91.4 cm diameter X 45.7 cm width drum with two Nos. 5 cm high lifters, fitted diametrically opposite to each other and rotating at 25 rpm. The material was then screened on a 1/4th in screen and the under-size was passed again through a 28 mesh screen, percentage of +1/4 mm. Size fraction was taken as the tumbling index and percentage -28 mesh size fraction as the abrasion index.

The tumbling index was used as the main criterion for comparing the qualities of sinter produced under various conditions. The values of the tumbling index, effects and co-efficients of variables are given in Table 3.

Table 3: Calculation of effects and co-efficients
Response : % Tumbling Index

Sl. No.	Tumbling Index	Response % I.D.- 60	Variable	Co-effi- cient	Effect
1.	64.5	4.5	Mean	63.625	63.625
2.	66.5	6.5	A	1.450	2.900
3.	62.2	2.2	B	- 0.025	- 0.050
4.	64.4	4.4	AB	- 0.300	- 0.600
5.	59.3	- 0.7	C	- 0.775	- 1.550
6.	64.3	4.3	AC	0.400	0.800
7.	62.7	2.7	BC	1.075	2.150
8.	65.1	5.1	ABC	- 0.350	- 0.700

So the regression equation can be developed in this form.

$$Y_{\text{strength}} = 63.625 + 1.45(A) - 0.025(B) - 0.30(AB) - 0.775(C) + 0.4(AC) + 1.075(BC) - 0.35(ABC) .$$

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Test for the significance of the co-efficients

Since the design matrix is orthogonal, all regression co-efficients can be estimated independently and the estimate of the co-efficient variance are the same

$$Sb_j^2 = \frac{Se^2}{\sum_{i=1}^N X_{ij}^2} = \frac{Se^2}{N}$$

$$\text{Or } Sb_j = \frac{Se}{\sqrt{N}}$$

Se, the experimental error is calculated in the following manner :

$$Se^2 = \frac{\sum_{i=1}^{n_0} (Y_i^0 - \bar{Y}^0)^2}{n_0 - 1}$$

Where Y_i^0 = Response at base level

\bar{Y}^0 = Average of responses at base level

n_0 = No. of observations at base level

N = Total no. observations at design point

Significance of the co-efficients can be tested using the student t- test

$$t_0 = \frac{|b_0|}{|Sb_0|} ; t_1 = \frac{|b_1|}{|Sb_1|} \dots\dots$$

For a significance level of $p = 0.05$ and $f = n_0 - 1$ degrees of freedom, if the t value is less than the tabulated value of the Student t distribution $t_p(f)$, the corresponding co-efficient is insignificant and must be removed from the equation.

Test for adequacy of equation
(Fisher, F - test).

Fisher F-test was adopted for the adequacy of the equation. For this Sr^2 was determined by the following :

$$Sr^2 = \frac{(Y_{\text{expt}} - Y_{\text{cal}})^2}{N - l}$$

Where Y_{expt} = Strength of each experiment.

Y_{cal} = Calculated strength.

N = Number of total experiment in the matrix.

l = Number of ^{significant} coeffs.

$$\text{Fisher } F = \frac{Sr^2}{Sy^2}$$

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Students. T-test

Combination	bo	A	B	AB	C	AC	BC	ABC
Co-efficient	63.625	1.45	0.25	0.3	0.775	0.4	1.075	0.35

Now three replicate observation at base level with responses. Tumbling indices at base level are 63.8, 62.6 and 63.0.

$$s_y^2 = \frac{(63.8 - 63.1)^2 + (62.6 - 63.1)^2 + (63.0 - 63.1)^2}{3 - 1}$$

$$= 0.3733$$

$$= 0.611$$

$$= \frac{0.611}{\sqrt{8}}$$

$$= 0.216$$

∴ s_y
∴ s_{b_j}

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Co-efficient values were divided by Sb_j value. Any value is less than the 4.3 ($t, 0.5, 2$) from statistical table value i.e. probability points of the T- distribution can be eliminated from the equation (i.e. the co-efficients in the equation as they are significant at the 5% point). Hence the significant co-efficients are as follows :

B_0, A and BC .

Fisher, F-test

% Strength, $Y_{cal} = 63.625 + 1.45A + 1.075 BC$
 calculative strength can be determined by this equation.

$$\begin{aligned} S_r^2 &= \frac{7.79}{8-3} \\ &= 1.558 \\ \text{Fisher, } F &= \frac{S_r^2}{S_y^2} = \frac{1.558}{0.3737} \\ &= 4.17 \end{aligned}$$

Where $4.17 < 5.79$.

The value 5.79 ($t, 0.5, 2, 5$) is the probability point of the variance ratio (F- distribution) as they are significant at 5.0% point.

Therefore model can be accepted in its given form.

plots were drawn to the effects and interactions of the factors. From the plots, it is clear that blue dust is not a significant factor in the ranges of study. Hence, it is possible to eliminate this factor so as to confine ourselves to a more compact and better design that would enable us to fit the model.

Hexagonal design and development of a mathematical model

Hexagonal design was the adopted to optimise the process variables.

A general model represented in the following form :

$$Y = B_0 + B_1X_1 + B_2X_2 + B_{11}X_1^2 + B_{22}X_2^2 + B_{12}X_1X_2$$

where X_1 and X_2 are the two variables. B_0 , B_1 and B_2 the first order least square co-efficients. B_{11} and B_{22} are the second order coefficients while B_{12} is the cross product co-efficient representing interaction between factors. An orthogonal experimental design by definition is one in which all of the main effects and interactions can be estimated without entanglement. The design points along with a geometrical representation are shown in Table 4. The range of the variables is also given and they are coded to cover the range of + 1

to -1. The experimental points derived after coding were tested at randoms at a fixed blue dust of 40%.

Table 4: Hexagonal design

X_1 = % Coke breeze, range 4.5-7.5

X_2 = % Moisture, range 5.0-9.0

<u>Test No.</u>	<u>Design level</u>		<u>Factor level</u>		<u>Response Strength</u>
	X_1	X_2	Coke	Moisture	
12.	1.000	0.0	7.50	7.0	66.8
13.	0.500	0.866	6.75	8.2	65.5
14.	- 0.500	0.866	5.25	8.2	63.0
15.	- 1.000	0.000	4.50	7.0	56.0
16.	- 0.500	- 0.866	5.25	5.8	63.5
17.	0.500	- 0.866	6.75	5.8	65.8
18.	0.000	0.000	6.00	7.0	64.1
19.	0.000	0.000	6.00	7.0	63.8
20.	0.000	0.000	6.00	7.0	63.0
21.	0.000	0.000	6.00	7.0	63.0

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$$\text{Code } X_1 = \frac{\text{Amount of coke} - 6.0}{1.5}$$

$$\text{Code } X_2 = \frac{\text{Amount of moisture} - 7.0}{2.0}$$

Regression equation was developed from hexagonal experiments.

$$\% Y_{\text{strength}} = 63.55 + 4.4 X_1 - 0.23 X_2 - 2.28 X_1^2 + 1.94 X_2^2 + 0.08 X_1 X_2$$

Differentiating the fitted models with respect to each of the variables and equating the derivatives to zero.

The points so obtain are given below :

$$X_1 = 7.20$$

$$X_2 = 7.24$$

The above points lie within the experimental regions of the design.

Sintering at optimised level

Sintering tests were then conducted with the level of coke breeze and water as predicted by optimisation. Blue dust was kept at 40% in the tests. Test results are given in Table 5.

Table 5: Results of sintering at optimised level

Test No.	<u>Factor level</u>		<u>Response</u>
	Coke	Moisture	% Strength
22.	7.20	7.24	66.4
23.	7.20	7.24	66.5

C o n c l u s i o n :

- 1) Statistical design of experiments is satisfactory method for quantifying the effect of various parameters in sintering.
- 2) Of the three variables, coke breeze contributes the most to the strength of sinter. Blue dust does not add to the strength properties in the range of composition studied.
- 3) Regression equations for strength have been developed for sinter. These equations can be utilised to design experiments to get desired properties.
- 4) Hexagonal design of experiments is a satisfactory method for quantifying the effect of various parameters.
- 5) The equation obtained by suitable choice of the various parameters, corrected by T-test are found to be adequate by the F-test.

R E F E R E N C E S

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