## OPTIMIZATION OF SOME PARAMETERS OF HIGH TENSION ROLL SEPARATOR TO RECOVER TITANIFERROUS PLACER MINERALS

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#### Abstract

In this study, a three-level Box-Behnken factorial design combined with response surface methodology (RSM) for modeling and optimizing of some process parameter of high tension roll separator for the separation of titanium bearing minerals (ilmenite and rutile) was developed. The three significant operational parameters of HTRS, which were feed temperature, feed rate and roll speed, were varied and the results evaluated with the Box-Behnken factorial design, Response Surface Methodology and also Quadratic Programming (QP). Second-order response functions were produced for grade and recovery and separation efficiency of the titanium bearing minerals in the conducting fraction. Taking advantage of the quadratic programming, it is observed that maximum grade of 98.76 % can be achieved at 123 rpm roll speed, 40 kg/h feed rate and 140°C temperature. Maximum recovery of 99.06 % can be achieved at 180 rpm, 60 kg/h feed rate and 140°C temperature. Similarly, maximum separation efficiency of 98.81% can be achieved at 120 rpm, 51.6 kg/h and 140 °C temperature using QP technique. Predicted values of responses obtained using model equations were in good agreement with the experimental values ( $R^2$  value of 0.98 for grade,  $R^2$  value of 0.97 for recovery and  $R^2$  value of 0.93 for separation efficiency).

**Keywords:** Box-Behnken design; response surface methodology; optimization; high tension roll separator; titanium bearing minerals.

# 1. Introduction

The bad lands topography of Ganjam Dist, Orissa, India, possesses red sediments. The red sediments are potential resources for heavy mineral concentration which contain maximum percentage of ilmenite followed by the other minerals such as rutile, zircon, monazite, sillimanite etc [1]. In most of mineral separation plants recovery of total heavy minerals is the first step by using spiral concentrators. This pre-concentrate is subjected to roll separator for recovery of conducting minerals. Electrostatic separation is based on differences in conductivity. There are three basic particle charging mechanisms used for electrical separation of minerals; induction, electrification and ion bombardment [2]. Once the particles charged with different polarities, they can be separated by applying an external electric field.

The parameters that affect the separation in high-tension roll separator are the roll speed, feed rate, temperature of the feed, intensity of the applied potential, splitter division plates, humidity, the feed characteristics such as mineral surface condition and feed size distribution. For a roll with a smaller diameter, a higher rotation speed is required in order to maintain a substantial feed rate. However, at higher rpm coarse non-conducting particles tend to leave the roll surface too early due to increase in the centrifugal force resulting in a large portion of misplaced non-conducting particles in the conductor stream. Similarly, under a low rpm condition, fine conductive particles do not gain enough inertia to be thrown off the roll, resulting in misplacement of the non-conductor stream [3]. Several authors have done research work on

high tension roll separator for understanding the factors that influence the separation [4-7]. Therefore a mixture consists of both conducting and non conducting minerals with variable feed size distribution, it is necessary to adjust the roll speed, temperature and feed rate and to some extent electrode position to optimize the process. The principle of separation of conducting and non conducting minerals in HTRS is shown in Fig. 1.



Fig.1. Principles of operations for High Tension Roll Separator to recover conducting minerals

High tension roll separators are commonly used to upgrade dry mineral mixtures on the basis of different electrical conductivities in which titanium minerals like ilmenite, rutile and leucoxene behave as conducting where as zircon, sillimanite, garnet and monazite behave as nonconducting minerals. The roll speed, where maximum mass flow and therefore maximum yield was produced on-grade, could therefore be rapidly determined [8]. The increase in separation efficiency has resulted in the reduction in the number of treatment stages required to produce final high grade mineral products [9]. The success of concentration by using high tension roll separator depends on the selection of suitable process variables at which the response reaches its optimum for recovery of titaniferrous minerals. RSM has been applied for optimization of yield at a desired ash level in coal flotation [10]. A three-level Box-Behnken factorial design combining with RSM was employed for the modelling and optimizing of three operation parameters on lead flotation [11]. RSM and Box-Behnken design have been applied in the grinding experiments of coal samples [12] and modeling of high tension roll separator for separation of titanium bearing minerals from beach sand [13]. Optimization of process parameters for producing graphite concentrate was also done using RSM by Aslan et al. [14]. Different experimental designs are used to develop models by varying process variables of multi gravity separator for celestite concentrate and chromite concentrate [15, 16]. Response surface methodology was implemented for optimization and modeling of sphalerite flotation from a low-grade Zn-Pb ore. In this case quadratic models were found for the prediction of response variables such as grade, recovery and separation efficiency of zinc [17].

In this present study an attempt is made for recovery of titaniferrous minerals (ilmenite and rutile) from the red sediments by using HTRS and the experimental results are analyzed by using software like MINITAB 6.0.1 and MATLAB 8.1.

#### 2. Materials and methods

#### 2.1. Response surface methodology

Response surface methodology is a collection of statistical and mathematical methods that are useful for the modeling and analyzing engineering problems. In this technique, the main objective is to optimize the response surface that is influenced by various process parameters. Response surface methodology also quantifies the relationship between the controllable input parameters and the obtained response surfaces [18].

The design procedure of response surface methodology is as follows [19]:

- (i) Designing of a series of experiments for adequate and reliable measurement of the response of interest.
- (ii) Developing a mathematical model of the second order response surface with the best fittings.
- (iii) Finding the optimal set of experimental parameters that produce a maximum or minimum value of response.
- (iv) Representing the direct and interactive effects of process parameters through two and three dimensional plots.

If all variables are assumed to be measurable, the response surface can be expressed as follows:

$$y = f(x_1, x_2, x_3, \dots, x_k)$$
(1)

where y is the answer of the system, and  $x_i$  the variables of action called factors.

The goal is to optimize the response variable *y*. It is assumed that the independent variables are continuous and controllable by experiments with negligible errors. It is required to find a suitable approximation for the true functional relationship between independent variables and the response surface. Usually a second-order model is utilized in response surface methodology [18, 19].

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^k \beta_{ij} x_i x_j + \varepsilon$$
(2)

where  $x_1, x_2,..., x_k$  are the input factors which influence the response y;  $\beta_0$ ,  $\beta_{ii}$  (*i*=1,2,...,*k*),  $\beta_{ij}$  (*i*=1,2,...,*k*) are unknown parameters and  $\varepsilon$  is a random error. The  $\beta$  coefficients, which should be determined in the second-order model, are obtained by the least square method. In general Eq. (2) can be written in matrix form.

$$Y = bX + \varepsilon \tag{3}$$

where Y is defined to be a matrix of measured values, X to be a matrix of independent variables. The matrixes b and  $\varepsilon$  consist of coefficients and errors, respectively. The solution of Eq. (3) can be obtained by the matrix approach [18, 19].

$$b = (X'X)^{-1}X'Y$$
(4)

where X' is the transpose of the matrix X and  $(X'X)^{-1}$  is the inverse of the matrix X'X.

### 2.2. Experimental design for HTRS tests

Experimental design is widely used for controlling the effects of parameters in many processes. Its usage decreases number of experiments, using time and material resources. Furthermore, the analysis performed on the results is easily realized and experimental errors are minimized. Statistical methods measure the effects of change in operating variables and their mutual interactions on process through experimental design way [20].

In this study, the Box-Behnken experimental design was chosen for finding out the relationship between the response functions and variables (roll speed, feed rate and temperature for grade, recovery and separation efficiency.

Box–Behnken design [20-25] is rotatable second-order designs based on three-level incomplete factorial designs. The special arrangement of the Box–Behnken design levels allows the number of design points to increase at the same rate as the number of polynomial coefficients. For three factors, for example, the design can be constructed as three blocks of four experiments consisting of a full two-factor factorial design with the level of the third factor set at zero [25].

Box–Behnken design requires an experiment number according to  $N = k^2 + k + c_p$ , where (k) is the factor number and ( $c_p$ ) is the replicate number of the central point [25].

For the three-level three-factorial Box–Behnken experimental design, a total of 15 experimental runs are needed.

The model is of the following form [23]:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3$$
(5)

Where *y* is the predicted response,  $\beta_0$  model constant;  $x_1$ ,  $x_2$  and  $x_3$  independent variables;  $\beta_1$ ,  $\beta_2$  and  $\beta_3$  are linear coefficients;  $\beta_{12}$ ,  $\beta_{13}$  and  $\beta_{23}$  are cross product coefficients and  $\beta_{11}$ ,  $\beta_{22}$  and  $\beta_{33}$  are the quadratic coefficients [23].

The coefficients, i.e. the main effect ( $\beta_i$ ) and two factors interactions ( $\beta_{ij}$ ) have been estimated from the experimental results by computer simulation programming applying least square method using MATLAB.8.1.

#### 2.3. Materials

Red sediment samples were collected from bad land topography showing concentration of heavy minerals derived from the top soil of Ganjam Dist, Orissa, India. These sediment samples were collected in a grid pattern up to the water table level during rainy season. All the samples were thoroughly mixed and prepared a composite sample. Two sub samples were prepared by coning and quartering methods for (i) size analysis and (ii) for scrubbing and de-sliming of large sample. Size analysis of scrubbed sample was carried out using standard sieves. The modal analysis of the red sediment sample shown in Fig. 2 indicate that the sample contain 15.3% slimes, 12.9% quartz , 62.1% ilmenite, 0.5% rutile and 9.2% other heavy minerals including sillimanite, zircon, garnet etc.



Fig.2. Model analysis of red sediment sample of Basanputti village, Ganjam Dist., Orissa

Initially, the representative sample was deslimed by using hydrocyclone. Mineralogical modal analysis of deslimed sample was carried out using a Lecia petrological optical microscope. Powdered scrubbed deslimed feed was subjected to X-ray diffraction (XRD) using PANalytical (X'pert) powder diffractometer, (scan speed-  $1.2^{\circ}$ /min from 6° to 40°, by Mo K $\alpha$  radiation) to identify the mineral phases.

The XRD pattern of deslimed feed sample is shown in Fig.3. The XRD data indicates that the deslimed feed sample contains maximum percentage of ilmenite followed by other minerals such as sillimanite, zircon, rutile, pseudorutile etc.



Fig.3. XRD of deslimed feed sample

Sink float test was carried out for deslimed feed with Bromoform (CHBr<sub>3</sub>; specific gravity 2.89), as a medium for separation of heavier fractions (total heavy minerals) from the lighter. The deslimed sample was subjected to rougher, cleaner and scavenging spirals for recovery of

99.1% total heavy minerals. The concentrate obtained from spiral was preheated to the desired temperature (100°C to 140°C) and then fed to the Carpco high tension roll separator [Model (HT 15, 25, 36) 111-15] of 60 inch. roll diameter and 6 inch length for recovery of conducting minerals (ilmenite and rutile). The variables such as roll speed, feed rate and temperature were maintained, keeping other parameters constant, such as voltage at 25 KV and the splitter position as per the experimental design. Each factor at three different levels was studied. The levels of variables chosen for the Box-Behnken design are given in Table 1.

Tuble 1. The level of variables chosen for the Dox Delinken design						
		Coded variable level				
Variables	Symbol		Centre	High		
		-1	0	+1		
Roll speed ( <i>v</i> ), rpm	$x_1$	120	150	180		
Feed rate ( <i>f</i> ), kg/h	$x_2$	40	50	60		
Temperature ( $t$ ), ° $C$	<i>X</i> 3	100	120	140		

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Table I.	The level	of variables	chosen for	the Box-I	Behnken design

The conducting and non-conducting minerals were collected separately from each experiment and weighed. The conducting fraction mainly contains titanium bearing minerals (ilmenite and rutile) and the non conducting fraction contains mainly zircon and sillimanite. Powdered scrubbed conducting fraction was subjected to X-ray diffraction (XRD) to identify the mineral phases present after HTRS operation.

In the present study, the Box-Behnken factorial design was chosen to find out the relation between the response functions (grade, recovery and separation efficiency) and three variables of HTRS (roll speed, feed rate and temperature). The separation efficiency of the process [18] was calculated using the following form.

$$E = \left(\frac{R - Y}{1 - \frac{f}{ma}}\right) \tag{6}$$

where, E = Separation efficiency in %

R = Recovery of titaniferrous minerals in %

Y = Yield of titaniferrous minerals in %,

f = Conducting minerals, % in the feed

ma = maximum titaniferrous minerals, % in the yield of conducting fraction.

The details of the experimental design of HTRS at different operating variables, levels and results are given in Table 2.

Run	Actual and coded level of variables			Observed results			
no:	$x_1(v)$ , rpm	$x_2(f), kg/hr$	$x_3(t), ^{\circ}C$	Grade,%	Recovery,	Separation	
					%	efficiency,%	
1	180 (+1)	50 (0)	140 (+1)	98.30	96.45	95.98	
2	180 (+1)	40 (-1)	120 (0)	97.25	97.79	95.51	
3	120 (-1)	50 (0)	140 (+1)	95.94	96.41	95.26	
4	150 (0)	50 (0)	120 (0)	98.20	96.26	96.23	
5	150 (0)	60 (+1)	140 (+1)	98.56	97.04	95.45	
6	120 (-1)	40 (-1)	120 (0)	88.45	97.97	96.71	
7	180 (+1)	60 (+1)	120 (0)	98.37	96.22	95.13	
8	150 (0)	40 (-1)	140 (+1)	98.50	96.07	96.07	
9	150 (0)	50 (0)	120 (0)	98.35	96.92	95.22	
10	150 (0)	50 (0)	120 (0)	96.23	97.84	95.96	
11	150 (0)	60 (+1)	100 (-1)	95.78	96.55	96.18	
12	180 (+1)	50 (0)	100 (-1)	98.52	96.07	95.30	
13	150 (0)	40 (-1)	100 (-1)	98.34	96.68	95.21	
14	120 (-1)	60 (+1)	120 (0)	98.78	95.75	94.88	
15	120 (-1)	50 (0)	100 (-1)	98.30	96.74	96.35	

Table 2. Box-Behnken design with actual/coded values and results

## 3. Results and discussion

A three-factor three-coded level Box-Behnken design was used to determine the response for the grade and recovery and separation efficiency of the titanium bearing minerals in the conducting fraction. The roll speed (v), feed rate (f) and temperature (t) were independent variables studied to predict the responses ( $Y_1$ ,  $Y_2$  and  $Y_3$ ).

Using the relationships in Table 2, the coded levels of the variables for each of the experiments in the design matrix were calculated and experimental results obtained as given in Table 3.

Table 3. Box-Behnken design with actual/coded values and results

Expt.	Grade, %		Recovery, %		Separation efficiency, %	
No.	Actual	Predicted	Actual	Predicted	Actual	Predicted
1	97,30	97,33	96,45	96,26	95,98	96,09
2	96,25	96,08	97,79	97,84	95,51	95,49
3	95,94	96,02	96,41	96,50	95,26	95,42
4	97,20	97,33	96,26	96,26	96,23	96,09
5	97,56	97,17	97,04	96,87	95,45	95,21
6	90,46	90,77	97,97	98,05	96,71	96,80
7	97,37	97,29	96,22	96,13	95,13	94,97
8	97,50	97,33	96,07	96,26	96,07	96,09
9	97,35	97,74	96,92	97,09	95,22	95,46
10	95,23	95,09	97,84	97,70	95,96	95,90
11	93,78	93,47	96,55	96,47	96,18	96,10
12	97,52	97,77	96,07	96,11	95,3	95,48
13	97,34	97,51	96,68	96,63	95,21	95,23
14	97,08	97,22	95,75	95,89	94,88	94,94
15	97,30	97,06	96,74	96,70	96,35	96,17

From the experimental results listed in Table 2 and Eq. (4), the second-order response functions representing the titaniferrous minerals grade and recovery and separation efficiency of process. This could be expressed as functions of the feed temperature, feed rate and roll speed. The model equations for grade, recovery and separation efficiency of titaniferrous minerals is given in Eqs. (7), (8) and (9).

Model equation for grade:

$$Y_{1} = 97.33 - 0.85x_{1} - 0.21x_{2} - 0.50x_{3} - 1.65x_{1}^{2} + 0.72x_{2}^{2} - 0.92x_{3}^{2} - 0.25x_{1}x_{2} - 2.65x_{1}x_{3} - 0.33x_{2}x_{3}$$
<sup>(7)</sup>

Model equation for recovery:

$$Y_{2} = 96.26 + 0.55x_{1} + 0.36x_{2} + 0.25x_{3} + 0.01x_{1}^{2} + 0.28x_{2}^{2} + 0.56x_{3}^{2} + 0.24x_{1}x_{2} + 0.43x_{1}x_{3} + 0.13x_{2}x_{3}$$
(8)

Model equation for separation efficiency:

$$Y_{3} = 96.09 + 0.35x_{1} + 0.13x_{2} - 0.001x_{3} + 0.001x_{1}^{2} - 0.79x_{2}^{2} + 0.04x_{3}^{2} + 0.11x_{1}x_{2} + 0.31x_{1}x_{3} + 0.01x_{2}x_{3}$$
(9)

The responses at any regime in the interval of our experiment design could be calculated from Eq. (7-9). Experimental results and predicted values obtained by using Eqs. (7-9) are tabulated in Table 3. The actual and predicted values of responses obtained using model equations (Eqs.7-9) are also presented in Fig. (4-6). Predicted values match with the experimental data points, indicating a good fitness ( $R^2$  value of 0.98 for the grade,  $R^2$  value of 0.97 for the recovery and  $R^2$  value of 0.93 for the separation efficiency.



Fig.4. Relation between experimental and predicted values for grade



Fig.5. Relation between experimental and predicted values for recovery



Fig.6. Relation between experimental and predicted values for separation efficiency

# 3.1. Optimization

The objective of response surface optimization is to find a desirable location in the design space. This could be a maximum, a minimum, or an area where the response is stable over a range of factors. In this research, quadratic optimization technique was used for optimization of response equations using MATLAB 8.1 software.

The results obtained by performing the above method are given below:

(i) Maximum grade of 98.76% of titaniferrous minerals achieved by optimizing the variables at:

- Roll speed: 123 rpm
- ➢ Feed rate: 40 kg/h
- $\blacktriangleright$  Temperature: 140 °C

(ii) Maximum recovery of 99.06% of titaniferrous minerals achieved by optimizing the variables at:

- ➤ Roll speed: 180 rpm
- Feed rate: 60 kg/h
- ≻ Temperature: 140 °C

(iii) Maximum separation efficiency of 96.81% achieved by optimizing the variables at:

- ➢ Roll speed: 120 rpm
- $\blacktriangleright$  Feed rate: 51.6 kg/hr
- ➤ Temperature: 140 °C

### 3.2. Effect of variables on the responses

In order to gain a better understanding of the results, Figs. 7(a-c), 8(a-c) and 9(a-c) show 3D response surface plots, which describe the effect of grade, recovery and separation efficiency with change in variable parameters.



Fig.7(a). Response surface plots showing the effect of roll speed (v) and feed rate (f) on the grade



Fig. 7(b). Response surface plots showing the effect of roll speed (v) and temperature (t) on the grade



Fig. 7(c). Response surface plots showing the effect of feed rate (f) and temperature (t) on the grade

Fig. 7(a) shows the effect of roll speed and feed rate on grade of titaniferrous minerals at the centre level of temperature. It is observed that higher grade is obtained at medium roll speed and the effect of feed rate has little on the grade. Fig. 7(b) shows the effect of roll speed and

temperature on grade of titaniferrous minerals at the centre level of feed rate. It is observed that the highest grade is obtained with at the lowest level of roll speed and the maximum level of temperature. Fig. 7(c) shows the effect of feed rate and temperature on grade of titaniferrous minerals at the centre level of roll speed. It is observed that higher grade is obtained at medium temperature and the effect of feed rate has little on the grade.



Fig.8(a). Response surface plots showing the effect of roll speed (v) and feed rate (f) on the recovery



Fig. 8(b). Response surface plots showing the effect of roll speed (v) and temperature (t) on the recovery



Fig.8(c). Response surface plots showing the effect of feed rate (f) and temperature (t) on the recovery

Fig. 8(a) shows the effect of roll speed and feed rate on recovery of titaniferrous minerals at the centre level of temperature. It is observed that higher recovery is obtained at higher level of feed rate and roll speed. It is due to increase in centrifugal force reduces the multilayer formation and increases the chance of discharging the attained surface charge at the ionization zone to the rotor. Fig. 8(b) shows the effect of roll speed and temperature on recovery of titaniferrous minerals at the centre level of feed rate. It is observed that higher recovery is obtained with increase in temperature and roll speed. Fig. 8(c) shows the effect of feed rate and temperature on recovery is obtained with increase in temperature and roll speed. It is observed that higher recovery is obtained with increase in temperature and roll speed. It is observed that higher recovery is obtained with increase in temperature and feed rate.

Fig. 9(a) shows the effect of roll speed and feed rate on separation efficiency of titaniferrous minerals at the centre level of temperature. It is observed that higher separation is obtained at the centre level of feed rate and higher level of roll speed. Fig. 9(b) shows the effect of roll speed and temperature on separation efficiency of titaniferrous minerals at the centre level of feed rate. It is observed that higher separation efficiency is obtained with increase in temperature and the roll speed has little effect on separation efficiency. Fig. 9(c) shows the effect of feed rate and temperature on separation efficiency at the centre level of roll speed. It is observed that higher separation is obtained at medium feed rate and any level of temperature.



Fig.9(a). Response surface plots showing the effect of roll speed (v) and feed rate (f) on the separation efficiency



Fig. 9(b). Response surface plots showing the effect of roll speed (v) and temperature (t) on the separation efficiency



Fig.9(c). Response surface plots showing the effect of feed rate (f) and temperature (t) on the separation efficiency



Fig.10. XRD pattern of conducting fraction achieved from HTRS

The titaniferrous minerals recovered by using HTRS can be used in pigment industries after suitable pyro-metallurgical/chemical processing methods. The XRD pattern of conducting fraction achieved from HTRS is given in Fig.10. The XRD data indicates that the conducting fraction contains maximum percentage of ilmenite followed by rutile, quartz and pseudorutile.

### 4. Conclusion

The following conclusions are drawn from the experimental and optimazation studies carried out on red sediments for recovery of magnetiferrous titanium placer minerals by optimizing different parameters of HTRS. (i) Three levels Box-Behnken factorial design with response surface methodology could employed successfully for modeling the HTRS. Different models are developed with varying three parameters such as, roll speed, feed rate and temperature.

(ii) In order to accomplish better understanding of the variables of the HTRS on grade, recovery and separation efficiency (process and equipment) in magnetic fraction, the predicted model values could be presented as 3D response surface graphs.

(iii) The regression analysis gives very good result between observed value and predicted value such as,  $R^2$  value of 0.98 for grade,  $R^2$  value of 0.97 for recovery and  $R^2$  value of 0.93 for separation efficiency.

(iv) Using optimization study, it is observed that maximum grade of 98.76% is achieved at 123 rpm, 40 kg/h feed rate and 104°C temperature. Maximum recovery of 99.06% is achieved at 180 rpm, 60 kg/h feed rate and 140 °C temperature. Similarly, maximum separation efficiency of 96.81% is achieved at 120 rpm, 51.6 kg/h and 140 °C temperature.

It is clear from this study, for all three responses such as, grade, recovery and separation efficiency could be increased using RSM and QP optimization techniques.

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