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Research Paper

DEVELOPMENT OF A STATISTICAL MODEL AND OPTIMIZATION OF PROCESS VARIABLES FOR COAL FINES PARTICLES USING OIL AGGLOMERATION TECHNIQUE

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Coal oil agglomeration can be a promising process for recovery of fine coal particles. In the present study, response to the coal-oil-agglomeration process was evaluated using Jamadoba Coal Preparation Plant, Jharkhand, India coal fines of size below 212 μm and 75 μm. The statistical model using two-level full factorial design has been developed to study the effect of process parameters on oil agglomeration process. The effect of the parameters including pulp density, oil dosage, particle size and agglomeration time on the % ash rejection (% AR) and % organic matter recovery (% OMR) were investigated on a laboratory scale using karanja oil as bridging oil. From the studies, it was adduced that low pulp density (3%), high oil dosage (15%), low particle size (75 μm) at 24 min 55 sec are optimum condition to recover organic matter as high as 96% with ash rejection of 60%.

Keywords: Karanja Oil, % Ash Rejection, % Organic matter recovery, Oil Agglomeration

INTRODUCTION

Many countries are dependent on coal for the fulfilment of their energy requirement because of its abundance and easy availability. To meet requirement of energy, lower seam coals has been excavated because of limited resources of high rank coal, using highly mechanized mining excavation of coal, which has led to the generation of large amount of coal fines (Sahinoglu and Uslu, 2013). Indian coals are of inferior quality and difficult to clean, containing high amount of mineral matter, which constitutes ash (Bandopadhyay, 1985).

For finer coal particles, conventional processes such as jigging, dense media cyclones, spiral concentrator, etc., are inefficient. Froth flotation, Flocculation, Oil agglomeration are known processes for the treatment of fines and ultra fines coal particles. Among these known processes, Oil agglomeration can be considered

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as appropriate for treating highly oxidized, low rank coal and particles below 75 µm because of its simplicity and high organic matter recovery (Chary and Dastidar, 2010; Gurses *et al*., 1996) .

Oil agglomeration is based on the differences in surface properties of organic matter (coal particles) and gangue minerals (mineral matter which constitutes ash). The oil selectively adheres to the carbonaceous surfaces and acts as a bridging liquid to gather other oil-coated coal particles and upon collision form into an agglomerate of larger size (Garcia *et al*., 1996). The size of the agglomerates increases with time and the difference in surface hydrophobicity causes the oil to agglomerate preferentially the hydrophobic organic coal material, thus enabling separation of the organic carbon portion from the hydrophilic inorganic mineral matter (Vanangamudi and Rao, 1984; Garcia *et al*., 1994).

The use of experimental design methods is accurate and advantageous in obtaining optimum conditions in a relatively small number of experiments. Design expert software is a powerful and easy to use program for design of experiments. This intuitive software is a most useful for optimization of a process and to determine the main effects and their interactions on a response. In present study, use of full factorial design of experiment where two levels of all variables (pulp density, oil dosage, agglomeration time, and particle size) has been used to investigate the influence of variables on response.

MATERIALS AND METHODS

Coal Sample

Coal sample was taken from Jamadoba Coal Washery, Dhanbad, India. A fine coal sample of approx. 120 kg was taken from coal deposit. The sample collected was below -0.5 mm and subjected to repeated cycle of riffling for proper mixing of sample. The sample was divided by repeated cycle of coning and quartering until one fourth of initial sample was obtained (approx. 30 kg). The sample was subjected to dry grinding

Table 1: Proximate and Ultimate Analysis of Coal Sample (as received basis)

Proximate Analysis				
	As Received Basis	Dry Basis	Dry ash free basis	Dry Mineral Matter Free Basis
% Moisture	2.33	—	—	—
% Ash	34.43	35.25	—	
% Volatile Matter	16.80	17.20	26.56	22.33
% Fixed Carbon	46.44	47.55	73.44	77.67

Ultimate Analysis	
% Carbon	59.18
% Hydrogen	2.08
% Nitrogen	1.79
% Sulphur	0.71

and sieved to obtain samples of coal fines of particles size less than 212 micron and 75 micron. Subsequently, the samples were stored in air tight plastic bags. The characteristic properties of coal as determined by proximate analysis and ultimate analysis are given in Table 1 and size-wise ash analysis of feed sample is given in Table 2. The characteristic properties of oil are tabulated in Table 3.

Experimental

Batch oil agglomeration experiments were performed in rectangular vessel equipped with 16 baffles placed above approx. 10 mm from bottom, with 1.5 litres capacity. An Agitair Flotation cell (Galigher, Model LA-500) was used for the experiments without air addition. For each oil agglomeration test, 1000 mL of freshly prepared slurry was taken in an agglomerating vessel of 1.5 litres capacity of desired pulp density (3% - 9%). The Karanja oil was used in the experiments to analyze the effect of the type of oil at different oil dosage and varying agglomeration time. The experiments were performed at ambient pH of the mixture (5.64). All the experiments were carried out at a fixed agitation speed of 800 rpm,

Table 2: Particle Size Distribution and Ash Analysis of Feed Sample

Size (mm)	Weight %	Cum. Wt.% Passing	% Ash
+0.5	3.57	96.43	37.58
-0.5+0.297	18.42	78.01	37.26
-0.297+0.212	12.61	65.40	36.05
-0.212+0.152	13.02	52.38	37.04
-0.152+0.104	12.48	39.90	35.38
-0.104+0.075	6.12	33.78	34.00
-0.075+0.053	21.32	12.46	31.43
-0.053	12.46	0	29.37
Cum. % Ash	34.43		

Table 3: Characteristics of Oil

Characteristics of oils	
Density (gm/cc)	0.934
Surface Tension (mN/m)	31.48
Oil Water Interfacial Surface Tension (mN/m)	8.48
Viscosity (mm^2/s)	120

Table 4: Experimental Ranges And Levels Of The Factors Used In The Factorial Design

Variables	Coded symbol	Level and Range	
		-1	1
Pulp Density (w/w)	A	3	9
Oil Dosage (w/w)	B	5	15
Agglomeration time (min)	C	10	25
Particle size (mm)	D	0.075	0.212

and conditioning time of 3 min. The agglomerates formed were washed and separated by wet screening. The recovered agglomerates were further washed with water and then with ethanol to ensure removal of oil and dried at 50-55^oC till constant weight was attained. The dried agglomerates were weighed and stored in the plastic bags for further analysis.

The efficiency of this process was based on % Ash Rejection (% AR) and % Organic matter recovery (% OMR) which were calculated by the formulae as below:

% Ash Rejection (% AR)

$$= 100 \times [A_2-(A_1 \times (W_1/W_2))/A_2] \quad \ldots (1)$$

% Organic Matter Recovery (% OMR)

$$= 100 \times (W_1/W_2) \times ((100-A_1)/(100-A_2)) \quad \ldots(2)$$

where, W_1 = weight of agglomerated coal, W_2 = weight of sample coal, A_1 = Ash of agglomerated coal and A_2 = Ash of sample coal.

RESULTS AND DISCUSSION

Statistical Design of Experiments

The use of statistical design is one of the best ways to study process behavior and is advantageous in obtaining optimum conditions in a relatively small number of experiments, low cost of laboratory tests and less time. From the design matrix a regression equation is obtained which highlights the effect of individual variables and their relative importance in the process. A number of factors influencing oil agglomeration such as pulp density, oil dosage, agglomeration time, particle size of feed are to be studied. In this investigation, the design was performed based on the $N=2^n$ equation, where N is the total number of tests; n is the number of variables. In other words, the effects of pulp density, oil dosage, and agglomeration time and particle size were evaluated using 2^4 (four variables at two levels) full factorial design. A total number of 16 experiments were conducted to complete the design.

Mathematical Model

A variance analysis was done to evaluate the significance of the effects and the interactions among the variables. The variable parameters and their range selected have been presented in Table 4 where actual and coded values have been tabulated. The process variables and their respective responses are mentioned in Table 5. experimental data and An effect was considered to be significant if the significance level was greater than 95%.

The regression equation can be represented as:

$$Y = X_0 + X_1A + X_2B + X_3C + X_4D + X_5AB + X_6AC + X_7BC + X_8ABC \quad \ldots (3)$$

Where, X_0 is the global mean or %Yield or %Ash when all parameters are at the base level, X_i is regression coefficients; shows the effect of corresponding parameters.

The effect of individual variables and interactional effect can be estimated from Equations (5-8). To test the significance of each coefficient, Fisher's test (F test) and corresponding P values were carried out at a 95% confidence level and insignificant coefficients were discarded. ANOVA analysis was carried out and as mentioned in Table 6 for % ash rejection and Table 7 for % organic matter recovery. Thus the final regression equation can be represented as:

$$\% AR = 52.99 - 5.04A + 0.36B - 0.34C - 0.39D + 0.62AB + 0.79AC + 0.11AD – 1.24BD – 0.54CD + 0.42ABD \quad \ldots(4)$$

Table 5: Factorial Design Matrix Of Four Variables In Coded Form And Respective Responses

Run No.	Coded Values of Variables				Response 2, % AR		Response 3, % OMR	
	A:PD	B:OD	C:AT	D:PS	Obs. Value	Pred. Value	Obs. Value	Pred. Value
1	1	1	1	-1	51.82	51.71	96.62	96.46
2	-1	1	1	-1	55.09	55.09	98.53	98.73
3	-1	-1	1	-1	58.12	58.15	72.76	72.58
4	1	1	1	1	48.31	48.33	91.95	91.97
5	-1	1	-1	1	56.76	56.45	78.25	78.68
6	-1	-1	-1	1	60.17	60.49	62.05	61.63
7	-1	1	1	1	54.45	54.75	87.37	86.94
8	1	-1	1	1	48.11	48.09	81.96	81.95
9	1	1	-1	-1	47.35	47.33	87.74	87.72
10	1	1	-1	-1	48.21	48.32	91.20	91.35
11	-1	-1	-1	-1	57.39	57.35	68.17	68.35
12	1	-1	-1	-1	44.79	44.70	82.04	81.89
13	1	-1	1	-1	48.06	48.16	86.89	87.04
14	-1	-1	1	1	58.71	58.40	69.26	69.68
15	-1	1	-1	-1	53.82	53.83	94.45	94.25
16	1	-1	-1	1	46.93	46.95	77.61	77.62

Table 6: Analysis of Variance (ANOVA) Table for % Ash Rejection

Source of Variation	Sum of Squares	Degree of Freedom	Mean Squares	F-Value	P-Value
Model	460.73	10	46.07	877.91	<0.0001
A: PD	406.22	1	406.22	7740.55	<0.0001
B: OD	2.04	1	2.04	38.97	0.0015
C: AT	1.82	1	1.82	34.73	0.0020
D: PS	2.39	1	2.39	45.48	0.0011
A*B	6.10	1	6.10	116.25	0.0001
A*C	9.99	1	9.99	190.27	<0.0001
A*D	0.21	1	0.21	3.94	0.1038
B*D	24.50	1	24.50	466.89	<0.0001
C*D	4.67	1	4.67	88.9	0.0002
A*B*D	2.79	1	2.79	53.14	0.0008
Residual	0.26	5	0.052		
Total	460.99	15			
R^2 = 0.9994; R^2 (adj.) = 0.9983, C.V.% = 0.43					

Table 7: Analysis of Variance (ANOVA) table for % Organic Matter Recovery

Source of Variation	Sum of Squares	Degree of Freedom	Mean Squares	F-Value	P-Value
Model	1752.41	11	159.31	3539.25	<0.0001
A: PD	283.75	1	283.75	6303.89	<0.0001
B: OD	947.72	1	947.72	21054.51	<0.0001
C: AT	108.26	1	108.26	2405.20	<0.0001
D: PS	201.07	1	201.07	4467.03	<0.0001
A*B	127.13	1	127.13	2824.23	<0.0001
A*C	0.97	1	0.97	21.55	0.0097
A*D	29.48	1	29.48	655.04	<0.0001
B*D	21.90	1	21.90	486.58	<0.0001
C*D	0.86	1	0.86	19.21	0.0118
A*B*D	28.09	1	28.09	624.05	<0.0001
A*C*D	3.17	1	3.17	70.39	0.0011
Residual	0.18	4	0.045		
Total	1752.59	15			
R^2 = 0.9999; R^2 (adj.) = 0.9996, C.V.% = 0.26					

Figure 1: Effect of process variables on % ash rejection

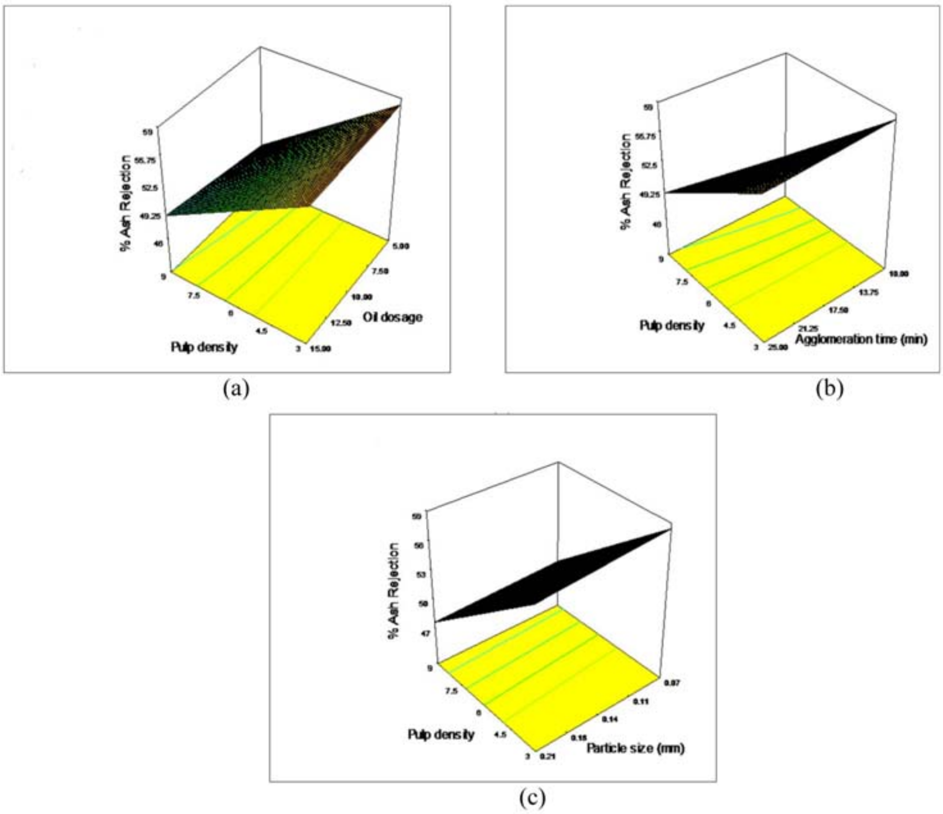
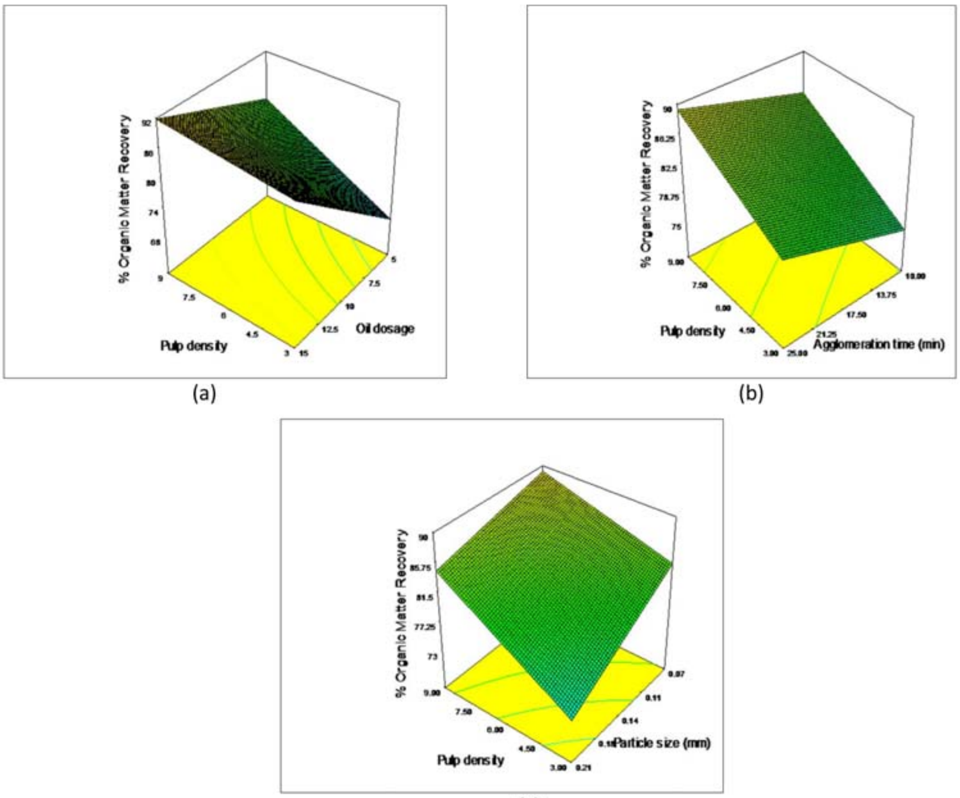
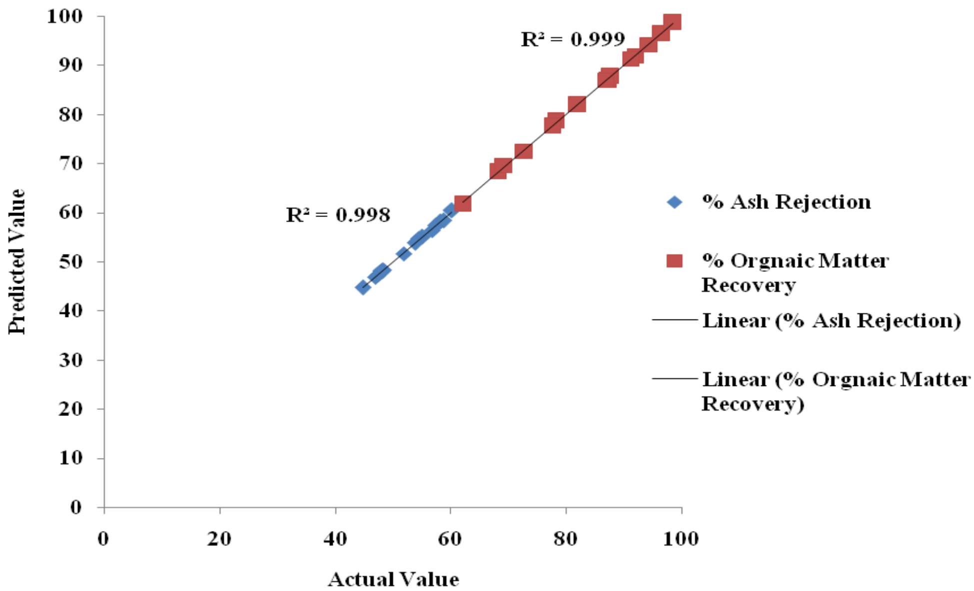


Figure 2: Effect of process variables on % Organic matter recovery



(a) (b) (c)

Figure 3: Comparison between Actual and Predicted values of responses



$$\% OMR = 82.79 + 4.21A + 7.7B + 2.6C - 3.55D - 2.82AB - 0.25AC + 1.36AD - 1.17BD + 0.23CD + 1.33ABD - 0.44ACD \quad ...(5)$$

Note: A: Pulp density; B: Oil dosage; C: Agglomeration time; D: Particle size

Positive relationships in Equations (4-5) are indicating their increase is having synergistic effect on response value. Since the difference between experimental and calculated values of responses is negligible it can be said that regression equation is adequate.

Analysis of Experimental Data and Model

3D response surface plots are helpful in deducing optimum conditions that will maximize/minimize the response variable. It is used to determine the potential relationships between three variables. It displays the 3-D relationships in 2-D dimensions, with x and y factors plotted on the x and y scales and response values represented by response plots. To evaluate an equation, the Fisher's F-test with a very low probability value [(P_{Model} value > F) = 0.0001] indicates that the model is reliable and meaningful.

With increase in pulp density % organic matter recovery is increasing at constant oil dosage and agglomeration time. This can be due to distance between the particle was greater at low solid concentration than that at high solid concentration and amount of oil required for agglomeration is more readily available at lower pulp density. Finer coal particle was found to be more agglomerated as more hydrophobic surface is available in system (Garcia *et al.*, 1995). From Figure 1 (a-c), it was observed that highest ash rejection is achieved at low pulp density, high oil dosage, high agglomeration time and minimum level of particle size. As the results indicates, ash rejection is steadily decreasing with increase in pulp density at constant oil dosage and agglomeration time because of increase in mineral content alongwith clean coal. With increase in particle size ash rejection is decreasing. Similar trend was reported by Garcia *et al.* (1996) and Chary *et al.* (2014).

As Figure 2 (a-c) shows the relationship between pulp denstiy, oil dosage, agglomeration time and paricle size with organic matter recovery. From the results it was adduced that organic matter recovery is increasing with decrease in pulp density, increase in oil dosage and agglomeration time and lower particle size. % OMR has decreased significantly with increase in particle size this may be due to low availability of hydrophobic surface with lower oil dosage availability (Alonso *et al*., 2002).

CONCLUSION

At constant pulp density and oil dosage with increasing agglomeration time following were concluded (1). Increase in pulp density lowers ash rejection, (2). Increase in oil dosage increases ash rejection, and (3). Increase in agglomeration time increase ash rejection. The highest % OMR is observed at lower pulp density (3%), followed by pulp density (6%) and pulp density (9%) when was done at constant oil dosage. As expected, increase in % OMR with decrease in particle size is found due to the better liberation of coal particle from gangue minerals. From study it was found that process variables affecting oil agglomeration are in following order: oil dosage > pulp density > particle size > agglomeration time. The high R^2 value represent that the model obtained is adequate to give response at given range of process variables as represented in Figure 3.

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