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# Optimization of coal flotation using statistical technique

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# Abstract

The flotation studies of non-coking coal fines containing 37.7% ash were carried out. The variables studied were pH, collector, frother, modifier (sodium silicate) and coal size. Experiments were carried out using  $2^5$  full factorial design. The main and interaction effects on recovery and grade were evaluated using Yates' analysis. The optimum flotation condition was found by the method of steepest ascent. A product with 72.62% combustibles (i.e. 25.38% ash) at 88.03% recovery was obtained at pH 7.5, diesel oil 0.42 g/kg, MIBC 0.09 g/kg, sodium silicate 0.02 g/kg and coal size -0.6 mm at optimum conditions. © 2003 Elsevier B.V. All rights reserved.

Keywords: Coal; Flotation; Statistical design; Optimization

# 1. Introduction

Indian coals in general have difficult washing characteristics and have high percentage of near gravity materials in association with their high ash contents due to drift origin. The yields of clean coal obtained by washing such coals are poor and the beneficiation involves high cost. Considerable percentages of co-products like middlings/rejects are also generated during washing. Another problem is that the mineral matter in the bulk of the coal is disseminated very finely. This necessitated the coals to be crushed to smaller sizes prior to washing to maximize recovery. Now with the progressive deterioration in the washability characteristic of the Run-of-Mine coal

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and due to depletion of reserve of better grade coal, crushing to finer sizes has become inevitable. The techniques of coal beneficiation based on static dense medium baths will not be effective at finer sizes. Washing should be carried out using improved techniques of jigging, heavy media cyclones, spirals and froth flotation. During the process of washing, plenty of fine coals (-0.5 mm) are being generated in conventional washery. But washeries adopting Jigging, fines below 1.0 mm are being generated, which are having high ash. These fines are to be beneficiated before mixing with the clean coal.

Commercial viability of fine coal flotation, slurry jigs, oil agglomeration, oleoflotation and spiral concentration are to be established for Indian coals [1]. With the deterioration of the quality of feed coal and increase in production of low quality fines, beneficiation of fines is essential. Froth flotation is the well-established process for fine coal cleaning. It has been reported that loss of good quality coal in fine coal washing is 10 times more than that of coarse coal washing [2]. Recovery of that good coal fines will be immensely helpful in improving economic performance of coal washery. Considerable research work has been done to improve the performance of coal flotation by different worker by using advanced techniques like packed column, Jameson cell, column cell and micro cell [3,4]. All these studies were concentrated to recover coal fines from coking coal washeries. However, not much work was reported on the flotation of non-coking coal fines generated during washing. Keeping in view of the latest developments in cleaning of non-coking coal, studies were initiated on the flotation of non-coking coal.

One of the most effective techniques to study process behavior is the factorial designed test with analysis of variance [5-9]. There are several advantages of statistical design of experiments over classical one variable at a time method, where one variable is varied at a time. In statistical design, experiments can be conducted in an organized manner and can be analysed systematically to obtain much needed information. The information can be utilized for optimization purpose.

The objective of the work was to determine the effects of variables using statistical techniques on flotation of high ash non-coking coal used in thermal power plant and find out optimum condition for its flotation. The different steps of optimization strategy used in this study are:

- 1. To design experimental tests (using factorial design).
- 2. To perform an analysis of the experimental results by ANOVA to determine the significant factors influencing the flotation process.
- 3. To find out optimum conditions for flotation of coal to reduce ash content.

# 2. Materials and methods

# 2.1. Materials

Non-coking coal forming feed to the existing thermal plants was collected from Talcher area for beneficiation studies. The fines (-1.0 mm) were generated during crushing of coal

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Size, mm	Weight, %	Ash, %
- 1.0+0.6	33.6	36.57
-0.6+0.3	29.4	36.87
-0.3 + 0.15	12.3	38.85
$-0.15 \pm 0.075$	11.9	39.53
$-0.075 \pm 0.045$	5.2	39.79
-0.045	7.5	39.88
Total	100	37.7

Table 1 Size and ash distribution of coal fines used

for preparation of feed (-13.0 to 1.0 mm) for jigging. The -13.0+1.0 fraction was treated by Jigging and the fines (-1.0 mm) were used in these studies. The size distribution and ash content of each fraction are given in Table 1. The proximate analysis and washability characteristics of the sample are given in Table 2 and Table 3 respectively. From the fines generated, two size fractions of -0.6 and -1.0 mm were prepared for the flotation studies. Commercial grade light diesel oil was used as collector and MIBC was used as frother. The pH was adjusted with NaOH and sodium silicate was used as dispersant/depressant of silica.

# 2.2. Methods

Denver D12 sub-aeration flotation machine with 1-l capacity cell was used for flotation studies. A total of 100 g of coal was mixed with 300 ml of water and conditioned in flotation cell for 2 min. The pH of the slurry was adjusted using NaOH before it was conditioned with sodium silicate and diesel oil. Another 600-ml make up water was added and conditioned further with predetermined quantity of MIBC (frother). The conditioning

Table 2Proximate analysis of sample

Sl. No.	Details	Weight, %
1	Moisture	7.5
2	Ash	37.7
3	Volatile matter	29.4
4	Fixed carbon	32.9

Table 3 Washability characteristics of sample

Specific gravity	Yield, %	Ash, %
1.5F	43.0	12.8
1.6F	53.2	16.8
1.7F	61.4	20.7
1.8F	70.8	25.8
1.9F	78.9	27.7
1.9S	21.1	76.0

F: float, S: sink.

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Variables	Code	Low level	Base level	High level	Step
		(-1)	(0)	(+1)	size
PH	А	7.0	7.5	8.0	0.5
Collector (diesel oil), g/kg	В	0.084	0.126	0.168	0.042
Frother (MIBC), g/kg	С	0.06	0.09	0.12	0.03
Modifier (sodium silicate), g/kg	D	0	0.02	0.04	0.02
Size, mm	Е	0.6	0.8	1.0	0.2

1 a					
2 <sup>5</sup>	factorial	design	for	coal	flotation

Cell rpm: 1500.

time for all the reagents was 2 min in each stage, then flotation was carried out by releasing the air at 1500 rpm for 5 min. Both the froth and tailings were collected separately, dried and analysed for ash percentage. Recovery was calculated as

Recovery, 
$$R = \frac{(\% \text{ wt product}*\% \text{ non ash material in the product})}{\% \text{ non ash material in the feed}}$$

The variables considered in the study are: pH, collector(diesel oil), frother (MIBC), modifier (sodium silicate) and feed particle size. The levels of variables studied are given in Table 4.

#### 3. Results and discussions

The statistical design of experiments is used when the effect of several factors are to be studied in order to determine the main and interaction effects. The effect of a variable is the change in response produced by varying the level of the factor. When the effect of a factor depends on the level of another factor, the two factors are said to interact. In the present work, five variables were taken into consideration to evaluate their main and interaction effects on the recovery of fine coal into float fraction and its grade in order to study the separation of ash forming material from coal. In other words, the main goal has been to establish best set of variables that could be used in floation to obtain maximum recovery in float fraction with acceptable grade.

A  $2^5$  full factorial unreplicated experiments were carried out in order to evaluate the main and interaction effects of variables in flotation of coal. Yates' notation has been used in this work to name each treatment [10]. For example, treatment abc is the experimental run in which the variables A, B and C are set at their high level whereas the variables D and E are at their low level. To study the main and interaction effects of the variables on the recovery and grade of coal, a Yates' analysis and analysis of variance have been carried out [10]. The total variance (total mean square) of a factorial experiment can be divided into several sources using Yates' analysis. In case of unreplicated experiments, all the variance is subdivided between the effects. A  $2^5$  experiment has  $(2^5-1)$  degree of freedom, and Yates' analysis divides the total variation in the results into the 31 effects. It follows that each effect has one degree of freedom;

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Table 4

hence, for any effect, the mean square equals the sum of squares. In Yates' analysis, the standard addition and subtraction in pairs is carried out by n times for *n* factors, then the numbers in the *n*th column of analysis are twice the effect. The Yates' analysis and analysis of variance for recovery and grade are given in Tables 5 and 6, respectively. The test of statistical significance of each effect necessitates estimation of experimental error. As the design did not incorporate replications, no direct estimate of standard error of an effect was available for Student's *t*-test. In this type of situation, the general practice is to use high order interactions or the interactions with relatively low values of the effects can be used to estimate the experimental error [10]. In this work, the highest (fifth) order interaction was assumed due to error; consequently, all the effects less than the fifth order interaction were considered due to error. The fifth order interaction and interactions less than that are squared and the squares are added and divided by the total of their degrees of freedom to give an estimate of  $S_e^2$  [11]. The calculation of  $S_e^2$  for recovery is given below.

$$S_{e}^{2} = (eff_{ABCDE}^{2} + eff_{BCDE}^{2} + eff_{ACE}^{2} + eff_{ABE}^{2} + eff_{ACD}^{2} + eff_{ABD}^{2} + eff_{ABC}^{2})/7$$
  
= 0.469

$$S_{\rm e} = 0.645$$

From this estimated value for the standard error of an effect, the standard error of an observation and the standard error of the average of observations were estimated.

The ANOVA was carried out to evaluate if the effect and the interaction among the investigated factors are significant (Tables 5 and 6). An effect is considered to be significant if its significance level is greater than 95%. The following was observed from the analysis results.

#### 3.1. Recovery

The main effect of all the variables on recovery is significant at 95% confidence level. The order of influence is E>B>A>D>C. The effect of size (E) is highly significant and negative. This indicates higher particle size (>0.6 mm) is not suitable for flotation. The variables pH, collector, frother and sodium silicate (variables A, B, C and D, respectively) have positive effect. They also increase recovery interacting positively (BD, CD, BCD, ABCD), but they decrease recovery interacting negatively (AB, AC, BC, AD). The variable E, which has negative effect, interacts with the variables A, B, C and D, and increases recovery with positive interaction of BCE, ADE and ABCE, and decreases with negative interaction of AE, BE, CE, DE, BDE, CDE and ADCE.

# 3.2. Grade

The main effect of all the variables on grade of coal produced by flotation is significant at 95% confidence level. The most important effect is C (frother). Its effect is negative. The effects of A and D are also negative, whereas that of C and E are positive. The order

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Table 5					
Results	of statistical	analysis	on res	sponse	(recovery)

TC	Recovery,	y, Yates' analysis					Divisor	Effects	Identification	t = effect/	Significan
%	%	1 <sup>a</sup>	2	3	4	5				Se	
(1)	17.77	55.27	139.56	286.01	662.81	1071.1	32	33.47	Ave		
a	37.50	84.29	146.45	376.80	408.30	105.85	16	6.62	А	9.66	99.9
b	29.22	78.35	155.81	207.00	66.43	118.51	16	7.41	В	10.82	99.9
ab	55.07	68.10	220.99	201.30	39.42	-48.43	16	-3.02	AB	-4.42	99.5
с	27.48	60.26	96.49	65.57	78.43	80.27	16	5.02	С	7.33	99.9
ac	50.87	95.55	110.51	0.86	40.08	-75.07	16	- 4.69	AC	-6.85	99.9
bc	35.75	98.31	103.56	8.12	-26.63	- 49.41	16	- 3.09	BC	-4.51	99.5
abc	32.35	122.68	97.74	31.3	-21.80	-15.79	16	-0.97	ABC	-1.44	NS
d	28.52	41.27	45.58	18.77	72.07	85.09	16	5.32	D	7.77	99.9
ad	31.74	55.22	19.99	59.66	8.20	- 41.53	16	-2.60	AD	-3.79	99.5
bd	46.77	52.01	5.17	20.44	-35.07	40.09	16	2.51	BD	3.66	99.5
abd	48.75	58.50	- 4.31	19.64	-40.00	8.67	16	0.54	ABD	0.79	NS
cd	49.06	48.93	11.63	-20.67	-50.19	38.45	16	2.40	CD	3.51	99.5
acd	49.25	54.63	- 3.51	- 5.96	0.78	6.39	16	0.40	ACD	0.58	NS
bcd	63.59	41.90	28.08	-7.88	- 36.33	44.05	16	2.75	BCD	4.02	99.5
abcd	59.09	55.84	3.22	-13.92	20.54	42.63	16	2.66	ABCD	3.89	99.5

е	16.28	19.73	29.02	6.89	90.79	-254.5	16	- 15.91	Е	- 23.23	99.9
ae	24.99	25.85	-10.25	65.18	-5.70	-27.01	16	- 1.69	AE	-2.46	95
be	26.15	23.39	35.29	14.02	-64.71	-38.35	16	-2.40	BE	-3.50	99.5
abe	29.07	-3.40	24.37	-5.82	23.18	4.83	16	0.30	ABE	0.44	NS
ce	26.36	3.22	13.95	-25.59	40.89	-63.87	16	- 3.99	CE	- 5.83	99.9
ace	25.65	1.95	6.49	-9.48	-0.80	-4.93	16	-0.31	ACE	-0.45	NS
bce	30.65	0.19	5.70	-15.14	14.71	50.97	16	3.19	BCE	4.65	99.5
abce	27.85	-4.50	13.94	-24.86	-6.04	56.87	16	3.55	ABCE	5.19	99.9
de	13.60	8.71	6.12	-39.27	58.29	-96.49	16	-6.03	DE	-8.81	99.9
ade	35.33	2.92	-26.79	-10.92	-19.84	87.89	16	5.49	ADE	8.02	99.9
bde	24.14	-0.71	-1.27	-7.46	16.11	-41.69	16	-2.61	BDE	- 3.81	99.5
abde	30.49	-2.80	-4.69	8.24	-9.72	-20.75	16	-1.30	ABDE	- 1.89	NS
cde	20.51	21.73	-5.79	-32.91	28.35	-78.13	16	-4.88	CDE	- 7.13	99.9
acde	21.39	6.35	-2.09	-3.42	15.70	-25.83	16	- 1.61	ACDE	-2.36	95
bcde	26.75	0.88	-15.38	3.70	29.49	-12.65	16	-0.79	BCDE	- 1.15	NS
abcde	29.09	2.34	1.46	16.84	13.14	-16.35	16	-1.02	ABCDE	-1.49	NS

<sup>a</sup> (1)+a, b+ab.....bcde+abcde are 1, 2....16th entry, respectively.

TC: treatment combination, Ave: average, NS: non-significant.

 $S_{\rm e} = 0.645$ , degrees of freedom: -7.

Treatment	Grade, carbon, %	Effects	Identification	Calculated $t = \text{effect}/S_{e}$	Significance
(1)	78.34	75.96	Average		
a	74.46	-1.14	A	- 11.85	99.5
b	77.85	0.45	В	4.66	97.5
ab	74.04	0.59	AB	6.18	97.5
с	75.31	-1.18	С	- 12.29	99.5
ac	74.39	0.77	AC	8.04	99
bc	78.11	0.90	BC	9.39	99
abc	77.02	-0.49	ABC	- 5.14	97.5
d	77.38	-0.84	D	-8.76	99
ad	77.52	0.58	AD	6.03	97.5
bd	74.02	-0.46	BD	-4.80	97.5
abd	76.24	0.05	ABD	0.50	NS
cd	72.95	-0.66	CD	-6.90	97.5
acd	73.17	-0.61	ACD	- 6.34	97.5
bcd	73.90	0.25	BCD	2.64	NS
abcd	73.80	-0.14	ABCD	- 1.49	NS
e	78.67	0.86	Е	8.96	99
ae	74.57	-0.23	AE	-2.44	NS
be	77.89	0.26	BE	2.76	NS
abe	77.30	0.39	ABE	4.02	95
ce	75.91	0.22	CE	2.30	NS
ace	75.77	0.34	ACE	3.56	95
bce	75.83	-0.67	BCE	-6.97	99
abce	76.64	-0.16	ABCE	-1.70	NS
de	78.71	0.48	DE	4.97	97.5
ade	74.57	-0.94	ADE	- 9.84	99
bde	77.18	0.49	BDE	5.07	97.5
abde	76.07	-0.18	ABDE	-1.92	NS
cde	75.55	0.77	CDE	8.06	99
acde	74.53	0.38	ACDE	3.98	95
bcde	76.87	0.26	BCDE	2.79	NS
abcde	76.19	0.13	ABCDE	1.32	NS

 Table 6

 Results of statistical analysis on response (grade)

NS: non-significant.

 $S_{\rm e} = 0.096$ , degrees of freedom: -2.

of their significance is C>A>E>D>B. The AB, AC, BC, AD, ABE, ACE, DE, BDE, CDE and ACDE interactions have positive effect, whereas that of ABC, BD, CD, ACD, BCE and ADE have negative effect on grade of float fraction.

The main effects of variables on recovery and grade are shown in Fig. 1. The surface charge of a substance changes with the change in pH. The increase in pH may be conducive for flotation of an intimately associated ash forming minerals or it may increase negative charge of both coal particles and slimes causing dispersion. The adsorption of collector on coal is possibly due to hydrophobic bonding [12]. The contact angle, which is related to hydrophobicity of coal particles, depends upon the ash-forming mineral content [13]. The particles having less ash forming minerals are likely to be enveloped first by collector. The increase in recovery and grade may be attributed to this. The sodium silicate



Fig. 1. Main effects of variables on recovery and grade. 1 = pH, 2 = collector, 3 = frother, 4 = sodium silicate, 5 = size.

is a well-known dispersant. It disperses slimes coated on coal surfaces and exposes hydrophobic surface to the system. This helps less hydrophobic particle to float, which otherwise would not have floated. The increase in recovery and decrease in grade is due to this. With the increase in particle size, the mass increases which decreases recovery drastically. Only a few high-grade coal particles float due to its high degree of hydrophobicity causing increase in grade.

The interaction of sodium silicate with collector (BD) and frother (CD) has positive effect on recovery. This is due to dispersion of slimes from coal surfaces and effective interaction of collector and frother, which increases the flotability. The decrease in grade is due to flotation of less hydrophobic high ash particles.

The interaction of pH with collector (AB) has negative effect on recovery an positive effect on grade. The industrial grade hydrocarbon oils are negatively charged with no observable isoelectric point [14,15]. Most of the coals are usually negatively charged or at most neutral at pH value above 5.0 [16]. The increase in pH increased the negative charge of both collector droplets and coal particles, and hindered interaction between them, which caused decrease in recovery. The increase in grade of the float indicated that the high ash particles are first to be affected. The same effect of pH and frother interaction (AC) on recovery and grade as that of AB interaction may be due to similar effect stated above. The decrease in recovery and increase in grade due to pH and sodium silicate interaction (AD) may be due to depression of high ash content particle at higher pH.

Coal can be floated only with the frother. In the presence of both collector and frother, the effect of each other may be less than what it would have been in the absence of other. The decrease in recovery due to collector and frother interaction (BC) may be attributed to this reason. The increase in grade indicates that the effect of frother is much limited in the presence of collector. It is to be mentioned that collector (B) has positive effect on grade, whereas frother (C) has negative effect on grade.

The decrease in recovery due to AE, BE, CE and DE interactions is due to increase in mass of particles. The AE, BE and CE interactions do not affect the grade significantly, whereas DE interaction has positive effect on grade. This increase in grade may be due to

dispersion of slimes from coarse particles and flotation of only highly hydrophobic highgrade particles.

Only the main effect of collector has positive effect on both recovery and grade. The BCD and ABCE interactions increase recovery without effecting grade significantly, whereas the ACE interaction increases the grade without effecting the recovery significantly. All the other significant effects increase the recovery with the decrease in grade or vice versa.

Statistical models can be built up for prediction of grade and recovery using Yates' analysis data (Tables 5 and 6). These models can be used to perform analysis of the residues to check the assumption on the experimental error distribution of the factorial designs [17]. The models formed for recovery and grade of coal using the effects of variable significant at 95% or more confidence level are given below:

$$Y_{\text{RE}} = 33.47 + 1/2(6.62\text{A} + 7.41\text{B} - 3.03\text{AB} + 5.02\text{C} - 4.69\text{AC} - 3.09\text{BC}$$
  
+ 5.32D - 2.60AD + 2.51BD + 2.40CD + 2.75BCD + 2.66ABCD  
- 15.91E - 1.69AE - 2.40BE - 3.99CE + 3.19BCE + 3.55ABCE  
- 6.03DE + 5.49ADE - 2.61BDE - 4.88CDE - 1.61ACDE) (1)

 $R^2 = 0.99$ 

$$Y_{GR} = 75.96 + 1/2(-1.14A + 0.45B + 0.59AB - 1.18C + 0.77AC + 0.90BC - 0.49ABC - 0.84D + 0.58AD - 0.46BD - 0.66CD - 0.61ACD + 0.86E + 0.39ABE + 0.34ACE - 0.67BCE + 0.48DE - 0.94ADE + 0.49BDE + 0.77CDE + 0.38ACDE)$$
(2)

 $R^2 = 0.96$ 

where  $Y_{\text{RE}}$  and  $Y_{\text{GR}}$  are recovery and grade of coal, respectively. A, B, C, D and E are expressed in coded form -1 and +1.

The coefficient of determination,  $R^2$ , is used to check the models ability to predict the response (recovery or grade) accurately. It is determined from the following equation:

$$R^{2} = 1 - \left[ \left\{ \sum (y_{i} - y_{i}^{\wedge})^{2} \left\{ \sum (y_{i} - y_{i}^{-})^{2} \right\} \right]$$

Where  $y^{\wedge}$  is the predicted response variable and  $y^{-}$  is the mean experimental value. If  $R^2$  is 1, then the prediction is nearly perfect. However, if  $R^2$  becomes zero, the model has little value. The empirical models were found to accurately estimate the response variable as indicated by  $R^2$  value (0.99 and 0.96 for recovery and grade, respectively). The residual analysis for grade and recovery is given in Fig. 2.

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Fig. 2. Residual analysis for recovery and grade.

# 3.3. Optimization

One of the techniques of optimization is the method of steepest ascent, in which the base point is assumed and the next set of values is selected, which is proportional to product of the coefficient and step size. The selected values are incremented

Table 7Evaluation of variables for optimum recovery

Variable	pH (A)	Collector (B), g/kg	Frother (C), g/kg	Sodium silicate (D), g/kg
Principal level, $Z_{i0}$	7.5	0.126	0.09	0.02
Increment, $\Delta Z_i$	0.5	0.042	0.03	0.02
Coefficient, $b_i$	3.31	3.705	2.51	2.66
$\Delta Z_i^* b_i$	1.655	0.15561	0.0753	0.0532
Normal steps	0.66	0.062	0.03	0.0212

Table 8Optimization of recovery for 0.6 mm coal

Variables		Response			
pH (A)	Collector (B), g/kg	Frother (C), g/kg	Sodium silicate (D), g/kg	Recovery, %	Grade, %
7.5	0.126	0.09	0.02	63.35	75.79
8.16	0.188	0.12	0.0412	78.57	75.85
8.82	0.25	0.15	0.0624	79.54	74.5
9.48	0.312	0.18	0.0836	80.22	74.27
10.14	0.374	0.21	0.1048	84.02	74.11

Grade=(100 - ash %).

Variable collector (B), g/kg	Response			
	- 0.6 mm coal		- 1.0 mm coal	
	Grade, %	Recovery, %	Grade, %	Recovery, %
0.126	75.79	63.35	74.98	44.87
0.252	74.83	72.19	73.22	53.44
0.420	74.62	88.03	73.00	66.65
0.588	73.64	86.50	72.92	64.21

Table 9	
Optimization of grade of coal	

pH: 7.5, frother: 0.09 g/kg, sodium silicate: 0.02 g/kg. Grade=(100 - ash %).

successively and objective function is evaluated each time till the optimum point is reached.

In this work, our objective was to maximize recovery at desired level of grade of coal. Eqs. (1) and (2) were used to determine increment size for recovery and grade, respectively. As the effect of size (E) on recovery is highly negative, the size is kept at low level for optimization experiment. The variables having positive effects were increased according to increment size (Table 7) and evaluated by carrying out successive experiments. The result obtained with their variables is given in Table 8. Eq. (2) indicates that the effects of collector (B) and size (E) are positive, whereas that of pH (A), frother (C) and sodium silicate (D) are negative. The variables having negative effects were kept at base level and optimization experiments were carried out for both low (-0.6 mm) and high (-1.0 mm) particle sizes. There is only one variable (B) left for optimization and the increment size was chosen accordingly and experiments were conducted. The results of the experiments are given in Table 9. The optimum condition was found to be at pH 7.5, frother 0.9 g/kg, sodium silicate 0.02 g/kg. At this condition, a product with 76.62% coal (i.e. 25.38% ash) at 88.03% coal recovery was obtained.

# 4. Conclusions

The fifth order interaction was assumed due to error. Consequently, all the other effects less than that were considered due to error and the experimental error was estimated to carry out *t*-test. The residual analysis and  $R^2$  value indicate that the assumption is quite satisfactory. The main effects of all the variables on recovery and grade are significant at 95% confidence level. The effect of size on recovery is highly significant and negative. The variables pH, collector, frother and sodium silicate have positive effects. The effects of pH, frother and sodium silicate on grade are negative, whereas that of size and collector are positive. The collector is the only variable having positive effect on both grade and recovery. A product having 25.38% ash with 88.03% recovery of coal could be obtained at pH 7.5, diesel oil 0.42 g/kg, frother 0.09 g/kg, sodium silicate 0.02 g/kg and size -0.6 mm.

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# References

- [1] J.P. Panda, J.P. Singh, Fine coal washing, Indian Mining and Engineering Journal 39 (9) (2000) 25–28.
- [2] P. Yeriswamy, D.P. Patil, J.P. Barnwal, B. Govindrajan, T.C. Rao, Evaluation of different flotation techniques treating Indian coal fines, Indian Mining and Engineering Journal 39 (9) (2000) 40–43.
- [3] P.S.R. Reddy, S.G. Kumar, K.K. Bhattacharya, S.R.S. Sastry, K.S. Narasimhan, Flotation column for fine coal beneficiation, IJMP 24 (1988) 161–172.
- [4] M.C. Harris, J.P. Franzidis, A.W. Breed, D.A. Degion, An on site evaluation of different flotation technologies for fine coal beneficiation, Minerals Engineering 7 (5/6) (1994) 699–714.
- [5] P.K. Naik, Quantification of induced roll magnetic separation of mineral sands, Scandinavian Journal of Metallurgy 31 (6) (2002) 367–373.
- [6] P.K. Naik, L.B. Sukla, S.C. Das, Aqueous SO<sub>2</sub> leaching studies on nishikhal manganese ore through factorial experiment, Hydrometallurgy 54 (2000) 217–228.
- [7] R.N. Sahoo, P.K. Naik, S.C. Das, Extraction of manganese from low grade manganese ore using oxalic acid as reductant, Hydrometallurgy 62 (3) (2001) 157–163.
- [8] P.K. Naik, L.B. Sukla, S.C. Das, Application of statistical design in the leaching study of low grade manganese ore using aqueous sulfur dioxide, Separation Science and Technology 37 (6) (2002) 1375–1389.
- [9] P.K. Naik, L.B. Sukla, S.C. Das, Extraction of manganese from low grade nishikhal ore using pyritiferous lignite in acidic medium, Minerals and Metallurgical Processing 19 (2) (2002) 110–112.
- [10] L. Davies, Efficiency in research, development, and production, The Statistical Design and Analysis of Chemical Experiments, The Royal society of Chemistry, Cambridge, UK, 1993, p. 180.
- [11] S. Agatzini, A.R. Burkin, Statistical approach to the precipitation of iron as goethite, Transactions-Institution of Mining and Metallurgy. Section C. Mineral Processing and Extractive Metallurgy 94 (1985) 105–114.
- [12] S.K. Mishra, Improved recovery of fine coal by flotation process, in: S.K. Mishra, R.R. Klimpel (Eds.), Fine Coal Processing, Noyes Publications, USA, 1987, pp. 110–135.
- [13] A.M. Gaudin, Flotation, 2nd ed., McGraw-Hill, New York, 1957, p. 573.
- [14] W.W. Wen, Electrokinetic behaviour of flotation of oxidized coal, PhD thesis, The Pennsylvania University, Cited in 12 (1978).
- [15] M. Salwaroski, Examination of adsorption of hydrocarbonfuel oil at coal and shale surfaces, Graduate Diploma thesis, W.A. Institute of Technology, Cited in 12 (1981).
- [16] F.F. Aplan, Coal flotation, chapter 45, in: M.C. Fuerstenau (Ed.), Flotation-A.M. Gaudin Memorial Volume, vol. 2, American Institute of Mining, Metallurgical and Petroleum Engineers, Inc., New York, 1976, pp. 1235–1264.
- [17] D.C. Montgomery, Design and Analysis of Experiments, 3rd ed., Wiley, Singapore, 1991.