Journal of Mining and Metallurgy, 41 A (2005) 11 - 25

FLOC FLOTATION STUDIES OF ULTRA FINE SILICEOUS MANGANESE ORE BY LINEAR ORTHOGONAL SATURATED DESIGN

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(Received 2005; accepted 10 October 2005)

Abstract

Effect of fifteen variables on hydrophobic floc flotation characteristics of ultra fine (-10 µm) siliceous manganese ore in mechanical flotation cell has been studied. Linear orthogonal saturated design was selected to study the main effect of 15 variables. The regression model was developed to quantify the effects of variables and attempt was made to optimize grade and recovery of beneficiation of low-grade siliceous manganese ore. The effect of independent variables on response has been explained wherever possible. The maximum grade obtained was 42.3 % Mn at 38.2 % Mn recovery. The maximum Mn recovery was 78.1 % at 34.9 % Mn grade from the feed having 27.8% Mn.

Key words: *Hydrophobic floc flotation, manganese ore, fractional factorial design.*

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1. Introduction

Flotation is one of the most efficient as well as complex minerals processing operation. It is affected by a very large number of variables and many of these are beyond the control of the mineral engineer, and some can not be even measured quantitatively. When the effect of variables is not well understood the statistical design of experiments is an important tool to understand and quantify the variables. The statistical design of experiments is dealt by many authors (Cochran & Cox, 1957; Daniel, 1976; Box et al, 1978; Akhanazarova & Kafarov, 1982) and these techniques have been used to study the flotation of minerals by Yalsin, 1999; Rao & Mohanty, 2002; Cilek & Yilmazer, 2003 and Martinez et al, 2003.

In Joda, Orissa huge quantity of finely disseminated low-grade siliceous manganese ore are produced as fines during the mining of high grade manganese ore which is being sold at nominal rate or dumped at the mine site for future use. Such ores require fine grinding for liberation and the available knowledge can not be utilized for the up gradation of ground low grade ore. In the last few decades, lot of work has been carried out on beneficiation of ultra fine particles. During this period different types of flotation techniques have been developed to treat ultra fine particles and slimes. Among these hydrophobic floc flotation technique has been considered to be a potential means for the beneficiation of ultra fine low-grade ores (Song, S. and Lopez-Valdivieso, A., 2002). Generally a special flotation cell is necessary to float fine (-10 μm) ore particles.

In the present work attempt is made to float the fine ore in the conventional mechanical flotation cell by flocculating the ore fines prior to flotation by floc flotation technique, as super fines could not be floated in conventional cells. The main objective was to quantify the effect of different operating variables and optimize the grade and recovery.

2. Experimental

2. 1. Materials

The low-grade siliceous manganese ore was collected from Roida area, Keonjhar district, Orissa, India. The ore was crushed by jaw crusher and roll crusher to obtain a product of -1.0 mm. This product was ground in a ball mill for 45 minutes. The milled product was subjected to hydrocyclone to separate minus 10 micron particles. The hydrocyclone overflow, which cannot be treated by conventional flotation technique, was taken for study. The sample of 50 gm each was prepared and kept in a sealed polythene packet for carrying out the experiments. The size analysis was carried out by MALVERN laser particle size analyzer. The size distribution and major components of the material is given in Table 1. The main minerals present are pyrolusite, hematite and quartz. The ore contains on average 27.8 % Mn, 6.23 %Fe2O3 and 43.35 % acid insoluble.

Table 1(a). Size distribution of the sample used
Specific surface area: 2.8671×106 m2/m3

Size µm	Wt, %
- 10.1 + 7.88	0.3
- 7.88 + 6.15	35.5
- 6.15 + 4.83	12.8
- 4.83 + 3.80	15.4
-1.3.8 + 0	36.2

Table 1(b). Chemical characteristics of the sample

Constituent	Wt (%)
MnO_2	43.99
Fe_2O_3	6.23
Acid Insoluble	43.35

2. 2. Flotation experiments

Flotation studies were carried out in Denver D12 subaeration flotation machines. Fifty gram of manganese ore was taken into the 1l flotation cell and 400 ml water of predetermined temperature was added into it. Conditioning was started and the solution containing $Na_2Si0_3.9H_2O$, $Fe(NO_3)_3.9H_2O$ and H_2SO_4 were added to the slurry and conditioned. Predetermined quantity of sodium hydroxide, manganese chloride tetra hydrate, sodium oleate and kerosene were added successively and conditioned. The temperature of the slurry was maintained through out the experiment by addition of hot water. The make up water was added, air was released and the concentrate was collected as float.

3. Results and discussions

The full factorial experiments are carried out with all the possible combination of factors at all levels involved. In such cases the number of observations (N) to be performed is given by (Akhanazarova and Kafarov, 1982)

$$N = n^k \tag{1}$$

Where n is the number of levels and k is the number of factors. In the present study 15 variables with two levels have been selected. The variables selected and their levels are given in Table 2. The full factorial design will require 32768 numbers of observations, which is practically difficult to carry out.

Table 2. Experimental conditions. Particle size : -10.1 µm

Sl. No.	Variable	Code	Low level (-)	Base level (0)	High level (+)
1	$Na_2SiO_3 . 9H_2O$, g/t	X_1	40	60	80
2	$Fe(NO_3)_3 . 9H_2O$, g/t	X_2	5	7.5	10
3	Sulphuric acid, g/t	X_3	16.56	24.84	33.12
4	Sodium oleate, g/t quantity	X_4	400	600	800
5	Sodium oleate, %, w/v concentration	X_5	0.2	0.3	0.4
6	Kerosene, g/t	X_6	1800	2700	3600
7	Sodium hydroxide, g/t	X_7	40	60	80
8	$MnCl_2 .4H_2O$, g/t	X_8	0	179.9	359.8
9	Temperature, ^0C	X_9	28	33	38
10	Agitation, rpm	X_{10}	900	1000	1100
11	Conditioning time for $X_1+X_2+X_3$, min	X_{11}	5	7.5	10
12	Conditioning time for collector, min	X_{12}	3	4.5	6
13	Conditioning time for kerosene, min	X_{13}	3	4.5	6
14	Conditioning time for sodium hydroxide, min	X_{14}	3	4.5	6
15	Conditioning time for $MnCl_2 .4H_2O$, min	X_{15}	3	4.5	6

The optimization by the method of steepest ascent necessitates only linear terms. If the linear approximation is adequate for the purpose for which the experiments are carried out, the amount of experimentation needed may be curtailed using a fractional factorial design. It is a design in which some level of combinations can be omitted. This leads to loss of some information on interaction of factors. The number of observations should be greater than or equal to that of unknown coefficients in the regression equation. When the number N of observations is equal to the number of coefficients (k+1) to be determined for a linear regression equation, the design is called linear saturated orthogonal design (Akhanazarova and Kafarov, 1982).

Linear saturated orthogonal design was selected to study the effect of fifteen flotation variables (Table 2) with defining relation of the design as $X_5 = X_1X_2X_3$, $X_6 = X_1X_2X_3X_4$, $X_7 = X_1X_4$, $X_8 = X_2X_3$, $X_9 = X_1X_2X_4$, $X_{10} = X_1X_3X_4$, $X_{11} = X_2X_3X_4$, $X_{12} = X_2X_4$, $X_{13} = X_3X_4$, $X_{14} = X_1X_3$, $X_{15} = X_1X_2$. This is nothing but 1/2048th replicate fractional factorial design of a 2^{15} full factorial design. The coded variables and the result showing recovery of manganese are given in Table 3.

Table 3. Design matrix for 15 factor linear orthogonal saturated design

Sl. No	X_0	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	X_{12}	X_{13}	X_{14}	X_{15}	R*
1	+	-	-	-	-	-	+	+	+	-	-	-	+	+	+	+	7.95
2	+	+	-	-	-	+	-	-	+	+	+	-	+	+	-	-	17.87
3	+	-	+	-	-	+	-	+	-	+	-	+	-	+	+	-	8.70
4	+	+	+	-	-	-	+	-	-	-	+	+	-	+	-	+	12.45
5	+	-	-	+	-	+	-	+	-	-	+	+	+	-	-	+	11.12
6	+	+	-	+	-	-	+	-	-	+	-	+	+	-	+	-	12.54
7	+	-	+	+	-	-	+	+	+	+	+	-	-	-	-	-	22.04
8	+	+	+	+	-	+	-	-	+	-	-	-	-	-	+	+	9.66
9	+	-	-	-	+	-	-	-	+	+	+	+	-	-	+	+	38.21
10	+	+	-	-	+	+	+	+	+	-	-	+	-	-	-	-	18.75
11	+	-	+	-	+	+	+	-	-	-	+	-	+	-	+	-	22.14
12	+	+	+	-	+	-	-	+	-	+	-	-	+	-	-	+	16.61
13	+	-	-	+	+	+	+	-	-	+	-	-	-	+	-	+	19.24
14	+	+	-	+	+	-	-	+	-	-	+	-	-	+	+	-	21.24
15	+	-	+	+	+	-	-	-	+	-	-	+	+	+	-	-	27.59
16	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	32.96

R : Recovery, * : Average of two experiment (i. e. average of column Y_{R1} and Y_{R2} of Table 4)

The experiments were conducted in replicate to estimate error. The regression equation for the matrix (Table 3) may be represented as:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 + b_6X_6 + b_7X_7 + b_8X_8 + b_9X_9 + b_{10}X_{10} + b_{11}X_{11} + b_{12}X_{12} + b_{13}X_{13} + b_{14}X_{14} + b_{15}X_{15} \quad (2)$$

Where Y = recovery/grade, b's = empirical model coefficients and X's = dimensionless coded factor of variables. The relation between the coded and actual variables are given as $X_1 = (x_1\text{-}60)/20$ …..etc
Where x_1 = actual variable ($Na_2Si0_3.9H_2O$) value

The regression coefficients were estimated by
$b_0 = (\Sigma Y\text{-}_i)/N$ and $b_j = (\Sigma X_{ji} Y\text{-}_i)/N$
Where i = index for observation, j = index for variable.

The regression equation developed for recovery of manganese is given below

$$Y_R = 18.674 - 0.949 X_1 + 0.345 X_2 + 0.840 X_3 + 5.918 X_4 - 1.119 X_5 - 0.201X_6 - 1.253 X_7 + 3.204 X_8 + 2.312 X_9 + 3.580 X_{10} + 1.518 X_{11} - 0.112X_{12} - 0.175X_{13} + 0.465 X_{14} - 0.150X_{15}$$

Where Y_R: Recovery of manganese and $X_1 \ldots X_{15}$: dimensionless coded variables correspond to variables given in Table 2.

Different terms were tested for significance. Some of the calculations adopted are given in Table 4.

Table 4. Results of fractional factorial experiments.

Sl No	Y_{R1}	Y_{R2}	Y^{-}_{R}	S_R^2	$Y^{\wedge}_{R}$	$(Y^{-}_{R}- Y^{\wedge}_{R})^2$	Y_{G1}	Y_{G2}	Y^{-}_{G}
1	7.42	8.47	7.95	2.21	8.58	0.41	38.38	34.00	34.19
2	17.57	18.17	17.87	0.72	17.81	0.00	35.30	35.70	35.50
3	8.37	9.03	8.70	0.87	8.41	0.08	32.88	32.38	32.63
4	12.80	12.10	12.45	0.98	12.86	0.17	34.40	34.86	34.63
5	11.50	10.74	11.12	1.16	11.01	0.01	38.49	38.85	38.67
6	12.54	11.98	12.54	0.63	12.25	0.00	37.60	38.18	37.89
7	22.37	21.71	22.04	0.87	21.81	0.06	40.70	41.02	40.86
8	9.94	9.38	9.66	0.63	9.32	0.11	33.40	33.78	33.59
9	38.52	37.90	38.21	0.77	37.87	0.12	42.20	42.80	42.27
10	19.00	18.5	18.75	0.50	18.51	0.06	37.40	37.86	37.63
11	22.5	21.78	22.14	1.04	22.13	0.00	39.13	39.37	39.25
12	16.87	16.35	16.61	0.54	16.50	0.01	38.40	38.60	38.50
13	19.42	19.06	19.24	0.26	19.65	0.17	31.75	31.10	31.43
14	21.50	20.98	21.24	0.54	20.95	0.08	36.00	36.26	36.13
15	27.88	27.3	27.59	0.67	27.53	0.00	38.60	38.90	38.75
16	32.66	33.26	32.96	0.72	33.60	0.41	41.05	41.45	41.25

Y: response, $Y\text{-}_R$: average response (recovery),
S_{Ri}^2: variances, Y^Ri : predicted results
S^2_{Rmax}= 2.21 and $_{I=1}^{N} S^2_{Ri} = 13.10$
S^2_{Rmax}: maximum variance for manganese recovery.

Cochran criteria (G) is

$G = (S^2_{Rmax} / \Sigma_{I=1}^{N} S^2_{Ri}) = 2.21/13.1= 0.168$

Where S^2_{Rmax}: Maximum variance of manganese recovery and S^2_{Ri}: Variances of manganese recovery.

For significance level $\alpha = 0.05$ and the number of degree of freedom 1 = 1, 2 = 16, the value for Cochran criterion is
$G_{0.95}(1,16) = 0.4709$
As $G < G_{0.95}(1,16)$, the variances are homogenous. Thus the error mean square (S^2_{Re} may be found as the mean.

$S^2_{Re} = 1/N * \Sigma_{I=1}^{N} S^2_{Ri} = 13.10/16 = 0.819$

The number of degree of freedom (γ) of error mean square is
$\gamma = N(m-1) = 16\ (2-1) = 16$, where m = number of replication
$S_{bj} = (S^2_{Re}/2*16)0.5 = 0.16$
t-value = coefficient/ S_{bj}

The tabulated value of Student t-test is $t_{0.05}(16) = 2.12$. The t-values less than 2.12 indicate that the corresponding terms are insignificant.

On deleting the terms not significant at 95 percent confidence level the equation developed for manganese recovery becomes

$$Y_{MnR} = 18.674 - 0.949\ X_1 + 0.345\ X_2 + 0.840\ X_3 + 5.918\ X_4 - 1.119\ X_5 - 1.253\ X_7 + 3.204\ X_8 + 2.312\ X_9 + 3.580\ X_{10} + 1.518\ X_{11} + 0.465\ X_{14} \quad (3)$$

The adequacy of fitting of the equation is tested by F-test (Akhanazarova and Kafarov, 1982). The adequacy mean square (S^2_g) is given by

$S^2_g = \{2\Sigma(y\text{-}_1\text{-}y\hat{}_1)^2\}/16\text{-}12 = 2*1.694/4 = 0.847$

The Fisher's variance ratio (F) is

$F = S^2_g / S^2_e = 0.847/0.819 = 1.035$

With $\alpha = 0.05$, $\gamma = 4$, the tabulated value of Fisher's F is $F_{0.95}$ (4,16) = 3.0, As $F < F_{0.95}$ (4,16) the equation fit the experimental data adequately.

The order of effect of variables on manganese recovery is
quantity of sodium oleate added > Agitation > MnCl2.4H2O > Temperature > Conditioning time for X_1, X_2 and X_3 > Sodium hydroxide > Concentration of sodium oleate > $Na_2SiO_3.9H_2O$ > Conditioning time for collector > $Fe(NO_3)_3.9H_20$ > Sulphuric acid

Similarly the equations for grade of manganese is found out.

$$Y_{MnG} = 37.087 +0.345\ X_2+0.233X_3+1.092\ X_4 - 0.844\ X_5 +0.395\ X_7 +0.946\ X_8 +0.482\ X_9 +1.512X_{10}+0.907X_{11} +0.913\ X_{12}-1.524\ X_{13}-0.243\ X_{15} \quad (4)$$

The regression coefficients are biased estimate of main effect and confounded interaction effects. The residual analysis of Eq 3 and 4 are given in Fig. 1. The significant effects on recovery and grade have been plotted in Fig. 2.

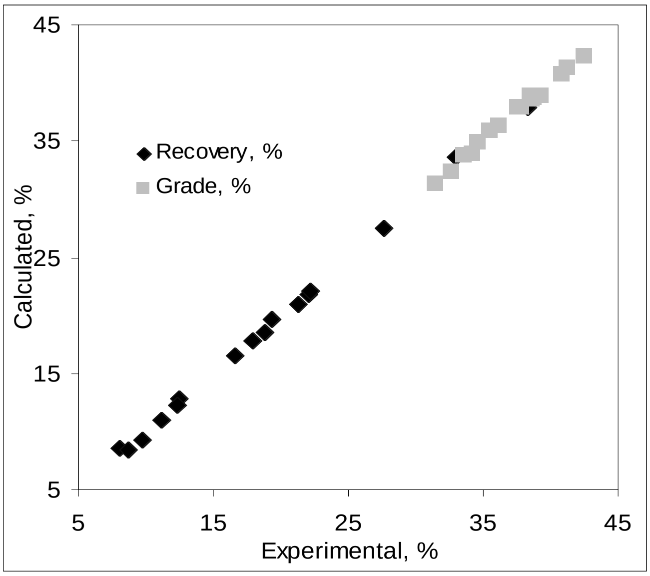

Fig. 1. Residual analysis

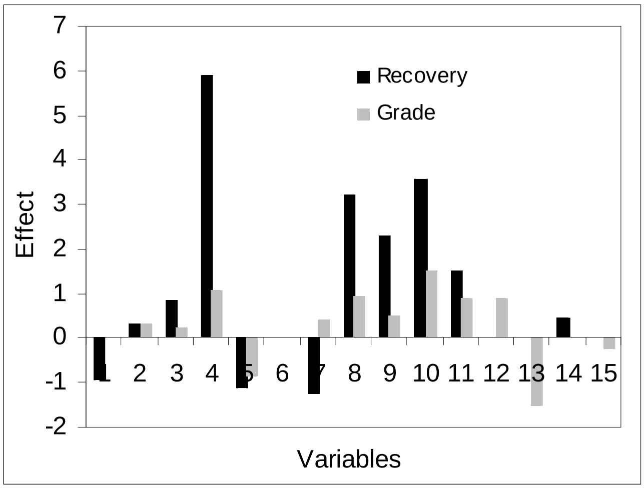

Fig. 2. Effect of variables on grade and recovery, 1…..15

Subscripts of variables given in Table 2.

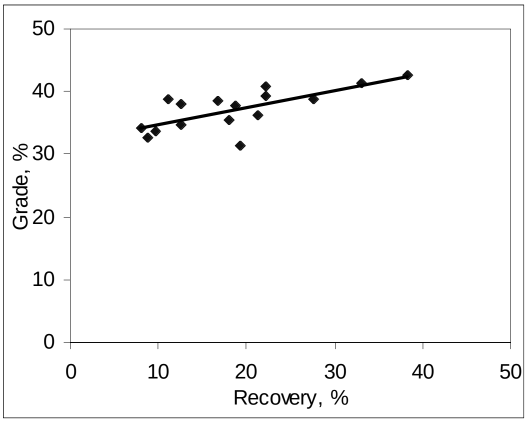

Fig. 3. Grade and recovery relation

3.1. Qualitative analysis

The grade and recovery relationship is depicted in Fig. 3. The increase in both grade and recovery indicated that the minerals are in liberated form. When the ore was subjected to sink-float analysis with bromoform as heavy medium everything is reported to sink. This indicates that the particles are intimately associated with each other due to electrostatic force of attraction. The dispersion of particles prior to flotation is essential. Even if the particles are dispersed effectively it cannot be floated in mechanical cell because the air bubbles generated are too large for effective particle-bubble collision and attachment. So it is necessary to aggregate the valuable minerals selectively to float them in mechanical cell. The role of variables in possible dispersion and aggregation has been discussed in following paragraphs. When both the grade and recovery of manganese increases or decreases (Fig. 2) it can be interpreted with certainty that the change in grade is due to change in recovery. When grade and recovery shows opposite effect the change in grade may be due to change in other components (gangue).

In the flotation cell agitation and high temperature helped to keep the minerals in dispersed state. The hydrophobic floc formation (Song and Lu, 1994) and chemisorption (Puri et al, 1985) is facilitated with the increase in temperature. There is an increase in both grade and recovery with the increase in temperature. To enhance the dispersion further a solution containing sodium meta silicate, ferric nitrate nonahydrate and sulphuric acid was added. In conventional flotation generally one dispersant is used. The $Na_2SiO_3{\cdot}9H_2O$ decrease the recovery of manganese. There is increase in both grade and recovery due to $Fe(NO_3)_3{\cdot}9H_2O$ and H_2SO_4. The manganese chloride tetrahydrate was added so that Mn^{2+} could adsorb on manganese minerals and hydrate which is suitable for adsorption of oleate anion. There is increase in both grade and recovery due to addition of manganese chloride tetrahydrate. The grade and recovery increase with the increase in quantity of sodium oleate and decrease with the increase in its concentration. The sodium oleate was diluted to 0.2 % (w/v) to prevent micelle formation, which favored adsorption of mono layer of oleate ion instead of multi layer. Kerosene was added to further improve the hydrophobicity of the oleate ion adsorbed manganese mineral particles and to promote floc formation and to increase floc strength (Song and Lopez-Valdivieso, 2002).

3.2. Optimization study

Attempt was made to optimize manganese recovery by the method of steepest ascent. The optimized process conditions were evaluated using eq (3). The non- significant variables and variables having negative effect were kept at low level. The increments decided are given in Table 5. The results of experiment are given in Table 6. Similarly optimization experiments were carried out for grade using eq (4) and the results are given in Table 7. The conditions of experiment No. 21 was considered optimum where 78.1 % recovery was obtained with 34.9 % Mn grade. The decrease in recovery beyond this condition (Expt. No. 22) is possibly due to high rate of agitation (1500 rpm) because increases of other variables are not likely to decrease recovery. This is supported by decrease in grade and recovery in optimization experiment of grade at 1500 rpm (Expt. No. 28). The best grade obtained is at conditions of experiment No. 9 of fractional factorial experiment where a grade of 42.3 % is obtained with 38.2 % recovery.

$Na_2SiO_3{\cdot}9H_2O$, g/t: 40
Sodium oleate concentration, %(w/v): 0.2
Kerosene, g/t: 1800
Sodium hydroxide, g/t: 40.0
Conditioning time for collector, min: 3
Conditioning time for kerosene, min: 3
Conditioning time for sodium hydroxide, min: 3
Conditioning time for $MnCl_2{\cdot}4H_2O$, min: 4.5

Table 5. Evaluation of optimized process conditions for recovery of manganese

	$Fe(NO_3)_3{\cdot}9H_20$, g/t	H_2SO_4, g/t	Sodium oleate, g/t	$MnCl_2{\cdot}4H_2O$, g/t	Temp., ^0C	Agitation, rpm	Cond. for X_1, X_2, X_3, min
Base level (Zj)	7.5	24.84	600	179.9	33	1000	7.5
Increment (ΔZj)	2.5	8.28	200	179.9	5	100	2.5
Coefficient (bj)	0.345	0.84	5.918	3.204	2.312	3.58	1.518
bj. ΔZj	0.8625	6.9552	1183.6	576.4	11.56	3.58	3.795
Normal steps	0.241	1.943	330.615	161.0	3.23	100	1.06

Cond.: Conditioning time

$Na_2SiO_3{\cdot}9H_2O$, g/t: 40
Sodium oleate concentration, %(w/v): 0.2
Kerosene, g/t: 1800
Sodium hydroxide, g/t: 40
Conditioning time for collector, min: 3
Conditioning time for kerosene, min: 3
Conditioning time for sodium hydroxide, min: 3
Conditioning time for $MnCl_2{\cdot}4H_2O$, min: 4.5

Table 6. Optimization experiment for recovery of manganese

Expt.No	$Fe(NO_3)_3{\cdot}9H_20$, g/t	H_2SO_4, g/t	Sodium oleate, g/t	$MnCl_2{\cdot}4H_2O$, g/t	Temp., ^0C	Agitation rpm	Cond. for X_1, X_2, X_3, min	Manganese	
								Recovery	Grade
17	7.5	24.84	600	179.9	33	1000	7.5	41.36	39.47
18	7.741	26.783	930.62	340.9	36.23	1100	8.56	50.15	38.32
19	7.982	28.726	1261.23	501.9	39.46	1200	9.62	59.54	37.63
20	8.223	30.669	1591.85	662.9	42.69	1300	10.68	69.79	36.25
21	8.464	32.612	1922.46	823.9	45.92	1400	11.74	78.06	34.88
22	8.705	34.555	2253.08	984.9	49.15	1500	12.80	72.20	32.63

Cond.: Conditioning time

$Na_2SiO_3{\cdot}9H_2O$, g/t: 40
Sodium oleate concentration, %(w/v): 0.2
Kerosene, g/t: 1800
Conditioning time for kerosene, min: 3
Conditioning time for sodium hydroxide, min: 3
Conditioning time for $MnCl_2{\cdot}4H_2O$, min: 3

Table 7. Optimization experiment for grade of manganese

Expt No	$Fe(NO_3)_3{\cdot}9H_20$, g/t	H_2SO_4 g/t	Sodium oleate, g/t	NaOH	$MnCl_2{\cdot}4H_2O$ g/t	Temp., ^0C	Agita-tion, rpm	Cond. X_1, X_2, X_3, min	Cond. Collect-or, min	Manganese	
										Grade	Recov-ery
23	7.50	24.84	600	60	179.9	33.0	1000	7.5	4.5	Not conducted	
24	8.07	26.116	744.444	65.225	292.46	35.53	1100	9.0	5.41		
25	8.64	27.392	888.888	70.45	405.01	36.07	1200	10.50	6.31		
26	9.21	28.668	1033.33	75.675	517.57	37.60	1300	12.00	7.23	32.63	63.78
27	9.78	29.944	1177.78	80.90	630.12	39.14	1400	13.50	8.12	33.25	70.37
28	10.35	31.220	1322.22	86.125	742.68	40.67	1500	15.00	9.03	33.0	51.88

4. Conclusions

The grade and recovery relationship found in hydrophobic floc flotation of ultrafine manganese ore (Fig. 3) is contrary to the normal grade and recovery relationship found in conventional separation techniques, which indicate that the dispersion of particles and aggregation of hydrophobic particles are important. The fractional factorial design is very useful when large numbers of variables are to be studied. In this case the amount of information available and its use is much more than the number of experiments conducted. The optimization experiments are only partially successful because the best grade is not obtained during optimization experiments. During optimization experiment it should be kept in mind that the regression models are valid within the range of variables in which it is studied and equal importance should be given to knowledge of pulp chemistry and hydrodynamic conditions. The maximum manganese recovery obtained was 78.1 % with 34.9 % Mn grade and the maximum obtainable grade was 42.3 % Mn with 38.2 % recovery from siliceous manganese ore (-10 µm) having 27.8 % manganese content.

5. Acknowledgements

The authors are thankful to Director of Regional Research Laboratory, Bhubaneswar for giving permission to present the paper.

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