ISSN NO 2320-5407 International Journal of Advanced Research (2013), Volume 1, Issue 5, 347-355

ISSN NO. 2320-5407

Journal homepage: http://www.journalijar.com

INTERNATIONAL JOURNAL OF ADVANCED RESEARCH

RESEARCH ARTICLE

OPERATION AND OPTIMIZATION OF SILLIMANITE BENEFICIATION FROM BEACH SANDS

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Manuscript Info

Manuscript History:

Received: 12 June 2013
Final Accepted: 27 June 2013
Published Online: July 2013

Key words:
Sillimanite,
Conventional Cells,
froth flotation,
Modeling,
Design of Experimentation

Abstract

Sillimanite is an alumino-silicate mineral with the chemical formula Al_2SiO_5. The Sillimanite mineral is subjected to classification, followed by flotation for separation of Ilmenite, Rutile, Zircon and Garnet. Rejects from which essentially consists of quartz, sillimanite with other in minor qualities needs to be concentrated to achieve higher grade of mineral sillimanite it is necessary to meet the stringent quality requirements of the market.

A typical ROM feed, comprising 8-9% sillimanite along with other heavy minerals, is treated in the Primary Concentration Plant (PCP) and further upgraded in WSC with flotation cells to produce froth concentrate containing 85% sillimanite. In the flotation process, Oleic acid is used as a collector cum frother, Sodium silicate as depressant and Soda ash to regulate pH.

The sillimanite concentrate is then treated in the Mineral Separation Plant (MSP) to produce a saleable final product with >96% sillimanite.

In Trimex Sands Private Limited, Conventional mechanical flotation cells were installed to float sillimanite. In addition to traditional problems associated with conventional cells, higher content of iron oxide (2% against 0.7%) in the final product due to presence of fine garnet complicated the flotation operation there by leading to lower recoveries coupled with inferior grades.

The main aim of the present investigation deals with the two fold objectives (i) To enhance the process operation thereby increasing the recovery of the sillimanite concentrate (ii) To reduce the iron content in the concentrate to produce marketable final product.

Introduction

India is gifted with abundant beach- and dune-placer deposits of strategic and economically important heavy minerals such as Ilmenite, Rutile, Zircon, Monazite, Garnet and Sillimanite. These deposits are mostly located in the coastal stretches of peninsular India. Silica sand deposits are commonly contaminated with various heavy minerals. Sillimanite concentration from other heavy mineral sands to achieve higher quality is gaining much attention because of rapid growth of refractory and ceramic industries.

Sillimanite is an alumino-silicate mineral with the chemical formula Al_2SiO_5. The Sillimanite mineral is subjected to classification, followed by flotation for separation of Ilmenite, Rutile, Zircon and Garnet. Rejects from which essentially consists of quartz, sillimanite with other in minor qualities needs to be concentrated to achieve higher grade of mineral sillimanite it is necessary to meet the stringent quality requirements of the market.

Trimex Sands Pvt Ltd (TSPL), a mineral wing of Trimex Industries, has set up a mining and mineral separation facilities based on the Srikurmam deposit in the district of Srikakulam in Andhra Pradesh in India. In TSPL, the ROM feed comprising 8-9% sillimanite along with other heavy minerals, is treated in the Primary Concentration Plant (PCP) comprises combination of spirals and up current classifier separates sillimanite / quartz (23% Sillimanite) which is further upgraded in Wet Sillimanite Circuit.

(WSC) uses spirals upfront- to remove residual Ilmenite, Rutile and Zircon (IRZ) and conventional flotation cells to produce froth concentrate containing 85% sillimanite. In the flotation process, Oleic acid is used as a collector cum frother, Sodium silicate as depressant and Soda ash to regulate pH. To separate Sillimanite from quartz/shell, differences in their surface properties are used in wet stage- Hydrophobicity (water repellent). In Trimex Conventional mechanical cells were installed to float sillimanite.

The sillimanite concentrate is then dried in the Mineral Separation Plant (MSP) and fed over Electrostatic plate separators and Rare Earth Roll magnetic separators to produce a saleable final product with >96% sillimanite ($Al_2O_3 \geq$ 57%) with iron content (as low as possible). The sample for the experimental studies was collected from Trimex Sands Private Limited (TSPL). The Sillimanite/quartz concentrate (feed to flotation) with 32% concentrate is collected from the plant.

Response surface methodology **(RSM)** is used with Fractional factorial design in the thesis. In order to determine the effect of different operating parameters such as dosage rates of the reagents, pulp density, pH, airflow rate etc., a test program was designed using **Full factorial Design (5 variables 3-level factors- 2 way interactions)** method - one of the popular tools in the Design of Experimentation (DOE) software given a set of 243 experiments with all the possible combinations of the effecting parameters. Based on the DOE output, flotation tests were conducted using a laboratory froth flotation cell and the results obtained were analyzed by ANOVA to determine the sillimanite recoveries.

Experimentation

The following are the operational issues faced at both the wet sillimanite circuit (WSC) and Dry sillimanite circuit:

a. Recovery of sillimanite in WSC is 51.6% at 81% sillimanite content in the froth against the designed recovery of 68% and a froth concentrate of 85% sillimanite.
b. High iron content (2% against 0.67%) in final product. This is due to the presence of fine garnet in the froth.

To address the above issues the following critical areas are identified:

a. Optimization of flotation reagents dosages.
b. Identification of parameters having the most significant impact on the flotation.

The main aim of the present investigation deals with the two fold objectives (i) Modeling and optimization of flotation process by conducting test works coupled with Design of Experimentation - a scientific tool to optimize the process parameters and to determine the more sensitive process variables. Optimization of operational parameters using response surface methodology (RSM) applying Central Composite Design (CCD) technique by factorial method; (ii) To reduce the iron content in the concentrate to produce marketable final product

The project work was undertaken at **Trimex Sands Private Limited Srikakulam Andhra Pradesh and Department of Chemical Engineering Andhra University**.

The test activities include:

Test Activities with Results and Discussion

The physical and surface properties, proportion, content and grain size of the minerals affect the floatability and grindability of the ore. Physical characteristics such as bulk density, true density, porosity and angle of repose of representative sample were determined and tabulated as below in Table 1:

Table 1 Physical Properties of Sillimanite Sample

Bulk Density, g/cm3	**2.5**
True Density, g/cm3	**3.5**
Apparent Porosity, %	**17**
Angle of Repose, Degree	**32**
d80 passing size, µm	**190**

A test program was designed using the Central Composite Design (CCD) method of FFD (Full Factorial Design), which is one of the popular tools in the Design of Experimentation (DOE). The variables (operational parameters of flotation)and levels (upper and lower limits) considered for the DOE studied are given below in Table 2.

Table 2 Levels of different process variables in coded and un-coded form

Variables	Factors	Range and levels		
		Lower limit (-1)	Center Point (0)	Upper Limit (1)
X_1	COLLECTOR DOSAGE (Oleic Acid) -(Kg / ton of feed)	0.60	0.80	1.00
X_2	PULP DENSITY – (% of Solids)	0.30	0.35	0.40
X_3	P^H	9.50	10.00	10.50
X_4	DEPRESSANT DOSAGE (SODIUM SILICATE) -(Kg / ton of feed)	0.57	0.71	0.85
X_5	AIR FLOW RATE – Liters per minute (LPM)	0.50	0.60	0.70

Based on the DOE output matrix 243 experimental runs, were carried out according to Central Composite design to check the reproducibility. Flotation tests were conductedusing laboratory froth flotation cell and the results obtained were analyzed to determine the sillimanite recoveries.

The ANOVA (Analysis of Variance) Technique of Statistical Software version 10.0 was used to analyze the test results.

The significance of regression co-efficient was determined by Student's t - test as a tool and 'P' values which signify the pattern of interaction among the factors. The larger the magnitude of the t - value and smaller the p - value, the more significant is the corresponding co-efficient.

By analyzing the 't' values and 'P' values, it is found that the x_1, x_2, x_3, x_4,x_5,x_{12}, x_{22}, x_{32}, x_{42}, x_{52}, x_1x_2, x_1x_3, x_1x_4, x_1x_5, x_2x_3, x_2x_4, x_2x_5, x_3x_4, x_3x_5, x_4x_5 have significance to explain the individual and interaction effect of operational variables on the sillimanite separation to predict the response. In the regression data table the first order main effects (represented as L) and the second order main effects (represented as Q) of parameters are given. The regression table is given below as Table 3.

Table 3 Estimated regression coefficients for Recovery of Sillimanite.

	Regression	StandardError	t(9)	P
Mean/Interc.	-1661.802	29.93265	-55.7220	0.000000
(1)ColDos (L)	1078.89	27.02133	39.9272	0.000000
ColDos (Q)	-531.46	11.50608	-46.1899	0.000000
(2)PulpDen (L)	26.09	0.54043	48.2735	0.000000
PulpDen (Q)	-0.24	0.00460	-52.3171	0.000000
(3)pH (L)	104.44	3.48370	29.9807	0.000000
pH (Q)	-4.79	0.14106	-33.9361	0.000000
(4)Dep. Dos (L)	403.18	19.81956	20.3424	0.000000
Dep. Dos (Q)	-275.73	7.19669	-38.3138	0.000000
(5)AFR (L)	690.66	26.27401	26.2869	0.000000
AFR (Q)	-512.46	11.50608	-44.5386	0.000000
1L by 2L	-10.41	0.29879	-34.8240	0.000000
1L by 3L	7.72	1.49394	5.1709	0.000586
1L by 4L	7.32	10.67101	0.6861	0.509934
1L by 5L	-34.75	14.93941	-2.3261	0.045037
2L by 3L	-0.20	0.02988	-6.7104	0.000087
2L by 4L	0.33	0.21342	1.5228	0.162136
2L by 5L	0.48	0.29879	1.5898	0.146353
3L by 4L	-2.08	0.93250	-2.2304	0.052660
3L by 5L	-5.17	1.49394	-3.4640	0.007116
4L by 5L	-25.54	10.67101	-2.3930	0.040358

Where:
t(9) – Students t- distribution – This is suitable for comparing two treatment means
p - The value obtained from the ratio at 5% level of significance

The best model for maximizing Sillimanite Recovery is fitted in the quadratic polynomial model by regression equation as below:

$$\mathbf{Y = -1661.802 + 1078.89X_1 + 26.09X_2 + 104.44X_3 + 403.18X_4 + 690.66X_5 - 531.46X_1^2 - 0.24X_2^2 - 4.79X_3^2 - 275.73X_4^2 - 512.46X_5^2 - 10.41X_1X_2 + 7.72X_1X_3 +}$$

$7.72X_1X_4$ $-34.75X_1X_5$ $-0.20X_2X_3+$ $0.33X_2X_4+$ $0.48X_2X_5$ $-2.08X_3X_4$ $-5.17X_3X_5$ -25.54

Where

Y = Recovery%; X_1 = Collector dosage; X_2 = Pulp density *(This parameter and its effect got redundant in the present flotation);* X_3 = pH; X_4 = Depressant Dosage; X_5 = Air Flow Rate.

The results of above regression model for Eq.'s - in the form of ANOVA is compiled in Table 4 below.

Table 4 ANOVA of Sillimanite Recovery for entire quadratic model

ANOVA OUTPUT DATA					
Source of variation	**SS**	**df**	**Mean square(MS)**	**F-value**	***P*> F**
Model	331.8188	20	16.59094	743.255	0.00000
Error	0.2009	9	0.022322		
Total	332.0197				
Legend:-d.f - Degree of freedom; SS – Sum of squares; MS – Mean sum of squares; F ratio – Mean sum of squares/Error mean sum of squares; P – The value obtained from the ratio at 5% level of significance.					

Where

SS – Sum of squares
Total SS = SST (sum of treatment) + SSE (Sum of error)
Df – degrees of freedom
MS – Mean Square – SS/df
MST = SST/DFT & MSE = SSE/DFE
DFT (degrees of freedom for treatment) = k-1 where k is number of experimental runs
DFE (degrees of freedom for error) = N-k where N is total number of observations
F is F-test statistics used in testing equality of treatment means & F = MST/MSE
p-value is test for homogeneity. If p value is <0.05 it is considered as the effect is significant.

The ANOVA results can be used to test the statistical significance of the ratio of mean square due to regression and mean square due to residual error. The higher f-statistics and lower p value (<0.05) indicates that the model is considered to be statistically significant at the 95% confidence level. The maximum sillimanite recovery i.e. **73.7%** (based on test results) is obtained at optimum/critical values of the parameters. Among the tested parameters, by the ANOVA table it has been found that all the variables have significant impact on the process.

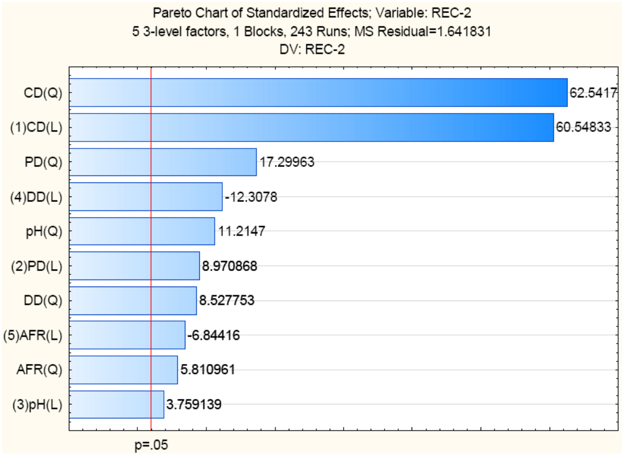
In general, the Fischer's '*F*-statistics' value ($=MS_{model}/MS_{error}$), where MS (-mean square) with a low probability '*p*' value indicates high significance of the regression model. The ANOVA of the regression model demonstrates that the model is highly significant, as is evident from the Fisher's *F*-test (F_{model}= 743.225) and a very low probability value ($p_{model}>F$=0.000000). Moreover, the computed *F*-value is greater than that of the tabular *F*-value at the 5% level, indicating that the conducted experiments are significant. The critical values of the parameters / variables obtained from the model are given in Table 5 below:

Table 5 Critical values of the parameters

Variables	Critical Values
Collector Dosage (kg/ton of feed)	0.728
Pulp Density (% solid by mass)	35.75
pH	10.05
Depressant Dosage (kg/ton of feed)	0.63
Air Flow Rate (Liter per minute)	0.58
Froth Grade	90.12
Predicted Recovery	**73.76**

The Pareto charts are given in Figure 1 below which show the operating variables and their interacting effects on the Sillimanite flotation process. A positive sign of the coefficient represents a synergistic effect which means sillimanite recovery increases with the increase in effect, while a negative sign indicates an antagonistic effect which means sillimanite recovery decreases with the increase in effect. The chart suggests that the individual effects of Collector dosage (synergistic) and Depressant dosage (antagonistic) are significant in the flotation operation.

Figure 1 Pareto Chart



Legend

(1)CD-Collector Dosage
(2)PD-Pulp Density
(3)pH
(4)DD-Depressant Dosage
(5)AFR-Air Flow Rate

The value of the regression coefficient obtained is $R^2 = 0.97449$, which shows that the observed (**o**) and predicted values (-) are in line with the design. The 5% sensitivity of the experiments conducted is also marked in the chart (-). The Observed and Predicted values chart is depicted below in Figure 2.

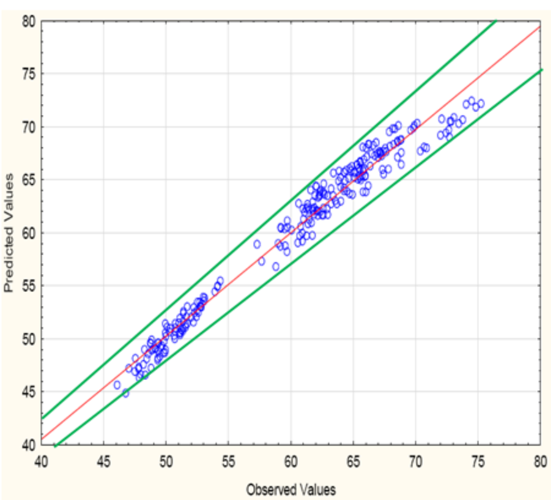


Figure 2 Observed Vs Predicted Chart

Interaction effects: The recovery of sillimanite over different combinations of independent variables is depicted through 3-D view of response surface plots which, explains the interaction effects of the variables. All the 3-D plots are represented as a function of two factors (variables), holding all other factors fixed at zero level. All the response surface plots reveals that at low and high levels of the variables the responses of sillimanite product are maximal, however, it is noted that there exist a region where neither an increasing nor a decreasing trend in the responses is observed. This phenomenon confirms that there is an existence of optimum for the Sillimanite variables in order to maximize the Sillimanite Recovery. The 3-D plots are given below in Figures 3 to 12

Among all the plots the significant interaction affecting the froth flotation process, is observed between Depressant dosage Vs Collector dosage; Air flow rate Vs pH and Air flow rate Vs Depressant Dosage.

Figure 3 pH Vs Collector Dosage

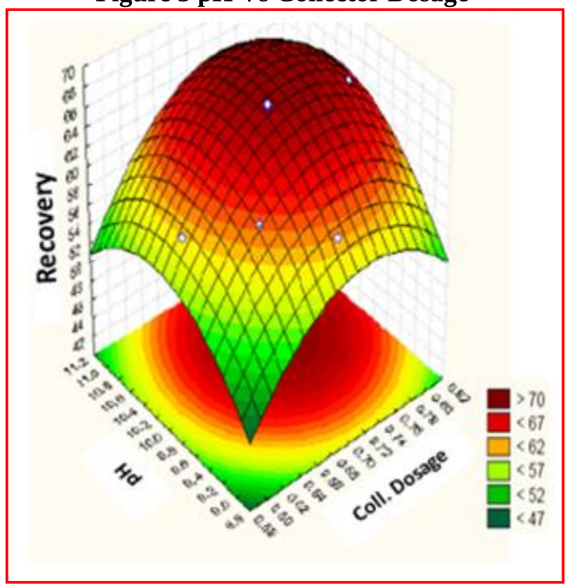


Figure 4 Air Flow Rate Vs Collector Dosage

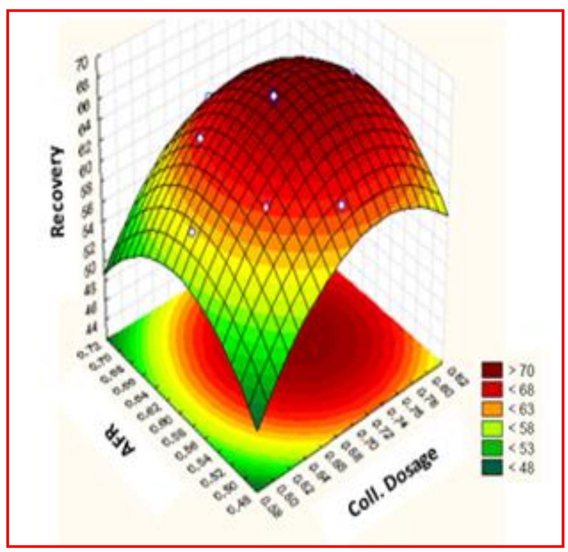


Figure 5 Depressant Dosage Vs Collector Dosage

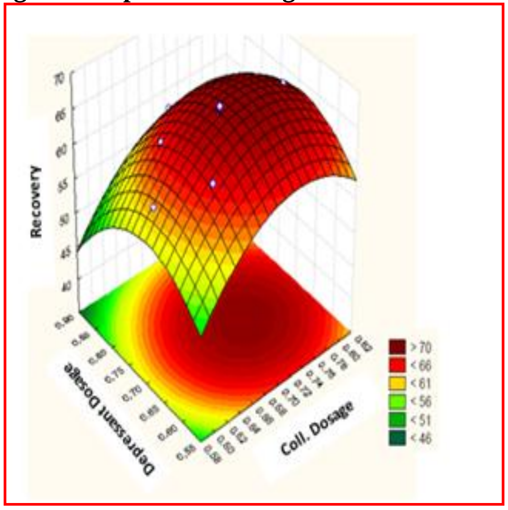


Figure 6 Pulp Density Vs Collector Dosage

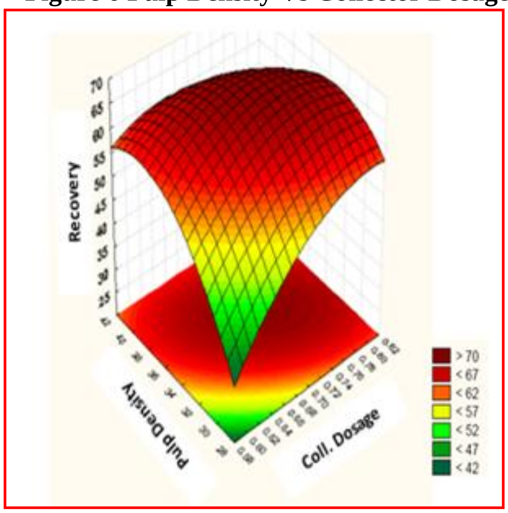


Figure 7 pH Vs Pulp Density

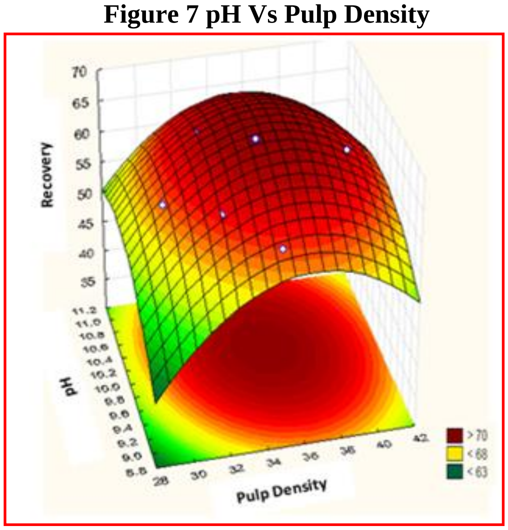


Figure 8 Air Flow Rate Vs pH

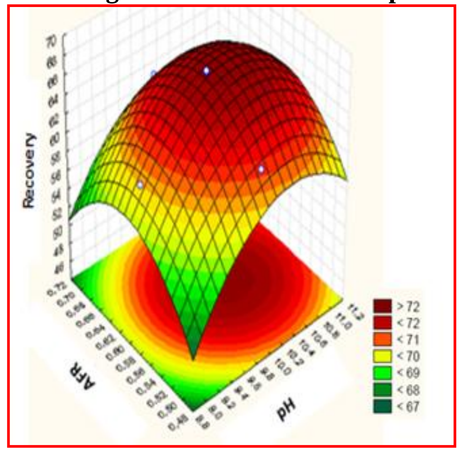


Figure 9 Depressant Dosage Vs pH

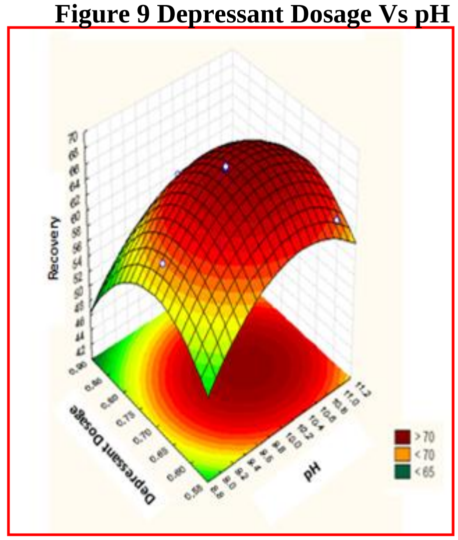


Figure 10 Air Flow Rate Vs Depressant Dosage

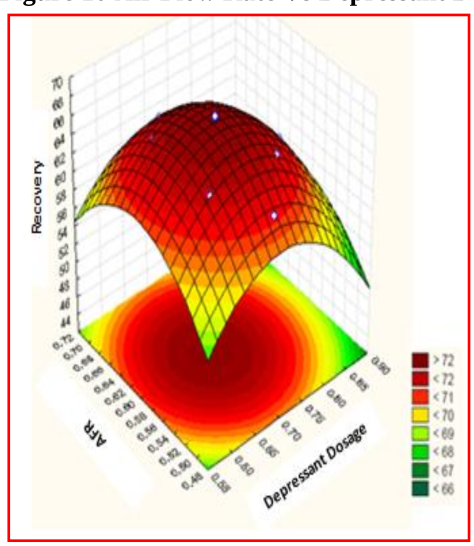


Figure 11 Air Flow Rate Vs Pulp Density

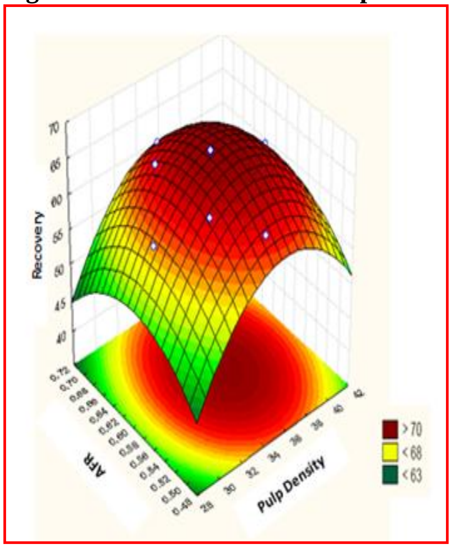
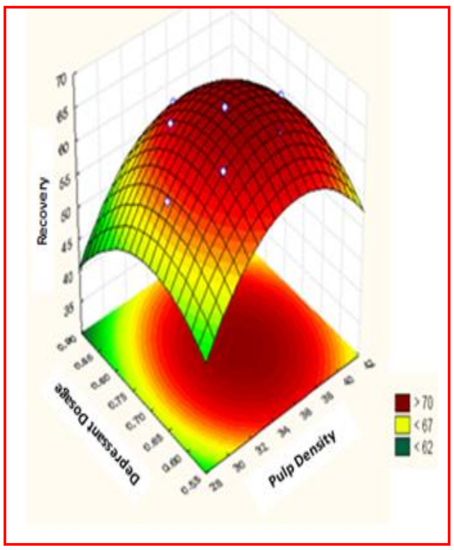


Figure 12 Depressant Dosage Vs Pulp Density



The obtained critical values of the parameters in the analysis are tested in laboratory and plant scale in order to validate the model. The results are tabulated as below in Table 6:

Table 6 Recovery & Grade Validation Results

	Recovery %	Grade%
As per analysis (ANOVA)	73.76	90.12
As per confirmatory tests at laboratory	72.5	90.0
As the tests at plant level (during continuous operation)	71-73	88-90

Granulometric Studies:

Effect of Sink-Float Studies: In the Bromoform and MI tests the minerals separation is obtained exploiting their specific gravity property. The representative samples were subjected to sink and float tests to assess the quality of the products and to calculate the THM, LHM & VHM percentages. When the Sillimanite froth and sink samples are treated with Bromoform having specific gravity of 2.89 g/cm^3, the heavy minerals (S.G > 2.89 g/cm^3) are reported in sink and light minerals (S.G < 2.89 g/cm^3) in float. Therefore, sillimanite with S.G 3.25 g/cm^3 reported in sink and float comprises mostly quartz with S.G 2.65 g/cm^3.

The sink material from Bromoform test is treated with Methylene iodide. Since the S.G of MI is 3.3 g/cm^3 the float comprises sillimanite (S.G 3.25 g/cm^3< MI) and sink comprises other minerals mostly garnet (S.G 4.3 g/cm^3> MI). The float-sink analysis is given below in Table 7

Table 7 Sink – Float tests with Sillimanite froth using Bromoform & Methylene Iodide

Sample	**Sillimanite Froth**
Bromoform Float	2.5
Bromoform Sink	97.5
MI Float	94.38
MI Sink	5.62
Sample	**Sillimanite Sink**
Bromoform Float	51.46
Bromoform Sink	48.54
MI Float	30.46
MI Sink	18.08

Effect of Size and Mineralogical Analysis: The sieve analysis of representative sillimanite feed, froth and sink samples are given in the Table 8. From the table it has been indicated that more than 80% of sillimanite mineral is retained in the size range of 80# to 140#. The results suggest that finer particles are recovered with loss of coarser particles to sink.

Table 8 size analysis of feed, froth and sink

Mesh (US)	Flotation Feed Wt%	Product (SL Froth) Wt%	Reject (SL Sink) Wt%
50	2.31	0.81	4.55
60	4.81	0.75	9.78
70	8.31	6.81	12.56
80	19.41	21.25	22.13
100	32.19	37.59	29.65
120	20.73	19.70	14.32
140	7.79	10.11	4.33
170	2.60	2.13	1.63
+200	0.47	0.44	0.46
-200	1.37	0.44	0.90
Total	100	100	100

The mineralogical analysis of the sieve fractions is given in the below Tables 9, 10 and 11. The mineralogical analysis indicates that in the feed sample about 80% of the sillimanite is distributed within the 80 to 140 mesh.

Table 9 Mineralogical Analysis of Sillimanite froth samples

SIZES	# 50	# 60	# 70	# 80	# 100	# 120	# 140	# 170	# -170	Total
ILMENITE %	0.0	0.0	0.0	0.1	0.5	0.9	0.4	0.1	0.1	2.1
RUTILE %	0.0	0.0	0.0	0.0	0.2	0.5	0.0	0.1	0.0	0.8
ZIRCON %	0.0	0.0	0.0	0.0	0.3	0.0	0.0	0.0	0.0	0.3
MONAZITE %	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.1
GARNET %	0.0	0.1	0.4	7.5	6.2	3.6	1.8	0.7	0.5	20.8
SILLIMANITE %	**1.9**	**2.2**	**3.5**	**7.2**	**10.2**	**4.7**	**1.5**	**0.5**	**0.5**	**32.1**
OTHERS %	0.0	0.1	0.2	0.1	0.8	0.6	0.2	0.0	0.5	2.6
QUARTZ %	0.4	2.4	4.2	4.5	14.0	10.4	3.8	1.3	0.3	41.3
Total %	2.3	4.8	8.3	19.4	32.2	20.7	7.8	2.6	1.9	100.0

Table 10 Mineralogical Analysis of Sillimanite froth samples

SIZES	# 50	# 60	# 70	# 80	# 100	# 120	# 140	# 170	# -170	Total
ILMENITE	0.0	0.0	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.3
RUTILE	0.0	0.0	0.1	0.1	0.1	0.0	0.1	0.0	0.0	0.4
ZIRCON	0.0	0.0	0.0	0.0	0.3	0.0	0.0	0.0	0.0	0.3
MONAZITE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
GARNET	0.0	0.2	0.5	3.1	3.4	0.0	0.0	0.2	0.0	7.4
SILLIMANITE	**0.7**	**0.4**	**5.5**	**16.9**	**33.4**	**19.5**	**9.9**	**1.8**	**0.8**	**88.8**
OTHERS	0.0	0.1	0.1	0.5	0.2	0.1	0.0	0.0	0.0	1.0
QUARTZ	0.1	0.1	0.7	0.4	0.2	0.1	0.1	0.0	0.1	1.8
Total	0.81	0.75	6.81	21.25	37.59	19.70	10.11	2.13	0.88	100.0

Table 11 Mineralogical Analysis of Sillimanite sink samples

SIZES	# 50	# 60	# 70	# 80	# 100	# 120	# 140	# 170	# -170	Total
ILMENITE	0.4	0.0	0.0	0.4	1.0	0.6	0.1	0.0	0.0	2.5
RUTILE	0.1	0.0	0.1	0.1	0.2	0.0	0.1	0.0	0.0	0.7
ZIRCON	0.0	0.0	0.0	0.0	0.2	0.0	0.0	0.0	0.0	0.2
MONAZITE	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.1
GARNET	2.8	0.3	0.8	3.5	5.6	2.8	0.3	0.0	0.0	16.2
SILLIMANITE	**0.2**	**0.3**	**0.3**	**1.0**	**3.8**	**2.5**	**0.8**	**0.3**	**0.8**	**9.9**
OTHERS	0.7	0.1	0.1	0.6	0.2	0.2	0.0	0.0	0.0	2.0
QUARTZ	0.3	9.1	11.3	16.5	18.7	8.2	3.0	1.3	0.2	68.5
Total	4.55	9.78	12.56	22.13	29.65	14.32	4.33	1.63	1.04	100.0

The Size and Mineralogical Analysis of sillimanite representative samples indicates that the sillimanite content of 32% in sillimanite intermediate (Sillimanite/quartz) is upgraded to >88% in sillimanite concentrate (Sillimanite froth) by flotation operation, giving a recovery of about 73%.

CONCLUSIONS

Sillimanite is industrially important mineral, Silica sand deposits are commonly contaminated with various heavy minerals. In Trimex Sands Private Limited, conventional mechanical cells were installed to float sillimanite. In a view to optimize the operational parameters of flotation at wet stage the present investigation carried out with an aim of (i) Modeling and optimization of flotation process by conducting test works coupled with Design of Experimentation and (ii) To reduce the iron content in the concentrate to produce marketable final product at dry stage.

The tests were conducted and found to be successful and achieved the sillimanite grade and recovery higher than the designed value. The results are validated by implementing in plant achieving recovery of 75% with grade of 88-90%. The garnet content also reduced from 20% in feed to about 7% in the froth which facilitates to have low iron content in final sillimanite product at dry stage and enhances the value of sillimanite in the market.

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