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RESEARCH ARTICLE

EFFECT OF ADDITION OF MIBC ON SILLIMANITE FLOTATION

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Abstract

Sillimanite is chemically named Aluminum Silicate (Al_2SiO_5) which is rare and one of the Trimorphs of alumino silicate polymorphs. Sillimanite is an important mineral among the mineral sand deposits and predominantly occurs along the costal line of India. The Sillimanite mineral occurs in beach and dune placer deposits along with strategic and economically important heavy minerals heavy minerals such as ilmenite, rutile, zircon, garnet and monazite. Trimex Sands Pvt Limited (TSPL), a mineral wing of Trimex Group is operating a heavy mineral sands mine based on Srikurmam Deposit in the district of Srikakulam in the state of Andhra Pradesh. At TSPL, froth flotation is employed to separate Sillimanite from quartz and the performance of the flotation circuit is not at par with that of the design. Earlier studies were successfully conducted to optimize the parameters to improve the performance of the flotation circuit using design of experimentation and ANOVA. This paper deals with various efforts made to enhance the performance of the sillimanite flotation further, with the use of additional frother such as Methyl Isobutyl Carbinol (MIBC) in combination with present collector cum frother Oleic acid. The results show that the usage of MIBC increased the recovery of sillimanite from 73% to >75% and reduced the consumption of oleic acid.

Introduction

Sillimanite is chemically named Aluminum Silicate (Al_2SiO_5) which is rare and one of the Trimorphs of alumino silicate polymorphs. Sillimanite is a refractory mineral indicating high-temperature metamorphism as its formation takes place at very high temperatures of 550^{O}C and above. Sillimanite is an important mineral among the mineral sand deposits and predominantly occurs along the costal line of India [1].

The sillimanite minerals decompose irreversibly to mullite when subjected to calcination [2], Sillimanite is essentially useful in high-alumina refractories forming the inner lining of furnaces and high-temperature vessels widely used in the production of metals, ceramics, glass and cement.

The Sillimanite mineral occurs in beach and dune placer deposits along with strategic and economically important heavy minerals heavy minerals such as ilmenite, rutile, zircon, garnet and monazite. India is gifted with such abundant beach- and dune-placer deposits [3]. These deposits are mostly located in the coastal stretches of peninsular India covering states of West Bengal, Odisha, Andhra Pradesh, Tamilnadu, Kerala, Karnataka, Goa, Gujarat and Maharashtra, where significant deposits of different minerals are available from beach sands, made this country one of the leading producers of Sillimanite in the world [4].

Trimex Sands Pvt Limited (TSPL), a mineral wing of Trimex Group is operating a heavy mineral sands mine based on Srikurmam Deposit in the district of Srikakulam in the state of Andhra Pradesh. Its operations include mining and processing of ROM sand to produce 200,000 tpa of ilmenite, 5,000 tpa each of rutile and zircon and 50,000 tpa each of sillimanite and garnet. At TSPL, froth flotation is employed to separate Sillimanite from quartz and the performance of the flotation circuit is not at par with that of the design [5]. Earlier studies were conducted to optimize the parameters to improve the performance of the flotation circuit using design of experimentation and ANOVA [6] , The findings of the studies were implemented successfully in the plant and a significant improvement in the performance was reported .

Traditionally Oleic acid/ sodium oleate is used as collector cum frother in sillimanite flotation. The molecular structure of sodium oleate is given below in Figure 1:

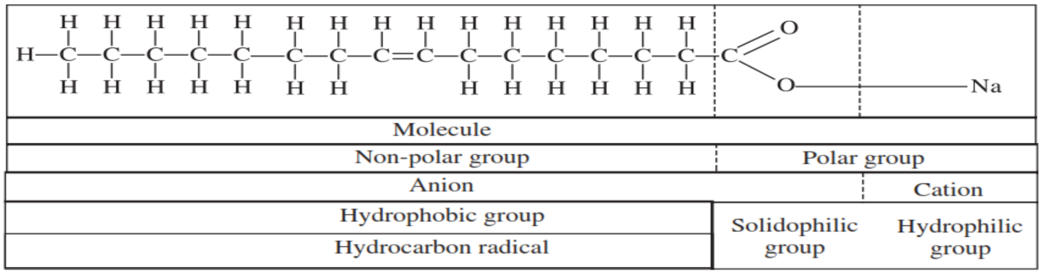


Figure 1: Molecular structure of Sodium Oleate

As shown in the Figure 1, Sodium oleate has polar group and hydrocarbon radical as like frothers having heteropolar surface-active compounds containing a polar group and a hydrocarbon radical, capable of adsorbing in the water–air interface. The frother molecules are arranged at the air–water interface such that the hydrophilic or polar groups are oriented into the water phase, and the hydrophobic or nonpolar hydrocarbon chain in the air phase. Sodium oleate comes under neutral frothers and can be used in both acidic and alkaline pulps. These types of frothers are widely used in flotation of base-metal ores, oxidic minerals and industrial minerals [7].

Several researchers reported the use of additional frothers such as MIBC to improve the performance sillimanite flotation.

This paper deals with studies conducted to evaluate the effect of additional frother such as Methyl Isobutyl Carbinol (MIBC) on Sillimanite flotation.

SILLIMANITE FLOTATION AT TSPL

As shown in Figure 3, an ROM feed of about 10% of sillimanite along with other heavy minerals, is treated in the Primary Concentration Plant (PCP). PCP employs gravity separation equipment such as spirals and hindered settling classifiers to separate Sillimanite-quartz stream from other heavy minerals such as ilmenite, rutile, zircon and garnet. The Sillimanite-quartz stream so produced is further upgraded in Wet Sillimanite Circuit (WCP) consists of flotation cells to produce a froth concentrate containing about 85-90% Sillimanite at a recovery of >73%. The sillimanite concentrate is then dried in the Mineral Separation Plant (MSP) and treated with Electrostatic and magnetic separators to produce a saleable final product with >96% sillimanite [8].

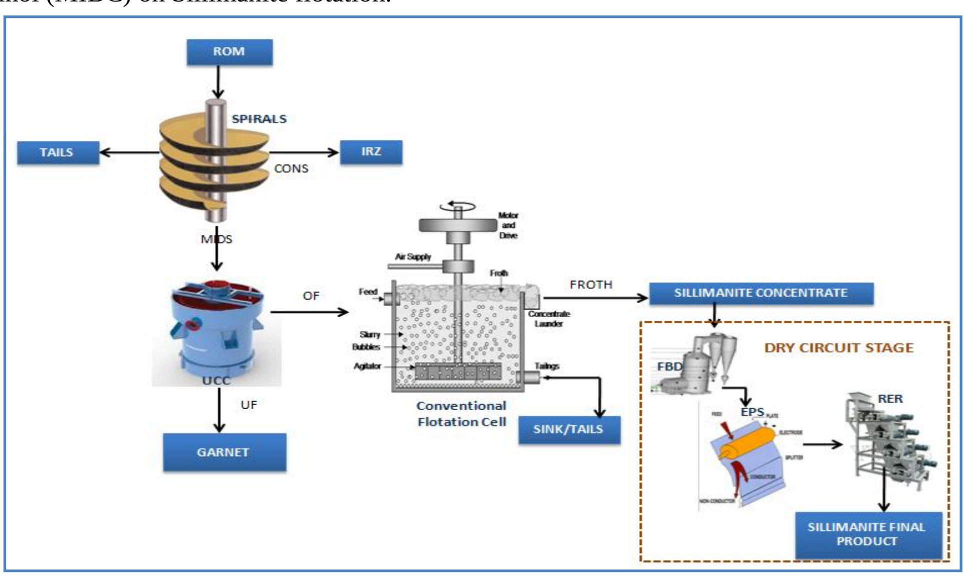


Figure 2: Processing of Sillimanite from Run of Mines

The details of plant operations are given below in Table 1.

Variables	Plant Operations
Number of flotation cells	5
Feed to flotation cells, Tons per hour	35
Flotation Time, min	10
Collector Dosage, kg/ton of feed	0.73
Pulp Density, % solid by mass	35.5
pH	10
Depressant Dosage, kg/ton of feed	0.63
Air Flow Rate, Liter per minute	0.58
Feed grade, %	32
Production from flotation cells, Tons per hour	9.08
Rejects from Flotation cells (Tons per hour)	25.92
Flotation Concentrate Grade, % Sillimanite	88-90
Flotation Rejects Grade, % Sillimanite	11-12
Recovery of Sillimanite in concentrate, %	71-73

Table1: Details of Plant operation of Flotation cell

EXPERIMENTATION

Laboratory studies were conducted in a laboratory flotation cell as shown in Figure 4

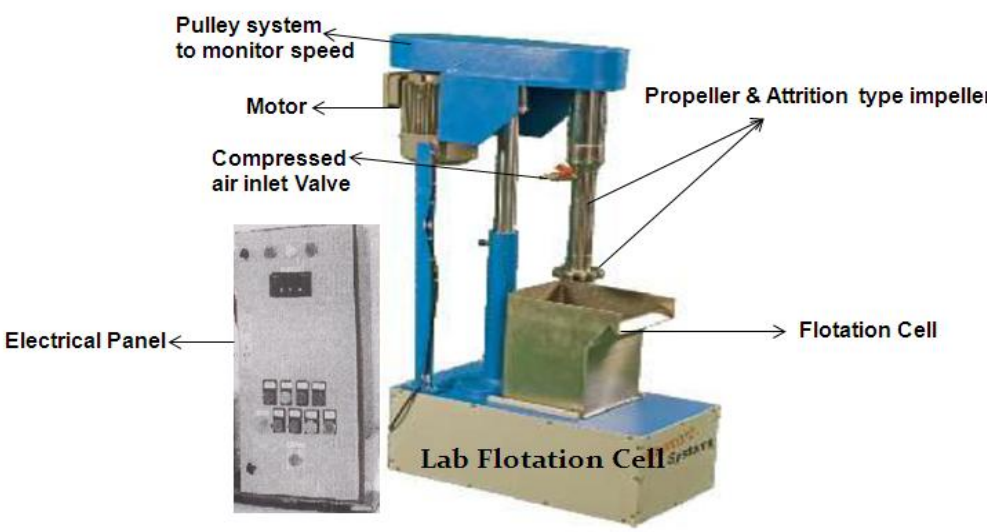


Figure 3: Flotation Cell used for experimentation in laboratory

Flotation Cell Specifications

- Cell capacity – 4.25 liters volume cell.
- Digital display of set speed.
- Provided with air inlet valve.
- Operated with - 4 pole, 10 amp MCB; 3 phase + neutral with earthing; 0.75 kW; 415 V AC; 50 / 60 Hz.
- The Unit is operated with 1 HP motor, 230 V, single phase, 50 cycles AC, 1415 rpm

Tests are conducted varying dosages of oleic acid and MIBC while keeping all other parameters (pulp density, Ph, air flow rate and depressant dosage) constant. By using DOE- Central Composite Design (CCD) a matrix is designed taking oleic acid and MIBC dosages as parameters. The variables (operational parameters of flotation) and levels (upper and lower limits) considered for the Design of Experimentation (DOE) studied are given below in Table 2.

Table 2: Levels of different process variables in coded and un-coded form for MIBC test works.

Variables	Factors	-1	0	1
X_1	Collector Dosage (Oleic Acid) -(Kg / ton of feed)	0.600	0.650	0.700
X_2	MIBC – (Kg / ton of feed)	0.015	0.040	0.065

The other parameters are maintained as per the plant operations as given in the below Table 3.

Table 3: Operational Parameters at Plant operations

pH	10.00
Pulp Density, % Solis by mass	35.50
Depressant Dosage, Kg/ton of feed	0.63
Air Flow Rate, Liter per minute	0.58
Feed grade, %	32.00

RESULTS AND DISCUSSIONS

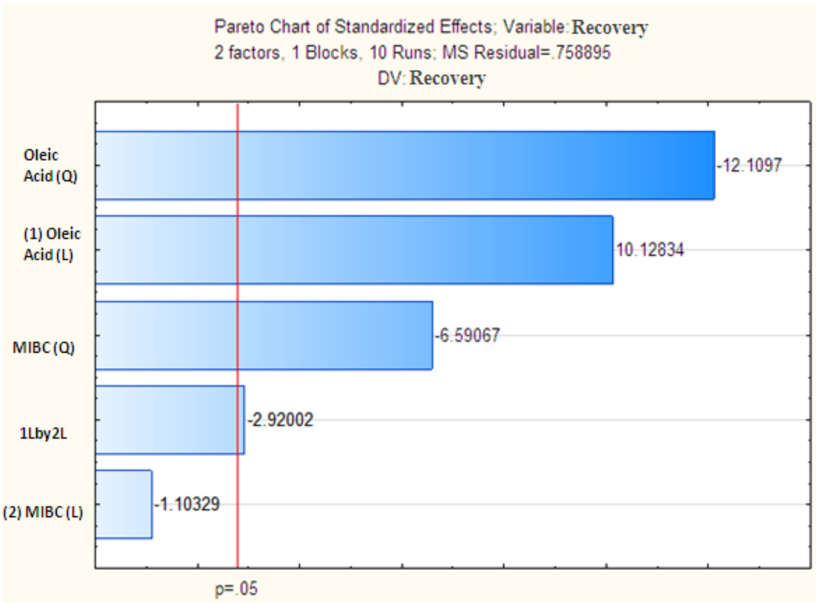
Based on the DOE output matrix, Flotation tests were conducted and the results obtained were analyzed to determine the sillimanite recoveries. The DOE matrix and Results obtained in terms of recovery is given below in Table 4

Table 4: DOE Matrix for experimental runs and results

Experiment Runs	Oleic Acid, kg/ton of feed	MIBC, kg/ton of feed	Recovery,%
01	0.600	0.015	58
02	0.600	0.065	66
03	0.700	0.015	72
04	0.700	0.065	67
05	0.650	0.040	74
06	0.600	0.040	65
07	0.700	0.040	73.2
08	0.650	0.015	75.1
09	0.650	0.065	73.1
10	0.650	0.065	73.1

The Pareto chart is depicted in Figure 4 and the individual and interacting effects of operating variables are presented in Table 5. 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 [8], which means sillimanite recovery decreases with the increase in effect. The chart suggests that the individual and quadratic effects of collector dosage are synergetic and antagonistic respectively in the flotation operation. The quadratic effect of MIBC is antagonistic and the linear effect of MIBC is not significant in the operation. The combined linear effects of Oleic acid and MIBC have moderately antagonistic effect on the operation.

Figure 4: Pareto Chart



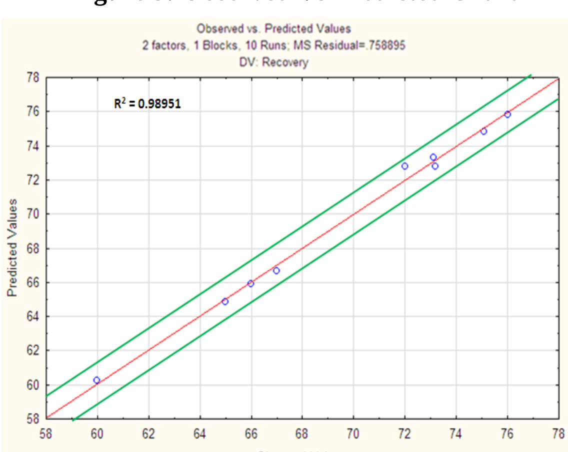
Legend: (1) CD-Collector Dosage; (2) MIBC-Methyl Isobutyl Carbinol

Table 5: individual and interacting effects of operating variables

Parameters	**Linear (Individual) Effect**	**Quadratic (interactive) Effect**
(1) Oleic Acid	High Synergetic (max)	High Antagonistic (max)
(2) MIBC	-	Moderately Antagonistic
Combined effect of Oleic Acid & MIBC (1Lby2L)	Low antagonistic	

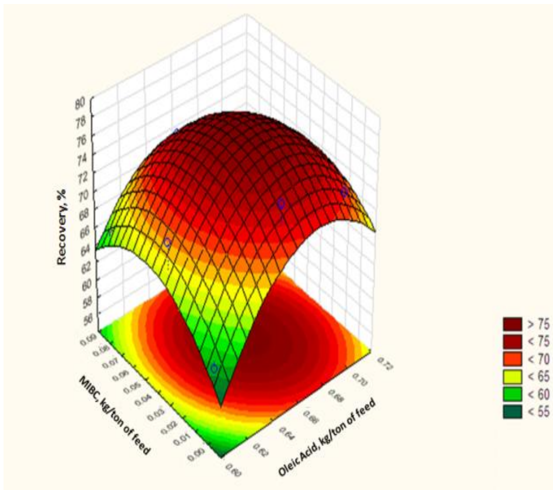
Observed Vs Predicted Values: The Observed and Predicted values are depicted below in Figure 5. The value of the regression coefficient obtained is $\mathbf{R^2}$ = **0.98951**, which shows that the observed (**o**) and predicted values (-) are in line with the design. The 5% sensitivity of the experiments conducted is also marked in the chart (-).

Figure 5: Observed Vs Predicted Chart



Interaction effects: The Results at different combinations of independent variables is depicted through 3-D view of response surface plots. The same explains the interaction effects of the variables. The 3-D plots are represented as a function of two factors (variables), holding all other factors fixed at zero level. Here a 3-D plot is determined for Collector dosage and MIBC dosage. The response surface plot 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 variables in order to maximize the Sillimanite Recovery. The 3-D plot is given below in Figures 6.

Figure 6: 3-D Response Surface Plot



The plot of **MIBC Vs Collector Dosage** shows that the defined shape obtained with maximum recovery range region (red color). This indicates that the interaction effect is highly significant and maximum **recovery of >75% is** obtained at **MIBC dosage of 0.025 kg/ton of feed & Collector Dosage: 0.65 kg/ton of feed**

Development of Correlation Equation:

The following quadratic equation – (1) was fitted to the above data using multiple linear regressions using Statistica Software version 10.

$$Y= b_0 + b_1 x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{11}x_1^2 +b_{22}x_2^2+ b_{33}x_3^2 + b_{44}x_4^2 + b_{55}x_5^2 + b_{12} x_1x_2 +b_{13} x_1x_3+ b_{23} x_2x_3+ b_{44} x_4^2+ b_{14} x_1x_4 + b_{24} x_2x_4+ b_{34} x_3x_4 \quad \text{--------------------------} \ (1)$$

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 [9].

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 6.

Table 6: Estimated regression coefficients for Recovery of Sillimanite

	Regression	Standard Error	t(9)	P
Mean/Interc.	-1306.44	98.928	-13.2060	0.000000
(1)ColDos (L)	4021.60	308.143	13.0511	0.000000
ColDos (Q)	-2925.52	241.586	-12.1097	0.000000
(2)MIBC (L)	2390.56	293.938	8.1329	0.000000
MIBC (Q)	-3230.06	1106.175	-2.9200	0.000000
1L by 2L	-3310.06	502.233	-6.5907	0.000000

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:

$$Y = -1306.44 + 4021.60X_1 + 2390.56X_2 - 2925.52X_1^2 - 3230.06X_2^2 - 3310.06X_1X_2$$

Where Y = Recovery%; X_1 = Oleic Acid; X_2 = MIBC dosage

ANOVA: The results of above regression model for Eq.'s - in the form of ANOVA are compiled in Table 7 below:

Table 7: ANOVA of Sillimanite Recovery for entire quadratic model

ANOVA OUTPUT DATA					
Source of variation	**SS**	**df**	**Mean square(MS)**	**F-value**	***P*> F**
Model	286.2094	5	57.24188	75.427	0.00000
Error	3.0356	4	0.7589		
Total	289.2450	9			
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. **75.52%** (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}=75.43) 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.

Critical Parameters: The critical values of the parameters / variables obtained from the model are given in Table 8 below:

Table 8: Critical parameters from ANOVA

Variables	Optimum/Critical Values by ANOVA
Collector Dosage (kg/ton of feed)	0.65
MIBC (kg/ton of feed)	0.025
Recovery, %	75.52

The obtained critical dosages of Oleic acid and MIBC are confirmed in laboratory and plant level. The results obtained under critical parameters are given in Tables 9 and 10. As shown in the Table, there is a significant improvement in the predicted performance results compared to that of existing operation in terms of recovery without affecting the concentrate grade of 90% the recovery increased from 73% to 75.5% (an increase of 2.5%) with the reduction of collector dosage from 0.728 kg/ton of feed to 0.65 kg/ton of feed. Further, the predicted performance was found to be superior to those of even designed values (The grade of 90% against 85% at a corresponding recovery of 73.76% against 68%).

Table 9: Comparison of Parameters (Predicted, Laboratory & Plant details)

Variables	Optimum/Critical Values by ANOVA	Laboratory Values	Plant Values
Collector Dosage (kg/ton of feed)	0.65	0.65	0.65
MIBC (kg/ton of feed)	0.025	0.025	0.025
Pulp Density (% solid by mass)	35.5	35.5	35.5
pH	10	10	10

Depressant Dosage (kg/ton of feed)	0.63	0.63	0.8
Air Flow Rate (Liter per minute)	0.58	0.58	0.58
Feed to Flotation cell (Tons per Hour)		1.964	35
Sillimanite Concentrate from Flotation cell (Tons per hour)		0.525	9.4
Rejects from Flotation cell (Tons per hour)		1.438	25.6

Table 10: Comparison of Parameters (Predicted, Laboratory & Plant details)

RESULTS			
	Optimum/Critical Values by ANOVA	Laboratory Values	Plant Values
Concentrate Grade, % sillimanite		90.1	89-90
Recovery, %	75.52	75.3	75-75.5

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. After the froth flotation operation at wet stage, the sillimanite concentrate obtained has oleic acid coatings. In the dry process of Sillimanite Beneficiation the flowability issues are high due to this oleate coating, which is leading to loss of final product and improper separation at electrostatic and magnetic separation stages. To address the above issues, the main aim of the present work deals with investigating additional frother that is suitable for usage in the sillimanite flotation operation along with the present collector (Oleic acid) in order to reduce the collector consumption. Tests are conducted to study the effect of MIBC on oleic acid consumption in the Sillimanite flotation operation.

The tests were conducted and found to be successful and achieved the recovery higher than the plant value without affecting the sillimanite concentrate grade. The results are validated by implementing in plant achieving recovery of >75% with grade of 90% with the reduction of collector dosage from 0.73 kg/ton of feed to 0.65 kg/ton of feed (reduction of 80 grms/ton of feed). Further, the predicted performance was found to be superior to those of even designed values (The grade of 90% against 85% at a corresponding recovery of 73.76% against 68%). The flowability of the sillimanite concentrate also increased in the dry circuit.

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