Abstract

Coal washery rejected coals and coal slurries have better options in froth flotation as a separation process. In this study, coal slurry received from Sudamdih coal washery, Jharkhand, India, examined for cleaning through fixed cell flotation. The Petrography study of the feed sample showed the presence of vitrinite, semi vitrinite and liptinite and inertinite as major minerals. General full factorial statistical design package (Minitab V17) was used to develop the regression models for the responses like froth height, froth density, recovery and ash content of clean coal. Results showed that experimental responses like froth height, froth density, recovery and ash content were found to be more sensitive to the frother dosage. The coefficient of correlation ($R^2$) values between the experimental and the predicted values of the flotation responses was found to be $>0.98$ for all the models. Further, flotation tests were conducted for varying pulp density and its effect on recovery, froth height, ash content, tests with five levels of pulp density (8, 9, 10, 11, 12 and 13% solids by weight). It found that the coal slurry sample from the study area could be cleaned with sufficient efficiency. The cleaned coal is suitable for powder coal consuming industries.

Keywords: coal, flotation, collector, frother, ash removal
Introduction:

A large amount of coal fines has been generating during modern mechanized coal mining techniques and coal washing plants, which has serious effects not only on the environment but also on sustainable mining practices [1]. Processing of finer size coal has always been a problem and costlier than the cleaning of coarse coal [2]. The best available technique for cleaning of fine size coal with particle size <0.5mm is the froth flotation technique [3-5]. The coal particles subjected to treatment with suitable liquid hydrocarbon reagents to alter the hydrophobicity, and enhance recovery and/or improve selectivity [6-7], but fine particles show lower flotation rate, resulting in low flotation recovery [8]. Generally, in the coal flotation, froth can affect both the recovery and the grade, because it promotes the selective drainage of minerals back to the pulp zone [9]. Drainage in the froth zone contributes significantly to the increase of selectivity of the flotation process. Therefore, the froth should have enough stability and height, to allow the drainage of the entrained hydrophilic particles back into the pulp and simultaneously support the hydrophobic particles, inhibiting their drainage and contributing to the total selectivity of the process. In most of the Indian coal washeries, diesel oils were used as a collector with a combination of different frothers. The type and dosage of collector and frother have significant effects on coal flotation performance [10]. Recovery of cleaner coal from coal fines will be an attractive approach for improving the economic performance of coal washery. Some researchers have done considerable researchers to improve the performance of coal flotation applying advanced techniques like packed column, Jameson cell, column cell and microcell flotation [11]. Many works attempted to recover coal fines from coal washeries. However, there is a wide scope to improve the flotation performance of coal fines generated during washing. Keeping in view of the latest developments in the cleaning of coal fines, studies were initiated on the flotation of coal fines.

One of the most effective techniques to study process behaviour is the factorial design of experiments with an analysis of variance [12-16]. The present study aims to recover clean coal from the discarded coal slurry of coal washery located in Jharkhand, India, by using the flotation technique. The general full factorial experimental design method was used to investigate the influence of collector and frother dosages on recovery and ash content of clean coal (froth product). Consequently, the effects of collector dosage and frother dosage, on the froth height, froth density, recovery and the ash content of the clean coal are discussed using 3D surface plots. Further, the study also attempted to understand the effect of pulp density on the froth height and froth density.
Experimental results are analyzed using response surface methodology (RSM) is a well-established technique, used to obtain the optimal conditions by using a regression model [17]. Works of literature indicate the use of RSM for modeling of processes such as coal processing and flocculation [18&19].

2. Materials and Methods

2.1 Materials:

The discarded coal slurry samples composed of coal fines (<0.5 mm) taken from a coal washery, Jharkand, India. The coal-slurry sample was filtered, dried and disaggregated for use in the flotation tests. Flotation experiments were carried out using commercially available diesel oil as a collector and pine oil as a frother. Figure 1 shows the size wise ash analysis of the dried coal sample. The feed sample was subjected to characterization studies such as proximate analysis and specific gravity analysis.

2.2 Flotation Experiments.

Wemco Fagergren flotation machine with 3.5L of volumetric capacity as shown in figure 2 was used for flotation studies. A total of 100 g of coal was mixed with 300 ml of water and conditioned with diesel oil (collector) in a flotation cell for 2 min. The pH of the slurry was adjusted to 8. Another 600-ml make-up water was added and conditioned further with a predetermined quantity of pine oil (frother). The conditioning time for all the reagents was 2 min in each stage, and then flotation was carried out by releasing the air at 1500 rpm for 5 min. Froth height is measured with a scale. Froth density was measured by weighing the 500ml beaker filled with froth. From Eq(1) froth density is calculated.

\[ \rho_f = \frac{W_1 - W_2}{V} \quad (1) \]

Where:

\( \rho_f = \) Froth density
\( W_1 = \) Weight of beaker filled with froth
\( W_2 = \) Weight of empty beaker
\( V = \) Volume of the beaker (500ml)

A general full 2 factors 4 level factorial design was used to obtain the response pattern and then to establish a model. The regression model can approximate a mathematical relationship between the two independent factors as follows:
\[ Y = a_0 + a_1 x_1 + a_2 x_2 + a_3 x_1 x_2 \]  \hspace{1cm} (2)

Where, \( Y \) is the predicted response function (froth height, froth density, recovery and ash content of float coal). \( a_0 \) is a model co-efficient. \( a_1, a_2, a_3 \) are the coefficients. \( x_1 \) and \( x_2 \) are coded values of flotation test variables (collector and frother dosage). The computer program (Minitab V.17) was used for the determination of the coefficients of Eq. (2) by regression analysis of the experimental data. The actual and coded values of the variables, collector dosage (kg/t) and frother dosage (kg/t), used in this design are given in Table 3. The responses are analyzed using 3D surface plots to know the effect of collector and frother dosages.

3. Results & Discussions:

3.1 Characterization studies

Sieve analysis carried out using 1000–75μmsieve aperture sizes. The feed particle size distribution with size-wise ash content is shown in figure 2. About 80% of the particles are found to be less than 150 μm size fractions. While about 40% of the particles found to be coarser than 500 μm particle sizes (Fig. 2). About 12% of particles were found to be of a size less than 75 μm. The proximate and ultimate analysis of the feed coal slurry sample on a dry basis is as shown in Table 1. The average density of coal slurry was found to be 1.63 g/cm³ and a gross calorific value of 5130 kcal/kg.

3.2 Flotation studies

The flotation studies on coal samples were conducted based on the statistical design to correlate the influence of collector and frother dosage on the responses (froth height, froth density, recovery and ash content of float clean coal). The effects of each parameter on the others were found. To determine these effects, experimental results have been analyzed by Minitab V.17 software through which the model can significantly be selected and influences of the variable were perfectly portrayed. In the present study, 16 experiments were conducted based on general full factorial regression experimental design as shown in table 3. Recovery and ash content of the clean coal (floats) were analyzed for flotation process efficiency. Coded factor values and respective values of responses for all the experiments are given in Table 4. The model developed using these factors and their interaction is given in Esq. (3-6):

\[
\text{Froth height (cm)} = 4.4420 - 1.575 x_1 + 1.331 x_2 - 0.117 x_1 x_2 \]  \hspace{1cm} (3)

\[
\text{Froth density (g/cm}^3) = 0.0092 + 0.3054 x_1 + 0.2666 x_2 - 0.3012 x_1 x_2 \]  \hspace{1cm} (4)

\[
\text{Ash} = 6.647 + 6.709 x_1 + 17.884 x_2 - 9.039 x_1 x_2 \]  \hspace{1cm} (5)

\[
\text{Recovery} = -29.10 + 78.09 x_1 + 149.25 x_2 - 92.77 x_1 x_2 \]  \hspace{1cm} (6)
The analysis of variance (ANOVA) and the estimated regression coefficient of the flotation parameters are tabulated in Table 5. It can be seen that the probability value (P-value) of the independent variables (collector and frother dosages) of the processing conditions is less than 0.05 in linear and interaction relationships. Meanwhile, the P-value for all the models developed nearing to 0.05 which shows the significance of the models (Eqs 3-6). The model adequacies were justified by the R² values. Figure 5(A-D) showed that the R² for all the influencing variables is >0.9, reasonably good agreement between the experimental values and values predicted by the models (Eqs. (3-6)).

3.2.1 Effect of collector and frother dosage on froth height

3D surface response graphs were used to analyze the effect of parameters (collector dosage and frother dosage) on responses like froth height, froth density, recovery and ash content of float coal. The variations in the values of responses with the collector and frother dosages have been presented in figures. 6, 7, 8 and 9. Recognising the two-phase structure of the flotation system, the rate constant is multiplied by the froth transmission co-efficient “T”. Using Harris and Rimmer model, the factor T is estimated as

\[ T = \frac{1}{1 + K_f t_f} \] (7)

Where \( K \) is the rate of transfer from froth to the pulp and \( t_f \) is the residence time in the froth phase.

Increasing froth height increases the value of \( t_f \), thus reducing T. Since \( K_f \) for ash forming mineral is much higher than \( K_f \) for clean coal, the net effect of increased froth height is to improve selectivity. The parameter \( K_f \) depends upon particle size and hydrophobicity. Hence, the study intended to know the effect of collector and frother dosage on the froth height. From figure 6, the maximum froth height of 5.0 cm was observed at a frother dosage of 0.80 kg/ton and a minimum collector dosage of 0.32 kg/ton. Whereas the minimum froth height, 3.6 cm is attained at a frother dosage and collector dosage of 0.32 kg/ton and 0.80 kg/ton respectively. Observations made from figure 6 that, the higher the collector dosage from 0.48 to 0.8 kg/ton tends to decrease the froth height through the increasing frother dosages. This may be attributed to the interaction of a large particle with the bubble and collision makes the decrease in froth height at higher collector dosage.
3.2.2 Effect of collector and frother dosage on froth density

Figure 7 depicts the effect of collector and frother dosage on the froth density, with the increase in the collector dosage the froth density increased to 0.27 g/cm³ and shows the optimum value at 0.64 kg/ton of collector dosage. Further, an increase in the collector dosages reduces the froth density drastically. It observed that an increase in frother dosage increases the froth density due to entrainment of the ash forming minerals with the coal. The maximum froth density of 0.28 g/cm³ is achieved at a collector and frother dosage of 0.8 kg/ton and 0.64 kg/ton respectively where as 0.32 Kg/t of both collector and frother dosage results minimum froth density of 0.16 g/cm³.

3.2.3 Effect of collector and frother dosage on ash content (%)

From figure 8, observed that the minimum ash of 13.6% with a recovery of 34% achieved at 0.32 kg/t of each collector and frother dosage. Further, an increase in the frother dosage caused to increase in the ash content in the clean coal product, which attributed to the poorly liberated macerals reporting to float. 17.2% ash content obtained with about 70% recovery for the clean coal of using collector and frother dosages of 0.8 kg/ton and 0.48 kg/ton, respectively.

3.2.4 Effect of collector and frother dosage on recovery (%).

The data presented graphically in figure 9 show the effect of collector and frother dosage on the recovery of coal during flotation experiments. From figure 9, the recovery of coal increases with the increase in the frother dosage. 93% recovery achieved at collector and frother dosage of 0.64 kg/ton and 0.80 kg/ton with a 20.6 ash (%). The recovery surface in figure 9 shows the linear relationship with the frother dosage with the fixed collector dosage of 0.48 kg/ton.

3.3 Effect of Pulp Density on Flotation Responses.

Thicker the pulp, the smaller is the cell volume required in flotation plants. In general flotation, industries prefer to carry out the process with pulp as dense as possible for their
economics with good selectivity and operating conditions. Usually, industrial flotation for minerals is carried out with pulps of densities between 20 and 40% solids by weight. However, the pulp density may be as high as 55% or more, and as low as 8% [20]. Depending upon the rank, type, ash content and size distribution of the coal feed. The best condition for coal flotation has been suggested as 10% solids by weight [21].

Subsequently, tests with six levels of pulp density (8, 9,10,11,12 and 13 % solids by weight) were carried out. Figures.10-12 shows the results of the effect of pulp density on the froth height, ash content and recovery of clean coal. Figure 10 shows the effect of pulp density on froth height. It confirms that with the increase in pulp density the froth height is decreasing irrespective of the reagent dosage. The froth height 4.6 cm for a pulp density of 8% percent solids by weight at a collector and frother dosage of 0.48 kg/ton each whereas the minimum value is 3.0 cm for pulp density of 13%. The change in pulp density leads to variation in the concentration of floatable material in the cell, the effects can increase the chance of collision between bubbles and particles, intensity of turbulence and requirement of aeration rate the froth height is decreasing with the increasing pulp density. For a collector and frother dosages of 0.32, 0.48 kg/t shows the optimum froth height of 4.0 cm at 10% pulp density.

Figure 11, depicts the effect of pulp density on the recovery of clean coal. It is evident from the figure that, increase in the pulp density increases the recovery of clean coal up to 10% of pulp density later the curve tends to reach its minimum. It is believed that the reason for the decrease of recovery with high pulp density is reduced turbulence, caused by dense pulp [22]. The absence of sufficient air bubbles to float the increased number of particles could be another reason for less recovery with denser pulp. However, the maximum recovery of >90% is achieved at minimum collector and frother dosages of 0.32kg/t lead to 24.32 ash%. Hence, collector and frother dosages of 0.32kg/t, 0.48 kg/t respectively at 10% pulp density attains the optimum recovery of 74%.

Figure 12 depicts the effect of pulp density on ash content (%) of clean coal. Figure 12 indicates that increasing frother concentration results in higher recoveries along with increases in the ash content value of clean. There is a clear separation of low ash products obtained from lower collector dosages i.e up to 0.32 Kg/t. The ash content of the cleaner product is increased when the collector dosage beyond the 0.32 Kg/t. However, the pulp density of the feed has not altered the ash content of cleaner coal. From the figure 12, shows the horizontal lines showing almost parallel to each test conditions with varying pulp density.
4. Conclusions

Coal slurry sample received from the coal washery, India with <0.5mm particle size containing ash content of 35.50% with volatile matter content of 20.80% and fixed carbon of 42.90% on dry basis analysis. Petrography study showed the presence of vitrinite, semi vitrinite and liptinite and inertinite as major minerals. General full factorial statistical design package (Minitab V17) was used to develop the regression models for the responses like froth height, froth density, recovery and ash content of clean coal. Response function predictions determined by the regression analysis were found to be in good agreement with the experimental results. Flotation responses like froth height, froth density, recovery and ash content were found to be more sensitive to the frother dosage. The coefficient of correlation ($R^2$) values between the experimental and the predicted values of the flotation responses was found to be >0.98 for all the models.

Subsequently, flotation tests were conducted to study the effect of pulp density on the recovery, froth height, ash content, tests with five levels of pulp density (8, 9, 10, 11, 12 and 13% solids by weight) were carried out. With increasing pulp density, the recovery increased but above optimum densities, approximately 10% for recovery, they decreased as the density increased. Due to the lack of good selectivity, the ash content of the clean coal also increased with higher frother dosage. However, pulp density appears less effect on ash content. It is believed that the reason for the decrease of recovery with high pulp density is reduced turbulence, caused by dense pulp. Insufficient air bubbles to float the increased number of particles could be another reason for less recovery with denser pulp. It found that the coal slurry sample from the study area could be cleaned with sufficient efficiency. The clean coal can be utilized effectively in powder coal consuming industries.
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