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# Ash Reduction of the Tailings of Agh-Darband Coal Washing Plant by Flotation Method

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*A B S T R A C T*

One of the major coal mines in the eastern part of Iran is Agh-Darband mine which is located in Sarakhs. Agh-Darband plant works with a content of less than 37% ash at inputs and about 12% ash in the product. Due to the equipment being depreciated and the lack of optimal conditions for the processing system, a large amount of the coal in feed is entered to the tailing part so that ash content in the tailing is about 75%. The purpose of this research is to investigate whether the flotation method can process the mine tailing with 75% ash content and reduce it to return to the processing circuit. For this purpose, various flotation tests were performed on 70 kg of tailing of Agh-Darband mine. The optimum conditions was obtained at neutral pH with 2000 g/t of oil collector, 30 g/t of pine oil as a frother, and a solid content of 18%, which the ash content of coal and recovery were 34% and 45%, respectively. In addition, using the experimental design software (DX7), the mechanical parameters of the cell such as mixer circulating speed, the distance of the mixer from the bottom, the aeration rate, skimmer circulating speed, and cell volume were optimized. Optimal values of parameters were mixer circulating speed= 1227.2 rpm, the distance of the mixer from the bottom= 0.13H, the aeration rate= 1 (completely open), skimmer circulating speed= 8.49 seconds, and the cell volume= 1 L, which resulted in the percentage of ash= 35.34% and the recovery= 52%.

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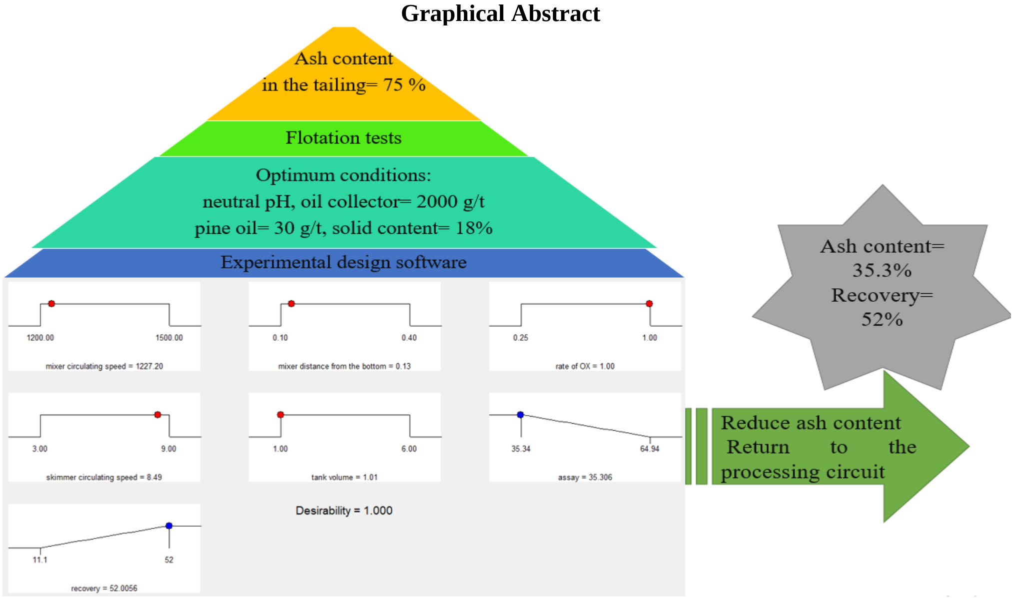
**Graphical Abstract**

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## 1. INTRODUCTION

The first fossil fuel that people used as their main source of energy was coal, one of the most significant energy sources in the world. The production of electricity, iron and iron alloy smelting furnaces, coke, liquefied gas, activated carbon production, and other processes like the production of graphite used in electric furnaces, as a pigment in glass production, hard rubbers, and sewage treatment are the most significant uses of coal (1).

**1. 1. Effective Factors in Coal Processing** Clay minerals comprise an average of 60 to 80 percent of all minerals in coal, making them the most important group. In the presence of water, clays, carbonaceous argillite, and clay shales swell. In the processing of coal, the swelling of clay and shale causes a drop in the apparent specific gravity of coal rejects and their proximity to the apparent specific gravity of their separation, leading to the connection of these materials with coal concentrate.

Sulfur is one of the most significant contaminants in coal that its value varies between 0.5 and 11%.

In gravity processing, the density can be altered by the particle size and shape. How particles flow in a fluid (water, air, or heavy media) is determined by their size and form.

However, massive production of coal is conducted by gravity methods (2-4) but flotation is a significant technique of coal processing for fines (<0.5 mm) (5-8).

**1. 2. Coal Flotation** The development and rapid growth of coal flotation are attributable to three factors (9, 10):

i. Inherent hydrophobicity of coal (organic phase) compared to clay and other minerals in coal

ii. Flotation by flocculation is one of the most effective methods to reduce water turbidity in a coal preparation plant.

iii. Relatively low cost of washing by flotation method in comparison to coal's rising value.

**1. 2. 1. Coal Ash from the Flotation Aspect** The amount of coal ash is one of the most important factors in determining the quality of coal (11). The non-combustible components of coal are known as ash. Before utilization, these materials should be cleansed and removed from the operational environment, as they play an undesirable impact. Increasing the ash content of coke has a negative impact on subsequent phases, increases coke consumption, and decreases its efficiency.

In addition to silicate compounds, oxides, and sulfates, coal ash also contains trace amounts of other chemicals. The chemical composition (XRD analysis) of ash in Agh-Darband mine is shown in Table 1.

**1. 2. 2. Inherent Hydrophobicity of Coal** The contact angle determines the hydrophobic properties of coals with various carbonization levels (12). The greater the proportion of the coal functional group (carboxyl, phenolic hydroxide, and acidic group), the more oxidized the coal surface and the less hydrophobic it is (13).

The zero charge point (ZPC) of coal, as determined by research on coals with a low degree of carbonization, is between pH=3.1 and 4.4. When a non-ionic collector is absorbed on the surface of coal, the zero-point charge of the coal is transported to the alkaline area. Thus, depending on the concentration of the collector, the adsorption process between the collector molecule and the coal surface makes coal flotation viable at a pH close to ZPC. Non-ionic collectors are chemically absorbed on the surface of coal with the assistance of covalently stable hydrogen bonds (14).

A significant volume of tailings produced from processing is generally left as permanent features of the landscape after the mining ceases (15). Coal mining is one of the mines that produces many tailings (ash).

The coal washing plant of the Agh-Darband mine is one of the oldest coal concentrate producing facilities in Iran, receiving roughly 60 tons of coal feed per day. This plant's coal processing system is inefficient due to an outdated and suboptimal method. Therefore, a large amount of the coal in feed is entered to the tailing part so that ash content in the tailing is about 75%. The purpose of this research is to investigate whether the flotation method can process the mine tailing with 75 percent ash content and reduce it. Priority was placed on increasing the circuit efficiency of this plant. The way to reach this goal was to analyze the effective chemical parameters in the flotation system and to determine the ideal values for each parameter, as well as to optimize the mechanical parameters of the cell, thereby increasing the flotation efficiency and decreasing ash.

**1. 3. Literature Review** By mechanical flotation, Naik et al. (16) studied the effect of 5 operational parameters, including the amount of collector, the amount of MIBC frother, pH, particle size, and the effect of sodium silicate, in order to optimize the

**TABLE 1.** The chemical composition of ash in Agh-Darband mine

| Chemical composition | % | Chemical composition | % |
|---|---|---|---|
| $SiO_2$ | 45.65 | Cao | 2.86 |
| $Al_2O_3$ | 28.3 | $Na_2O$ + $K_2$ O | 5.02 |
| $TiO_2$ | 1.95 | $So_2$ | 0.057 |
| $Fe_2O_3$ | 8.07 | MgO | 1.39 |

flotation of non-coking coals with an ash content of 37.7%. In this study, the volume of the collector had the largest impact among the variables, and optimal conditions resulted in 88% recovery and a 25.4% ash percentage .

Tahmasebi et al. examined the decrease of ash in an Agh-Darband coal feed sample in 2006. The sample contained 30% ash on average. Experiments involving vibrating tables were undertaken, including calculating the flow rate of water entering the table and its slope. This technique reduced coal ash by 12.5%. The optimum specific gravity for ash reduction was determined through the use of heavy liquid testing. A concentrate containing 12 percent ash required a medium with a specific gravity of 1.5 g/cm$^3$ in the fraction -850+212 microns. To identify the best state of the effective parameters, flotation tests were performed. Coal ash decreased by 11.5% in the ideal state.

In 2007, Hacifazlioglu and Sutcu (17) conducted research on the optimization of column flotation operational parameters. Their goal was to accomplish optimal separation as well as a comparison of the column and mechanical cells for the Zonguldak coal mine in Turkey, which has 47.5% ash less than 130 microns in size. Their investigation yielded a product with 15.6% ash and 50.92% recovery by column flotation, and a product with 19.52% ash and 82.32% recovery by mechanical flotation (17).

In the same mine, Hacifazlioglu and Toroglu (18) conducted an additional study on the optimization of Jameson flotation cell design parameters and flotation operational parameters, such as the amount of collector, the amount of frother, and the percentage of solids, in order to achieve the optimal separation of bituminous coal containing 45.3% ash. Under optimal conditions, the influence of the examined parameters resulted in 14.9% ash and 74.2% recovery (18).

Jena et al. (19) compared the fatty alcohols ethanol and butanol. They examined the effect of decreasing coal surface oxidation on the flotation ability of sub-bituminous coals oxidized with 42% ash and particles smaller than 1 mm. This study yielded a product containing 31% ash and 80.4% recovery via mechanical flotation, as well as a product containing 26.6% ash and 66.5% recovery via column flotation (19).

Cinar (20) used column and mechanical flotation cells to investigate the effect of low temperature and no temperature heat treatment on the flotation ability of Turkish Soyak lignite coals in 2009. In this investigation, low-temperature heat treatment was used in both column and mechanical cells to increase recovery and the percentage of ash, even without the use of a collector (20).

Anderson et al. (21) explored flotation in an oscillatory baffled column (OBC). Using a sequence of oscillating sinusoidal baffle plates in the liquid and within the flotation cell, they investigated a new stirring mechanism. This technique of stirring demonstrated the ability to generate a uniformly distributed shear rate within the cell. The results demonstrated that OBC could increase the flotation speed constant by as much as 60% for fine particles (less than 30 µm) and by 30% to 40% for larger particles. Therefore, OBC can greatly enhance the flotation rate in typical cells (21).

Shukla et al. (22) evaluated the effect of process variables on gas holdup in a column flotation cell for coal, including feed rate, gasification rate, frother concentration, and solid content. The gas holdup in the collection area was shown to be impacted by both the aeration rate and feeding rate. However, the influence of the feeding rate is negligible in comparison to the gas delivery rate, the gas holdup altered by more than 6 percent when the feeding rate increased from 1 cm/s to 2 cm/s. Furthermore, the addition of frother had a favorable effect. However, as the solids percentage increased, gas holdup dropped (22).

Vapur et al. (23) explored the optimization of a coal flotation system employing modified parameters in a Jameson cell. Using the optimal variables in a Jameson cell, a percentage of recovered combustibles was calculated. The best cell variables were 0.25 mm particle size, 1:1 ratio of vegetable oil to kerosene, 20% solids percentage, and 0.6 l/min washing water flow rate. Finally, in ideal circumstances and an 8-minute flotation time, 94.83% combustible concentrate or 94.86% ash and 17.86% ash were derived (23).

Dashti and Eskandari Nasab (24) studied the impact of hydrodynamic factors on the flotation of coarse coal particles in 2012. The findings of their experiment demonstrated that by increasing the stirrer's speed to 700 rpm, the particles receive sufficient energy to collide and interact with the air bubble, and their recovery rate increases. In addition, the recovery of coarse particles diminishes as the distance of the stirrer from the bottom of the cell increases, as the dead zone under the stirrer expands and the particle suspension becomes less intense. Increasing the concentration of the frother decreased the size of the bubbles and enhanced the stability of the foam phase, resulting in a greater recovery of coarse particles (24).

Pineres and Barraza (25) studied the effect of pH, air velocity, and frother concentration on the recovery of combustible components and the amount of sulfur and ash removed from four samples of Colombian bituminous coal by means of column flotation. The results indicated that the most ash and sulfur were removed from coal from the Guachinate and Nechi regions at acidic pH values of 4 and 5 and air velocities of 1.4 and 1.8 cm/s, respectively. The maximum amount of ash (85.4% and 84.7%) and sulfur (81.1% and 85.1%) removal was obtained in alkaline conditions (pH 9 and 10) with air velocity ranging from 1 to 1.4 cm/s in Creejon and Jagua coals (25).

Chamnet et al. examined the gravimetric and flotation procedures for the Agh-Darband coal mine feed sample. The separation density for the heavy medium test was determined to be 1.53 g/cm$^3$, and the coal concentrate obtained from the vibrating table test included 34.5 weight percent and 15.50 percent ash with a recovery rate of 47.2%. The concentrate contains oil collector at a rate of 1,000 grams per ton, pine oil at a rate of 30 grams per ton as a frother in neutral pH with 20% solids, coal with a weight percentage of 56.2% and ash at a rate of 17.59%, with a recovery of 78.63%. Integrated gravity-flotation tests were also conducted, and flowcharts were produced for this purpose in order to improve accuracy and avoid coal waste in tailings. 75.7% of the coal was recovered, yielding a concentrate with a weight percentage of 56.2% and an ash grade of 16.59%.

Other research in recent years resulted in optimizing the flotation process from different aspects. For example, the study of flotation kinetics (26), determination relationships between operating variables and metallurgical responses of industrial coal column flotation circuits (27), examining the performance of feature engineering for concentrate ash content of coal flotation by froth image feature engineering-based prediction method (28) also based on deep learning algorithms and attention mechanism (29, 30). In addition, the optimization of column flotation for fine coal using Taguchi method (31), and using response methodology and central composite rotatable design (CCRD) (32) are other cases of these optimizations.

## 2. MATERIALS AND METHODS

**2. 1. Instruments** The Denver D-12 flotation laboratory cell was used in this study's flotation tests, and the ash content of the sample was measured after coal flotation. Utilizing an oven, moisture was extracted from the sample. The sieves were used to determine the particle-size distribution. The digital scale was applied to measure the weight of samples with a 0.001-gram resolution. The pH meter was used to determine the pulp's pH. The thermometer is used to measure and control the temperature of coal pulp throughout the flotation process. A filter was also employed in this experiment to extract the liquid, dry the sample after the fine-grinding step, and to dry the flotation concentrate. The sample was reduced to diameters smaller than 2 mm using a roll crusher, and then divided into 2 kg, 1 kg, and head sample bags using a rifle. The sample of approximately 1 mm in size, which is unsuitable for flotation, is crushed to approximately 150 microns using a bond rod mill. Using a planetary ball mill, the sample must be reduced to a particle size below 75 microns in order to determine its ash content following the flotation test.

**2. 2. Sample Preparation** The collected sample weighed approximately 70 kg, and the ash content of the tailings measured at approximately 75%. The sample was crushed, the particle size distribution of the crusher product was determined, and the sample was divided and homogenized. The coal tailings were crushed with a roll crusher and sieved through a 2 mm control screen. Half of the 70 kg sample was crushed to less than 600 microns in size. A quarter of the sample was crushed to less than 2 mm, and another quarter was crushed to less than 1 mm. For each fraction, the weight of the remaining material on each sieve was determined, and then the percentage of these materials and cumulative percentage of the remaining materials on each sieve were calculated.

The half of the sample, whose its d80 were smaller than 600 microns, was divided using a riffle, and 2 kg packages were made from it for use in flotation experiments. Another quarter of the samples that were crushed to less than 2 mm and another quarter of the samples that were crushed to less than 1 mm were packaged in 2 kg quantities so that, if necessary, they can be crushed to sizes smaller than 600 micron.

In order to get the needed optimal particle size for flotation, the sample with size smaller than 600 microns was crushed in a bond rod mill at three different times (3, 5 and 10 minutes) and the ash content and recovery was calculated for each time to determine the optimal time.

**2. 3. Flotation Experiment** After wet grinding of 1 kg of the sample by a bond rod mill to the optimal dimensions, and after placing it in a vacuum filter, 0.5 kg of it was used for the flotation tests. Using the Denver D-12 flotation cell, the experiments were conducted. Thus, pulp containing 19% solids is agitated for 5 minutes at a specific speed, and its pH and temperature were monitored at the same time. Then the collector is inserted, followed by the frother two minutes later. After one minute, the air valve was opened and foaming is performed for three minutes at 5-second intervals. At the completion of the foaming period, a 20-gram sample was made from each of the concentrates and tailings acquired after placing each in a vacuum filter and dryer and sending the sample to be ground for 5 minutes until it is ready to be placed in the furnace. In order to calculate the percentage of ash, 1 gram of the sample is heated in a furnace at 950°C for one hour and then the sample taken out of the furnace was weighed. If the discrepancy between the measurements is greater than 0.005 grams, the procedure must be repeated until the requirements are fulfilled. The percentage of ash is determined by comparing the sample's weight before and after being placed in the furnace (Equation 1):

$$A=\frac{(D-B)}{(C-B)}\times 100 \quad (1)$$

A : Ash content (%)
B : Crucible weight (g)

C : Crucible and sample weight (g)
D: Weight of crucible and sample after burning (g)

The experiments were carried out in duplicate, some were randomly repeated for a third time, and the results were then averaged. Error or standard deviation of repeating experiments was logical.

**2. 4. Flotation Requirements** There are several requirements in coal flotation that are considerably effective in increasing the efficiency of the test if they are fulfilled:

1. The operating temperature should be between 22 and 23°C.
2. The water used is the same water that was used in the laboratory and was determined by pH measurement to be 7.
3. The pulp surface is maintained in every experiment between 12.7 mm and 15.6 mm below the cell edge, which is managed by stirring and aerating.
4. Soaking the coal: Because the sample is dry (not in slurry form), it is necessary to stir it for 5 minutes after mixing it with water and before adding chemicals so that it will float better during the flotation process.
5. Before opening the air flow, chemicals used in flotation are added to the flotation cell. Addition of chemicals (collector and frother) is performed with precision using a pipette or microsyringe.
6. The pH of the pulp is measured prior to the addition of the collector, frother, or any other pre-treatment material.

## 2. 5. Taguchi Test Design for Mechanical Variables

All five investigated parameters are quantitative and each is examined at four distinct levels. The levels of these five parameters are displayed in Table 2.

Using the Taguchi approach, the software developed 16 trials ($L_{16}$) based on the effective parameters. These tests are listed in Table 3 according to their respective levels. All of these trials were conducted in a mechanical laboratory flotation cell. In these studies, the percent of solids, type of frother, type of collector, amount of frother, and amount of collector, etc., were calculated beforehand and considered optimal and consistent.

**2. 6. Optimal Sizes for Flotation** In order to achieve this objective, one kg of the sample with diameters smaller than 600 microns with one liter of

**TABLE 2.** The examined mechanical parameters and their levels

| Parameter level | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Mixer circulating speed (rpm) | 1200 | 1300 | 1400 | 1500 |
| Mixer distance from the bottom | 0.1H | 0.2H | 0.3H | 0.4H |
| Rate of oxygenation | one quarter | half | Three quarters | Full |
| Skimmer circulating speed (rpm) | 20 | 12 | 8.6 | 6.6 |
| Tank volume (lit) | 1 | 2 | 4 | 6 |

Where, H is the cell height.

**TABLE 3.** 16 trials designed by the software (experiment design $L_{16}$)

| Test number | Mixer circulating speed | Mixer distance from the bottom | Rate of oxygenation | Skimmer circulating speed | Tank volume |
|---|---|---|---|---|---|
| 1 | 1 | 1 | 1 | 1 | 1 |
| 2 | 1 | 2 | 2 | 2 | 2 |
| 3 | 1 | 3 | 3 | 3 | 3 |
| 4 | 1 | 4 | 4 | 4 | 4 |
| 5 | 2 | 1 | 2 | 3 | 4 |
| 6 | 2 | 2 | 1 | 4 | 3 |
| 7 | 2 | 3 | 4 | 1 | 2 |
| 8 | 2 | 4 | 3 | 2 | 1 |
| 9 | 3 | 1 | 3 | 4 | 2 |
| 10 | 3 | 2 | 4 | 3 | 1 |
| 11 | 3 | 3 | 1 | 2 | 4 |
| 12 | 3 | 4 | 2 | 1 | 3 |
| 13 | 4 | 1 | 4 | 2 | 3 |
| 14 | 4 | 2 | 3 | 1 | 4 |
| 15 | 4 | 3 | 2 | 4 | 1 |
| 16 | 4 | 4 | 1 | 3 | 2 |

water was crushed using a bond rod mill at three different times (2, 5 and 10 minutes). After filtering the crushed samples, the remaining part on the filter was wet-screened. Figures 1-3 de pict the particle size distribution of the product of a bond rod mill over periods of 10 minutes, 5 minutes, and 2 minutes, respectively. According to the obtained results and with the aid of interpolation, d80 of samples with a crushing duration of 10 minutes, 76.68 microns, 5 minutes, 151 microns, and 2 minutes, 237.22 microns were derived.

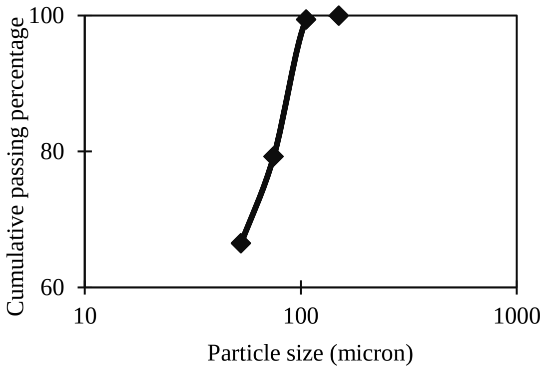


**Figure 1.** Particle size distribution of the product of a bond rod mill (crushing time: 10 minutes)

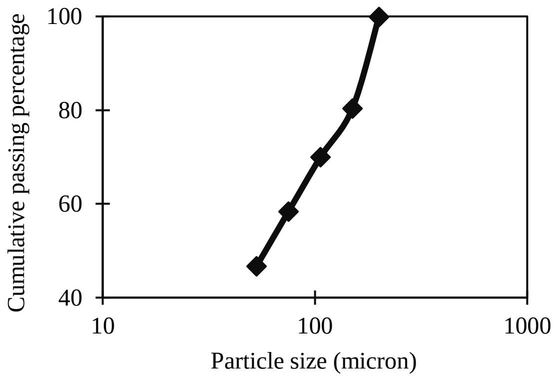


**Figure 2.** Particle size distribution of the product of a bond rod mill (crushing time: 5 minutes)

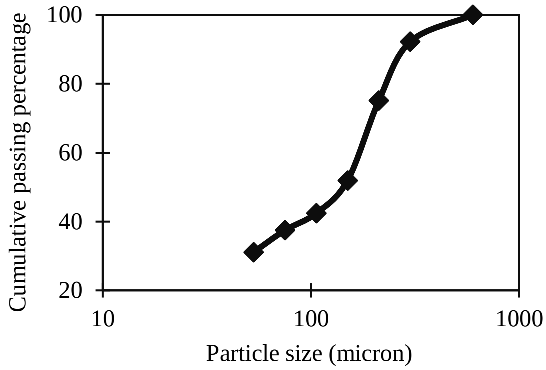


**Figure 3.** Particle size distribution of the product of a bond rod mill (crushing time: 2 minutes)

## 3. RESULTS

### 3. 1. Effect of Particle Size on Ash Content and Coal Recovery

To perform flotation tests, it is necessary to determine the optimal particle size. Based on this, flotation tests were performed for samples with d80s of 1000, 600, 212, 150 and 75 microns. In all experiments, the percentage of solids for coal and coal tailings were chosen as 19% according to the sources and the tests performed. The chemical parameters used are pH = 7.7, 2000 g/t of oil collector, and 0 g/t of pine oil frother.

Figure 4 compares ash content and coal recovery in concentrate part for various particle size. According to this figure, decreasing the size of the sample increases coal recovery in flotation operations, however the ash content is varied. Coal recovery is extremely poor in samples with d80s of 1000 and 600 microns, and the flotation process is not practicable or possible in these sample sizes.

According to Figure 4 and the calculation of the separation efficiency of various sizes, among the sizes of 212, 150, and 75 microns, 150 microns was chosen as the optimal size due to the decrease in ash content to approximately 35% and the increase in recovery to approximately 45%.

### 3. 2. Effect of Collector Dosage on Ash Content and Coal Recovery

Following the measurement of the sample's sizes, the effect of collector concentration was investigated at 1000, 2000, 3000, 4000, and 5000 g/t. Oil collector, solid percentage 19%, pH = 7.7, d80= 150

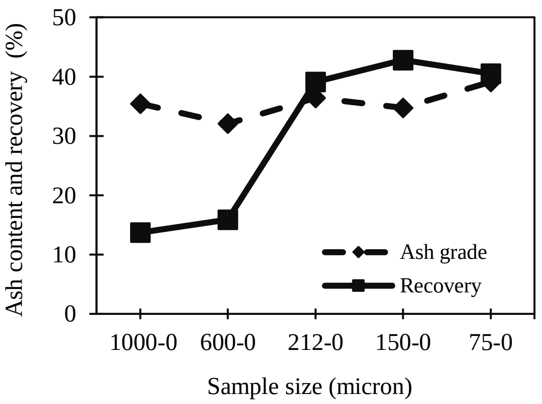


**Figure 4.** Ash content and coal recovery in concentrate part for different d80s

microns, and pine oil frother in the amount of 0 g/t were used in this series of experiments.

According to Figure 5, the grade of the ash increases as the collector concentration rises. Because when the concentration of the collector increases, some waste are also floating with the coal. At a concentration of 1,000 g/t, coal recovery is poor, but it increases by around 10% at a concentration of 2,000 g/t. From a concentration of 2,000 to 5,000 g/t, the range of recovery is essentially the same and there are no discernible alterations.

In accordance with the graph and the calculation of the separation efficiency for various concentrations, the oil collector with a concentration of 2,000 g/t was selected due to the lower grade of ash (approximately 34%) and higher coal recovery (approximately 43%) in comparison to other options.

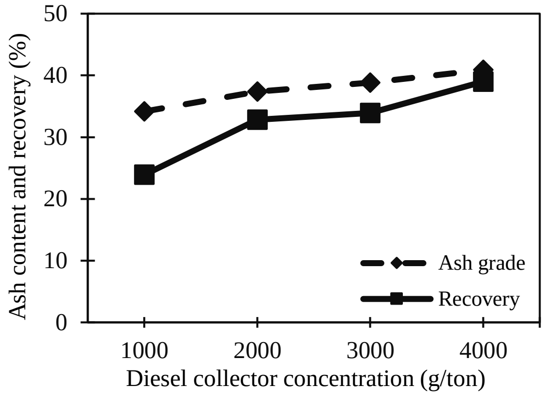


**Figure 6.** Ash content and coal recovery in concentrate for various diesel collector concentrations

**3. 3. Effect of Collector Type on Ash Content and Coal Recovery** The type of collector was explored after determining the dimensions of the sample and the concentration of the collector. The diesel collector was employed in this series of experiments at concentrations of 1000, 2000, 3000, and 4000 g/t (oil was estimated in the previous step), solid percentage 19%, pH = 7.7, dimensions 150 microns, and 0 g/t of pine oil as a frother.

Figure 6 demonstrates that when the concentration of diesel collector increases, so does the recovery, but to a lesser extent than with the oil collector. As concentration rises, the percentage of ash grade also tends to rise, which is more so than with the oil collector.

Comparing the results of oil and diesel collectors reveals that oil collectors have superior performance.

**3. 4. Effect of Frother Concentration** In this set of tests, pine oil frother with concentrations of 10, 20, and 30 grams per ton, 19% solids, pH = 7.7, and 150 microns in size, as well as oil collector with a concentration of 2,000 g/t, were utilized.

Figure 7 shows that raising the concentration of frother to 20 and 30 g/t has no noticeable effect on the ash grade, although the recovery follows a downward and upward trend.

Comparing the results and calculating the separation efficiency led to the selection of 30 g/ton for the pine oil frother.

**3. 5. Effect of Frother Type** In this set of tests, MIBC frother with concentrations of 10, 20, and 30 grams per ton, 19% solids, pH = 7.7, and 150 microns in size, as well as oil collector with a concentration of 2,000 g/t, were utilized. Results of these tests are depicted in Figure 8.

Comparing the results of pine oil frother and MIBC frother reveals an increase in ash grade and a decrease in recovery. Consequently, pine oil frother yields superior results in terms of both ash content and coal recovery.

**3. 6. Effect of Soluble Salts** In this series of studies, the influence of soluble salts on coal flotation was explored using pine oil frother, 19% solids, pH = 7.7, 150 micron diameter samples, and a 2000 g/t oil collector concentration. Table 4 demonstrates that as the concentration of soluble salts increases, so do the ash

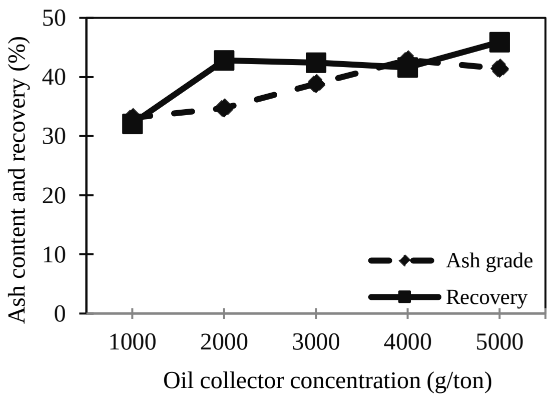


**Figure 5.** The ash content and coal recovery in concentrate for oil collector at various concentrations

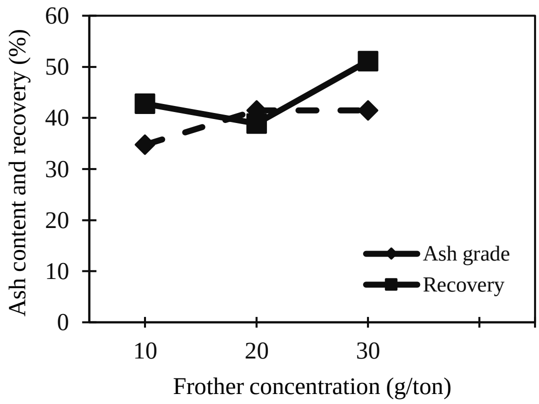


**Figure 7.** Ash content and coal recovery for various concentrations of pine oil frother

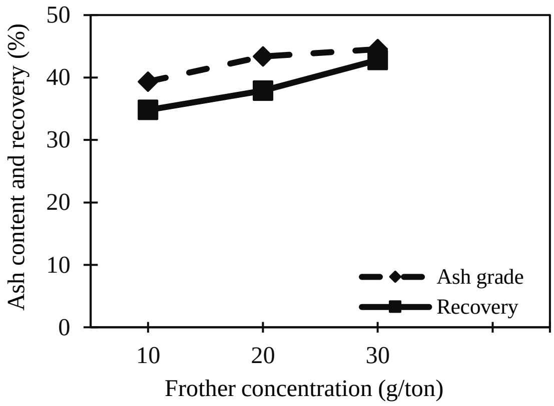


**Figure 8.** Ash content and coal recovery in concentrate as a function of the amount of MIBC frother

grade and coal recovery. However, it can be argued that its function in coal flotation is irrelevant.

**3. 7. Optimizing the Flotation Cell's Mechanical Parameters** Once the chemical parameters of the cell have been determined through a series of tests, the mechanical parameters are optimized. Experiment design by Taguchi method of Design Expert (DX7) software can be used to determine the percentage of effect of each parameter, the influence of each parameter at each level, the optimal settings for each of these parameters, and the mutual influence of each of these parameters. The software obtained the lowest percentage of the ash in the concentrate and the greatest coal recovery with the fewest tests. The mechanical properties of the cell and its surfaces were determined for this purpose using Table 2 in Section 2.5.

**TABLE 4.** Effect of soluble salts on ash production and coal recovery

| The concentration of dissolved salts (g/ton) | Concentrate (%) | | | Tailing (%) | | | Coal content in feed (%) |
|---|---|---|---|---|---|---|---|
| | Weight | Ash content | Coal recovery | Weight | Ash grade | Coal recovery | |
| NaCl (1000) | 17.9 | 38.32 | 40.4 | 82.1 | 80.18 | 59.6 | 72.69 |
| NaCl (2000) | 20.8 | 42.37 | 44.4 | 79.2 | 81 | 55.6 | 72.95 |
| $CaCl_2$(1000) | 21 | 46.88 | 45.6 | 79 | 81.06 | 54.4 | 73.12 |
| $CaCl_2$(2000) | 24 | 47.31 | 48.1 | 76 | 80.04 | 51.9 | 71.56 |
| $FeCl_3$(1000) | 21.5 | 42.95 | 42.7 | 78.5 | 80.85 | 57.3 | 72.56 |
| $FeCl_3$(2000) | 22.5 | 44.32 | 44.2 | 77.5 | 81.31 | 55.8 | 73.36 |

Based on the effective parameters, software (DX7) generated sixteen tests using the Taguchi method (L 16). These tests are listed in Table 3 according to their respective levels.

**3. 8. Statistical Analysis of Data** It might be advantageous to present the results of experiments in the form of an experimental model, or a model that expresses the relationship between the response variable and relevant design parameters (33). The technique employed is response surface (historical data). Given that the conducted tests yielded two results (recovery and percentage of ash), the software analyses were also based on these two variables.

**3. 8. 1. Variance Analysis of the Proposed Ash Content Model** Table 5 displays the significance of the model and recovery model factors.

According to Table 5, the model's P and F values are 0.0001 and 24.89, respectively, which are statistically significant at the 95% confidence level. The greatest value of F is connected to the parameters E (cell volume), BE (the interaction between the distance of the stirrer from the cell bottom and the volume of the cell), and B(the distance of the stirrer from the cell bottom) as depicted in Figure 9. The remaining F values, from highest to lowest, are connected to the parameters D

**TABLE 5.** ANOVA analysis of variance of ash percentage model

| Source | Sum of squares | df | Mean square | F value | Prob>F | |
|---|---|---|---|---|---|---|
| Model | 1467.11 | 7 | 209.59 | 24.89 | <0.0001 | significant |
| A-mixer circulating | 135.25 | 1 | 135.25 | 16.06 | 0.0039 | |
| B-mixer distance | 161.64 | 1 | 161.64 | 19.2 | 0.0023 | |
| C-rate of ox | 28.14 | 1 | 28.14 | 3.34 | 0.1049 | |
| D-skimmer circulating | 153.11 | 1 | 153.11 | 18.19 | 0.0027 | |
| E-tank volume | 501.92 | 1 | 501.92 | 59.61 | <0.0001 | |
| BD | 126.81 | 1 | 126.81 | 15.06 | 0.0047 | |
| BE | 167.51 | 1 | 167.51 | 19.9 | 0.0021 | |
| Residual | 67.35 | 8 | 8.42 | | | |
| Cor total | 1534.46 | 15 | | | | |

(rotation speed of froth collector), A (rotation speed of agitator), BD (mutual impact of agitator distance from the cell bottom and froth collector rotation speed), and C (aeration rate).

Table 6 also displays the model's statistical data. The value of the correlation coefficient derived from the model demonstrates the model's high accuracy.

According to the graph, the projected values and actual values of ash percentage in Figure 10, the distribution of data around the line shows the model's normality and acceptability.

The following is the final equation for the ash percentage of the model based on the parameters of the software code factors:

Grade (%) = +42.23 +4.25A +4.31B -1.79C +4.05D -7.30E +5.23BD -6.3BE

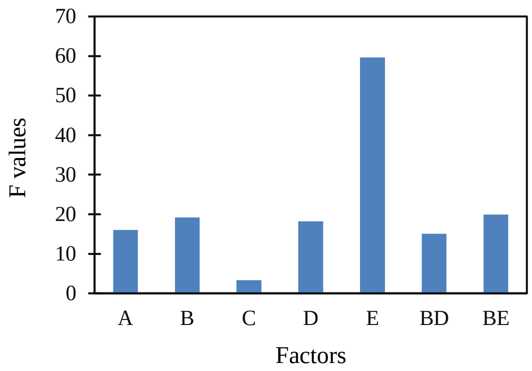


**Figure 9.** The model's effect of factors in terms of F value

**TABLE 6.** Statistical criteria of assessing model validity

| STD. Dev. | 2.9 | R-Squared | 0.9561 |
|---|---|---|---|
| Mean | 42.48 | Adj R-squared | 0.9177 |
| C.V.% | 6.83 | Pred R-Squared | 0.8025 |
| PRESS | 303.1 | Adeq Precision | 15.931 |

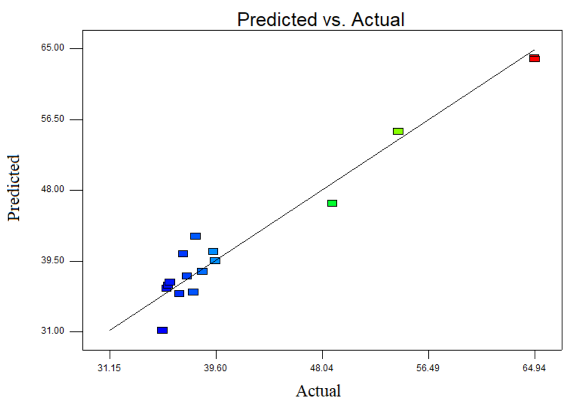


**Figure 10.** The percentage of actual ash relative to the amount anticipated by the model

### 3. 8. 1. 1. Analysis of Geometric Graphs Provided by the Model

Due to the adequacy of the model, the geometric diagrams generated by the model have been evaluated in a single-factor and synchronous manner based on the response-influencing parameters.

### 3. 8. 1. 2. The Effect of Single-factor Parameters

A. Ash's response to stirrer rotating speed.

A linear relationship between stirrer speed and ash quality is depicted in Figure 11; as the speed of the stirrer is increased, so is the ash content.

B. The effect of aeration rate on ash content

As demonstrated in Figure 12, the aeration rate has a minimal impact on the process.

### 3. 8. 1. 3. Interaction of Design Parameters with Coal Ash Content

The software has the capability to examine the effect of parameters under varying circumstances.

A. Mutual effect of the stirrer's distance from the cell bottom and the froth collector's rotation speed on ash (BD)

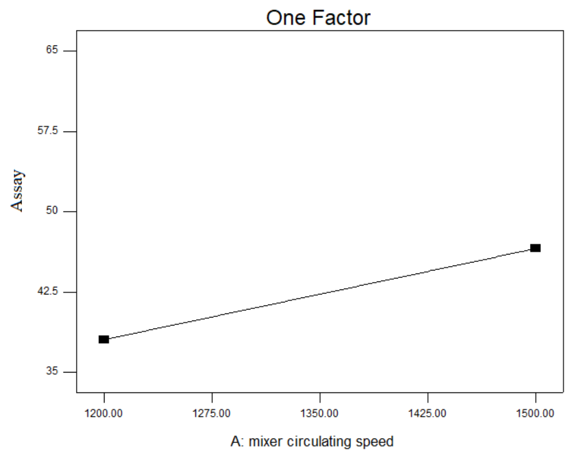


**Figure 11.** Effect of stirrer rotating speed on coal ash

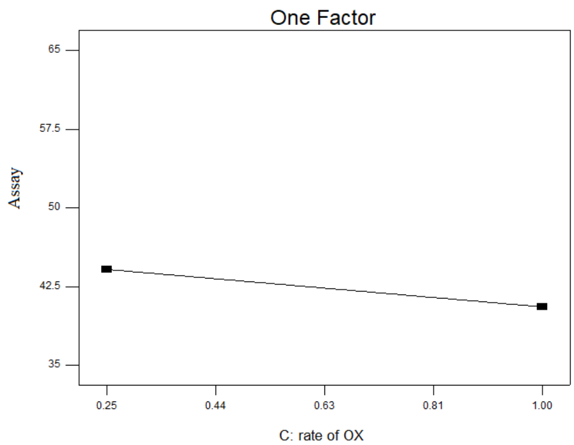


**Figure 12.** Effect of aeration on coal ash

According to the three-dimensional diagram and contour line in Figure 13, the proportion of ash increases as the stirrer's distance from the cell bottom and the froth collector's rotation speed increase. In such a way that the maximum ash percentage occurs at a stirrer distance of 0.4 H and a rotation speed of froth removal every 9 seconds, and the minimum ash percentage occurs at a stirrer distance of 0.1 H and froth removal every 3 seconds.

B. The mutual effect of the stirrer's distance from the froth and the cell's volume (BE)

Figure 14's three-dimensional diagram and contour line indicate that the distance of the stirrer from the cell bottom and the volume of the cell are inversely correlated. The lowest content of ash is found in cells with the highest volume and the stirrer closest to the bottom.

**3. 8. 2. Variance Analysis of the Proposed Model for Recovery** Table 7, Displays the significance of the model and recovery model parameters.

According to Table 7, the model's P and F values are 0.0009 and 11.49, respectively, which are significant at the 95% confidence level. As shown in Figure 15, the maximum value of F corresponds to the parameters C (aeration rate). After that, the highest values are related to $B^2$ (enhanced effect of stirrer distance from the bottom), E (cell volume), D (rotating speed of skimmer), B (stirrer distance from the bottom), and BD (the mutual effect of the distance of the stirrer from the bottom and the rotation speed of the skimmer), respectively.

In Table 8, the model's statistical data are also reviewed. The value of the correlation coefficient derived from the model demonstrates the model's high accuracy.

Figure 16 shows a graph of predicted and actual ash percentage values, and the data distribution around the line proves the model's normality and appropriateness.

Following is the final model recovery equation based on the conditions of software code factors:

Recovery (%) = +35.44 -3.78B +8.92C +4.28D -5.08E -4.38BD -10.3$B^2$

**3. 8. 2. 1. Analysis of Geometry Diagrams Generated by the Model** Due to the adequacy of the model, the geometry diagrams generated by the model have been evaluated in a single-factor and simultaneous manner based on the response-influencing variables.

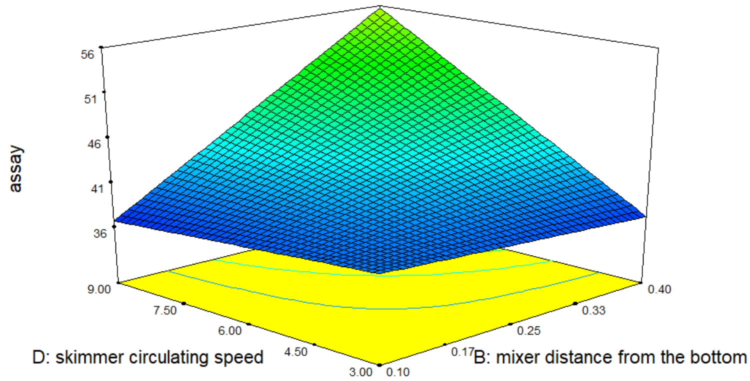


**Figure 13.** The mutual effect of the stirrer's distance from the bottom and the skimmer rotation speed on the percentage of coal ash. A: 3-dimensional B: Contour line

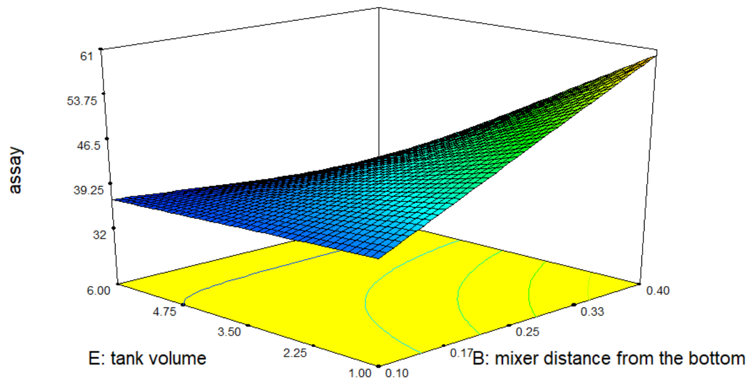


**Figure 14.** The mutual effect of the stirrer's distance from the bottom and the cell volume. A: 3-dimensional B: Contour line

**TABLE 7.** ANOVA analysis of variance recovery model

| Source | Sum of squares | df | Mean square | F value | Prob>F | |
|---|---|---|---|---|---|---|
| Model | 1657.35 | 6 | 276.22 | 11.49 | 0.0009 | significant |
| B-mixer distance | 126.51 | 1 | 126.51 | 5.26 | 0.0475 | |
| C-rate of ox | 705.27 | 1 | 705.27 | 29.34 | 0.0004 | |
| D-skimmer circulating | 173.84 | 1 | 173.84 | 7.23 | 0.0248 | |
| E-tank valume | 243.19 | 1 | 243.19 | 10.12 | 0.0112 | |
| BD | 95.97 | 1 | 95.97 | 3.99 | 0.0768 | |
| $B^2$ | 330.86 | 1 | 330.86 | 13.76 | 0.0048 | |
| Residual | 216.36 | 9 | 24.04 | | | |
| Cor total | 1873.71 | 15 | | | | |

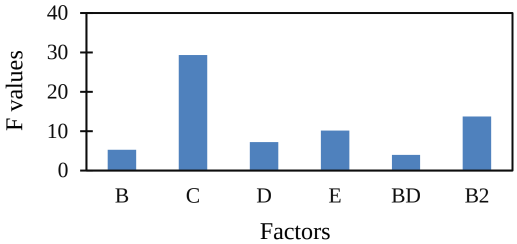


**Figure 15.** The model's influence of factors in terms of F value

**TABLE 8.** Statistical evaluation criteria for model validity

| STD. Dev. | 4.9 | R-Squared | 0.8845 |
|---|---|---|---|
| Mean | 29.99 | Adj R-squared | 0.8075 |
| C.V.% | 16.35 | Pred R-Squared | 0.5658 |
| PRESS | 813.54 | Adeq Precision | 11.105 |

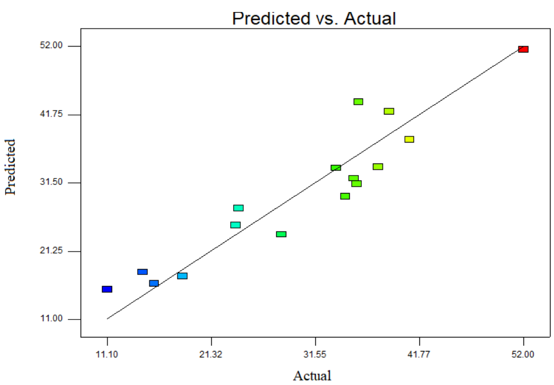


**Figure 16.** Actual recovery compared to the recovery predicted by the model

### 3. 8. 2. 2. Effect of Single Variables

A. Aeration rate

As shown in Figure 17, there is a clear correlation between the aeration rate and the recovery, and by raising the aeration rate and fully opening the aeration valve, a substantial improvement in recovery is observed.

B. Cell volume

According to Figure 18, the relationship between cell volume and recovery is inverse, with recovery decreasing as cell volume increases.

### 3. 8. 2. 3. Mutual Effects of Design Factors on Coal Recovery

The software includes the capability to examine the effect of parameters under varying circumstances.

A- The mutual effect of the rotation speed of the skimmer and the distance of the stirrer from the froth (BD)

The three-dimensional graphic and contour lines in Figure 19 show that the maximum recovery occurs when the stirrer is about 0.2 H away from the tank bottom and the skimmer rotates once every 9 seconds.

### 3. 8. 3. Optimal Values of Parameters in the Design of Experiments

In order to optimize the software parameters in line with the research objectives, the

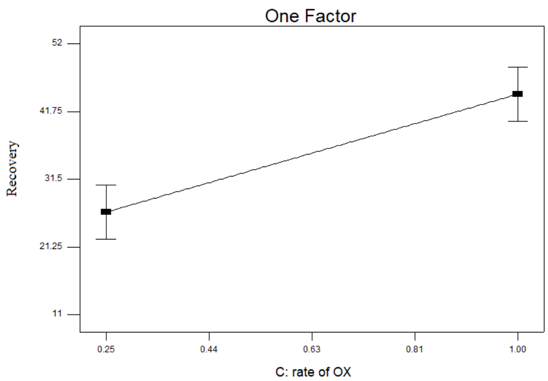


**Figure 17.** Implications of aeration rate on coal recovery

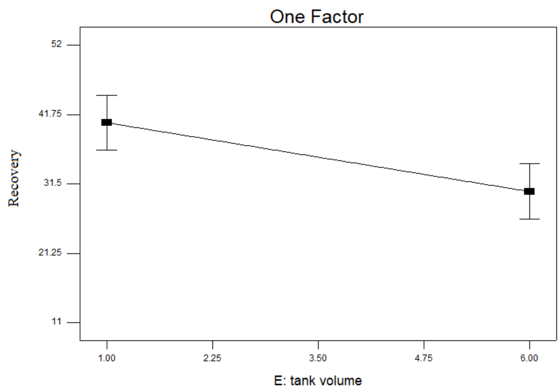


**Figure 18.** Implications of cell volume on coal recovery

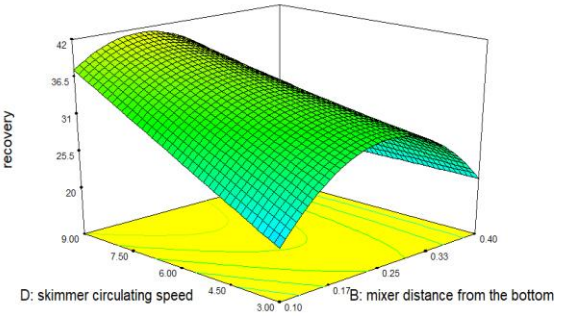


**Figure 19.** The mutual effects between skimmer rotating speed and stirrer distance from tank bottom on coal recovery. A: 3-dimensional B: Contour line

percentage of ash in the minimum state and recovery in the maximum state were considered. Other variables such as the speed of the stirrer, the stirrer distance from the tank bottom, the aeration rate, the speed of the skimmer, and the volume cell were placed within the appropriate range. Figure 20 demonstrates the best mode. The following are the results obtained with a level of utility of 1:
Stirrer rotation speed = 1227.2 rpm
The distance of the stirrer from the tank bottom= 0.13 H
Aeration rate = 1 (fully open)
Rotation speed of skimmer= every 8.49 seconds
Cell volume = 1 liter
Ash percentage = 35.34%
Recovery = 52%

Four experiments were done under the aforementioned conditions to validate its accuracy. In these four experiments, the average recovery and grade of ash were approximately 54% and 33%, respectively.

Ministry of Environment Forest & Climate Change (MoEF&CC) allows use of coal with ash content less than 34% in thermal power plants (34). Therefore, even producing a 34% ash concentrate is valuable and marketable without creating environmental pollution; however, the ash produced in this research returns to the processing circuit.

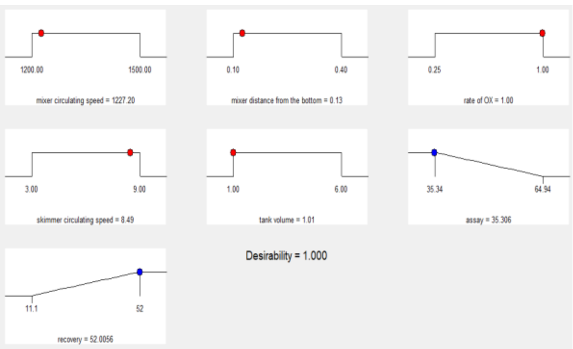


**Figure 20.** Optimal conditions predicted by the statistical model

## 4. CONCLUSIONS

The coal tailings of Agh-Darband mine with 75% ash so far include a volume of 140,000 tons in the place of the tailings depot from the coal washing plant. Therefore, this research was done in order to reach a product with a grade of 30-35% to return it to the processing circuit. In this regard, flotation tests were performed and the chemical and mechanical parameters of the cell were optimized.

Following are the optimal results of flotation tests (chemical parameters) for achieving the lowest ash content and highest recovery:
Particle size range (d80): 150 microns
Solid percentage: 18%
Oil collector: 2000 grams per ton
Pine oil frother: 30 grams per ton
pH: in the neutral range

After optimizing the chemical parameters, the design expert software was used to optimize the mechanical parameters of the cell, including the rotation speed of the stirrer (A), the distance of the stirrer from the tank bottom (B), the aeration rate (C), the rotation speed of the skimmer (D), and the volume of the cell (E). Software opti/mization led to a recovery of approximately 52% and an ash content of approximately 35%:

1. Stirrer rotation speed = 1227.2 rpm
2. The distance of the stirrer from the froth = 0.13 H
3. Aeration rate = 1 (completely open)
4. Rotation speed of the skimmer = every 8.49 seconds
5. Cell volume = 1 liter

Following is the final model recovery equation based on the conditions of software code variables:
Recovery (%) = +35.44 -3.78B +8.92C +4.28D -5.08E -4.38BD -10.3$B^2$

The following is the final equation of the model's ash content according to the requirements of the software code variables:
Grade (%) = +42.23 +4.25A +4.31B -1.79C +4.05D -7.30E +5.23BD -6.3BE

Using the optimal parameter values, the average recovery and ash content were approximately 54% and 33%, respectively.

Based on the reported experiments and results, the following recommendations can be made to continue the work:

- Using positive laboratory results, it is required to undertake semi-industrial tests so that the responses obtained are as similar as possible to industrial settings, making it easier to make the correct decision.
- In accordance with the ash grade (about 33%) produced by optimizing the chemical and mechanical parameters of the cell, the coal tailing washing product may be used as the input of the processing plant's current

circuit, and consequently, the mine coal tailings can be returned to the circuit.

Therefore, producing a 34% ash concentrate is valuable and marketable without creating environmental pollution.

## 5. REFERENCES


1. Leonard III JW. Coal preparation. 1991.
2. Yüce E, Güney A, Onal G, Kangal O, Kökkiliç O, Ozer M, et al., editors. Fine coal evaluation by gravity, methods. XXV International Mineral Processing Congress 2010, IMPC 2010; 2010.
3. Mijał W, Tora B, editors. Development of dry coal gravity separation techniques. IOP Conference Series: Materials Science and Engineering; 2018: IOP Publishing. 10.1088/1757-899X/427/1/012003
4. Phengsaart T, Srichonphaisan P, Kertbundit C, Soonthornwiphat N, Sinthugoot S, Phumkokrux N, et al. Conventional and recent advances in gravity separation technologies for coal cleaning: A systematic and critical review. Heliyon. 2023. https://doi.org/10.1016/j.heliyon.2023.e13083
5. Özbayoğlu G. Coal flotation. Mineral processing design. 1987:76-105. https://doi.org/10.1007/978-94-009-3549-5_4
6. Li L, Lu X, Qiu J, Liu D. Effect of microemulsified collector on froth flotation of coal. Journal of the Southern African Institute of Mining and Metallurgy. 2013;113(11):877-80.
7. Dong Z, Wang R, Fan M, Fu X. Switching and optimizing control for coal flotation process based on a hybrid model. Plos one. 2017;12(10):e0186553. https://doi.org/10.1371/journal.pone.0186553
8. Feng Q, Wang M, Zhang G, Zhao W, Han G. Enhanced adsorption of sulfide and xanthate on smithsonite surfaces by lead activation and implications for flotation intensification. Separation and Purification Technology. 2023;307:122772. https://doi.org/10.1016/j.seppur.2022.122772
9. Taşdemir T, Taşdemir A. Optimization of floc-flotation process in the removal of suspended particles from wastewater in a Jameson cell using central composite design. Journal of Water Process Engineering. 2023;52:103552. https://doi.org/10.1016/j.jwpe.2023.103552
10. Sokolović J, Miskovic S. The effect of particle size on coal flotation kinetics: A review. Physicochemical Problems of Mineral Processing. 2018;54(4):1172-90. https://doi.org/10.5277/ppmp18155
11. Tian Q, Wang H, Pan Y. Associations of Gangue Minerals in Coal Flotation Tailing and Their Transportation Behaviors in the Flotation Process. ACS omega. 2022;7(31):27542-9. https://doi.org/10.1021/acsomega.2c02988
12. Leonard JW, Hardinge BC. Coal Preparation, Society for Mining, Metallurgy, and Exploration. Inc Littleton, Colorado. 1991:467-70.
13. Vamvuka D, Agridiotis V. The effect of chemical reagents on lignite flotation. International Journal of Mineral Processing. 2001;61(3):209-24. 10.1016/S0301-7516(00)00034-X
14. Akdemir Ü, Sönmez İ. Investigation of coal and ash recovery and entrainment in flotation. Fuel Processing Technology. 2003;82(1):1-9. 10.1016/S0378-3820(02)00248-5
15. Kumar CL M, KG S, Sunagar P, Noroozinejad Farsangi E. Studies on Contaminated Mine Soil and Its Remediation Using Soil Washing Technique-A Case Study on Soil at Kolar Gold Fields. International Journal of Engineering, Transactions A: Basics. 2022;35(1):201-12. 10.5829/IJE.2022.35.01A.19
16. Naik PK, Reddy P, Misra VN. Optimization of coal flotation using statistical technique. Fuel Processing Technology. 2004;85(13):1473-85. https://doi.org/10.1016/j.fuproc.2003.10.005
17. Hacifazlioglu H, Sutcu H. Optimization of some parameters in column flotation and a comparison of conventional cell and column cell in terms of flotation performance. Journal of the Chinese Institute of Chemical Engineers. 2007;38(3-4):287-93. 10.1016/j.jcice.2007.03.006
18. Hacifazlioglu H, Toroglu I. Optimization of design and operating parameters in a pilot scale Jameson cell for slime coal cleaning. Fuel processing technology. 2007;88(7):731-6. https://doi.org/10.1016/j.fuproc.2007.03.003
19. Jena MS, Biswal S, Rudramuniyappa M. Study on flotation characteristics of oxidised Indian high ash sub-bituminous coal. International Journal of Mineral Processing. 2008;87(1-2):42-50. https://doi.org/10.1016/j.minpro.2008.01.004
20. Çınar M. Floatability and desulfurization of a low-rank (Turkish) coal by low-temperature heat treatment. Fuel Processing Technology. 2009;90(10):1300-4. https://doi.org/10.1016/j.fuproc.2009.06.017
21. Anderson CJ, Harris M, Deglon D. Flotation in a novel oscillatory baffled column. Minerals Engineering. 2009;22(12):1079-87. 10.1016/j.mineng.2009.04.007
22. Shukla SC, Kundu G, Mukherjee D. Study of gas holdup and pressure characteristics in a column flotation cell using coal. Minerals Engineering. 2010;23(8):636-42. 10.1016/j.mineng.2010.03.005
23. Vapur H, Bayat O, Uçurum M. Coal flotation optimization using modified flotation parameters and combustible recovery in a Jameson cell. Energy Conversion and management. 2010;51(10):1891-7. 10.1016/j.enconman.2010.02.019
24. Dashti A, Nasab ME. Optimization of the performance of the hydrodynamic parameters on the flotation performance of coarse coal particles using design expert (DX8) software. Fuel. 2013;107:593-600. 10.1016/j.fuel.2012.11.066
25. Piñeres J, Barraza J. Effect of pH, air velocity and frother concentration on combustible recovery, ash and sulphur rejection using column flotation. Fuel Processing Technology. 2012;97:30-7. https://doi.org/10.1016/j.fuproc.2012.01.004
26. Bao X, Liu J, Wang S, Chen D, Xu W, Zhang D, et al. New Insight into Temperature Effects on Low-Rank Coal Flotation Using Diesel as a Collector. ACS omega. 2023;8(17):15479-87. 10.22103/JSSE.2023.3816
27. Chelgani SC, Nasiri H, Alidokht M. Interpretable modeling of metallurgical responses for an industrial coal column flotation circuit by XGBoost and SHAP-A "conscious-lab" development. International Journal of Mining Science and Technology. 2021;31(6):1135-44. https://doi.org/10.1021/acsomega.3c00774
28. Wen Z, Zhou C, Pan J, Nie T, Jia R, Yang F. Froth image feature engineering-based prediction method for concentrate ash content of coal flotation. Minerals Engineering. 2021;170:107023. https://doi.org/10.1016/j.mineng.2021.107023
29. Wen Z, Zhou C, Pan J, Nie T, Zhou C, Lu Z. Deep learning-based ash content prediction of coal flotation concentrate using convolutional neural network. Minerals Engineering. 2021;174:107251. https://doi.org/10.1016/j.mineng.2021.107251
30. Yang X, Zhang K, Ni C, Cao H, Thé J, Xie G, et al. Ash determination of coal flotation concentrate by analyzing froth image using a novel hybrid model based on deep learning algorithms and attention mechanism. Energy. 2022;260:125027. https://doi.org/10.1016/j.energy.2022.125027
31. Sachinraj D, Kopparthi P, Samanta P, Mukherjee A. Optimization of Column Flotation for Fine Coal Using Taguchi Method.

Transactions of the Indian Institute of Metals. 2022;75(5):1255-67. https://doi.org/10.1007/s12666-021-02512-2

32. Hazare G, Pradhan SS, Dash N, Dwari RK. Studies on low-grade coking coal characterisation, flotation response and process optimisation. International Journal of Coal Preparation and Utilization. 2023:1-23. 10.1080/19392699.2022.2164572

33. Hosseini Nasab M, Noaparast M, Abdollahi H. Dissolution optimization and kinetics of nickel and cobalt from iron-rich laterite ore, using sulfuric acid at atmospheric pressure. International Journal of Chemical Kinetics. 2020;52(4):283-98. https://doi.org/10.1002/kin.21349

34. NITI Aayog. 2020 - Economic and Environmental Impact of Coal Washing in India National Institution for Transforming India (NITI Aayog). India2020 [Available from: https://policycommons.net/artifacts/2421097/2020/3442689/.



## Persian Abstract

**چکیده**

یکی از معادن بزرگ زغال‌سنگ در شرق ایران، معدن آق‌دربند است که در سرخس قرار دارد. کارخانه آق‌دربند با محتوای کمتر از ۳۷ درصد خاکستر در ورودی و حدود ۱۲ درصد خاکستر در محصول کار می کند. به دلیل مستهلک بودن تجهیزات و نبود شرایط بهینه برای سیستم فرآوری، مقدار زیادی از زغال‌سنگ خوراک وارد قسمت باطله می‌شود به طوری که میزان خاکستر در باطله حدود ۷۵ درصد است. هدف از این تحقیق بررسی این موضوع است که آیا روش فلوتاسیون می‌تواند باطله معدن با محتوای خاکستر ۷۵ درصد را فرآوری کرده و محتوای آن را برای بازگشت به مدار فرآوری کاهش دهد. برای این منظور آزمایش‌های مختلف فلوتاسیون بر روی ۷۰ کیلوگرم باطله معدن آق‌دربند انجام شد. شرایط بهینه در pH خنثی با ۲۰۰۰ گرم در تن کلکتور نفت، ۳۰ گرم در تن روغن کاج به عنوان کف ساز و محتوای جامد ۱۸ درصد به دست آمد که محتوای خاکستر زغال‌سنگ و بازیابی به ترتیب ۳٤ و ٤۵ درصد بود. همچنین با استفاده از نرم افزار طراحی آزمایش (DX7)، پارامترهای مکانیکی سلول مانند سرعت گردش همزن، فاصله همزن از کف سلول، نرخ هوادهی، سرعت گردش کفگیری و حجم سلول بهینه شد. مقادیر بهینه پارامترها عبارت بودند از: سرعت گردش همزن = ۱۲۲۷/۲دور در دقیقه، فاصله همزن از کف سلول= ۰/۱۳ ساعت، نرخ هوادهی = ۱ (کاملا باز)، سرعت گردش کفگیر= هر ۸/٤۹ ثانیه، حجم سلول= ۱ لیتر، درصد خاکستر= ۳۵/۳٤ درصد و بازیابی ۵۲ درصد بود.