

Article

The Recovery of Unburned Carbon from Coal Bottom Ash Using Froth Flotation: The Taguchi Optimization Method

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Abstract: The high consumption of coal in thermal power plants generates a significant amount of bottom ash, most of which is currently disposed of in landfills. However, the presence of unburned carbon in bottom ash limits its potential applications. To enable its use in construction materials, it is essential to reduce the unburned carbon content. Flotation is a promising technique for removing or recovering unburned carbon due to its high throughput and efficiency. This study aimed to optimize key parameters in the flotation process for recovering unburned carbon from bottom ash. The target was to achieve a tailing loss on ignition (LOI) of less than 6%, in accordance with ASTM C618 standards for concrete applications. The parameters evaluated in this study included the frother dosage, collector dosage, air flow rate, and pH. Optimization was conducted using the Taguchi method, which identified frother dosage as the most influential factor in flotation recovery, contributing 80.55%. Additionally, the air flow rate was found to have the greatest impact on combustible recovery (73.34%) and carbon content in the concentrate (41.05%). The optimized process resulted in a tailing LOI of 5.3%, meeting ASTM C618 requirements. These findings demonstrate the effectiveness of the Taguchi method in optimizing the flotation process for bottom ash treatment.

Keywords: flotation; bottom ash; unburned carbon; Taguchi



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1. Introduction

As reported by the International Energy Agency and World Coal Association, coal is the world's single largest source of fuel for electricity production [1]. For many countries coal provides affordable and reliable energy. Coal has been found to be an important source of fuel in Malaysia, where it is widely used in the production of electricity, steel, and cement manufacturing [2]. The efficiency of coal combustion varies depending on the technology used in power plants. The thermal efficiency of a coal power plant can reach 45.5% [3].

The coal-fired power plants currently operating in Peninsular Malaysia include the Manjung power plant (2100 MW capacity) in Perak, the Tanjung Bin power plant (2100 MW capacity) in Johor, the Sultan Salahuddin Abdul Aziz power plant (2420 MW capacity) in Selangor, and the Jimah power plant (1400 MW capacity) in Negeri Sembilan [4]. Collectively, these plants supply for approximately 40% of Malaysia's coal-based electricity generation [5].

Coal ash is the residue from burning coal in power plants; it consists of bottom ash and fly ash. Depending on the product in Malaysia, fly ash is used to produce cement [5]. But bottom ash is generally stored in ponds at the plant, and disposal of it is an ongoing crucial issue to plant operators, which ultimately become a source of pollution [1,3]. Studies on bottom ash are rather limited. But, in the context of circular economy, it has become a necessity rather than a desire to reuse the bottom ash. The unburned carbon in the bottom ash could be recovered [6], and the residue (called tailing ash) could be potentially used as construction materials [7].

Bottom ash appears as porous, shiny, and dark gray particles. It is light, fragile, and highly similar to cement clinker [8]. At the end of combustion, bottom ash, which accumulates in the basement of the combustion chamber, will be directed to a hopper that is initially filled with water. It is then eliminated by high-pressure water jets to decant [9]. Bottom ash is of limited use now due to its high carbon content as compared to fly ash [10]. In order to remove unburned carbon from the bottom ash, a few techniques could be used, such as gravitational separation, electrostatic separation, or froth flotation [11].

Froth flotation has been shown to be an effective method for the recovering of unburned carbon [11]. Uçurum et al. employed a full two-level factorial design to assess the main and interaction effects of variables in the recovery of unburned carbon [12].

The aim of this study is to find the optimum condition for the flotation of bottom ash on recovery of the unburned carbon to improve its application as a constituent in concrete production by reducing the LOI to less than 6% in accordance with ASTM C618. The bottom ash was generated by a power plant in Malaysia. To achieve this, the Taguchi method was applied to identify the optimal operating conditions for the flotation process in recovering unburned carbon and to determine the most influential parameters through analysis of variance (ANOVA). The Taguchi method offers advantages over other optimization techniques by simultaneously optimizing multiple factors that influence performance characteristics. It provides deeper insights with fewer trials, making it a cost-effective strategy [13].

2. Material and Methods

2.1. Materials

The sample used in the present study was collected from the bottom ashes of TNB Janamanjung Power Plant, which is taken directly from the bottom of the combustion chamber. It was then dried in an oven at 60 °C. The samples received were grinded in a ball mill grinder and sieved to various sizes using sieves. The sample size used in this study is 63 µm. Kerosene, a mixture of neutral hydrocarbons, was used as the collector, while MIBC (98% purity, Sigma-Aldrich, USA), a synthetic alcohol commonly used in coal flotation, served as the frother.

2.2. Methods

Nine flotation tests were required for this analysis using the Taguchi method, which is a statistical optimization approach. The column flotation experiments were performed at pH levels of 6, 7.5, and 9, with air flow rates varying from 4 to 10 L/min during a 10 min conditioning phase. The reagents used included kerosene at 1000, 3000, and 5000 g/ton, and MIBC at 1400, 1600, and 1800 g/ton. Kerosene functioned as the collector, while MIBC acted as the frother. To facilitate fundamental research, these flotation tests were conducted in a glass column that had a diameter of 6 cm and a volume of 2.0 L. A schematic diagram is shown in Figure 1. The bottom ash was sieved to a size of 63 µm and loaded at 50 g/L. Each sample was conditioned in water for 5 min to ensure that it was fully wetted at the desired pH before the reagents were added. The conditioning time for kerosene was set at

2 min, while MIBC was conditioned for 30 s. Both reagents were introduced in droplets. The total conditioning period for each test was 10 min. At the end of the experiment, the flotation products (the concentrate) were filtered, dried, and weighed. The recovery (R) and combustible recovery (CR) were calculated using the following equations:

$$R(\%) = \frac{M_c}{M_f} \times 100 \quad (1)$$

$$CR(\%) = \left[\frac{M_c(100 - A_c)}{M_f(100 - A_f)} \right] \times 100 \quad (2)$$

where A_c is ash content of concentrate; A_f is ash content of feed; M_c is mass of concentrate; and M_f is mass of feed.

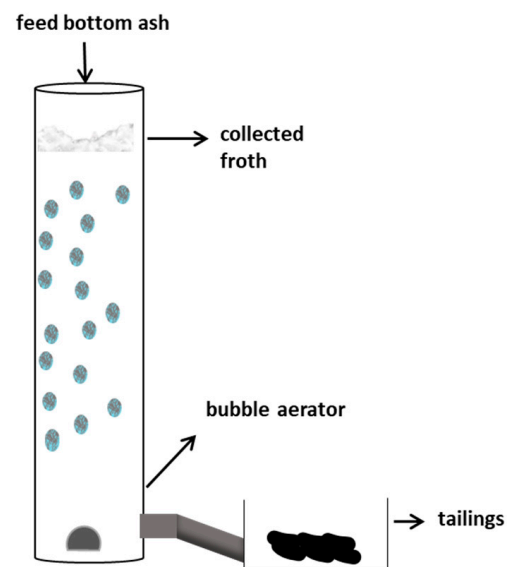


Figure 1. Schematic diagram of flotation process.

Particle size is a critical factor influencing the recovery and entrainment behavior of coal and ash materials. Higher flotation efficiency can be achieved through an initial low-energy input for the recovery of easily floatable materials, followed by a higher energy input for the recovery of difficult-to-float materials [14]. Composite collectors were shown to enhance flotation performance and efficiency, particularly for low-rank coal. Effective interaction between the collector and coal particles is essential for improving flotation performances [15].

Analytical Methods

Chemical analysis of the coal bottom ash samples was conducted using an X-ray fluorescence (XRF) (ZSX Primus IV), with the findings summarized in Table 1. The elements are reported as their oxides, including SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , and CaO , since the sample is a byproduct of combustion. The XRF analysis indicated that the total content of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ in the bottom ash is 69.07%, with a ratio surpassing 50%, and the loss of ignition was measured at 15%. Based on ASTM C618-08a, this chemical composition classifies the ash as “C” type ash, which possesses pozzolanic and cementitious properties, making it appropriate for use in the concrete industry [12].

Table 1. Results of XRF analysis of coal bottom ash.

Component	Coal Bottom Ash (%)
SiO ₂	43.61
Al ₂ O ₃	14.47
Fe ₂ O ₃	10.99
CaO	8.82
MgO	3.45
K ₂ O	0.85
Na ₂ O	0.83
TiO ₂	0.76
SO ₃	0.75
Cl	0.28
P ₂ O ₅	0.17
BaO	0.12
MnO	0.09
SrO	0.08
ZrO ₂	0.06
V ₂ O ₅	-
Cr ₂ O ₃	0.01
CeO ₂	0.01
ZnO	0.01
NiO	0.01
LOI	15

The carbon content was determined using the loss of ignition (LOI) test, which is a standard analytical technique method. Loss of ignition is frequently used to evaluate volatile compounds, unburned carbon, and moisture in solid materials [16]. This method provides a practical way to determine what might have burned, provided that the combustion residue has not absorbed water after being removed from the furnace.

2.3. Taguchi Design of the Experiment

The Taguchi design method employs fractional factorial test designs known as orthogonal arrays (OAs), which help reduce the number of experiments required. The selection of an appropriate OA is based on the number of control factors and their respective levels. By utilizing OA designs, multiple process variables can be evaluated at the same time for their impact on performance characteristics, while minimizing the number of test runs. Orthogonal arrays significantly decrease the number of experimental procedures needed for investigation [17]. The design of experiments (DOE) in the Taguchi method provides a clear, efficient, and systematic approach to identifying optimal conditions [15–18]. In this study, the L9 OA (four parameters, each at three levels) was implemented. To ensure consistent results, each test was conducted three times under identical conditions. The L9 OA was used to establish the instrumental parameters shown in Table 2, where each row indicates a trial condition along with the corresponding factor levels.

Table 2. Selected reaction control factors and respective level.

Level	Factor			
	Frother Dosage (g/t)	Collector Dosage (g/t)	Air Flow Rate (L/min)	pH
	A	B	C	D
1	1400	1000	4	6
2	1600	3000	7	7.5
3	1800	5000	10	9

2.4. Signal-to-Noise Ratio

In the Taguchi method, the term ‘signal’ refers to the desired value (mean), while ‘noise’ indicates the undesirable value (standard deviation) for the output characteristic. Therefore, the signal-to-noise (S/N) ratio is the relationship between the mean and the standard deviation. This ratio is utilized to convert the quality characteristics [19]. The equation for the S/N ratio changes depending on the optimization criteria for these characteristics. As illustrated in Table 3, there are three types of S/N ratios: (1) smaller is better, (2) nominal is best, and (3) larger is better. Since this study aims to maximize the recovery of unburned carbon and carbon content, the S/N ratio for the “larger is better” scenario was used. The influence of each process parameter on the S/N ratio at various levels for each sample is shown based on the orthogonal experimental equations [20].

Table 3. S/N ratio experimental equations.

Characteristic	S/N
Nominal is the best	$-\log_{10}\left(\frac{1}{n}\sum_{i=1}^n(y^2 - y_0^2)\right)$
Larger is better	$-10\log\left(\frac{1}{n}\sum_{i=1}^n\frac{1}{y_i^2}\right)$
Smaller is better	$-\log_{10}\left(\frac{1}{n}\sum_{i=1}^n y_i^2\right)$

2.5. ANOVA

Analysis of variance (ANOVA) is a statistical technique used to evaluate the average response level (% contribution) of each parameter in orthogonal experiments. It was applied to analyze the results from the designed L9 OA (L9 (34)) and to indicate the most significant parameters [21]. The impact of each experimental factor (frother dosage, collector dosage, air flow rate, and pH) was assessed using one-way ANOVA. A one-dimensional static analysis allows for a more straightforward evaluation of the individual effects of each parameter on the flotation process. The one-dimensional approach, as applied in the Taguchi method, efficiently identifies the most influential factors with a reduced number of experiments. This ensures a balance between accuracy and practicality in optimizing the flotation parameters. The goal of ANOVA is to determine which process parameters affected the flotation performance of bottom ash [19].

3. Results and Discussion

The Taguchi orthogonal array, illustrated in Table 4, was created using Design Expert, a statistical design software package. The optimization results of bottom ash by flotation using Taguchi are shown in Table 5. In this work, the experimental outcomes were recovery (R), combustible recovery (CR), and carbon content in concentrate (CC), which are 49.84%, 97.46% and 69.65%, respectively.

ANOVA for (a) recovery, (b) combustible recovery, and (c) carbon content of concentrate with the percentage of contribution of each factor and their interactions are presented in Table 6. The finding revealed that the frother dosage had the maximum impact on recovery. The highest variance and contribution percentage was at 80.55%, followed by pH (17.70%), air flow rate (1.14%), and collector dosage (0.52%). For combustible recovery, air flow rate was the most influential parameter contributing 73.33%, followed by frother dosage (20.67%), collector dosage (5.18%), and pH (0.812%). For carbon content in the concentrate, air flow rate was the most significant factor, contributing 41.05%. The next most important factors were frother dosage (40.60%), pH (17.18%), and collector dosage (1.17%), respectively.

Table 4. Orthogonal array of process variables for optimization.

	Frother Dosage (g/t)	Collector Dosage (g/t)	Air Flow Rate (L/min)	pH
Run	A	B	C	D
1	1400	1000	4	6
2	1400	3000	7	7.5
3	1400	5000	10	9
4	1600	1000	7	9
5	1600	3000	10	6
6	1600	5000	4	7.5
7	1800	1000	10	7.5
8	1800	3000	4	9
9	1800	5000	7	6

Table 5. Taguchi orthogonal experimental results for recovery, combustible recovery, and carbon content of concentrate (mean $\pm$ SD, replicated 3 times) as well as the respective calculated S/N values.

Factors					Results					
Run	Frother Dosage (g/t)	Collector Dosage (g/t)	Air Flow Rate (L/min)	pH	R (%)	S/N (R)	CR (%)	S/N (CR)	CC (%)	S/N (CC)
1	1400	1000	4	6	30.71 $\pm$ 0.64	29.75	94.59 $\pm$ 0.16	39.52	46.20 $\pm$ 0.06	33.29
2	1400	3000	7	7.5	33.40 $\pm$ 0.61	30.47	95.11 $\pm$ 0.07	39.56	42.72 $\pm$ 0.27	32.61
3	1400	5000	10	9	26.14 $\pm$ 0.00	28.31	88.21 $\pm$ 0.06	38.91	50.63 $\pm$ 0.20	34.09
4	1600	1000	7	9	20.13 $\pm$ 0.03	26.08	93.48 $\pm$ 0.25	39.41	69.65 $\pm$ 0.33	36.86
5	1600	3000	10	6	25.44 $\pm$ 0.32	28.11	85.94 $\pm$ 0.66	38.68	50.67 $\pm$ 0.12	34.11
6	1600	5000	4	7.5	25.2 $\pm$ 0.27	28.05	94.66 $\pm$ 0.43	39.52	56.18 $\pm$ 0.66	34.99
7	1800	1000	10	7.5	49.84 $\pm$ 1.00	33.95	86.87 $\pm$ 0.32	38.78	26.15 $\pm$ 0.05	28.35
8	1800	3000	4	9	35.78 $\pm$ 0.40	31.07	93.44 $\pm$ 0.80	39.41	39.17 $\pm$ 0.20	31.86
9	1800	5000	7	6	41.09 $\pm$ 0.05	32.28	97.46 $\pm$ 0.43	39.78	35.5 $\pm$ 70.09	31.02

Table 6. Analysis of variance (ANOVA) for recovery, combustible recovery, and carbon content of concentrate.

Source	Degree of Freedom	Sum of Squares	Variance	Percentage Contribution
frother dosage (g/t)	2	38.11	19.05	80.55
collector dosage (g/t)	2	0.24	0.12	0.52
air flow rate (L/min)	2	0.54	0.27	1.14
pH	2	8.42	4.21	17.79
Error	0	0	0	0
Total	8	47.31	5.91	100

Source	Degree of Freedom	Sum of Squares	Variance	Percentage Contribution
frother dosage (g/t)	2	0.25	0.12	20.67
collector dosage (g/t)	2	0.06	0.03	5.18
air flow rate (L/min)	2	0.88	0.43	73.33
pH	2	0.01	0.005	0.812
Error	0	0	0	0
Total	8	1.19	0.15	100

Source	Degree of Freedom	Sum of Squares	Variance	Percentage Contribution
frother dosage (g/t)	2	18.99	9.49	40.60
collector dosage (g/t)	2	0.55	0.27	1.17
air flow rate (L/min)	2	19.20	9.6	41.05
pH	2	8.04	4.02	17.18
Error	0	0	0	0
Total	8	46.77	5.85	100

The S/N ratio graph in decibel (dB) for response parameters is shown in Figure 2 for recovery, Figure 3 for combustible recovery, and Figure 4 for the carbon content of concentrate for the effects of (a) frother dosage (g/t), (b) collector dosage (g/t), (c) air flow rate (L/min), and (d) pH. The purpose of this work is to maximize the response (quality of flotation) with the highest S/N ratio. The higher S/N ratio indicates better quality characteristics [21]. It is reported that the parameter is more significant when the noise factor is low [22].

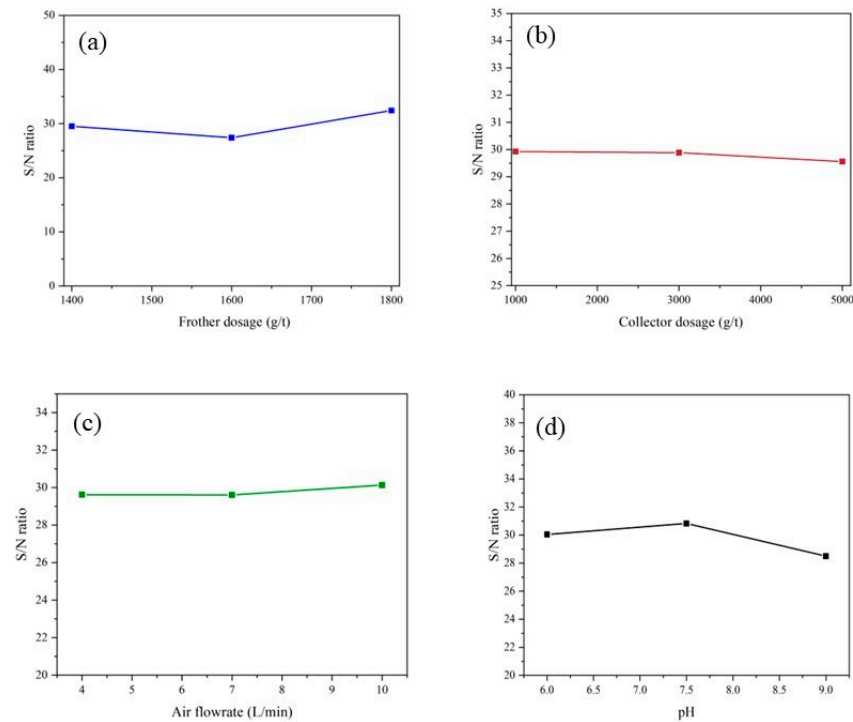


Figure 2. S/N ratio plots for the effects of (a) frother dosage (g/t), (b) collector dosage (g/t), (c) the air flow rate (L/min), and (d) pH for recovery (R).

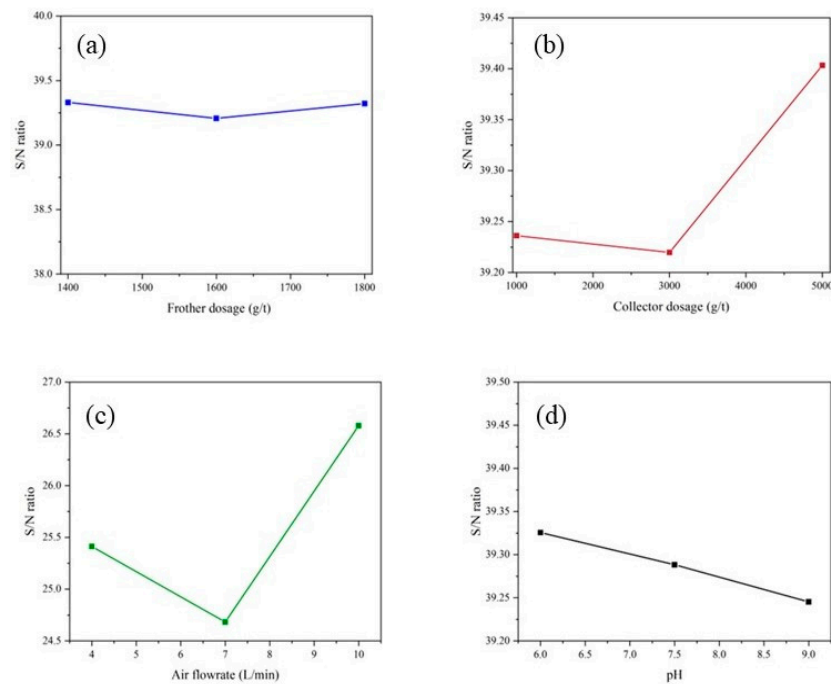


Figure 3. S/N ratio plots for the effects of (a) frother dosage (g/t), (b) collector dosage (g/t), (c) the air flow rate (L/min), and (d) pH for combustible recovery (CR).

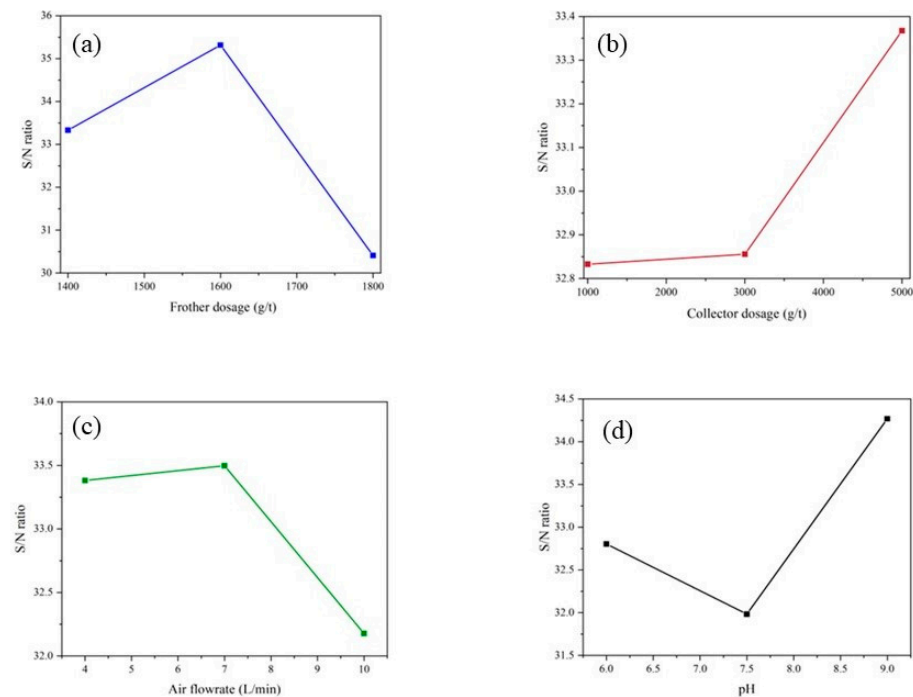


Figure 4. S/N ratio plots for the effects of (a) frother dosage (g/t), (b) collector dosage (g/t), (c) the air flow rate (L/min), and (d) pH for carbon content of concentrate (CC).

Table 7 shows the comparison of actual experimental data with predicted values. Based on the results, the experimental values were quite close and comparable to the predicted values. Thus, these results revealed that the Taguchi methodology could be applied effectively for optimizing the recovery of unburned carbon by flotation process.

Table 7. Experimental and predicted optimal conditions for the flotation.

				Prediction		Experiment	
Factor				S/N Ratio Predicted	Recovery	S/N Ratio Actual	Recovery
A	B	C	D	dB	%	dB	%
1800	1000	10	7.5	33.95	49.84	33.99	50.11
				Prediction		Experiment	
Factor				S/N Ratio Predicted	Combustible Recovery	S/N Ratio Actual	Combustible Recovery
A	B	C	D	dB	%	dB	%
1800	5000	7	6	39.78	97.46	39.69	96.57
				Prediction		Experiment	
Factor				S/N Ratio Predicted	Carbon Content	S/N Ratio Actual	Carbon Content
A	B	C	D	dB	%	dB	%
1600	1000	7	9	36.86	69.65	36.83	69.45

4. Conclusions

This study employed the Taguchi orthogonal experimental design to optimize the froth flotation process for bottom ash treatment, aiming to maximize both carbon recovery and carbon content. The effects of key process parameters—frother dosage, collector dosage, pH, and air flow rate—were systematically investigated. Signal-to-noise (S/N) ratio plots identified the optimal parameter combination: a frother dosage of 1800 g/t, a collector dosage of 1000 g/t, an air flow rate of 10 L/min, and pH 7.5. Additionally, ANOVA analysis

provided valuable insights into the most influential parameters. The results revealed that frother dosage had the greatest impact on flotation recovery (80.55%), while air flow rate was the most critical factor for combustible recovery (73.34%) and carbon content in the concentrate (41.05%).

These findings highlight the key governing factors in the flotation process and demonstrate the effectiveness of the Taguchi method in optimizing bottom ash treatment. This approach also enhances waste management and byproduct utilization by enabling secondary applications for recovered carbon, such as fuel reuse or activated carbon production. Furthermore, the low-LOI bottom ash can be repurposed in construction materials, reducing landfill disposal and promoting sustainability.

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