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Development of magnetic flotation hybrid separation process for cleaner coal preparation

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A R T I C L E I N F O

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A B S T R A C T

The magnetic flotation hybrid separation process, which combines column flotation and magnetic separation, was anticipated to beneficiate a representative coal sample acquired from the El-Maghara coal mine with a relatively high ash percentage of 27.21%. The system was designed to introduce a high gradient magnetic field to a flotation column to hinder the flotation of magnetic particles even if attached to flotation bubbles, improving the coal demineralization process and producing high quality coal. The Box-Behnken design and response surface methodology were employed to investigate and optimize the combined influence of various operating parameters on the process performance. The effects of the main operating parameters such as magnetic field strength, slurry circulation flow rate, air flow rate, collector dosage, and frother concentration were investigated. Ash percentage, combustible recovery, and separation efficiency were defined as process responses. Upgrading the coal sample under the optimum conditions of 1.2 T magnetic field strength, 1500 ml/min circulation flow rate, 11.64 ml/min air flow rate, 0.87 kg/ton diesel collector, and 42 ppm methyl isobutyl carbinol frother, the predicted and two confirmation experiments data were 7.78%, 7.89%, 7.71% ash percentages with maximum recoveries of 75.86%, 76.37%, 75.94%, and maximum separation efficiencies of 77.17%, 78.51%, 78.71% respectively.

1. Introduction

Coal is regarded as one of the most important non-renewable energy sources. It plays an essential role in power generation, supplying more than 40% of electricity for the world (Xia et al., 2015; Yassin et al., 2022). Before coal burning, a considerable amount of the ash-forming mineral compounds such as carbonates, sulfates, phosphates, oxides, and sulfides that are often found in the structure of coal have to be removed (Lakhmir et al., 2022; S. Yahaya Babatunde and A. A. Adeleke, 2014). This mineral matter is a non-burning contaminant that reduces the handling and combustibility of coal. Therefore, cleaning coal before use is essential for producing cleaner fuels and reducing environmental impacts (Bykov et al., 2001; Jones et al., 2002; Uslu and Atalay, 2004). Demineralization/desulfurization of coal before combustion was accomplished using physical methods (Baruah et al., 2000; Çelik and Yildirim, 2000; Das et al., 2010; Özgen et al., 2011), bio-processing (Koyunoğlu and Karaca, 2023), microwave technique (Bykov et al., 2001; Jones et al., 2002; Uslu and Atalay, 2004), and chemical methods (Karaca and Yildiz, 2007; Meshram et al., 2015). It was reported that combining physical beneficiation with the chemical cleaning of coal has the potential for significant mineral matter reduction with less cost and wastewater generation (Meshram et al., 2015). On the other hand, the flotation process has been considered one of the most effective physical–chemical procedures to purify fine and ultra-fine coals and produce coal concentrate with low ash and sulfur contents (Dong et al., 2017; Hacifazlioglu, 2011; Onel and Tanriverdi, 2020; Xia et al., 2015).

Coal could be de-sulfurized and de-ashed using a magnetic separation process (Bancrjec and Dixit, 2007; Trindade and Kolm, 1973). For instance, a wet high-gradient magnetic separator was investigated for the separation of pyritic sulfur from Egyptian coal (Ibrahim et al., 2016), and the separation of pyritic sulfur from ultra-fine coal was assessed in another study using pyrolysis and a wet high magnetic separation procedure (Yassin et al., 2022).

The magnetic separation as well as froth flotation has long been

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Table 1
Proximate analysis of the original coal sample.

Analysis	(%)
Moisture	1.12
Ash in coal	27.20
Volatile matter	14.20
Fixed carbon	57.50

utilized to upgrade minerals and materials. Combining both can treat a wide range of types of materials from colloidal to large size, from non-magnetic to strongly magnetic, and from hydrophilic to hydrophobic in many applications such as steel production, coal preparation, kaolin de-colorization, wastewater treatment and metal recovery, food processing, protein and DNA purification, and bio catalysis (Yavuz et al., 2009).

The floatability of magnetic particles may be reduced by using a magnetic field during the flotation process (López-Valdivieso et al., 2018). The magnetic field generated by the coils of the C-shaped magnet of the Davis tube apparatus was employed to hinder magnetite flotation when floating chromite which improves the separation selectivity (Yousef et al., 2013). Another technique utilizing a similar concept includes using the high-intensity magnetic field to remove pyrrhotite from the froth phase as it exited the flotation cell (Yalçın et al., 2000). Similarly, a modification was conducted to a conventional WEMCO flotation cell and utilized for magnetic flotation experiments by inserting magnetic grids into the flotation cell to create a magnetic field; the magnetic grids cover the whole flotation surface and are in contact with the pulp/froth (Ersayin and Iwasaki, 2002). A similar study has been carried out to evaluate the influence of an external magnetic field produced by three solenoid coils wrapped around a micro-flotation column to inhibit magnetite flotation in quartz cationic flotation (Birinci et al., 2010). Although a magnetic field enhanced separation efficiency during quartz flotation from magnetite, with a large magnetic field, magnetic flocs consisting of magnetite particles adhere to the column wall, causing material transport difficulty. Although the magnetic flotation technique is well known, it has received little attention and is still being considered.

This study aims to design and fabricate a new magnetic flotation hybrid system that has the capability of beneficiating a complex ore containing more than two minerals based on the differences in magnetic and hydrophobic characteristics. The developed system is utilized for obtaining low-ash coal with high combustible recovery and separation efficiency from El-Maghara coal. The effect of operating parameters in this new system is fully investigated. Then, the performance of this system is evaluated through experiments test design to determine the influence of significant operation parameters, including magnetic field strength, air flow rate, slurry circulation flow rate, collector dosage, and frother concentration. The presented five variables are considered in designing the experiment sequence using the Box-Behnken experimental design and response surface technique by conducting Design Expert 13 software to identify the optimal operational conditions.

2. Experimental

2.1. Coal sample

A representative coal sample used for this study was acquired from the El-Maghara coal mine located in Sinai, Egypt. The original coal was firstly crushed to −3.0 mm using a Denver jaw crusher, followed by rod milling to produce 95% less than 100 μm ground coal. For better liberation between combustible material and mineral matter, the rod mill has been used since it is beneficial for grinding coarser particles compared with ball mill grinding (Ma et al., 2022). The ground coal was mixed, divided into smaller portions, and saved for later use. A representative coal sample was analyzed using proximate and X-ray diffraction (XRD). The proximate analysis of the sample is shown in Table 1. The XRD pattern shown in Fig. 1 indicates that the coal mineral matter contains mainly quartz, calcite, dolomite, and pyrite.

2.2. Reagents

Analytical grade of diesel oil and Methyl Isobutyl Carbinol (MIBC) were purchased from Sigma-Aldrich. Diesel oil was used as a collector to increase the hydrophobicity of coal, and MIBC was used as a frother to stabilize the froth and make it stable enough to hold accumulated minerals in the froth zone.

2.3. Fabricating magnetic flotation hybrid system

A canister made of plexiglass of 2 cm width, 9 cm length, and 20 cm height is placed vertically between two specific geometry poles made of electromagnet coils on a U-shaped iron enclosure. The poles are made of highly pure soft iron material and their dimensions from the U-shaped iron enclosure gradually decrease to 8 cm width and 16 cm height to increase the magnetic field strength up to 1.8 Tesla. Whereas, the direct electric current of different intensities passes through the energizing

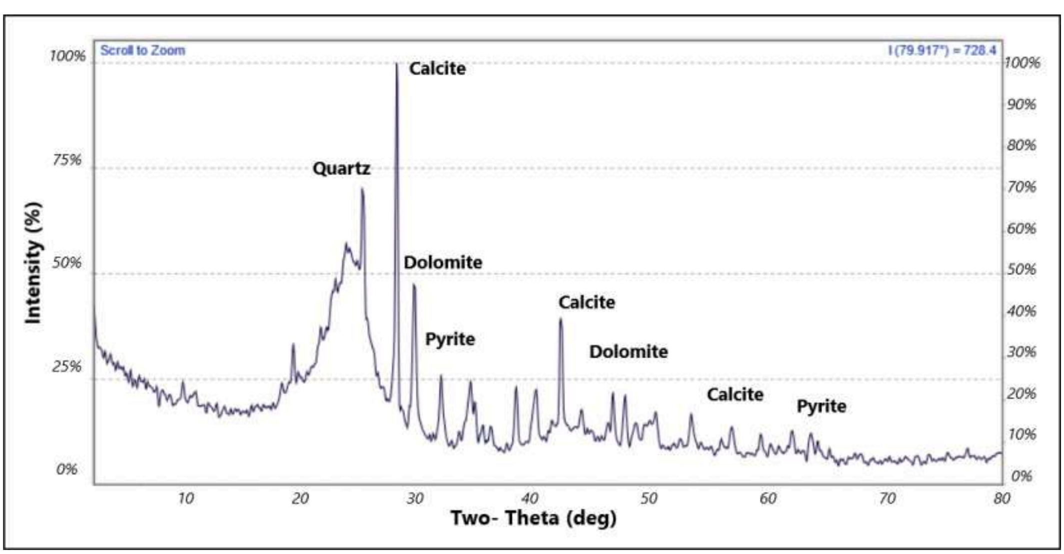

Fig. 1. XRD pattern of the coal sample.

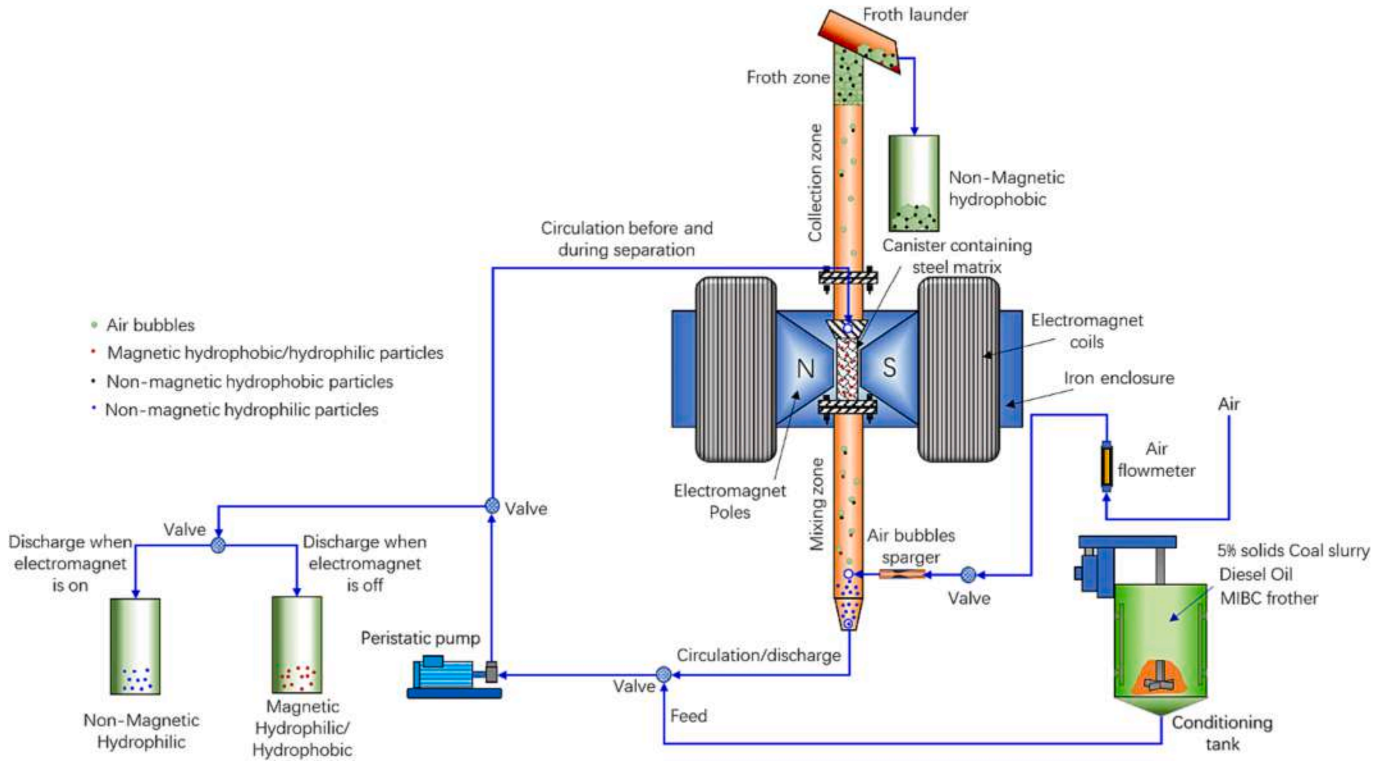

Fig. 2. Schematic diagram of magnetic flotation hybrid separation process.

coils to establish magnetic field intensities of high gradient, where the produced magnetic field strength can be easily adjusted from 0.2 to 1.8 T.

The canister is filled vertically with 8 mesh-type sheets of the steel matrix, side by side, staggered, and reversed. The sheets' dimensions are 0.1 cm in width, 8 cm in length, and 18 cm in height. The inserted matrix was positioned in the separating zone between the electromagnetic poles to generate a high magnetic field gradient and to capture weak magnetic particles from flow during the separation process.

Upper and lower columns were fabricated and connected with Plexiglas canister. The upper flotation column was made of plexiglass of 6.0 cm diameter and 76 cm height. It consists of collection and froth zones. There is a plexiglass froth launder at the top of the column of 6.0 cm diameter with 30° angle inclination to allow the flow of the nonmagnetic hydrophobic particles/froth to the froth receiver.

The inlet downcomer is a plexiglass column with a diameter of 6 cm and a length of 28 cm connected upward with the rectangular canister. The lower column has an air inlet slightly above a circulation/discharge outlet at the bottom. It is utilized for slurry mixing with air bubbles, for collecting hydrophilic nonmagnetic particles during the separation process, and for collecting magnetic particles after the separation process. At the air inlet, a sparger is instilled to generate bubbles by aerating the particle suspension under severe shear forces. There is a feed/circulation inlet slightly above the canister for feeding the slurry then circulating it from the circulation outlet at the bottom of the lower column before and during the separation process. The magnetic flotation system with a 124 cm overall height was made from plexiglass for the optical visualization of the separation process. Fig. 2 shows the specially designed magnetic flotation hybrid system used in this work.

2.4. Methodology

The magnetic flotation hybrid process is a froth flotation integrated with magnetic separation. The experiments were performed to demonstrate the effectiveness of the magnetic flotation hybrid system for coal cleaning. One of the benefits of the hybrid system is that the magnetic particles regardless of their degrees of hydrophobicity even if attached to a flotation air bubble are attached to the magnetic matrix during the separation process. The recovery in flotation starts with the collision and adhesion of hydrophobic particles to the air bubbles in the lower column followed by transportation of the nonmagnetic hydrophobic particle-bubble aggregate to the upper column then from the collection zone to the froth zone. Major process parameters, including magnetic field strength, slurry circulation flow rate, air flow rate, collector dosage, and frother concentration were examined to investigate their effects on magnetic flotation hybrid separation performance. Prior to each test, the feed slurry containing 75 g of a coal sample is mixed with a diesel collector (0.5–1.5 kg/t) for 4 min and MIBC frother (15–55 ppm) for 1 min in 1.5 L conditioning cell at 1500 rpm agitation speed.

The slurry from a conditioning cell is then fed to the system through a feed/circulation inlet designed slightly above the canister using a peristaltic pump "Master flex-model 7518-00″ at different feeding/circulation flow rates (800–1500 ml/min). The slurry is circulated from the discharge/circulation outlet at the bottom of the lower column into the circulation inlet before and during the separation process. The circulating flow promotes the suspension of particles in the pulp and enhances the probability of attaching the magnetic particles to the magnetic matrix during the magnetic flotation hybrid process.

The circulation increases the probability of particle-bubble collision and attachment of the descending solid particles and rising air bubbles to enhance reporting of the hydrophobic particles to the upper column through the magnetic field, especially the nonmagnetic hydrophobic particles, while the magnetic-bubble aggregates trap in the magnetic matrix.

It should be noted that after the slurry was fed and circulated for several minutes, the magnetic field was turned on, and the air bubbles were introduced to the system. Different magnetic field strengths were used for the separation process (0.2 to 1.6 T), and different air flows were injected into the system's lowest part inlet air opening at a flow rate of 400 to 1200 ml/min. The air injection causes bubbles to form and

Table 2
Operational variables and their levels for Box-Behnken design of magnetic-flotation.

Parameters	Symbols	Unit	Coded variable levels		
			−1	0	+1
Magnetic field strength	A	T	0.2	0.9	1.6
Feed/circulation flow rate velocity	B	ml/min	800	1150	1500
Air flow rate	C	ml/min	400	800	1200
Collector dosage	D	kg/t	0.5	1	1.5
Frother concentration	E	ppm	15	35	55

encourage their contact with the solid particles, which causes hydrophobic particles to adhere to the air bubble.

As a result of the device's design, the hydrophobic particle-carrying bubbles ascend upward, forming a thick froth layer from hydrophobic-nonmagnetic particles at the top of the column, as depicted in Fig. 2. Froth washing process was used to skim the hydrophobic particle/froth from the top of the system by spraying water onto the froth. It also drained the entrained gangue, hydrophilic, particles by displacing the entrained liquid that transmits gangue particles. In contrast, the hydrophilic and nonmagnetic particles that do not adhere to the air bubbles or are attracted to the magnetic matrix flow downward and discharge from the discharge point at the bottom of the system. When the hydrophilic-nonmagnetic particles completely discharged through the system, the magnetic field was turned off, and the magnetic fraction was collected by flushing water until the effluent was clear. All products were dewatered, dried, weighed and analyzed.

2.5. Multivariable design of separation experiment

The design of experiments (DOE) and response surface methodology (RSM) utilizing Design-Expert 13 software developed by "Stat-Ease" company was conducted to investigate the influence of magnetic field strength, slurry circulation flow rate, air flow rate, collector dosage, and frother concentration and to optimize the separation process. The experiments were conducted with a five-factor and three-level Box-Behnken experimental design. Table 2 shows the five operational variables and their upper and lower levels. Table 3 shows the design of Forty-three experiments together with the observed responses.

Analysis of variance (ANOVA) was used to investigate and optimize the process parameters and assess their interactions. The validity of the optimization process has been confirmed by running two additional

Table 3
Box-Behnken experimental design and observed response.

No	Levels of parameters					Response				
						Ash (%)			Combustible recovery (%)	Separation efficiency (%)
	(A)	(B)	(C)	(D)	(E)	Mag	Non-magnetic	Float		
1	0	0	1	0	1	74.2	43.2	15.00	80.70	72.23
2	0	1	0	0	−1	70.6	48.5	10.20	85.21	76.59
3	0	0	0	−1	1	50	36.8	21.60	68.70	61.00
4	−1	0	1	0	0	78.9	42.6	13.30	77.34	69.93
5	0	1	0	0	1	74.1	50	12.30	83.99	75.77
6	−1	0	0	−1	0	74.2	43.2	13.00	78.76	70.18
7	1	−1	0	0	0	59.6	28.2	10.50	63.73	69.43
8	0	0	−1	0	−1	65	28.8	10.60	62.26	69.69
9	0	0	1	0	−1	71.7	42.5	20.40	71.72	67.22
10	0	0	0	0	0	70	39.2	11.40	74.37	73.05
11	0	−1	0	1	0	64.2	30	14.20	45.47	63.30
12	−1	0	−1	0	0	72.1	30.9	8.50	61.20	69.81
13	0	−1	0	0	−1	56	27.5	10.20	47.13	65.16
14	0	0	1	1	0	73.6	54.1	12.50	79.82	76.02
15	1	0	0	1	0	57.6	26.8	11.10	60.48	68.29
16	0	0	0	0	0	71.5	34.8	10.90	72.30	72.05
17	1	0	0	−1	0	64.2	42.4	14.40	77.85	71.84
18	−1	0	0	1	0	81.1	53	13.30	84.20	72.86
19	1	0	1	0	0	66.3	36.8	11.30	73.33	72.67
20	0	0	−1	0	1	66.8	38.6	9.80	70.93	73.14
21	0	0	1	−1	0	69.8	26.5	10.60	63.20	70.27
22	0	0	−1	−1	0	63.8	47.2	11.00	80.49	75.46
23	0	−1	−1	0	0	58.8	25.7	11.60	32.52	60.44
24	0	0	0	1	1	64.6	32.1	12.30	72.23	69.24
25	0	−1	0	0	1	65.4	25.6	12.00	47.48	64.24
26	0	1	−1	0	0	76.7	51.1	11.50	85.51	75.83
27	0	1	0	−1	0	70.6	46.8	13.40	80.78	73.42
28	0	−1	1	0	0	67	29.8	11.50	60.95	68.82
29	−1	−1	0	0	0	58.9	24.7	11.80	44.63	62.71
30	1	0	0	0	1	71.8	33	10.40	68.37	71.81
31	0	0	0	1	−1	65.3	29	10.10	58.60	68.92
32	0	1	0	1	0	77.2	48.3	14.20	87.76	71.11
33	0	0	0	−1	−1	70.1	29.7	12.10	66.68	68.60
34	0	0	0	0	0	71.2	37.7	9.30	73.51	75.98
35	1	1	0	0	0	68.8	41.7	12.10	79.30	73.86
36	1	0	−1	0	0	61.6	29.8	12.80	67.31	68.21
37	−1	0	0	0	1	86.3	42.6	23.00	70.70	62.35
38	1	0	0	0	−1	63	31.6	7.80	65.65	73.79
39	0	−1	0	−1	0	55.8	25	9.40	46.62	65.43
40	0	1	1	0	0	73.3	40.7	9.30	80.18	75.99
41	−1	1	0	0	0	79.7	54.5	16.00	85.97	70.15
42	0	0	−1	1	0	66.4	37.2	9.40	66.95	73.60
43	−1	0	0	0	−1	77.3	44.8	11.50	74.41	72.98

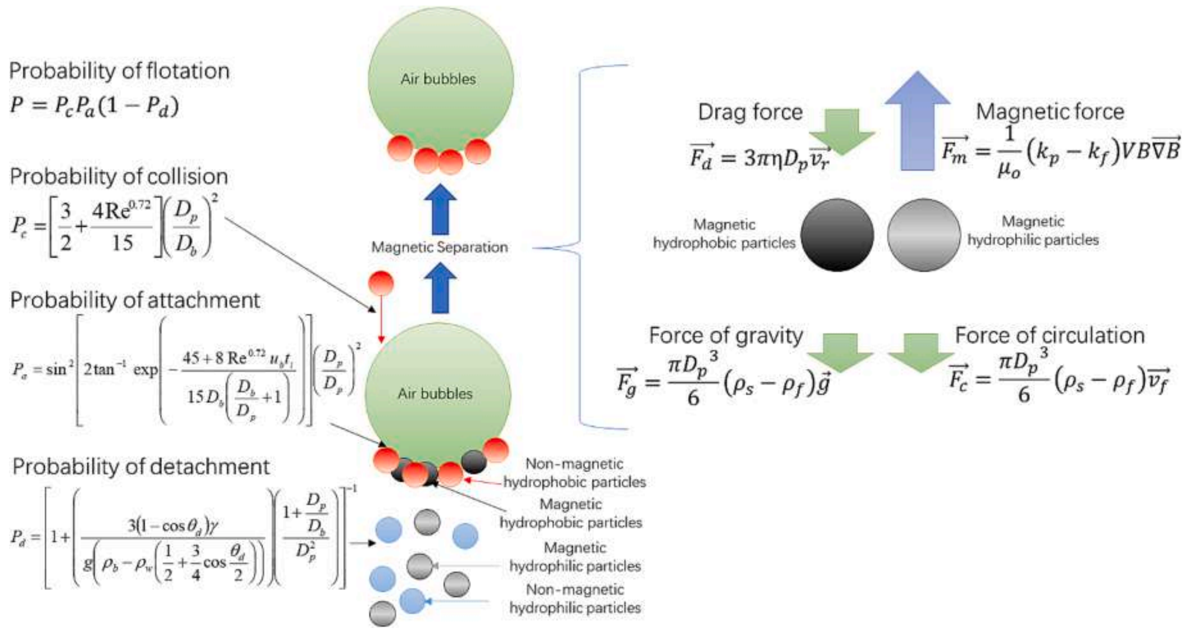

Fig. 3. Schematic diagram of the principals of magnetic flotation hybrid separation process.

experiments at the optimum conditions generated from the statistical experimental design.

The ash percentage of the magnetic flotation products was analyzed for each flotation experiment. Additionally, the ash and combustible recoveries in the floated portion were determined to assess the separation efficiency of the magnetic flotation hybrid process for coal cleaning. The combustible material recovery, ash recovery, and separation efficiency were calculated using Eq. (1), Eq. (2), and Eq. (3) respectively.

$$Combustible recovery = 100 \cdot \frac{M_c(100 - A_c)}{M_f(100 - A_f)} \quad (1)$$

$$Ash recovery = 100 \cdot \frac{M_c A_c}{M_f A_f} \quad (2)$$

$$Separation efficiency = \frac{Combustible recovery + Ash recovery}{2} \quad (3)$$

where A_c and A_f are the ash percentage in clean coal and feed respectively; M_c and M_f are the weight percentage of clean coal and feed respectively.

3. Results and discussion

3.1. Principals of magnetic flotation hybrid separation process

By conducting the developed process, it is possible to separate more than two components of the ore by exploiting the differences in both degrees of hydrophobicity and magnetic properties such as magnetic susceptibility between different minerals.

Coal is known to be weakly diamagnetic, or essentially nonmagnetic, while most of the mineral material included in coal, particularly iron-containing material such as pyrite, is paramagnetic. As a result, mineral impurities in coal require a high magnetic field intensity to attract weak paramagnetic material that can be removed from coal (Seferinoglu and Duzenli, 2022). Therefore, increasing the magnetic field strength can cause more paramagnetic minerals to accumulate on the matrix that needs to be separated from the mixture. As a result, the amount of mineral matter in the floating section is indirectly reduced by increasing the attraction of magnetic minerals to the matrix as a magnetic product. The combined effect of the forces operating on the particles, namely the magnetic force F_m, the fluid drag force F_d, the circulation force F_c, and the gravity force F_g as illustrated in Fig. 3, determines the building of particles on the matrix in the magnetic separation process (Luborsky and Drummond, 1975; Svoboda and Fujita, 2003).

Furthermore, when the magnetic force is larger than the sum of competing forces for magnetic particles and vice versa for non-magnetic particles, the separation takes place. Where μ_o is the magnetic permeability of the space, k_p and k_f are the magnetic susceptibility of the particle and the fluid respectively, $V = \pi D_p^3/6$ is the particle volume, D_p is the particle diameter, $B = \mu H$ is the magnetic flux density, $\mu = \mu_o(1 + x_v)$ is the degree of magnetization that a material obtains in response to an applied magnetic field H, $(1 + x_v)$ is the relative magnetic permeability of the material, $x_v = M/H$ is the volumetric magnetic susceptibility, M is the magnetization of the material, ∇B is the magnetic field gradient, ρ_s and ρ_f are solid (particle) and fluid densities respectively, g is acceleration due to gravity, η is dynamic viscosity of the fluid, $v_r = v_f - v_p$ is the relative particle velocity with respect to the fluid velocity. In addition, integrating the matrix in the magnetic field leads to extend of the applicability of system to materials that were previously considered too fine and too weakly magnetic due to the significant increase in the magnetic field gradient.

The magnetic separation selectivity is determined by the relative magnitudes of competing and magnetic forces acting on the particles, and these are influenced by the developed process itself and its operating settings. These relative magnitudes of the forces are also impacted by the particle diameter where F_m, F_g, F_c are directly proportional with D_p^3 while F_d is directly proportional with D_p. For example, 1 μm and 10 μm strongly and weakly magnetic particles of 1000 m^3/kg and 1 m^3/kg magnetic susceptibility respectively under the same magnetic force of 1000 N, they will appear in the same products unless the competing forces significantly influence the particles of different sizes in various ways (Svoboda and Fujita, 2003).

Because this is a combination process (magnetic and flotation), there is injected air at different flow rates producing air bubbles by using a sparger. Thus, the collision, attachment, and detachment of the particles with the air bubbles are accomplished, where only the hydrophobic particles are captured selectively by the air bubbles and only nonmagnetic hydrophobic particles will be carried from the collection zone to the froth zone and then to the product stream, while the magnetic hydrophobic/hydrophilic particles will be captured by the magnetic forces

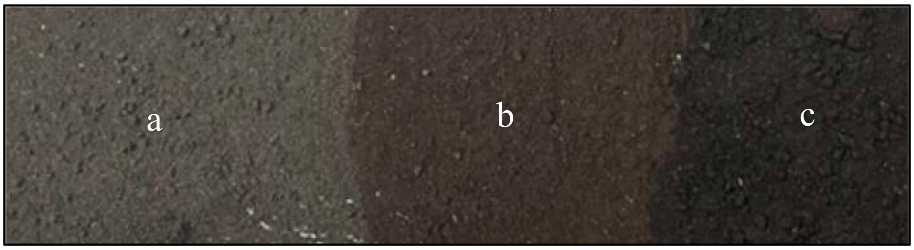

Fig. 4. Three separation products produced from the developed magnetic flotation system; (a) hydrophilic/non-magnetic product, (b) magnetic product, (c) floating product.

Table 4
Summaries of variance analysis (ANOVA).

	Ash % (Mag)	Ash % (Non-mag)	Ash % (Float)	Combustible recovery (%)	Separation efficiency (%)
p-value	<0.0001 (significant)	< 0.0001 (significant)	0.0005 (significant)	< 0.0001 (significant)	0.0021 (significant)
F-value	9.02	7.40	5.66	18.57	4.38
Lack of p-value Fit	0.0523 (Not significant)	0.1815 (Not significant)	0.3537 (Not significant)	0.0699 (Not significant)	0.4739 (Not significant)
Mean	68.49	37.51	12.27	69.29	70.31
Std. Dev.	3.24	4.82	1.57	3.54	2.43
C.V. %	4.74	12.86	12.81	5.10	3.45
R^2	0.9161	0.8199	0.9107	0.9789	0.8874
Adjusted R^2	0.8146	0.7091	0.7499	0.9262	0.68466
Adeq Precision	11.8527	11.2599	11.5724	18.3131	7.9896

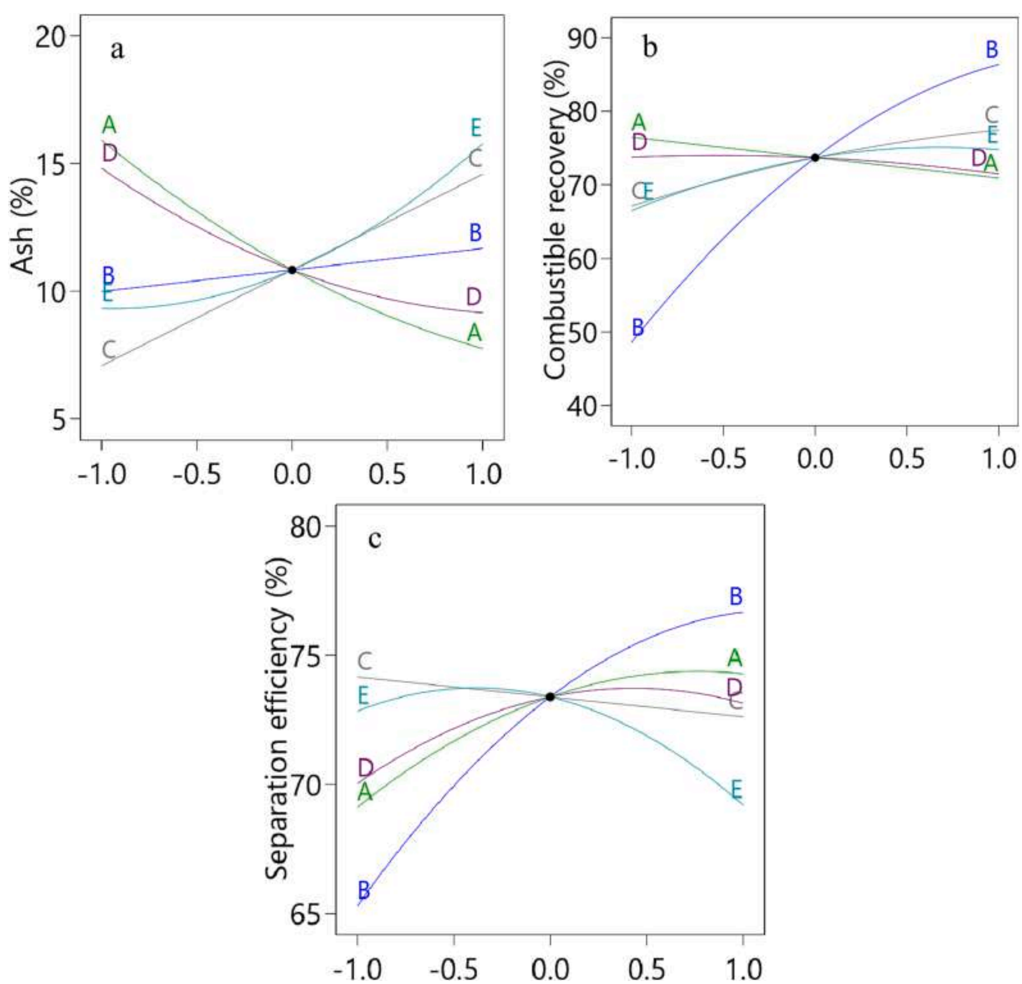

Fig. 5. Perturbation plots of (a) Product ash percentage, (b) combustible recovery, and (c) separation efficiency.

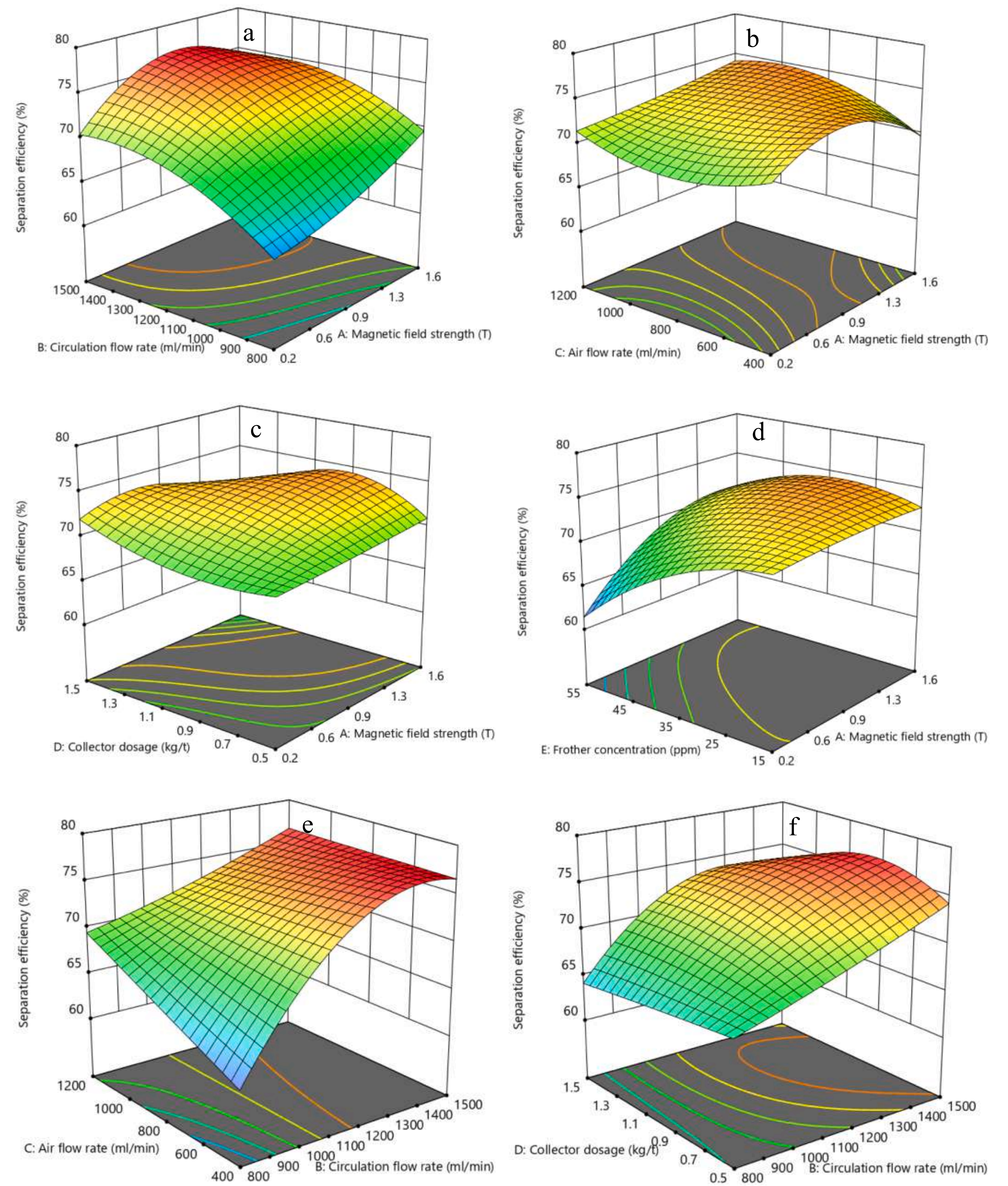

Fig. 6. Effect of each two variables at the central level of other parameters on the separation efficiency of the magnetic flotation hybrid process.

in the magnetic zone, and the nonmagnetic hydrophilic particles will settle down to the tailing stream. The flotation P, collision P_c, attachment P_a, and detachment P_d probabilities are shown in Fig. 3 where D_b is the bubble diameter, Re is the Reynolds number, U_b is the bubble rising velocity, t_i is the induction time required for the particle to get attached to the air bubble, θis the contact angle, γis the liquid surface tension, ρ_b

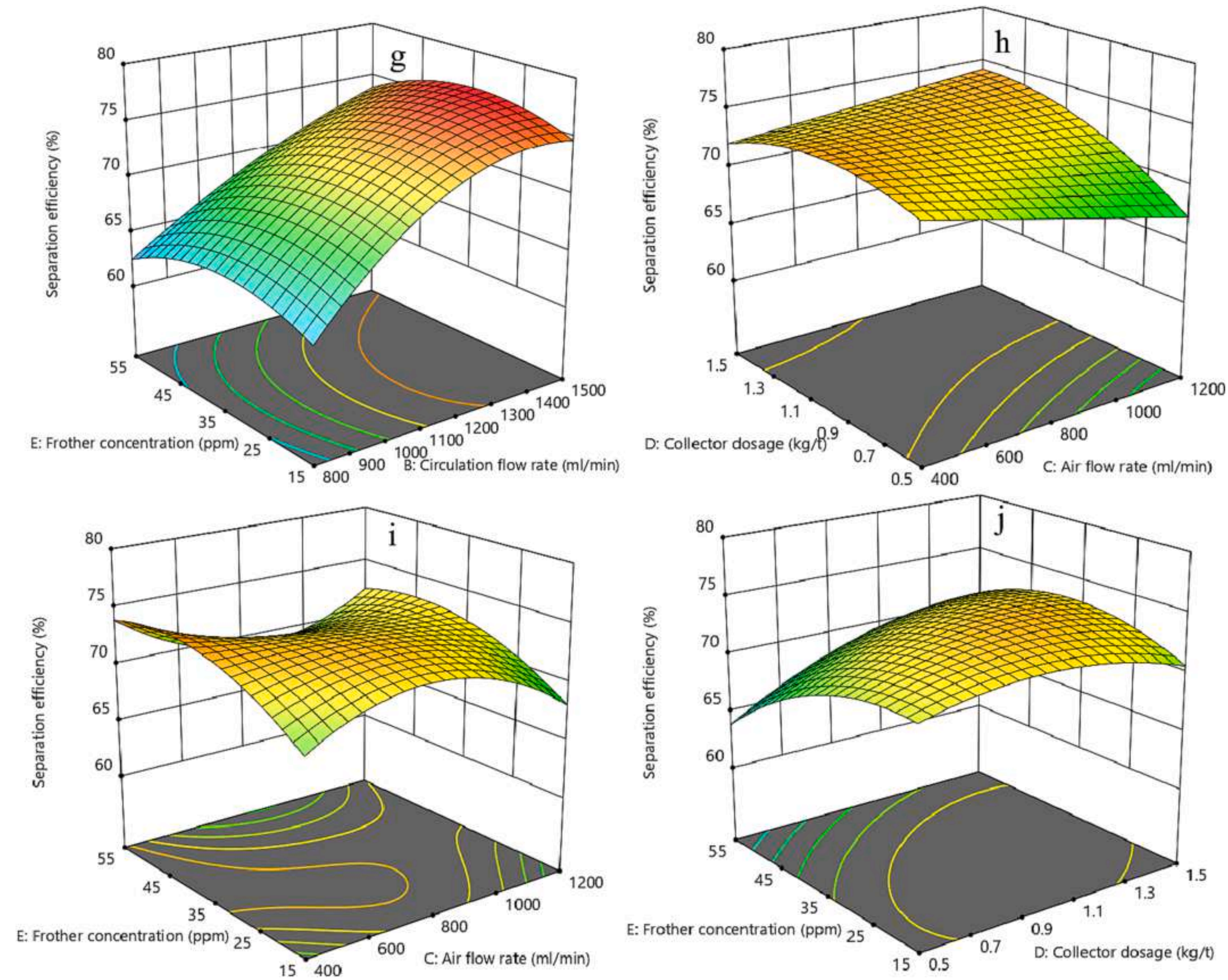

Fig. 6. (*continued*).

is the bubble density, and ρ_w is the water density (Sobhy and Tao, 2013; Tao and Sobhy, 2019). Furthermore, flotation probability is mainly affected by the particle dimeter, bubble diameter, and particle hydrophobicity.

The additional reactions caused by utilizing reagents in the conditioning cell and the slurry circulation force F_c in the lower column create a relatively complicated process. Thus, future studies on other ores will be carried out using the developed process. According to the numerous conflicting interactions that occur in the magnetic flotation hybrid system, the interactive effects were accounted for by utilizing experimental design and optimization processes.

Based on the results shown in Table 3, the experiments confirmed the superiority of the magnetic flotation hybrid techniques in achieving significant separation of the feed sample into three different products. For example, from experiment number 5, the lowest ash percentage of 7.8% was obtained from feed material of 27.21% ash achieving 83.99% combustible recovery and 75.77% separation efficiency. Thus, this developed magnetic flotation hybrid separation technique could improve the conventional flotation process for demineralizing coal and produce clean coal with reasonable separation efficiency. The advantage of the developed process is the use of the magnetic field as an alternative to depressants or inhibitors for magnetic minerals such as pyrite, in addition to the separation of particles based on the degree of hydrophobicity. As a result, this technique has the potential to separate individual minerals from complex ores, improving the conventional flotation process, and eliminating the use of chemical reagents such as depressants for ores containing magnetic minerals. Therefore, this hybrid process is recommended for future work to beneficiate complex ores such as sulfide and iron-manganese ores.

Overall, the beneficiation of the coal sample using the developed magnetic flotation hybrid system was achieved with a noticeable difference between the three products, as shown in Fig. 4. The three products are floating product (clean coal), magnetic product attributed to the occurrence of pyrite, and hydrophilic/non-magnetic product.

3.2. Statistical analysis

The summary of ANOVA is given in Table 4. The determined R^2 and adjusted R^2 close to unity indicate the predicted values are significantly correlated to the experimental data. According to other statistical parameters of all responses, p-values less than 0.05, adequacy precision greater than 4, small standard deviations, and insignificant lack of fit, the plots of experimental results and predicted responses for ash percentage, combustible recovery, and separation efficiency can be used to guide the design space.

3.3. Perturbation analysis

A perturbation analysis was performed to clarify the influences of the main independent variables. In this section, the effect of the main operating variables that influence the quality of the product from the magnetic flotation hybrid separation process was explained using the perturbation plots shown in Fig. 5. The lines in the graphs reflect the individual significant effects and sensitivity of the parameters for ash, combustible recovery, and separation efficiency at the central level of all variables. Magnetic field strength (A) and collector dosage (D) reduced significantly the product ash percentage Fig. 5*(a)*, but slightly reduced the combustible recovery Fig. 5*(b)*, and in sequence had a significant positive impact on the separation efficiency Fig. 5*(c)*. On the opposite, air flow rate (C) followed by frother concentration (E) increased both product ash percentage Fig. 5*(a)* and combustible recovery Fig. 5*(b)* but reduced the separation efficiency Fig. 5*(c)*. Whereas slurry circulation flow rate (B) slightly increased the product ash percentage Fig. 5*(a)*, but significantly improved the combustible recovery Fig. 5*(b)*, and in sequence significantly enhanced the separation efficiency Fig. 5*(c)*.

3.4. Interaction effect of variables on separation efficiency

A three-dimensional response surface was developed to investigate the interaction effects of independent variables on the separation efficiency, as shown in Fig. 6. These graphs assisted in recognizing the relationship between the dependent and independent variables.

The separation efficiency increased by increasing the magnetic field strength (A) with increasing the slurry circulation flow rate (B) at the central level of other parameters as shown in Fig. 6*(a)*. This may attribute to the enhanced selectivity by using both higher magnetic field strength and higher circulation rate. Besides, increasing the magnetic field strength may have assisted in the accumulation of paramagnetic minerals in the matrix as a magnetic product, and in sequence, the amount of mineral matter reported to the froth zone was reduced improving the separation efficiency.

At a higher level of magnetic field strength, reducing the air flow rate to a value less than 800 ml/min negatively impacted the separation efficiency as shown in Fig. 6*(b)*, while the separation efficiency slightly decreased at both higher levels of air flow rate and magnetic field strength, but at a lower level of magnetic field strength, only the middle level of air flow rate of 800 ml/min provided a lower separation efficiency. The slurry aeration rate, which substantially impacts the flotation response, depends on the amount of air introduced to the process. It also plays a vital role in the formation of froth. Thus, increased air flow rate results in shorter froth residence time and increased gangue entrainment (Tao et al., 2000) because higher gas flow rates create conditions that allow hydrophilic particles to ascend to the collection zone surface (Bedekovic, 2016). In addition, a high air flow rate increases gas hold-up and surface area flux, which assists in increasing the recovery of combustible material (Ling et al., 2018).

With increasing the magnetic field strength, the collector dosage can be reduced to maintain a high separation efficiency as revealed in Fig. 6*(c)*. It is well known that the collector improves particle floatability by increasing their hydrophobicity. The increase in the mineral matter could be due to collector overdosage, which caused the froth's physical entrapment of ash-forming minerals.

At a higher level of magnetic field strength, the frother concentration can be increased to a value of up to 45 ppm to produce a high separation efficiency as shown in Fig. 6*(d)* but reducing the frother concentration at magnetic field strength above 0.4 T is needed to obtain maximum separation efficiency. Kimpel and Hansen (Klimpel and Hansen, 1989) found that increasing the frother dosage to increase recovery generally results in less selective flotation regardless of frother type.

The slurry circulation flow rate impacts the probabilities of magnetic particle attachment to the magnetic field, hydrophobic/hydrophobic particles collision with the air bubbles, hydrophobic particles attachment to the air bubbles, and hydrophilic particles detachment from the air bubbles. Which is not similar to the feed flow rate. As is well known, the feed flow rate is related to residence time and flotation time, which impacts concentration quality. The flotation time decreases as the feed rate increases. Without enough flotation time, only the easiest to float minerals float into the concentrate, increasing selectivity while decreasing recovery (Ma et al., 2021). Thus, fewer hydrophobic particles (slow-floating fraction) require more retention time to be reported to the floating stream (Sobhy et al., 2020). In the case of coal, it is well-accepted that good separation does not necessitate long residence times (Vasumathi et al., 2016). At a lower level of circulation flow rate, the air flow rate should increase to enhance the separation efficiency, and the maximum separation efficiency was negligibly impacted by the air flow rate at a higher level of circulation flow rate as shown in Fig. 6*(e)*.

The circulation flow rate at a value less than 1200 ml/min had a significant positive influence on the separation efficiency regardless of the collector dosage value, but increasing the circulation flow rate required a collector dosage of less than 1.3 kg/t to maintain a high separation efficiency as shown in Fig. 6*(f)*. The middle level of frother concentration enhanced the separation efficiency which was again significantly enhanced by increasing the circulation flow rate as indicated in Fig. 6*(g)*.

To obtain a high separation efficiency, a higher level of air flow rate should be used at a higher level of collector and vice versa, while the maximum separation efficiency was provided at a higher level of both as given in Fig. 6*(h)*.

The interaction between frother concentration and air flow rate at the central level of other parameters was a little complicated. The separation efficiency increased at the range from middle to lower level of air flow rate and the range of a middle to a higher level of frother concentration as shown in Fig. 6 (i). The air flow rate should be enough to achieve high throughput. However, if the airflow rate is too great, the flow pattern will be disrupted, and the bubbly swarm will likely be lost causing less separation efficiency (Vasumathi et al., 2016). Moreover, it was discovered that the air flow rate had two conflicting impacts. One is that more bubbles are produced as the gas flow rate increases, increasing the flotation rate. Increasing the gas flow rate, on the other hand, results in more giant bubbles, which lowers the flotation rate, and makes the performance of the process very sensitive to different variables.

Whereas the interaction between frother concentration and collector dosage shown in Fig. 6*(j)* indicates that increasing the collector dosage with reducing the frother concentration at the central level of other parameters enhanced the separation efficiency, but at the higher level of collector dosage and lower level of frother concentration, the separation efficiency slightly decreased.

Overall, the optimization goal's chosen criteria were to produce a lower ash percentage with higher combustible recovery and separation efficiency in the final concentration (floating product). The ideal variable values based on the best combination of factor levels were produced by varying the inputs. Thus, the concentrate had a minimum ash percentage of 7.78%, a maximum combustible recovery of 75.86%, and a separation efficiency of 77.14% at the optimum conditions of 1.2 T magnetic field strength, 1500 ml/min slurry circulation flow rate, 1164 ml/min air flow rate, 0.87 kg/t collector dosage, and 42 ppm frother concentration. Whereas the ash percentages of both magnetic and non-magnetic/hydrophobic products were 73.49% and 40.93% respectively.

The performance of the developed method is more efficient than the traditional flotation method of the same type of coal. The traditional method in a previous study produced a concentrate of 9–11.5 % ash based on the operation conditions, and even with employing nano-bubbles to column flotation, only the combustible recovery was improved with slight increase in the ash percentage (Sobhy et al., 2023).

The results of the confirmation experiments at the optimum condition were similar to the predicted data obtained from the experiment design and optimization processes as indicated in Table 5.

Table 5
Results of confirmation experiments at optimum conditions.

Ash % (Mag)	Ash %(Non-mag)	Ash % (Float)	Combustible recovery (%)	Separation efficiency (%)
75.1	39.99	7.89	76.37	78.51
74.3	40.51	7.71	75.94	78.71

To sum up, the results of this work revealed that the magnetic flotation hybrid separation technique significantly improved coal separation performance. In the future, more research should be done to properly evaluate this system on a wide range of minerals to determine the benefits that can be achieved by employing this developed technology.

4. Conclusions

In this study, a magnetic flotation hybrid separator was designed and fabricated to improve the coal beneficiation process. The hybrid magnetic flotation separation process demonstrated that the mineral matter in the El-Maghara coal sample could be reduced effectively. The experiments were carried out using a response surface approach (Box-Behnken design) to evaluate the influence of operating conditions such as magnetic field strength, slurry circulation flow rate, air flow rate, and collector dosage on the separation efficiency for the coal beneficiation processes. The ANOVA confirmed that the experimental results and predicted responses for ash percentage, combustible recovery, and separation efficiency can be used to guide the design space. The individual effects and sensitivity of the parameters examined using perturbation analysis indicate that magnetic field strength, slurry circulation, and collector dosage significantly enhanced the separation efficiency, while air flow rate followed by frother concentration reduced the separation efficiency. In addition, the optimization of these interaction effects using a three-dimensional response surface of separation efficiency recognized the best conditions of 1.2 T magnetic field strength, 1500 ml/min slurry circulation flow rate, 1164 ml/min air flow rate, 0.87 kg/t collector dosage, and 42 ppm frother concentration. These optimum conditions produced a concentrate of 7.78% ash with maximum combustible recovery and separation efficiency of 75.86% and 77.14% respectively. Whereas the ash percentages of both magnetic and non-magnetic/hydrophobic products were 73.49% and 40.93% respectively. Besides, the confirmation experiments at the optimum condition were approximately similar to the predicted data.

CRediT authorship contribution statement

Ahmed Sobhy: Conceptualization, Investigation, Methodology, Supervision, Data curation, Formal analysis, Software, Validation, Writing – review & editing. **Jing Lu:** Writing – review & editing. **luzheng Chen:** Writing – review & editing. **Nourhan Ahmed:** Methodology, Software, Validation, Writing – original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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