BJET
(Bayero Journal of Engineering and Technology)
https://bjet.ng/index.php/jet

Research Article
2026, Vol. 21 (3), 80-91

RESPONSE SURFACE OPTIMISATION OF A MAGNETIC ACTIVATED CARBON FIXED-BED COLUMN FOR CHROMIUM AND LEAD REMOVAL FROM WASTEWATER

Abdulmalik Musa[1], Badruddeen Saulawa Sani[2], Umar Abubakar Alfa[3], Donatus Begianpuye Adie[4]

[1]Department of Chemical Engineering, Nasarawa State University, Keffi, PMB. 1022, Nigeria
[1,2,3,4]Department of Water Resources and Environmental Engineering, Ahmadu Bello University, Zaria, PMB. 1045, Nigeria

*Corresponding author email: musaabdulmalik@nsuk.edu.ng

Received: 13-07-2026
Revised: 17-08-2026
Accepted: 28-08-2026
Published: 21-09-2026

Abstract: *Fixed-bed systems are more suitable for practical wastewater treatment than batch processes; however, their performance strongly depends on operating conditions. This study optimised a magnetic activated carbon (MAC) fixed-bed column for the simultaneous removal of chromium (Cr) and lead (Pb) from wastewater using response surface methodology (RSM) based on central composite design (CCD). Bed height (8-18 cm) and influent metal concentration (5-30 mg/L) were investigated under two flow rate scenarios (6.30 mL/min and 10.50 mL/min), while adsorption capacity and breakthrough time served as response variables. The developed models showed good agreement with the experimental data, with coefficients of determination (R^2) ranging from 0.9445 to 0.9892. Bed height and influent concentration significantly influenced adsorption performance ($P < 0.05$). Increasing the flow rate from 6.30 to 10.50 mL/min reduced Cr adsorption capacity from 1.96 to 1.17 mg/g and Pb adsorption from 1.61 to 1.11 mg/g, with substantial reductions in breakthrough time. Numerical optimisation identified a model predicted optimum of 18 cm bed height, 11 mg/L influent concentration, and 6.30 mL/min flow rate. The findings demonstrate the potential of MAC for continuous Cr and Pb removal under the investigated operating conditions and provide predictive models and model derived operating conditions for the MAC fixed-bed adsorption system.*

Key words: Magnetic activated carbon; Fixed-bed adsorption; Central composite design; Heavy metal removal; Wastewater treatment

1 Introduction

Heavy metal contamination of aquatic environments remains a major environmental challenge due to persistence, toxicity, bioaccumulative nature, and non-biodegradability of these pollutants. Among the most hazardous contaminants, chromium (Cr) and lead (Pb) are frequently discharged into wastewater from industries such as leather tanning, mining, and metal processing (Nemeș *et al.*, 2026). Their accumulation in surface and ground water poses serious risks to human health and aquatic ecosystems, necessitating the development of efficient and sustainable treatment technologies.

Adsorption is widely regarded as one of the most effective methods for heavy metal removal because of its simplicity, high efficiency, operational flexibility, and cost effectiveness (Burakov *et al.*, 2018). Activated carbon (AC) remains the most extensively used adsorbent owing to its high surface area, well developed pore structure, and strong affinity for a broad range of contaminants (Ali and Gupta, 2006). Recent advances have led to the development of magnetic activated carbons (MACs), which can combine the adsorption properties of activated carbon with magnetic separability, thereby facilitating adsorbent recovery and regeneration (Mohan *et al.*, 2011). These characteristics make MACs attractive candidates for wastewater treatment applications

Although adsorption has been extensively investigated, most studies involving activated carbons and MACs have been conducted used batch conditions. While batch adsorptions provide valuable information on adsorption equilibrium and kinetics, they do not adequately represent the dynamic conditions encountered in practical treatment operations. Continuous fixed-bed columns are generally preferred for practical wastewater treatment because they offer higher treatment capacity, improved process control, and easier integration into existing treatment facilities (Dichiara and Weinstein, 2015). However, column performance is governed by complex interactions among hydraulic residence time, mass transfer processes, contaminant loading, and adsorbent utilisation (Patel, 2019). This makes optimisation of column operating conditions essential.

Response surface methodology (RSM) has emerged as a powerful statistical tool for modelling and optimisation of adsorption processes because it enables simultaneous evaluation of multiple factors and their interactions while minimising experimental effort (Bezerra *et al.*, 2008). Among the available experimental designs, Central Composite Design (CCD) is widely employed for developing quadratic models and identifying optimum operating conditions (Ferreira *et al.*, 2007; Szpisznik-Gulyás *et al.*, 2023). The applicability of RSM for adsorption optimisation has been demonstrated for various heavy metal removal systems (Garba *et al.*, 2016; Bahrami *et al.*, 2019; Adamu *et al.*, 2023; Mohseni *et al.*, 2024).

Despite these advances, comparatively few studies have focused on the optimisation of continuous fixed-bed adsorption systems employing MAC, particularly for the simultaneous removal of multiple heavy metals. Furthermore, the interactive effects of key operating variables such bed, height, influent concentration, and flow conditions are often inadequately quantified, limiting the translation of laboratory findings into practical treatment applications.

This study employed RSM based on CCD to optimise a MAC fixed-bed column for the simultaneous removal of Cr and Pb from wastewater. Bed height and influent concentration were investigated under two flow rate scenarios, while adsorption capacity and breakthrough time were used as performance indicators. The objectives were to develop predictive models, evaluate the individual and interactive effects of operating variables, and identify optimum conditions for Cr and Pb removal using a MAC fixed-bed adsorption system.

2 Methodology

2.1 Adsorbent

The MAC employed in this study was synthesized by chemical co-precipitation and characterised prior to use in the column experiments. The previously determined characteristics of the MAC included a Brunauer-Emmett-Teller (BET) surface area of 254.61 m^2/g, total pore volume of 0.13 cm^3/g, point of zero charge of 5.30, and bulk density of 0.601 g/cm^3. Elemental composition indicated an iron content of 85.5 at. % based on scanning electron microscopy-coupled with energy-dispersive X-ray spectroscopy (SEM-EDX). While Fourier Transform Infrared Spectroscopy (FTIR) characterisation established the presence of Fe-OH and Fe-O functionalities.

The MAC was sieved using a 1.18 mm sieve placed over a 600 µm sieve and a collection pan. After mechanical agitation, the fraction retained on the 600 µm sieve (particle size range of 0.60-1.18 mm) was collected and stored in an airtight container until use.

2.2 Synthetic Wastewater Preparation

Synthetic wastewater was prepared in accordance to OECD (2001) synthetic sewage formulation by dissolving 160 mg of peptone, 110 mg meat extract, 30 mg urea, 7 mg NaCl, 4 mg $CaCl \cdot 2H_2O$, 2 mg $MgSO_4 \cdot 7H_2O$, and 28 mg K_2HPO_4 in 1000 mL of distilled water. The solution was autoclaved at 120 °C for 15 min and stored in the dark for one week. An aliquot of the prepared stock synthetic wastewater was analysed for Biochemical oxygen demand (BOD_5). The stock solution was subsequently diluted with tap water to obtain a target BOD_5 (25 mg/L) and the dilution factor was determined. Thus, the volumes of stock synthetic wastewater (V_{SWW}) and tap water were calculated using Eq. 1 and 2, respectively.

$$V_{SWW} = 0.1282V_t \quad (1)$$

$$V_{tap\ water} = 0.8718V_t \quad (2)$$

Where V_t is the total volume required for each experiment.

The tap water supplied by the Ahmadu Bello University water treatment plant, was used directly without further characterisation. Cr and Pb stock solutions were prepared using K_2CrO_7 and $Pb(NO_3)_2$, respectively, and added to the synthetic wastewater by dilution to obtain the required influent metal concentrations. The pH was adjusted to approximately 7.0 using 0.5 M HCI or 0.5 M NaOH and measured using a pH meter (PH-2601, Sinotester, China).

2.3 Fixed-Bed Column Experimental Setup

Continuous flow adsorption experiments were conducted using a fixed-bed column made from plastic tubing (height: 22.5 cm, internal diameter: 1.0 cm) as shown in Plate 1.

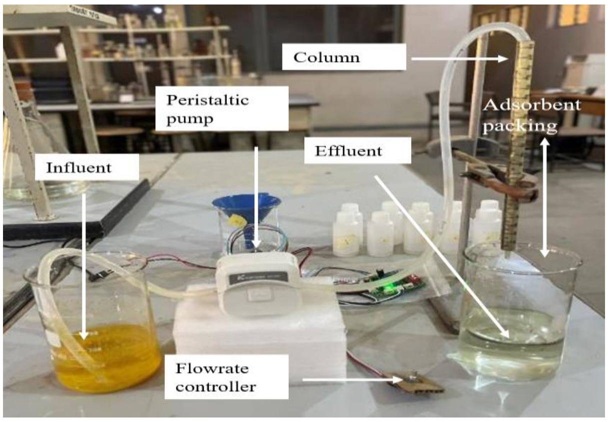

Plate 1: *Fixed-bed column adsorption experimental setup*

The column was packed with MAC to the specific bed height, and the actual mass of adsorbent packed at each bed height was recorded and used in the corresponding adsorption capacity calculations. The packed column was rinsed with distilled water to remove entrapped air and ensure uniform packing. The wastewater containing known influent concentrations (C_0) of Cr and Pb was introduced in a downward flow mode using a peristaltic pump (KK300-S25, Kamoer, China) at the desired investigated flow rate. Effluent samples were collected at specific time intervals (t) and analysed for pH and metal concentrations (C_t) during column operation. Column performance was evaluated through breakthrough curve analysis, obtained by plotting C_t/C_0 against time.

2.4 Heavy Metal Analysis

Samples were digested following the method of Ibrahim *et al.* (2024) using mixed acid solutions (HNO_3, H_2SO_4, and $HClO_4$). The Cr and Pb concentrations were determined using an atomic absorption spectrophotometer (280FS AA, Agilent Technologies, USA). Prior to analyses, calibration curves with high linearity were prepared from each metal standards. The samples absorbances were quantified and concentrations were determined based on the calibration curves.

2.5 Experimental Design and Optimisation

RSM based on CCD was employed to investigate and optimize the effects of operating variables on fixed-bed adsorption performance. Experimental design and statistical analyses were performed using Design-Expert software (Version 13.0.1). Bed height (A) and influent metal concentration (B) were selected as the optimisation factors while adsorption capacity (q_e) and breakthrough time (t_b) were considered as response variables. Flow rate was treated as a scenario-based parameter rather than a continuous optimisation variable because the peristaltic pump could be operated reliably only at two discrete flow rates (scenario 1: 6.30 mL/min, Scenario 2: 10.50 mL/min). Consequently, separate CCD optimisation was conducted for each flow rate scenario using the factor levels presented in Table 1.

Table 1: *Coded and actual levels of the factors used in the central composite design*

Factors	-α	-1	0	+1	+α
A	5.93	8.00	13.00	18.00	20.10
B	-0.18	5.00	17.50	30.00	35.18

2.6 Evaluation of Breakthrough Time and Adsorption Capacity

Column performance was evaluated using breakthrough time (t_b) and adsorption capacity (q_e). Breakthrough time was defined as the time corresponding to $C_t/C_0 \leq 0.10$ while exhaustion time (t_e) corresponded to $C_t/C_0 \leq 0.90$. The mass of metal adsorbed at exhaustion was determined by integrating the area above the breakthrough curve in accordance to Eq. 1 (Mani and Bhandari, 2022) and adsorption capacity was subsequently calculated as mass of metal adsorbed normalised with the adsorbent mass according to Eq. (2) Juela *et al.*, 2021).

$$m_{ads,t_e} = QC_0 \sum_{i=1}^{n} \int_0^{t_e} \left(1 - \frac{C_t}{C_o}\right) dt \qquad (3)$$

$$q_e = \frac{m_{ads,t_e}}{m} \qquad (4)$$

Where m_{ads,t_e} represents the mass of metal adsorbed (mg), Q is the volumetric flow rate (L/min), q_e is the adsorption capacity (mg/g), and m is the mass of adsorbent packed in the column (g).

2.7 Statistical Analysis

Model adequacy was evaluated using analysis of variance (ANOVA), coefficient of determination (R^2), adjusted coefficient of determination (Adj. R^2), predicted coefficient of determination (Pred. R^2), and prediction error sum of squares (PRESS). Statistical significance was evaluated at a 95% confidence level ($P < 0.05$). Numerical optimisation based on the desirability function in the Design-Expert software was employed to identify operating conditions that simultaneously maximised q_e and t_b.

3 Results and Discussion

3.1 Experimental Responses

The CCD experimental matrix and corresponding responses for Cr and Pb fixed-bed column adsorption at flow rates of 6.30 and 10.50 mL/min are presented in Table 2. Across both scenarios during the column operation, the pH of the collected effluent samples remained within 6.0-7.5, with no substantial variation. Also, Cr adsorption capacity varied from 0.18 to 2.46 mg/g and Pb adsorption capacity from 0.31 to 2.66 mg/g, while breakthrough times ranged from 0.12 to 33.00 min for Cr and 0.31 to 114.94 min for Pb. The observed variation in response values across the experimental domain indicates the influence of operating conditions on fixed-bed performance and provided the basis of subsequent response surface modelling and optimisation.

Table 2: *CCD matrix, packed mass, and experimental responses for Scenarios 1 and 2*

Run	m (g)	A (cm)	B (mg/L)	Cr			Pb
				q_e (mg/g)	t_b (min)	q_e (mg/g)	t_b (min)
	Scenario 1 (6.30 mL/min)						
1*	–	13.00	-0.18	–	–	–	–
2	6.14	13.00	17.50	2.38	6.72	1.94	0.59
3	3.78	8.00	30.00	0.76	0.34	1.51	0.49
4	6.14	13.00	17.50	2.38	6.72	1.91	0.72
5	6.14	13.00	17.50	2.39	6.00	1.91	0.69
6	6.14	13.00	35.18	2.06	1.61	2.33	0.82
7	9.50	20.10	17.50	2.16	15.85	2.23	81.42
8	6.14	13.00	17.50	2.30	6.98	1.84	0.74
9	8.50	18.00	30.00	2.33	3.40	2.66	4.78
10	3.78	8.00	5.00	0.18	0.61	0.36	31.56
11	2.80	5.93	17.50	0.43	0.36	0.60	0.39
12	6.14	13.00	17.50	2.46	6. 76	1.73	0.87
13	8.50	18.00	5.00	0.71	33.00	0.88	114.94
	Scenario 2 (10.50 mL/min)						
1*	–	13.00	-0.18	–	–	–	–
2	6.14	13.00	17.50	1.98	0.91	1.19	0.30
3	3.78	8.00	30.00	0.73	1.18	1.00	0.21
4	6.14	13.00	17.50	1.94	0.94	1.13	0.33
5	6.14	13.00	17.50	1.92	0.99	1.0	0.28
6	6.14	13.00	35.18	2.12	0.90	1.62	0.50
7	9.50	20.10	17.50	1.64	1.86	1.49	2.52
8	6.14	13.00	17.50	1.98	0.94	1.09	0.31
9	8.50	18.00	30.00	2.24	0.90	2.07	0.57
10	3.78	8.00	5.00	0.32	10.83	0.28	0.34
11	2.80	5.93	17.50	0.37	0.12	0.43	0.08
12	6.14	13.00	17.50	1.86	0.90	1.12	0.29
	8.50						

Note: Run 1* (axial point at coded level -1.414) was excluded from the design matrix and experimental evaluation because the calculated actual concentration was negative (-0.18 mg/L), which is physically impossible to prepare.

3.2 Model Adequacy and Statistical Evaluation

For both Cr (Table 3) and Pb (Table 4), the developed models showed good agreement with the experimental data, with R^2 values ranging from 0.9445 to 0.9892, the lower value corresponded to the three-term model for Cr breakthrough time under scenario 1. These values indicate that over 94% of the variation in the adsorption performance was explained by the fitted equations. The close agreement between Adj. R^2 and Pred. R^2 further confirms the predicted reliability of the models within the investigated design space.

Table 3: *Model fitting summary statistics for Cr adsorption under scenario 1*

Model Summary: Adsorption Capacity						
Source	Std. Dev.	R^2	Adj. R^2	Pred. R^2	PRESS	Status
Linear	0.7480	0.4318	0.3056	-0.0828	9.60	Inadequate
2FI	0.7717	0.4623	0.2607	-1.0816	18.45	Inadequate
Quadratic	0.1543	0.9839	0.9705	0.8472	1.35	Suggested
Cubic	0.0567	0.9960	0.9960	-	*	Aliased
Model Summary: Breakthrough Time						
Linear	5.44	0.7121	0.6481	0.2197	722.02	Inadequate
2FI	2.53	0.9445	0.9237	0.7471	233.98	Suggested
Quadratic	2.01	0.9737	0.9518	0.4788	482.27	Acceptable
Cubic	0.3716	0.9994	0.9984	-	*	Aliased

Table 4: *Model fitting summary statistics for Pb adsorption under scenario 1*

Model Summary: Adsorption Capacity						
Source	Std. Dev.	R^2	Adj. R^2	Pred. R^2	PRESS	Status
Linear	0.3168	0.8346	0.7978	0.6702	1.80	Inadequate
2FI	0.3170	0.8527	0.7975	0.4655	2.92	Inadequate
Quadratic	0.1147	0.9855	0.9735	0.8009	1.09	Suggested
Cubic	0.0844	0.9948	0.9856		*	Aliased
Model Summary: Breakthrough Time						
Linear	26.21	0.6170	0.5318	0.2260	12494.25	Inadequate
2FI	24.03	0.7138	0.6065	0.0744	14942.07	Inadequate
Quadratic	5.38	0.9892	0.9803	0.8382	2611.07	Suggested
Cubic	0.1008	1.0000	1.0000		*	Aliased

ANOVA results indicated that all quadratic models were statistically significant ($P < 0.05$), confirming the strong influence of the investigated factors on adsorption capacity and breakthrough time. For Cr under Scenario 2 (Table 5), bed height, influent concentration, their interaction, and quadratic effects significantly affected adsorption capacity, while breakthrough time was primarily influenced by bed height, concentration, and their interaction. Similar trends were observed for Pb (Table 6), indicating that column performance was governed by balance between adsorbent bed capacity and contaminant loading.

Table 5: *ANOVA results for Cr adsorption capacity and breakthrough time (Scenario 1)*

ANOVA Analysis: Adsorption Capacity						
Source	Sum of Squares	df	Mean Square	F-value	p-value	Significance
Model	8.72	5	1.74	73.26	< 0.0001	Significant
A-Bed height	2.58	1	2.58	108.55	< 0.0001	
B-Initial concentration	1.84	1	1.84	77.31	0.0001	
AB	0.2704	1	0.2704	11.36	0.0150	
A^2	2.28	1	2.28	95.64	< 0.0001	
B^2	2.04	1	2.04	85.86	< 0.0001	
Residual	0.1428	6	0.0238			
Cor Total	8.86	11				
ANOVA Analysis: Breakthrough Time						
Model	873.89	3	291.30	45.37	< 0.0001	Significant
A-Bed height	411.22	1	411.22	64.05	< 0.0001	
B-Initial concentration	247.61	1	247.61	38.57	0.0003	
AB	215.06	1	215.06	33.50	0.0004	
Residual	50.81	8	6.42			
Cor Total	925.25	11				

Table 6: *ANOVA results for Pb adsorption capacity and breakthrough time (Scenario 1)*

ANOVA Analysis: Adsorption Capacity						
Source	Sum of Squares	df	Mean Square	F-value	p-value	Significance
Model	5.38	5	1.08	81.77	< 0.0001	Significant
A-Bed height	1.98	1	1.98	150.10	< 0.0001	
B-Initial concentration	2.58	1	2.58	195.97	< 0.0001	
AB	0.0992	1	0.0992	7.54	0.0335	
A^2	0.3308	1	0.3308	25.14	0.0024	
B^2	0.3468	1	0.3468	26.35	0.0021	
Residual	0.0790	6	0.0132	-	-	
Cor Total	5.46	11	-	-	-	
ANOVA Analysis: Breakthrough Time						
Model	15968.71	5	3193.74	110.40	< 0.0001	Significant
A-Bed height	5113.83	1	5113.83	176.77	< 0.0001	
B-Initial concentration	5370.32	1	5370.32	185.64	< 0.0001	
AB	1563.81	1	1563.81	54.06	0.0003	
A^2	2287.01	1	2287.01	79.06	0.0001	
B^2	1869.61	1	1869.61	64.63	0.0002	
Residual	173.53	6	28.93	-	-	
Cor Total	16142.28	11	-	-	-	

Under scenario 2, the adsorption capacity models for Cr and Pb remained highly significant (Tables 7 and 8), with bed height, influent concentration, and their interaction contributing significantly to response variation. For breakthrough time, the significance of the concentration quadratic term (B^2) indicates stronger concentration depended on effects of higher hydraulic loading, suggesting that reduced time increased the sensitivity of column performance to influent contaminant concentration.

The significance of the quadratic terms across most models confirms the presence of curvature in the response surfaces and demonstrates that adsorption performance was governed by nonlinear factor interactions. Although cubic models occasionally produced higher R^2 values, they were aliased and statistically unreliable due to insufficient degrees of freedom for independent coefficient estimation. Consequently, quadratic models were selected for subsequent analysis and optimisation as they provided the best balance between statistical significance, predictive capability, and model robustness.

Table 7: *Model fitting summary statistics for Cr adsorption under Scenario 2*

Model Summary: Adsorption Capacity						
Source	Std. Dev.	R^2	Adj. R^2	Pred. R^2	PRESS	Status
Linear	0.5795	0.5095	0.4005	0.0508	5.85	Inadequate
2FI	0.5679	0.5813	0.4243	-0.4454	8.90	Inadequate
Quadratic	0.1190	0.9862	0.9747	0.8864	0.7001	Suggested
Cubic	0.0498	0.9984	0.9956		*	Aliased
Model Summary: Breakthrough Time						
Linear	4.48	0.4994	0.3881	-0.1819	426.95	Inadequate
2FI	4.51	0.5495	0.3805	-1.3153	836.34	Inadequate
Quadratic	1.28	0.9728	0.9501	0.6352	131.79	Suggested
Cubic	0.0351	1.0000	1.0000		*	Aliased

Table 8. *Model fitting summary statistics for Pb adsorption under Scenario 2*

Model Summary: Adsorption Capacity						
Source	Std. Dev.	R^2	Adj. R^2	Pred. R^2	PRESS	Status
Linear	0.1907	0.8882	0.8634	0.7285	0.7947	Inadequate
2FI	0.1248	0.9574	0.9414	0.8586	0.4139	Inadequate
Quadratic	0.0763	0.9881	0.9781	0.9085	0.2680	Suggested
Cubic	0.0695	0.9934	0.9819		*	Aliased
Model Summary: Breakthrough Time						
Linear	0.9325	0.6006	0.5118	0.0714	18.19	Inadequate

2FI	0.7143	0.7917	0.7136	0.2329	15.03	Acceptable
Quadratic	0.1920	0.9887	0.9793	0.8026	3.87	Suggested
Cubic	0.0192	0.9999	0.9998		*	Aliased

For Cr, ANOVA (Table 9) confirmed a highly significant quadratic model (P < 0.0001) for adsorption capacity, indicating a curved response surface with optimum region. Conversely, the quadratic bed height term (A^2) for breakthrough time was non-significant (P = 0.4757), demonstrating that response variation was driven primarily by concentration effects rather than nonlinear bed height effects.

Table 9: *ANOVA results for Cr adsorption capacity and breakthrough time (Scenario 2)*

Source	Sum of Squares	df	Mean Square	F-value	p-value	Significance
ANOVA Analysis: Adsorption Capacity						
Model	6.08	5	1.22	85.86	< 0.0001	Significant
A-Bed height	1.52	1	1.52	107.34	< 0.0001	
B-Initial concentration	1.69	1	1.69	119.21	< 0.0001	
AB	0.4422	1	0.4422	31.25	0.0014	
A^2	1.65	1	1.65	116.72	< 0.0001	
B^2	0.6925	1	0.6925	48.93	0.0004	
Residual	0.0849	6	0.0142			
Cor Total	6.16	11				
ANOVA Analysis: Breakthrough Time						
Model	351.40	5	70.28	42.90	0.0001	Significant
A-Bed height	13.55	1	13.55	8.27	0.0282	
B-Initial concentration	266.76	1	266.76	162.85	< 0.0001	
AB	18.11	1	18.11	11.05	0.0159	
A^2	0.9476	1	0.9476	0.5785	0.4757	
B^2	149.76	1	149.76	91.42	< 0.0001	
Residual	9.83	6	1.64			
Cor Total	361.23	11				

For Pb (Table 10), bed height, influent concentration, and their interaction significantly influenced adsorption capacity (p < 0.0001). The significance of B^2 (p = 0.0181) and non-significance of A^2 (p = 0.0891) indicate that response curvature was driven primarily by influent concentration. Additionally, the highly significant breakthrough time model (p < 0.0001) confirmed nonlinear systems behaviour at the higher flow rate.

Table 10: *ANOVA results for Pb adsorption capacity and breakthrough time (Scenario 2)*

Source	Sum of Squares	df	Mean Square	F-value	p-value	Significance
ANOVA Analysis: Adsorption Capacity						
Model	2.89	5	0.5785	99.34	< 0.0001	Significant
A-Bed height	0.9378	1	0.9378	161.03	< 0.0001	
B-Initial concentration	1.55	1	1.55	265.57	< 0.0001	
AB	0.2025	1	0.2025	34.77	0.0011	
A^2	0.0239	1	0.0239	4.11	0.0891	
B^2	0.0605	1	0.0605	10.38	0.0181	
Residual	0.0349	6	0.0058			
Cor Total	2.93	11				
ANOVA Analysis: Breakthrough Time						
Model	19.37	5	3.87	105.09	< 0.0001	Significant
A-Bed height	8.08	1	8.08	219.22	< 0.0001	
B-Initial concentration	4.64	1	4.64	125.96	< 0.0001	
AB	3.74	1	3.74	101.56	< 0.0001	
A^2	1.40	1	1.40	37.95	0.0008	
B^2	2.21	1	2.21	60.08	0.0002	
Residual	0.2212	6	0.0369			
Cor Total	19.59	11				

3.3 Response Surface Models

The quadratic regression models developed for adsorption capacity and breakthrough time are presented in Table 11. These models quantitively describe the combined linear and nonlinear interactive influence of hydrodynamic and concentration driven effects on column performance at low and elevated flow conditions.

Table 11: *Developed quadratic regression models for adsorption capacity and breakthrough time*

Response	Scenario 1 (6.30 ml min^{-1})	Scenario 2 (10.50 mL min^{-1})
Cr q_e	$q_{(Cr)} = 2.38 + 0.5683A + 0.6192B + 0.2600AB - 0.5924A^2 - 0.6967B^2$	$q_{(Cr)} = 1.94 + 0.4358A + 0.5929B + 0.3325AB - 0.5047A^2 - 0.4055B^2$
Cr t_b	$t_{b(Cr)} = 8.13 + 7.17A - 6.52B - 7.33AB$	$t_{b(Cr)} = 0.9360 + 1.30A - 7.45B - 2.13AB + 0.3822A^2 + 5.96B^2$
Pb q_e	$q_{(Pb)} = 1.87 + 0.4969A + 0.7330B + 0.1575AB - 0.2258A^2 - 0.2870B^2$	$q_{(Pb)} = 1.11 + 0.3424A + 0.5676B + 0.2250AB - 0.0607A^2 - 0.1198B^2$
Pb t_b	$t_{b(Pb)} = 0.7220 + 25.28A - 33.45B - 19.77AB + 18.78A^2 + 21.07B^2$	$t_{b(Pb)} = 0.3020 + 1.01A - 0.9836B - 0.9675AB + 0.4644A^2 + 0.7253B^2$

3.4 Effects of Process Variables on Adsorption Performance

3.4.1 Performance of Chromium

The response surface plots (Fig. 1 and 2) show that Cr adsorption in the MAC fixed-bed column was significantly influenced by bed height and influent concentration. Adsorption capacity increased with bed height and influent concentration up to an intermediate region, beyond which further increases did not improve the response. Whereas breakthrough time increased with bed height but decreased with influent concentration.

Under Scenario 1, Cr adsorption capacity increased from 0.18 mg/g to a maximum of 2.46 mg/g (Fig. 1a). Greater bed depths improved performance by increasing adsorbent bed capacity and mass transfer zone (MTZ) development, while higher influent concentrations enhanced Cr instantaneous uptake due to stronger mass transfer driving force but service time is reduced due to repaid adsorption site occupation (Maiti *et al.*, 2020). The significant interaction between the two factors indicates that deeper beds accommodated higher loadings more effectively than shallow beds.

Breakthrough time exhibited an opposite trend (Fig. 1b), reaching a maximum of 33 min at 18 cm bed height and 5 mg/L influent concentration. Increasing bed height delayed breakthrough by extending the MTZ and available adsorption sites, whereas increasing influent concentration accelerated bed saturation (Kalavathy *et al.*, 2009; Garba *et al.*, 2016).

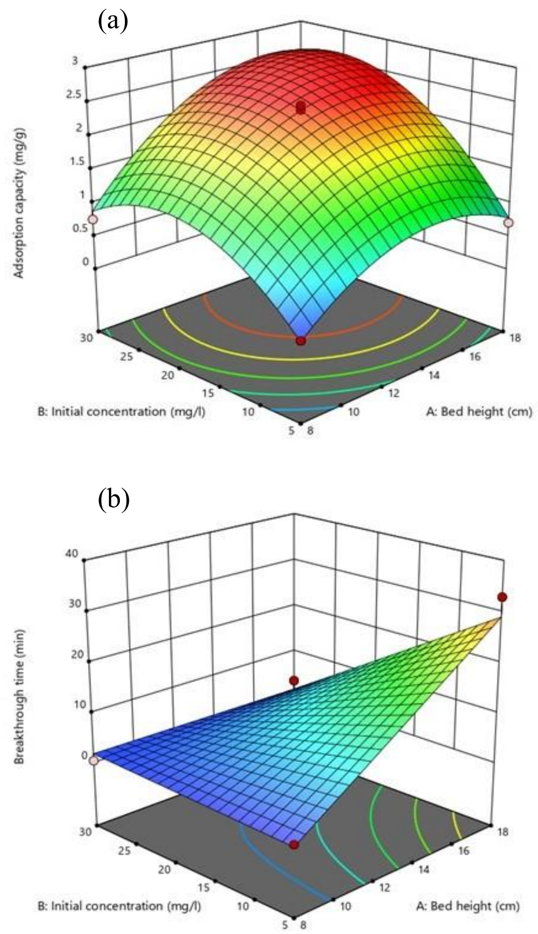

Figure 1: *Response surface plots showing the effect of bed height and initial concentration on (a) adsorption capacity and (b) breakthrough time for Cr under scenario 1*

A similar response pattern was observed under Scenario 2 (Fig. 2), although both adsorption capacity and breakthrough time were lower. The maximum

BJET, 2026, Vol. 21(3), 80-91

adsorption capacity decreased to 2.24 mg/g, while breakthrough occurred considerably earlier than under Scenario 1. This reduction is attributed to the shorter hydraulic residence time at the higher flow rate, which limited mass transfer and bed utilisation, leading to more rapid saturation of the adsorption bed.

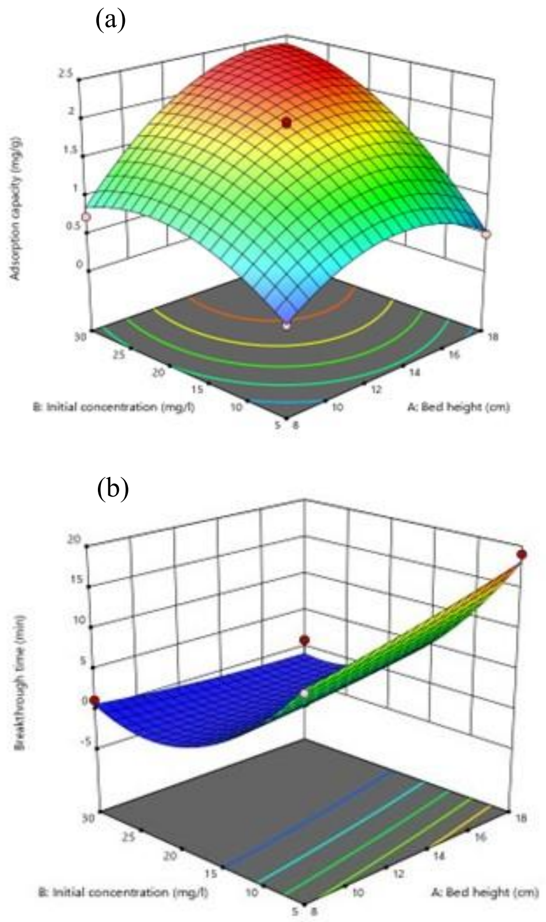

Figure 2: *Response surface plots showing the effect of bed height and initial concentration on (a) adsorption capacity and (b) breakthrough time for Cr under scenario 2*

3.4.2 Performance of Lead

The response surface plots (Fig. 3 and 4) show that Pb adsorption was strongly influenced by bed height and influent concentration under both flow rate scenarios. For Pb, adsorption capacity increased with bed height and influent concentration, while breakthrough time generally increased with bed height but decreased with influent concentration.

Under scenario 1, adsorption capacity increased from 0.36 mg/g at 8 cm bed height and 5 mg/L influent concentration to a maximum of 2.66 mg/g at 18 cm bed height and 30 mg/L (Fig. 3a). the significant interaction between the two factors indicates that deeper bed accommodated higher contaminant leadings more effectively. Breakthrough time exhibited the opposite trend with influent concentration (Fig. 3 b), reaching a maximum of approximately 115 min at 18 cm bed height at 5 mg/L.

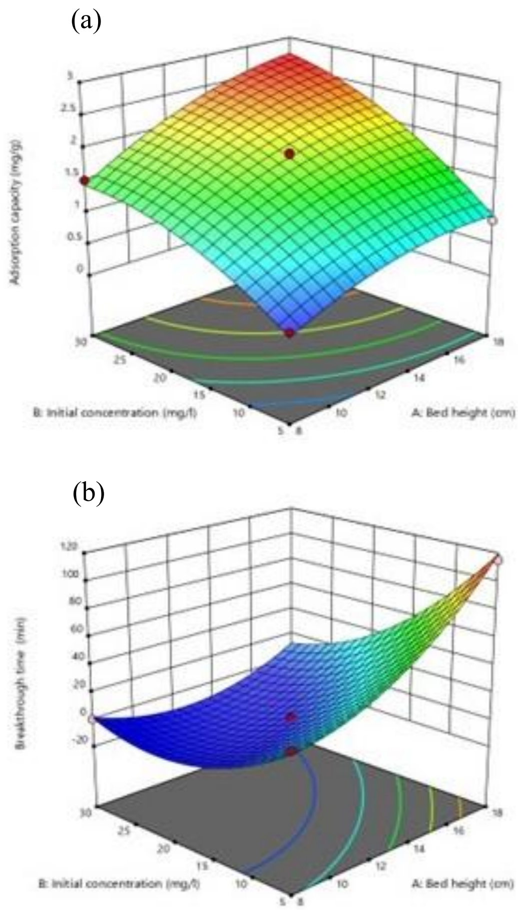

Figure 3: *Response surface plots showing the effect of bed height and initial concentration on (a) adsorption capacity and (b) breakthrough time for Pb under scenario 1*

A notable observation was the superior adsorption performance of Pb relative to Cr. Under comparable operating conditions, Pb exhibited a breakthrough time of approximately 115 min compared with 33 min for Cr, while its maximum adsorption capacity (2.66 mg/g) exceeded that of Cr (2.46 mg/g). The longer breakthrough times observed for Pb under several operating conditions indicate greater retention within the MAC bed compared to Cr.

Under Scenario 2, similar response trends were observed (Fig. 4), although adsorption capacities and breakthrough times were lower because of the shorter hydraulic residence time. Nevertheless, Pb maintained higher adsorption capacities and longer breakthrough times than Cr across the experimental domain. This shows its preferential adsorption on the MAC fixed-bed column.

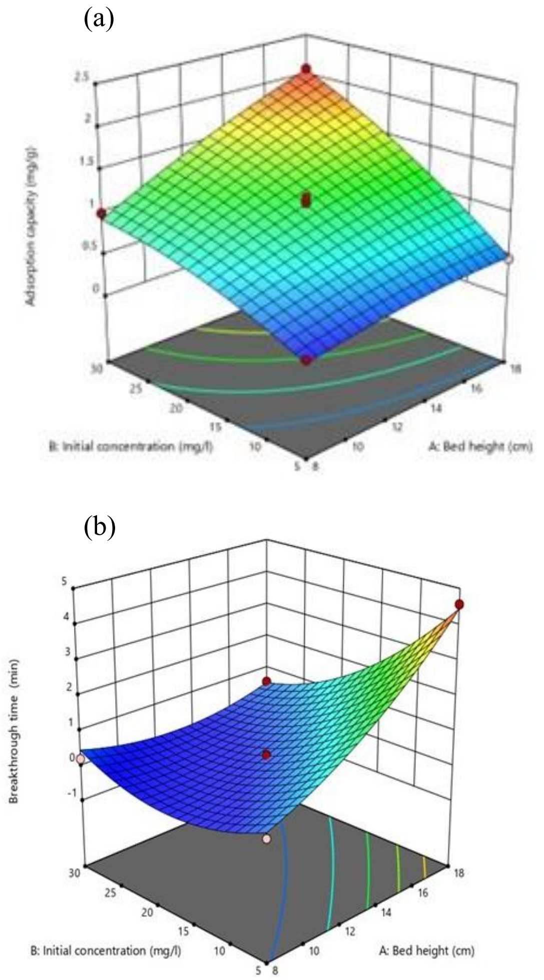

Figure 4: *Response surface plots showing the effect of bed height and initial concentration on (a) adsorption capacity and (b) breakthrough time for Pb under scenario 2*

3.5 Influence of Flow regime on Adsorption Performance

Flow rate exerted a significant influence on the performance of the MAC fixed-bed column. Comparison of the optimised responses under the two flow regimes (Table 12) showed that increasing the flow rate from 6.30 to 10.50 mL/min reduced both adsorption capacity and breakthrough time for Cr and Pb. Representative breakthrough curves for both Cr and Pb are shown in Fig. 5 and 6, respectively.

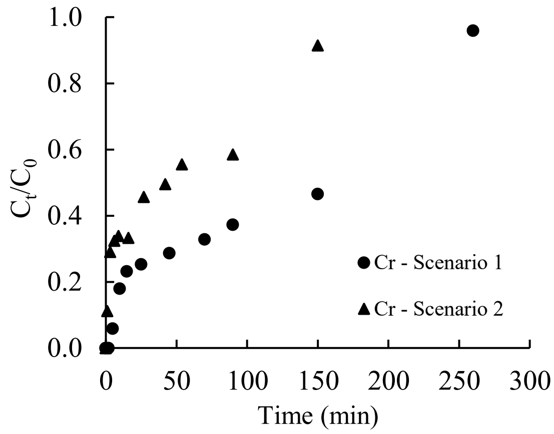

Figure 5: *Representative breakthrough curves for Cr at 13 cm bed height and 17.5 mg/L influent concentration under scenarios 1 and 2*

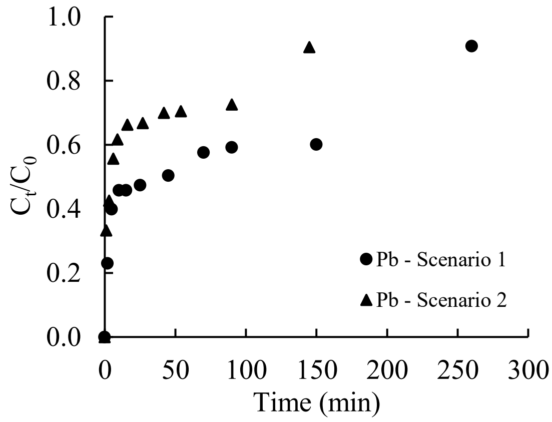

Figure 6: *Representative breakthrough curves for Pb at 13 cm bed height and 17.5 mg/L influent concentration under scenarios 1 and 2*

Under the optimised RSM solutions, Cr adsorption capacity decreased from 1.96 to 1.17 mg/g (40.3%), while Pb adsorption capacity decreased from 1.61 to 1.11 mg/g (31.1%). The effect was more pronounced for breakthrough time, which decreased from 20.0 min to 6.54 min for Cr and 76.0 min to 1.88 min for Pb, corresponding to reductions of 67.3% and 97.5%, respectively. These results indicate accelerated bed saturation and substantial decline in effective column service life at higher hydraulic loading.

The reduced performance at higher flow rate is attributed to shorter hydraulic residence time, limited mass transfer due to increased axial dispersion resulting to broader MTZ. Liu *et al*. (2016) and Bahrami *et al*. (2019) reported that such broader MTZ reduces bed utilisation and column performance. The results demonstrate that lower flow rates provide more favourable conditions for simultaneous Cr and Pb removal in the MAC fixed-bed column.

3.6 Optimisation and Selection of Operating Conditions

The optimisation results (Table 12) identified scenario 1 as the most favourable operating regime, producing higher adsorption capacities, longer breakthrough times, and greater desirability values than scenario 2 for both Cr and Pb.

Table 12: *Comparison of optimised RSM solutions for scenarios 1 and 2*

Factor/ Response	Scenario 1 (Cr, Pb)		Scenario 2 (Cr, Pb)	
A (cm)	18.00	18.00	14.40	18.00
B (mg/L)	13.20	11.00	6.80	12.97
q_b (mg/g)	1.96	1.61	1.17	1.11
t_b (min)	20.00	76.00	6.54	1.88
Desirability	0.70	0.62	0.59	0.51

Numerical optimisation identified a bed height of 18 cm, influent concentration of 11 mg/L, and flow rate of 6.30 mL/min as the model-predicted optimum within the investigated design space. The choice of these conditions was guided by Pb removal, which demonstrated comparatively lower adsorption capacity and desirability relative to Cr. In multi-component systems, optimisation based on the least effectively removed species provide a conservative basis for ensuring satisfactory simultaneous removal of both species (Es-Said *et al*., 2023). The selected conditions represent the optimum balance between adsorption capacity and breakthrough time within the investigated design space.

4 Conclusions

The developed RSM models adequately described the MAC fixed-bed column responses for the simultaneous removal of Cr and Pb from wastewater using RSM based on CCD. Bed height and influent concentration significantly influenced adsorption capacity and breakthrough time, while significant quadratic terms indicated the nonlinear responses within the experimental domain. Numerical optimisation identified bed height (18 cm), influent concentration (11 mg/L), and flow rate (6.30 mL/min) as the model predicted optimum. Flow rate substantially influenced column performance, increasing the flow rate from 6.30 to 10.50 mL/min reduces Cr and Pb adsorption capacities by 40.3% and 31.1%, respectively, accompanied by substantial reductions in breakthrough time (67.3% for Cr and 97.5% for Pb). The lower flow rate therefore favoured greater adsorption capacity and delayed breakthrough under the investigated conditions. The developed models provide a basis for predicting column responses and optimising operating conditions within the investigated experimental domain, while the identified operating conditions provide a practical guidance for operation of the MAC fixed-bed adsorption system.

References

Adamu, A. D., Sani, B. S., Muhammad and Ahmad, A. O., 2023. Modelling and optimization of cadmium removal from wastewater onto sugarcane bagasse activated carbon. Nigerian Journal of Engineering, 30(1), pp.36–42.

Ali, I. and Gupta, V. K. (2006). Advances in water treatment by adsorption technology. Nature Protocols, 1(6): 2661–2667.

Bahrami, Amiri, M., M. J. and Bagheri, F., 2019. Optimization of the lead removal from aqueous solution using two starch-based adsorbents: Design of experiments using response surface methodology (RSM). Journal of Environmental Chemical Engineering, 7(1), p.102793.

Bezerra, M. A., Santelli, R. E., Oliveira, E.P., Villar, L. S. and Escaleira, L. A., 2008. Response surface methodology (RSM) as a tool for optimization in analytical chemistry. Talanta, 76(5), pp.965–977.

Burakov, A. E., Galunin, E. V., Burakova, I. V., Kucherova, A. E., Agarwal, S., Tkachev, A. G., and Gupta, V. K., 2018. Adsorption of heavy metals on conventional and nanostructured materials for wastewater treatment purposes: A review. Ecotoxicology and Environmental Safety, 148, 702–712.

Dichiara, A. B. and Weinstein, S. J., 2015. On the choice of batch or fixed-bed adsorption processes for wastewater treatment. Industrial & Engineering Chemistry Research, 54(34), pp.8579–8586.

Es-Said, A.; Nafai, H., Zerki, N., and Bchitou, R., 2023. Chemometrics approach for multi-response optimisation of heavy metals Zn(II), Cu(II) and Cd(II) removal by phosphogypsum in a ternary aqueous solution. International Journal of Environmental Analytical Chemistry, 103(13), pp.3044–3058.

Ferreira, S. L. C., Bruns, R. E., Ferreira, H. S., Matos, G. D., David, J. M., Brandão, G. C., da Silva, E. G. P., Portugal, L. A., dos Reis, P. S., Souza, A. S., and dos Santos, W. N. L., 2007. Box–

Behnken design: An alternative for the optimization of analytical methods. Analytica Chimica Acta, 597(2), pp.179–186.

Garba, Z. N., Bello, I., Galadima, A., and Lawal, A. Y., 2016. Optimization of adsorption conditions using central composite design for the removal of copper (II) and lead (II) by defatted papaya seed. Karbala International Journal of Modern Science, 2(1), pp.20–28.

Juela, D., Vera, M., Cruzat, C., and Alvarez, X., 2021. Adsorption properties of sugarcane bagasse and corn cob for sulfamethoxazole removal in a fixed-bed column. Sustain Environ Res, 3, p.27.

Kalavathy, H., Regupathi, I., Pillai, M. G., and Miranda, L. R., 2009. Modelling, analysis and optimization of adsorption parameters for H_3PO_4-activated rubber wood sawdust using response surface methodology. Colloids and Surfaces B: Biointerfaces, 70(1), pp.35–45.

Liu, Y., Liu, F., Ni, L., Meng, M., Zhong, G., and Qiu, J., 2016. A modeling study by response surface methodology on Sr(II) ion dynamic adsorption optimization using a novel magnetic ion-imprinted polymer. RSC Advances, 6, pp.54679–54692.

Maiti, S., Prasad, B., and Minocha, A. K., 2020. Optimization of copper removal from wastewater by fly ash using central composite design of response surface methodology. SN Appl. Sci., 2, p.2151.

Mani, S. K., and Bhandari, R., 2022. Efficient fluoride removal by a fixed-bed column of self-assembled Zr(IV)-, Fe(III)-, Cu(II)-complexed polyvinyl alcohol hydrogel beads. ACS Omega, 7(17), pp.15048–15063.

Mohan, D., Sarswat, A., Singh, V. K., Alexandre-Franco, M., & Pittman Jr., C. U. (2011). Development of magnetic activated carbon from almond shells for trinitrophenol removal from water. Chemical Engineering Journal, 172(2–3), 1111–1125.

Mohseni, E., Rahmani, A., and Hamdi, Z., 2024. Efficiency of response surface methodology based on central composite design in optimizing cadmium adsorption from aqueous solutions using activated charcoal prepared from walnut shell. Desalination and Water Treatment, 317, p.100250.

Nemeș, N. S., Negrea, A., Ciopec, M., Negrea, P., Duţeanu, N., & Duda-Seiman, D. M., 2026. Heavy metal ion removal: A global review of wastewater treatment technologies. International Journal of Molecular Sciences, 27(4), 1741.

Organisation for Economic Co-operation and Development (OECD), 2001. OECD Guideline for the Testing of Chemicals: Simulation Test – Aerobic Sewage Treatment: 303 A: Activated Sludge Units – 303 B: Biofilms. Paris: OECD Publishing.

Patel, H., 2019. Fixed-bed column adsorption study: A comprehensive review. Applied Water Science, 9, 45.

Szpisznik-Gulyás; N., Al-Tayawi, A. N., Horváth, Z. H., László, Z., and Kertész, S. 2023. Methods for experimental design, central composite design and the Box–Behnken design to optimise operational parameters: A review. Acta Chromatographica, 52(4), pp.521–537.