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Waste-derived activated carbons for effective adsorptive removal of strontium, barium, and binary pollutants: A response surface methodology study

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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

A double-pronged approach to pollution management is proposed by sustainably managing solid wastes and converting them to activated carbons that are then utilized for water treatment. In this study, gas-to-liquids (GTL) derived biosolids, carboard and their mixed samples are used to produce the activated carbons. In a laboratory batch study, a Box-Behnken experimental design was used with four factors and three levels to optimize the removal of single component strontium and barium, and the binary system of pollutants from an aqueous solution. The design incorporated response surface modeling (RSM) techniques with a total of 29 different experimental data points collected and analyzed in this study. The study was conducted considering four parameters: the initial pH of the solution (ranging from 4.0 to 8.0), temperature (ranging from 20 to 40 ℃), the percentage of cardboard (fixed at 0 %, 50 %, and 100 %), and the amount of adsorbent (between 0.05 and 0.5 g). These factors were assigned three levels, represented as −1, 0, and 1. A second-order polynomial regression equation was then developed to estimate the responses. The statistical significance of the independent variables and their interactions was assessed using analysis of variance (ANOVA) with a 95 % confidence level (α = 0.05). The results revealed that only temperature and dosage show significant effects on the responses and that optimum values of the selected parameters were obtained by solving the prediction equations, which were validated with less than 4 % error %. In an attempt to optimize the factors, a pH of 5.5, temperature of 40 ℃ and dose of 0.3 g is found for all three samples. Validation results for optimization also proved that varying the percentage cardboard showed little difference in the percentage removal of all the pollutants. The results from this study can be directly applied for any such systems trying to optimize these parameters and the prediction equations can be utilized effectively.

1. Introduction

Among the many problems leading to instability in the world, waste management is one of most concern. Waste is generally generated more in high income countries every year amounting to about 34 % of the total world generated MSW even though these countries only make up to 16 % of the global population. Furthermore, an expected 3.6 Gt/yr of municipal solid waste will be produced in the year 2050 [1]. The global problem of increased waste generation is now being addressed by utilizing it in different ways that support a circular economy [2]. Solid waste management techniques mainly include open dumping, landfilling, and incineration; all with their associated environmental implications. In recent times, research efforts focus on assessing connections between waste, resource use, climate change, air and water pollution [2]. Thermochemical conversion methods, such as pyrolysis, are often explored to convert wastes to beneficial products such as biochar, bio-oil and combustible gases in inert atmospheres [3]. This not only reduces the volume of waste produced but also increases its economic value. In general, pyrolysis studies have moved from using one feed to more than one, termed 'co-pyrolysis' creating a more ideal scenario for waste

Abbreviations: KBS, Activated biosolid; AC, Activated carbon; KCB, Activated cardboard; KM, Activated mixed; ASTM, American Society for Testing and Materials; ANOVA, Analysis of variance; BS, Biosolid; BET, Brunauer-Emmett-Teller; CB, Carboard; FTIR, Fourier Transform Infrared Spectroscopy; GTL, Gas-to-Liquids; RSM, Response Surface Methodology; SEM-EDS, Scanning electron microscopy SEM and energy dispersive X-ray spectroscopy EDS; XPS, X-ray photoelectron spectroscopy; XRD, X-ray Powder Diffraction.

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management [4]. Biochar, often considered a by-product, is known to have several agricultural and water treatment applications.

There is an abundance of research being carried out to apply and use biochar for water treatment applications; however, whenever biochar is used directly the adsorption capacities are frequently low and therefore, activation is carried out to produce activated carbons with improved biochar qualities. Activation is mainly done by physical and chemical methods and in both cases, the carbon is shown to have increased surface area, pore properties and functionality- these properties are known to significantly enhance the ability to remove pollutants (both organic and inorganic) from water [4]. Recently, there has been an increasing focus on a novel approach known as 'single-stage activation,' which involves directly adding chemicals to the feeds at high temperatures without prior pyrolysis. The aim of this approach is to enhance the environmental sustainability of the system by reducing the energy, time, and cost required for the activation process [5].

When it comes to wastewater production, just like waste production, an increasing population has projected it to increase to 24 % in 2030 and 51 % in considering the current annual global volume of 380 billion cubic meters [6]. There are several pollutants that have been targeted as requiring removal, including pharmaceuticals, personal care products, pesticides, dyes, heavy metals and others from various sources (agricultural, industrial, and municipal). Among the various wastewater treatment methods such as coagulation, fenton/advanced oxidation, biological, electrochemical, membrane filtration and others, adsorption is considered the most simple, cost-effective, sustainable and environmentally friendly method [7]. However, this treatment technology still has a long way to go towards full circular implementation.

Generally, adsorption studies mainly include parametric analysis, modelling, thermodynamics and mechanistic analysis [8–14]. However, research that optimizes the parameters based on the various parameters is lacking. It is important as it is difficult to observe the effects of several parameters on responses as it requires many extra experiments. Traditionally, parametric studies keep one of the factors constant while changing the other, however, this is a tedious task with less accurate results. Response surface methodology (RSM), a multivariate optimization tool works on modelling based on factorial techniques and the Analysis of Variance (ANOVA) is useful as they extend the tools for a more detailed modeling of the parametric effects. RSM can also be used for prediction of responses based on the parameters once the analysis is complete.

Activated carbons are known to recover 100 % of the produced water (from the natural gas production) and 85 % of the heavy metals can be removed through the adsorption process [15]. Heavy metals by nature are known to be toxic or dangerous and there have been numerous adsorption attempts to remove them from water applying RSM analysis [16–18]. However, there is a lack of waste-derived activated carbons being used for such studies. The targeted heavy metals in this case are strontium and barium, both known to be pollutants present in relatively high concentrations in produced water [19]. Although RSM has been applied to water treatment studies [20–22], this is the first time it is being used to study the effect of biomass percentage (and other parameters) on the adsorption of single component strontium and barium - and the lesser studied- binary system pollutant removal. Generally, the disadvantage of adsorption in terms of cost and maintenance is resolved by using activated carbons, especially with waste origins.

For this project, the investigation into feed sources primarily focused on examining Qatar's context and the significant waste generated within the country. Specifically, domestic solid waste production exceeds 2.5 million tons annually, primarily comprised of organic materials (~60 %) along with recyclable materials like paper, glass, plastics, and metal [23]. A separate study on residential waste generation in Qatar revealed that approximately 69.1 % of respondents produce paper and cardboard waste at home [24].

Solid waste management in Qatar primarily relies on landfilling, with a small portion being incinerated or composted. Apart from environmental and cost-related concerns, the lack of available space in the country makes avoiding landfilling a priority [25]. Additionally, there is a need to significantly reduce the production of biosolids (bio sludge) stemming from biological processes in both domestic and industrial wastewater treatment, as the dewatering and disposal of this waste are costly. The wastewater treatment process in the oil and gas industry also generates substantial amounts of biosolids. For example, a single Gas-to-Liquids (GTL) plant's wastewater treatment facility in the North of Doha produces around 6000 tons/year of dry biosolids [26]. Given Qatar's heavy reliance on the oil and gas sector, finding alternative uses for the industry's waste products is imperative. Finally, initial characterization of the samples regarding surface area, pore volume and morphology showed promise for water treatment applications. Therefore, this study uniquely produces activated carbons are GTL-derived biosolids and cardboard.

The following objectives are aimed for this study: (1) The waste materials: GTL-derived biosolids (BS), cardboard (CB), and combined samples (50:50) were chemically activated using potassium carbonate, K_2CO_3, in a single activation phase (2) In order to comprehend the impacts of activation on the morphology, contents, and composition of the samples: both un-activated (BS, CB and mix) and activated (KBS, KCB and KM respectively) are characterized (4). The Design of experiments approach has been applied with RMS, based on several parameters, namely: biomass (cardboard) %, pH, temperature, adsorbent dose (5) conduct adsorption studies for strontium, barium and binary solutions for all the parameters (6) using ANOVA analysis, optimization for the prediction and validation of results.

2. Materials and methods

2.1. Production of activated carbons

The cardboard was obtained from one of Qatar's major marketplaces, and the dried biosolid sample was provided by the Pearl Shell GTL industry in Qatar, currently the world's largest GTL facility. The BS, CB, and combined (1:1) samples are activated in a single-stage activation process. Both methods involve impregnating the samples with potassium carbonate (1 M) (Reagent ACS Anhydrous, Granular, Merck, Millipore, CAS number: 584–08–7), at a ratio of 1:3 (sample: activating agent). The samples are then mixed at 100 rpm for 24 hours in an auto shaker before being dried at 105 °C for 8 hours. After being crushed, the samples are placed in a furnace, where they are gradually heated to 700 °C at a rate of 10 °C per minute in the presence of nitrogen. This process activates the samples, but they are then treated with distilled water, adjusted to a neutral using a pH probe (Orion Star A121 and A329, Thermo Scientific); the neutralizing chemical used is 1 M hydrochloric acid (Analytical reagent, 37 %, Fisher Scientific, CAS number: 7647–01–0), and dried before they can be used for subsequent adsorption test purposes. The yield is calculated using the following equation:

$$Yield\ \ of\ \ activated\ \ carbon\ \ (\%) = \frac{Weight\ \ of\ \ activated\ \ carbon(g)}{Weight\ \ of\ \ unactivated\ \ feed(g)}\ \ x100 \tag{1}$$

2.2. Characterization of samples

The samples, before and after activation, are characterized using BET (Brunauer–Emmett–Teller) instrument (Tristar3200, Micromeritics, Norcross, GA, USA) for analyzing surface area and pore volume. Then, the surface chemical bonds, specifically the carbon and oxygen bonds are studied using X-ray photoelectron spectroscopy (XPS) (ESCALAB250Xi, Thermo Fisher Scientific, East Grinstead, UK). To understand the crystallinity and the structure of the samples using a Bruker D8 Advance X-ray diffractometer (XRD) is used and for the surface charge analysis, zeta potential using (Zetasizer Nano-ZS, Malvern P analytical, Malvern, UK) is utilized. The surface morphology and composition of elements are studied by Scanning electron microscopy (SEM) (Quanta650FEG FEI SEM, Hillsboro, OR, USA) connected with energy dispersive X-ray spectroscopy (EDS) (Bruker Quantax EDS detector, Billerica, MA, USA). The detailed preparation and methodology of using all instruments can be obtained in a previous publication [3].

2.3. Experimental design by Box-behnken (BBD)

RSM is a statistical and mathematical method that, as previously mentioned, is used to optimize processes by minimizing the number of runs while concurrently establishing a relationship between the variables and responses. The Design Expert 13 software (Stat-Ease, Inc., Minneapolis, MN) was used to design the experiment, tabulate the responses, and analyze the experimental results. Optimization for maximum removal of strontium, barium, binary solutions including strontium and barium was employed using a Box-Behnken Design (BBD). The Box-Behnken design (BBD) stands out among the array of response surface methodology (RSM) techniques as a highly effective multivariate statistical approach for optimizing multiple factors in diverse processes. Its efficiency and minimal experimentation requirements have made it widely adopted. Notably, BBD surpasses other designs such as the 3-level factorial design, central composite design, and Doehlert design due to its rotatability and the absence of extreme factor combinations at highest and lowest levels, enhancing its efficiency further [27].

Four numeric factors are included in the design: pH (A), temperature (B) in °C, dose in g (C) and cardboard % (D). Table 1 shows the levels (low, middle and high levels) were designated as −1, 0 and +1, (lower and higher) numerical factors that came to a total of around 29 runs. Replication was integrated into the design to account for experimental errors. To anticipate the responses in coded values within the study's used ranges, individual linear, quadratic, cubic, and interactive mathematical empirical models were developed using the BBD approach (Table 1). To determine the regression coefficients, the experimental data were fitted to the second order polynomial model. By computing F and p values of 0.05, the statistical significance level of each polynomial equation term was examined. To understand how the process factors affected the responses, three-dimensional surface plots were created. Then, ANOVA was then used to evaluate the results. The matching F and p-values of the constructed models were used to evaluate them. Significant models correlated with higher F values and lower p values. After optimization was concluded, the model equation was validated by comparing predicted and experimental values. The following sections will include detailed analysis of the results.

The provided quadratic polynomial equation represents the function of the response variable. Typically, the function of the response is influenced by the encoded variables:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} \beta_{ij} x_i x_j + \varepsilon \quad (2)$$

Where Y is the response (heavy metal removal %) and ε is the random error. The polynomial model utilizes various coefficients, including β_0 (representing the constant term), β_i, β_{ii}, β_{ij} representing linear, quadratic, and interaction effects. The natural uncoded variables that are independent are x_i to x_k. To determine their importance in the regression equation, an investigation was conducted to assess the significance of each sentence in the equation. The study examined the significance of each sentence within the regression equation, and the identification of significant expressions in the model was achieved through the analysis of ANOVA for each response.

Table 1
Levels of parameters for BBD experiments.

Variable	Unit	Code	Level		
			-1	0	1
Temperature	°C	B	20.0	30.0	40.0
Dose	g	C	0.0500	0.275	0.50
Cardboard	%	D	0.00	50.0	100
pH	-	A	4.00	6.00	8.00

2.4. Adsorption experiments

Adsorption experiments were carried out following the BBD (Table 1). The other important parameters were maintained constant, such as the contact time at 9 hours and auto shaker speed at 150 rpm for all experiments. Additionally, the initial pollutant concentration was prepared at 100 ppm from 500 ppm stock solutions for the single strontium (strontium nitrate, Merck, CAS number:10042–76–9), barium (barium nitrate, SCP Surechem, CAS number: 10022–31–8) and binary solutions in 300 mL of deionized water. For the pH adjustments, 1 M hydrochloric acid (Analytical reagent, 37 %, Fisher Scientific, CAS number: 7647–01–0) or 1 M sodium hydroxide (SIGMA, CAS number: 1310–73–2) was used.

The initial and final concentrations of MB in the samples were determined using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), specifically the Agilent 5800 ICP-OES instrument located in Palo Alto, CA, USA. After the adsorption process, the solutions were filtered, and the concentration of MB in the supernatant was measured. The calibration of strontium and barium can be seen in Figure S1. The removal percentage was calculated using Equation 3.

Heavy metal removal% $= \frac{C_0 - C_e}{C_0} x100$ (3) where C_o = initial concentration in ppm and C_e = final equilibrium concentration in ppm

3. Results and discussion

3.1. Characterization

Following activation due to elevated temperature, proximate analysis reveals decreased moisture and increased ash in the feeds (Table 2). Due to devolatilization at high temperatures, it has also been seen that the volatile content has decreased. Regarding the final study, the findings unequivocally demonstrate that the biochar sample carbon content has decreased (Table 2). When potassium carbonate is used to activate the AC, a rise in oxygen is noticed, which may imply an increase in oxygen-containing functional groups. Nitrogen levels in the BS and mixed samples were notable due to its origin (Table 2).

Surface area (SA) and pore volume (PV) of the feeds are less, with values for SA and PV for both samples less than 1.745 m^2/g and 0.04 cm^3/g, respectively. Activation causes the SA to significantly rise, rising to 156 m^2/g for BS, 515 m^2/g for CB, and 527 m^2/g for mixed samples (Table 3). The fact that the KM has the highest surface shows that the mixed feeds work best together suggesting a synergistic effect.

Table 2
Proximate and ultimate analysis of samples.

Ultimate analysis*					
Sample	**C**	**H**	**O****	**N**	**S**
BS	33.69	6.18	29.02	5.03	-
KBS	20.80	0.89	35.47	2.91	-
CB	48.87	4.77	36.30	0.05	-
KCB	21.61	1.22	39.52	0.20	-
KM	30.12	2.17	30.17	2.01	-
Proximate analysis***					
Sample	**Moisture**	**Fixed carbon**	**Volatile matter**	**Ash**	
BS	12.29	14.16	47.47	26.08	
KBS	4.13	40.87	15.07	39.93	
CB	5.53	72.60	11.84	10.03	
KCB	3.32	51.67	7.55	37.46	
KM	3.09	40.57	20.81	35.53	

* dry basis
** by difference.
*** air-dried basis

Table 3
Surface properties of samples.

Sample	Surface area (m^2/g)	Pore volume (cm^3/g)	Surface charge
BS	0.010 ± 0.00	0.0210 ± 0.00	-17.35 ± 0.2
KBS	156.5± 25.5	0.235 ± 25.5	-20.31 ± 0.4
CB	1.745 ± 0.08	0.040 ± 0.01	-20.42 ± 0.2
KCB	515.7 ± 20.1	0.196± 25.5	-24.45 ± 0.8
Mix	0.56 ± 0.01	0.010 ± 0.00	-19.33 ± 0.2
KM	527.0 ± 29.3	0.357± 25.5	-25.61 ± 0.5

The pore volume also experienced a significant improvement (Table 3). Surface area and pore volume increase is also evident from the SEM images in Fig. 1. Each sample has a minor negative charge change after being pyrolyzed and activated (Table 3).

The XRD analysis highlights the importance and benefits of activation, as all activated carbons are known to have increased functionality. Because magnetite was present after activation, the BS and mixed samples exhibit magnetic characteristics (Fig. 2). This might be because iron coagulation was used to treat the wastewater, which could have led to the iron presence in the BS feed- this is apparent from the EDS results (Figs. 2 and Fig. 3). On the contrary, the carboard samples only detected cellulose and calcite due to its natural presence. Table 4

The XPS results showing the chemical state analysis of C1s of the feeds: BS and CB denoted peaks C-C/C-H, C-O/C-N, C=O and -CO3/O-C=O (Fig. 4 A and C). Upon activation, KBS, KCB and KM demonstrated peaks C-C/C-H, C-O/C-N, and increased in C=O, and $-CO_3$/O-C=O peaks in addition to $K2p_{3/2}$ and $K2p_{1/2}$ – this is due to activation using potassium carbonate (Fig. 4).

3.2. Box-Behnken Design (BBD) analysis

In both single and binary systems, the removal of strontium is less effective compared to barium. The use of the tested carbons in this study proves to be beneficial because their adsorption capacities are significantly higher than what has been reported in existing literature. In the random points provided by the RSM BBD design, the maximum removal of all pollutants was revealed using 0.5 g of ACs with operating conditions of 40 °C and a pH of 6 with removal % of 91.22, 99.80, 82.45 and 89.55 for Sr, Ba, Bin-Sr and Bin-Ba respectively.

Besides the positive prediction model outcomes in the statistics, the Design Expert Software offers additional diagnostic tools for further analysis using the experimental data and model. This discussion focuses on four key diagnostic tools: the normal plot of residuals, residuals versus predicted, Box-Cox plot for power transformations and Cook's distance (Fig. 5). These graphs aid in identifying any outliers within the experimental results. The normal plot of residuals exhibits a mostly straight line, indicating the model's accurate predictions. With an R^2 value of greater than 0.941 in all cases, approximately 94 % of the data points align closely with the fitted regression line, leaving only 6 % as residues. The following are the un-adjusted R^2 values: 0.96, 0.96, 0.95, 0.94 for Sr, Ba, binary-Sr and binary-Ba respectively. Both the residual versus predicted and residual versus run plots demonstrate consistent variations without any noticeable trends or outliers (no points above the red line). A similar conclusion can be made from the Cook's distance plots as well. The Box-Cox plot, derived from the normal probability plot and computed correlation coefficient, suggests that no transformation is necessary to normalize the data in the model. This confirms the assumption of normality for this particular experiment.

Fig. 6 illustrates the correlation between the predicted and actual removal %, and the linearity of the graph indicates that the model is effective in efficiently predicting the yields. Additionally, Fig. 7 displays perturbation curves for all the responses based on different parameter conditions in this study (A, B, C, and D). The slope of each curve indicates the sensitivity of the responses to the respective factors. Notably, factors B (temperature) and C (dose) exhibit the highest slopes, indicating that they have the most significant impact on all the responses. Conversely, factors A (pH) and D (cardboard percentage) display the least sensitivity. It can be inferred that the pH effect may not be pronounced due to the narrow pH range selected, which might hinder the detection of significant effects. The fact that the biomass percentage or cardboard percentage does not affect the removal rates highlights the effectiveness of modification using potassium carbonate. In other words, the functionality of the carbons likely played a more prominent role in the adsorption process compared to other characteristics such as surface area and pore volume.

The ANOVA was used to evaluate the data on the interactive effects of the operational characteristics on the process. Based on the F-test and p-values, the effective parameters for modelling the response were examined using ANOVA. Table 8 provides the BBD for the experimentally obtained findings and the model-predicted results linked to the removal of heavy metal contaminants by activated KBS, KCB, and KM. The ANOVA findings of the quadratic model for the elimination of strontium, barium, and binary solutions are also shown in Table 5. The F-value of 58.62 for the model suggests that the model has a considerable impact on the response, as made clear by ANOVA.

According to F-values, the tested parameters had the following effects in order on the adsorption process: For every pollutant removal (barium is an exception), dose > temperature > cardboard % > pH was

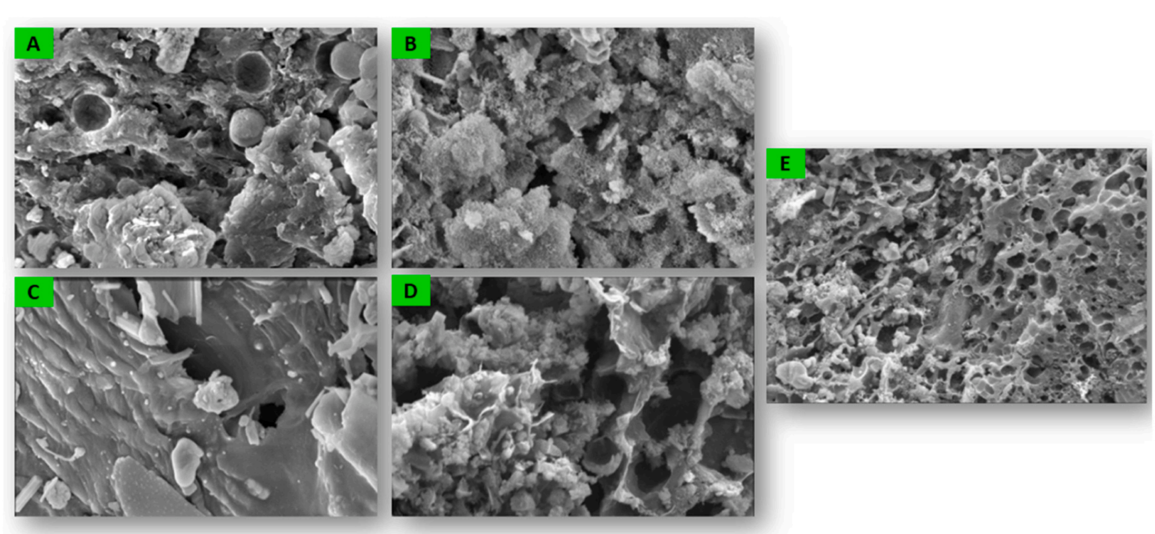

Fig. 1. SEM images at 50,000 magnification (A)BS (B) KBS (C) CB (D) KCB (E) KM.

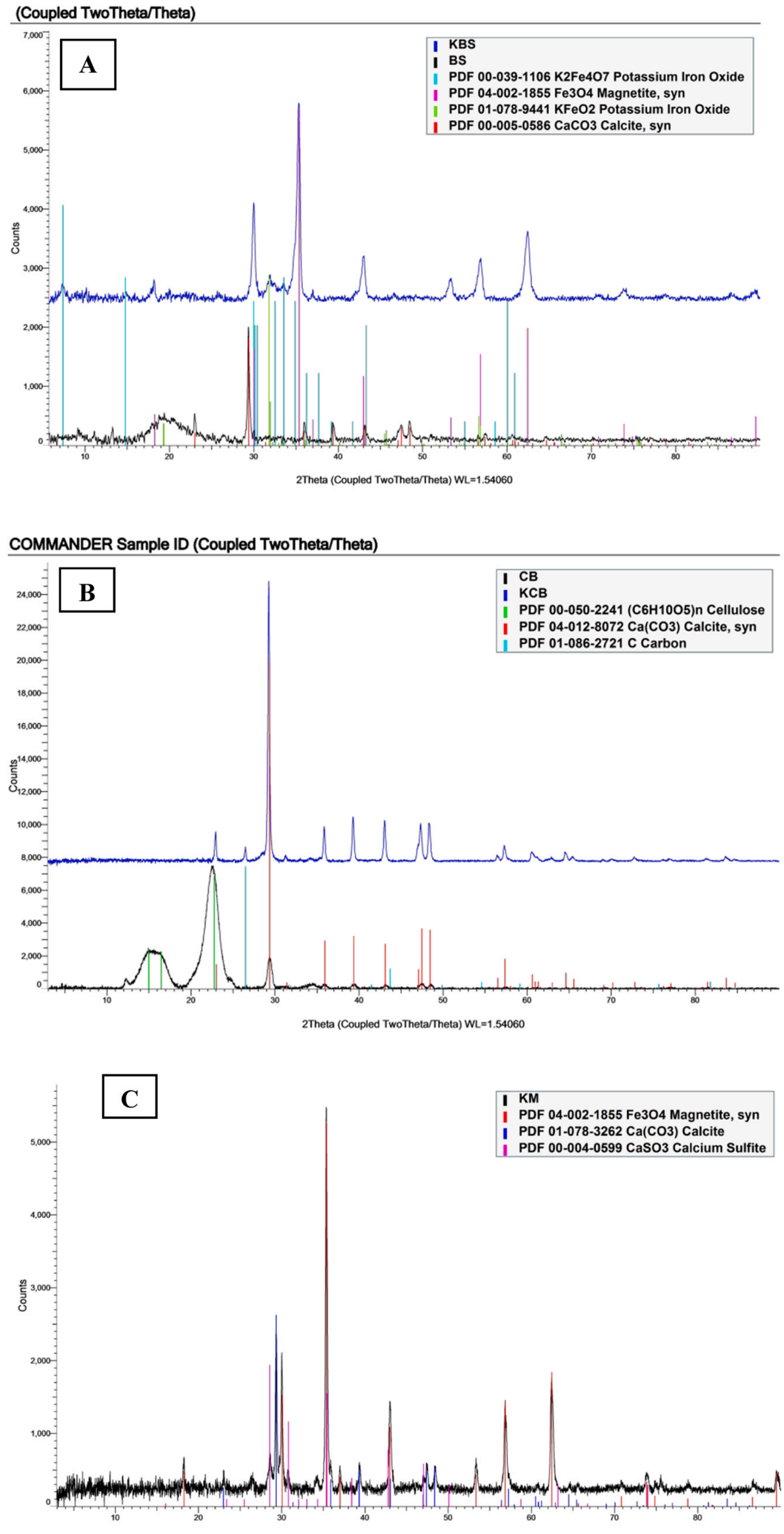

Fig. 2. XRD results (A)BS & KBS (B) CB & KCB (C) KM.

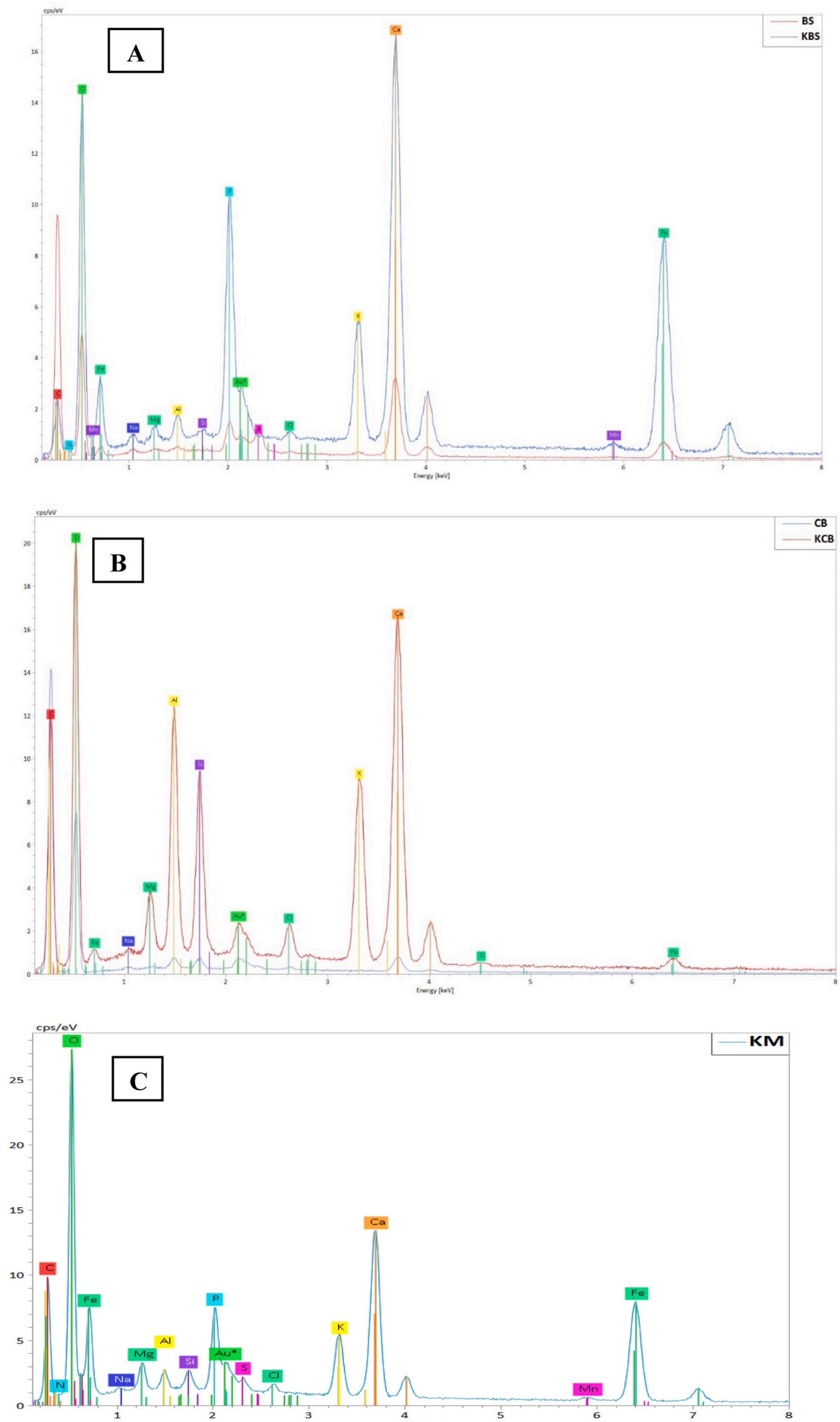

Fig. 3. EDS results (A) BS & KBS (B) CB & KCB (C) KM.

Table 4
BBS design runs with responses (heavy metal removal).

Run	Factor A	Factor B	Factor C	Factor D	Response 1: Sr removal (%)	Response 2: Ba removal (%)	Response 3: Bin-Sr removal (%)	Response 4: Bin- Ba removal (%)
1	6	40	0.275	0	80.77	92.98	78.66	87.55
2	4	30	0.05	50	20.54	28.45	15.89	20.76
3	6	30	0.05	0	25.18	31.46	10.23	24.44
4	6	20	0.5	50	80.66	88.66	52.46	60.83
5	6	30	0.5	100	89.82	97.68	82.01	87.54
6	6	40	0.275	100	83.98	93.08	76.29	85.10
7	8	30	0.275	100	53.40	70.55	46.86	62.44
8	6	30	0.275	50	80.75	85.75	61.00	72.60
9	6	20	0.275	0	72.54	80.91	56.98	64.43
10	6	30	0.275	50	89.13	94.35	75.28	80.21
11	4	40	0.275	50	56.24	94.94	61.93	72.43
12	4	30	0.5	50	83.45	95.53	72.55	88.19
13	6	20	0.05	50	22.44	29.87	14.94	21.44
14	6	40	0.05	50	33.49	47.79	22.19	34.44
15	6	30	0.05	100	38.76	47.45	20.97	32.54
16	6	30	0.5	0	88.22	98.22	76.56	76.55
17	6	30	0.275	50	86.76	94.32	78.24	84.32
18	6	30	0.275	50	90.93	96.52	72.23	86.33
19	8	20	0.275	50	49.12	70.34	35.23	62.82
20	4	30	0.275	0	58.33	82.22	44.71	75.44
21	8	40	0.275	50	80.55	80.09	63.38	71.12
22	4	30	0.275	100	79.96	86.68	68.22	74.33
23	6	30	0.275	50	89.24	95.87	79.12	82.22
24	4	20	0.275	50	50.67	68.13	34.79	50.66
25	6	20	0.275	100	71.97	89.97	56.66	84.40
26	8	30	0.5	50	77.22	85.96	64.22	77.88
27	8	30	0.275	0	61.15	70.76	58.10	65.77
28	6	40	0.5	50	91.22	99.80	82.45	89.55
29	8	30	0.05	50	28.97	47.11	13.97	37.54

* 0 % CB = KBS, 50 % CB = KM, 100 % CB = KCB

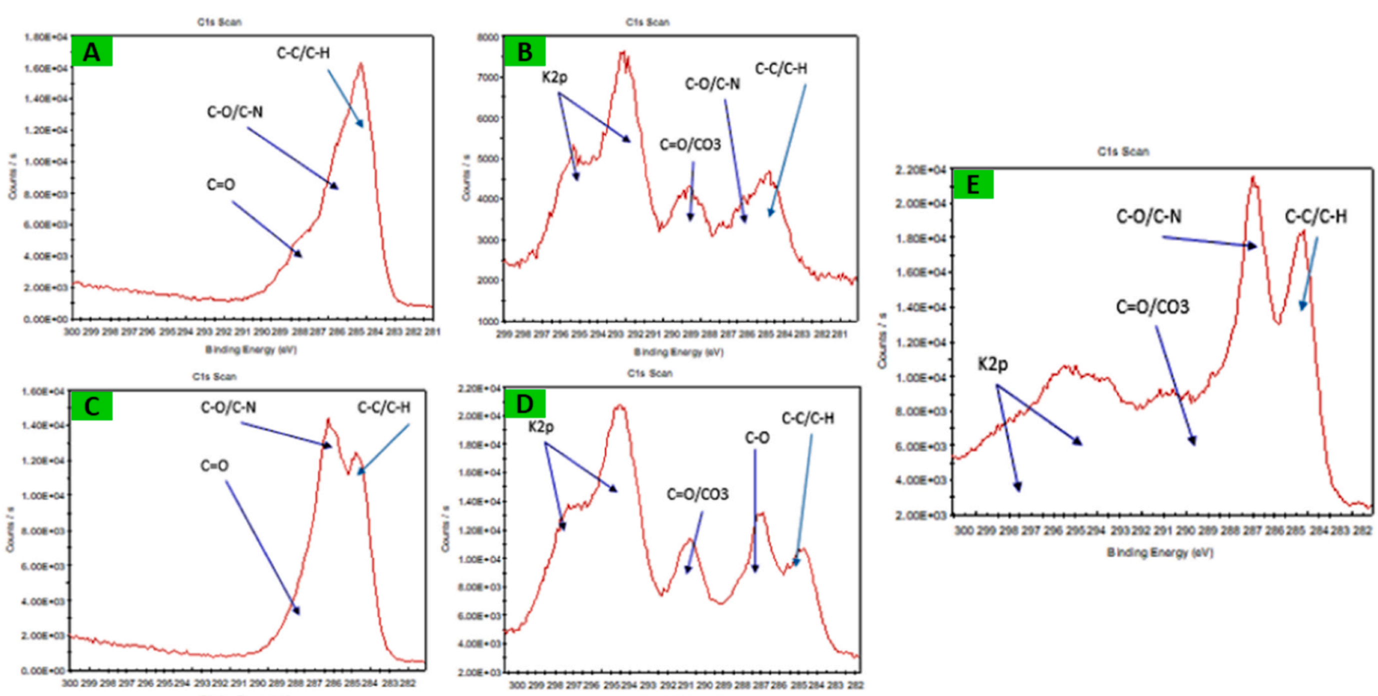

Fig. 4. XPS results (A) BS (B) KBS (C) CB (D) KCB (C) KM.

followed. Regarding barium, pH shows more notable impacts than cardboard %. The "Lack of Fit F-value" for strontium (Sr), barium (Ba), binary-strontium (Bin-Sr) and binary-barium (Bin-Ba) is 0.1515, 0.2895, 0.657, and 0.2061 respectively. This value denotes that the Lack of Fit is not significant compared to the pure error and the associated p-value, indicating the significance of this model for predicting the experimental data. The values of the R^2 and "R^2 adjusted" were observed to be 0.9413 and 0.8826 for Bin-Sr, 0.9413 and 0.9591 for Bin-Ba, and 0.965 and 0.96 for Sr and Ba, respectively.

Furthermore, the overall model showed significance at 95 % confidence in all cases (Table 5). The only two factors that showed significance ($P < 0.00001$) with all metal removal is temperature and

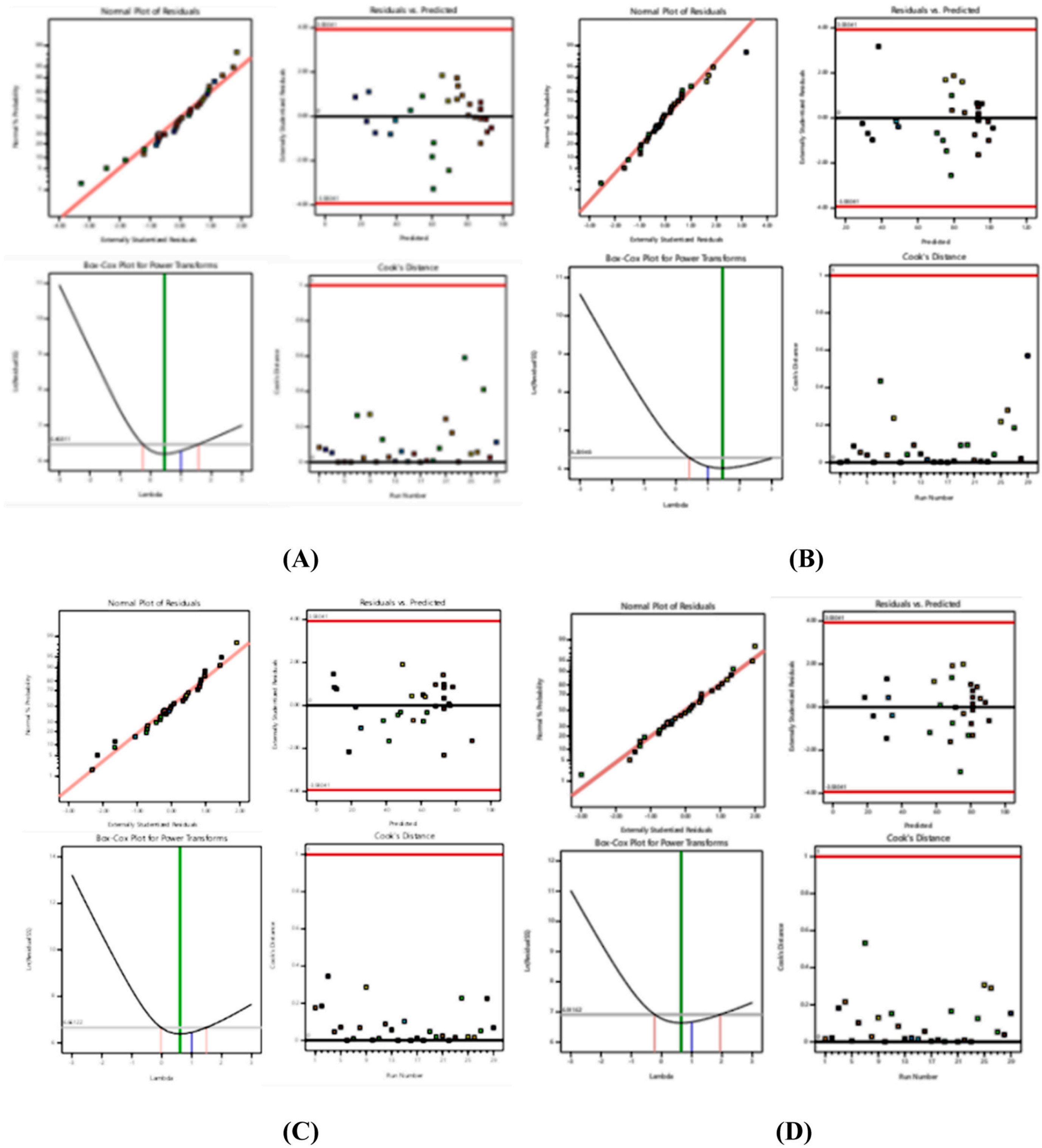

Fig. 5. ANOVA results including Normal plot of residuals, Residuals vs. predicted, Box-Cox plot for transformation and Cook's distance for (A) Sr (B) Ba (C) Binary-Sr (D) Binary-Ba.

adsorbent dosage. Additionally for Sr and Bin-Sr, AD, A^2, B^2, C^2 interactions were shown to be significant and as for Ba and Bin-Ba, AC, A^2, C^2 - this could be a possible indication as the similarity in adsorption patterns in both single and binary systems.

Different models were used for data fitting, such as linear, quadratic, and cube models, to create regression equations. For all pollutants subjected to adsorption using activated carbons, the quadratic model exhibits the highest degree of fitness (Table 6). The final model is presented in Table 7 to predict the removal percentage values of all the pollutants. A, B, C, and D were indicative of pH, temperature, dose, and cardboard, respectively.

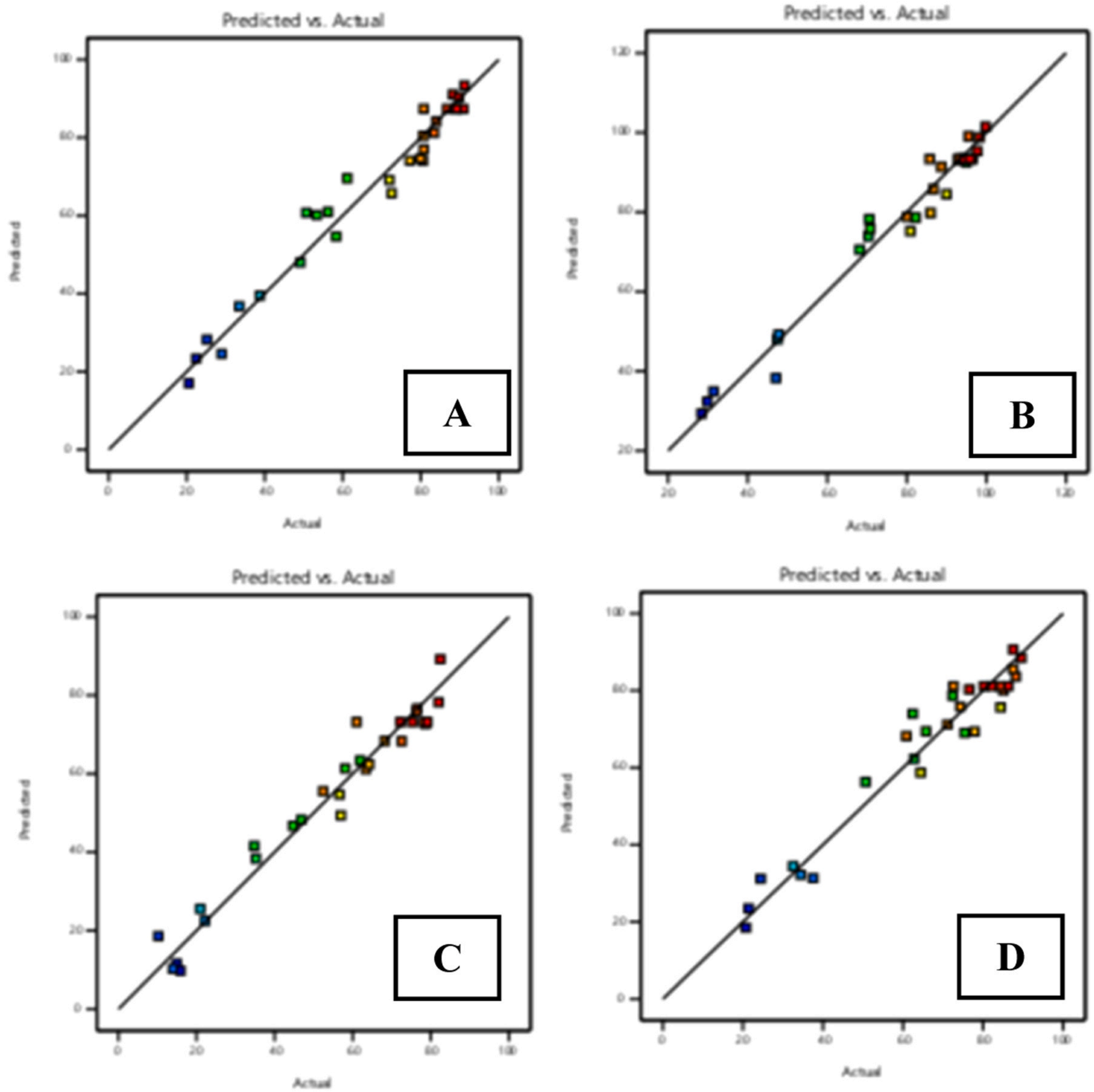

Fig. 6. Predicted Vs Actual (A) Sr (B) Ba (C) Binary- Sr (D) Binary-Ba.

3.3. Effect of parameters

Fig. 8 shows the effect of temperature and dose, the significant parameters on heavy metals removal. Reducing temperature and dosage is known to decrease the heavy metal removal from water. Regarding temperature for heavy metals adsorption processes, it is a crucial factor that impacts both the capacity and behavior of the adsorption. Typically, in most adsorption processes, the efficiency of adsorption tends to rise as the temperature increases, reaching a peak at a certain point after which the efficiency begins to decrease due to the occurrence of desorption [28]. This is observed to be true in both single and binary systems- in all cases, adsorption increased until it reached 40 ℃. Since the adsorption increased with rise in temperature (Fig. 8), increased temperature improves the efficiency by promoting greater movement of the molecules toward the active sites of the adsorbent. Similarly, increased dose is known to be favorable to adsorption due to the increase in active sites for the pollutants to adsorb, this like the effect of temperature, becomes constant after a certain amount [28]- in this case, adsorption increased till adsorbent dose reached 0.5 g (Fig. 8).

The potential mechanism for the removal of strontium, barium and binary pollutants from water using ACs is discussed in this section. The possible interactions include the attraction of the positively charged metals to the negatively charged activated carbons. Additionally, the stretching vibration of -OH and C-O group, with the involvement of most acidic groups such as ketone, carboxylic, and ester based on the XPS analysis (Fig. 4); the major adsorption sites for metal pollutants could be the C═C and C═O present on the ACs. The interaction between the metals and the carbons is mostly through electron withdrawing of π-electrons from AC surface layer. The adsorption mechanism can also be related to hydrophobic interactions induced by Van der Waals forces and the π-acceptor cloud interactions between activated carbon and heavy metals. Other possible mechanisms such as ion exchange need to be explored further. Both temperature and dosage would have played a key role in facilitating the above discussed mechanism of adsorption.

3.4. Predictability and optimization

Since the difference between the predicted and adjusted R^2 values is less than 0.2 in each case, it can be concluded that the statistical model accurately predicted removal rates for all contaminants and aligned well with the experimental data (error % below 4 in all cases). The model effectively establishes the relationship between the response

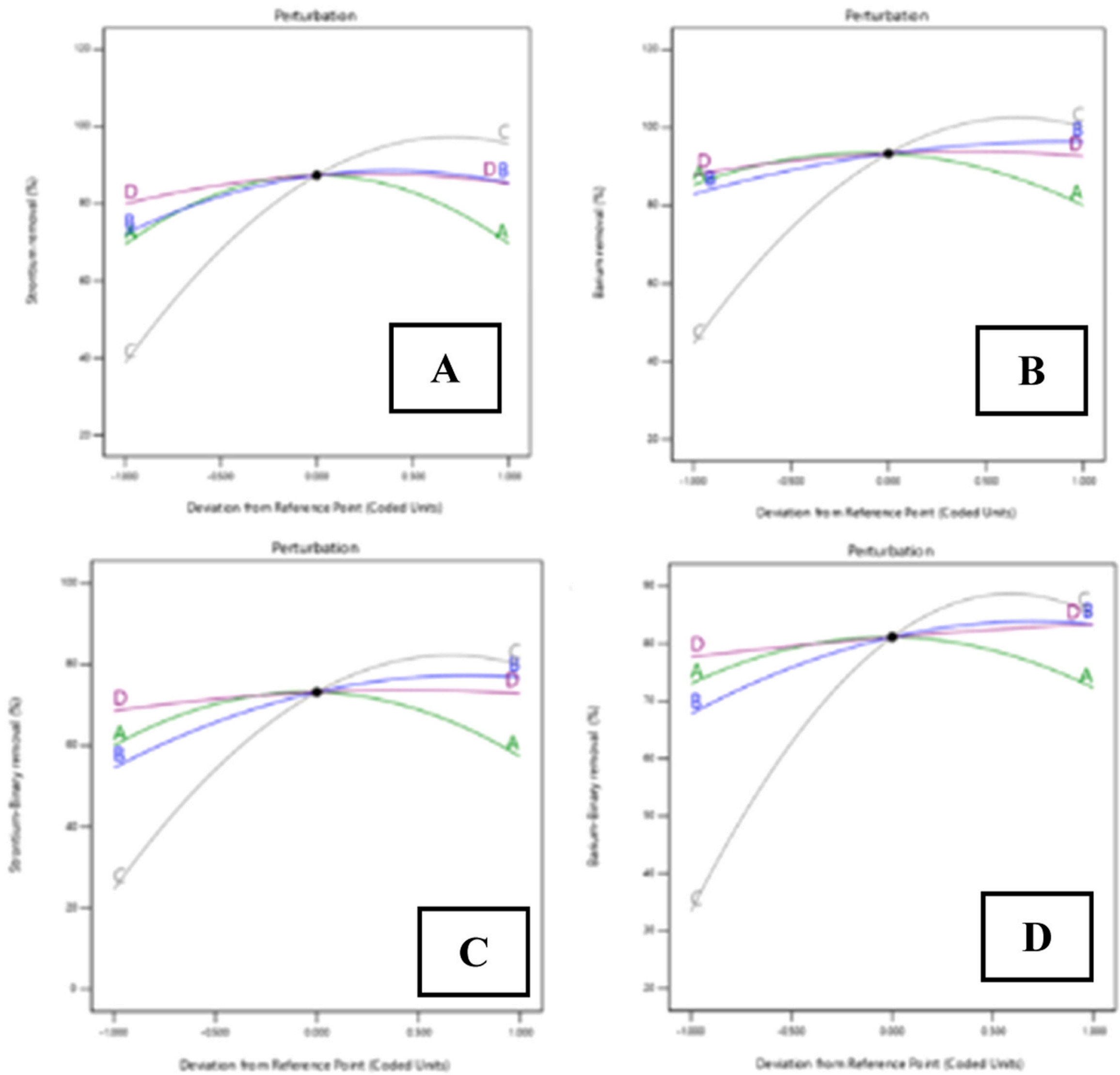

Fig. 7. Perturbation plots (A) Sr (B) Ba (C) Binary- Sr (D) Binary-Ba.

(percentage of pollutants removed) and the relevant variables, as determined by ANOVA. The prediction equations presented in Table 7 were validated for all removal responses, where the cardboard percentage was fixed at 0, 50, and 100 for KBS, KM, and KCB, respectively. The remaining parameters (pH: 6, temperature: 40 °C, dosage: 0.3 g) were optimized based on the results and choosing the case with least dosage for economic benefits. Table 7 shows that the experimental results closely matched the predicted values for all three samples and pollutant removal cases. Table 8

To optimize the process, two scenarios with the highest desirability were examined for maximum pollutant removal. The first scenario involved using 67.56 % cardboard, while the second scenario used 14.94 % (Table 7). In both cases, the pH, temperature, and dosage were set to 6, 40 °C, and 0.3 g, respectively. These cases yielded strontium, barium, and binary removal results that were similar to the mixed AC-KM values. However, it should be noted that the optimization results overestimated the values with percent error between 4 % and 11 %, as the focus was on maximizing pollutant removal. Additionally, since cardboard % is shown to have no significant impact based on the statistical analysis, the difference between the adsorption capacities utilizing KM and the two scenarios mentioned here showed minimal difference. Based on the validation results, it is advisable to optimize the process while keeping the responses within an acceptable range, rather than maximizing them, to obtain realistic solutions. Mixing the wastes has been shown to be more favorable for adsorption probably to do the increased surface area and pore volume of the mixed activated carbon (Table 3). The adsorption capacities are better some literature studies [10,11,13] (Table S1)- for instance, a previous study using spent coffee grounds biochar and activated carbon reported strontium removal rates of 51.81 and 32.79 mg/g, which are both much lower than the values obtained in this work. Additionally, another study utilizing biobased chitosan derivatives removed 64 mg/g of barium from water. Also, several studies have compared the removal of strontium and barium from water and have concluded that barium exhibits better selectivity and higher adsorption capacities. One such study utilized dolomite (with reported values of Sr: 1.172 mg/g, Ba: 3.958 mg/g) [29], while another study utilized expanded perlite (with reported values of Sr: 1.14 mg/g, Ba: 2.486 mg/g) [30].

The findings obtained indicated a higher adsorption of barium onto KM compared to strontium. This variation in adsorption capacity may stem from a range of factors, including the experimental conditions, adsorption mechanism, and metal selectivity. Although the adsorption of metal ions with higher electronegativity tends to be greater, in this scenario, the electronegativities of the metals (Sr: 1.0 and Ba: 0.9) are quite close by, necessitating the consideration of additional factors. The results of this study suggest that the preference of the tested activated

Table 5
ANOVA for quadratic model based on RSM design results.

Source	Sum of squares	df	Mean square	F-value	P-value	
Strontium removal						
Model	14833.64	14	1059.55	27.6	< 0.0001	significant
A-pH	0.1248	1	0.1248	0.0033	0.9553	not significant
B-Temperature	518.12		518.12	13.5	0.0025	
C-Dose	9702.24		9702.24	252.76	< 0.0001	
D- Cardboard %	83.72		83.72	2.18	0.1619	
AB	167.18		167.18	4.36	0.0557	
AC	53.73		53.73	1.4	0.2565	
AD	215.85		215.85	5.62	0.0326	
BC	0.0602		0.0602	0.0016	0.969	
BD	3.57		3.57	0.0931	0.7648	
CD	35.88		35.88	0.9347	0.35	
A^2	2064.64		2064.64	53.79	< 0.0001	
B^2	478.14		478.14	12.46	0.0033	
C^2	2671.87		2671.87	69.61	< 0.0001	
D^2	148.26		148.26	3.86	0.0695	
Residual	537.4	14	38.39			
Lack of Fit	473.94	10	47.39	2.99	0.1515	
Pure Error	63.46	4	15.87			
Cor Total	15371.04	28				
Barium removal						
Model	13563.74	14	968.84	31.65	< 0.0001	significant
A-pH	80.81	1	80.81	2.64	0.1265	
B-Temperature	544.09		544.09	17.78	0.0009	
C-Dose	9281.14		9281.14	303.22	< 0.0001	
D- Cardboard %	69.34		69.34	2.27	0.1545	
AB	72.76		72.76	2.38	0.1454	
AC	199.17		199.17	6.51	0.0231	
AD	5.44		5.44	0.1778	0.6797	
BC	11.45		11.45	0.3742	0.5505	
BD	20.05		20.05	0.6551	0.4318	
CD	68.31		68.31	2.23	0.1574	
A^2	742.15		742.15	24.25	0.0002	
B^2	89.78		89.78	2.93	0.1088	
C^2	2872.29		2872.29	93.84	< 0.0001	
D^2	60.19		60.19	1.97	0.1826	
Residual	428.52	14	30.61			
Lack of Fit	352.46	10	35.25	1.85	0.2895	not significant
Pure Error	76.06	4	19.02			
Cor Total	13992.26	28				
Binary – strontium						
Model	14915.44	14	1065.39	23.46	< 0.0001	significant
A-pH	22.21	1	22.21	0.4889	0.4959	
B-Temperature	1492.8		1492.8	32.87	< 0.0001	
C-Dose	9188.28		9188.28	202.3	< 0.0001	
D- Cardboard %	55.34		55.34	1.22	0.2883	
AB	0.2591		0.2591	0.0057	0.9409	
AC	10.24		10.24	0.2255	0.6422	
AD	301.78		301.78	6.64	0.0219	
BC	129.38		129.38	2.85	0.1136	
BD	1.06		1.06	0.0232	0.881	
CD	6.98		6.98	0.1538	0.7009	
A^2	1369.91		1369.91	30.16	< 0.0001	
B^2	366.77		366.77	8.08	0.0131	
C^2	2849.36		2849.36	62.74	< 0.0001	
D^2	39.99		39.99	0.8805	0.364	
Residual	635.86	14	45.42			
Lack of Fit	421.26	10	42.13	0.7852	0.657	not significant
Pure Error	214.59	4	53.65			
Cor Total	15551.3	28				
Binary – barium						
Model	12600.91	14	900.07	16.03	< 0.0001	significant
A-pH	1.5	1	1.5	0.0267	0.8726	
B-Temperature	730.1		730.1	13.01	0.0029	
C-Dose	7976.07		7976.07	142.09	< 0.0001	
D- Cardboard %	97.38		97.38	1.73	0.2089	
AB	45.35		45.35	0.808	0.3839	
AC	183.52		183.52	3.27	0.0921	
AD	1.23		1.23	0.0219	0.8845	
BC	46.97		46.97	0.8368	0.3758	
BD	125.63		125.63	2.24	0.1568	
CD	5.99		5.99	0.1068	0.7487	
A^2	467.19		467.19	8.32	0.012	
B^2	200.78		200.78	3.58	0.0795	

(*continued on next page*)

Table 5 (*continued*)

Source Strontium removal	Sum of squares	df	Mean square	F-value	P-value	
C^2	3124.15		3124.15	55.66	< 0.0001	
D^2	2.28		2.28	0.0405	0.8433	
Residual	785.86	14	56.13			
Lack of Fit	673.84	10	67.38	2.41	0.2061	not significant
Pure Error	112.02	4	28.01			
Cor Total	13386.77	28				

Table 6
Fit summary of responses.

Source	Sequential p-value	Lack of Fit (p-value)	Adjusted (R^2)	Predicted (R^2)	
Strontium removal %					
Linear	< 0.0001	0.008	0.6154	0.5585	
2FI	0.9228	0.0051	0.5354	0.3498	
Quadratic	**< 0.0001**	**0.1515**	**0.9301**	**0.816**	**Suggested**
Cubic	0.3827	0.1049	0.9405	-0.2478	**Aliased**
Barium removal %					
Linear	< 0.0001	0.0176	0.6651	0.5977	
2FI	0.923	0.0112	0.5954	0.3525	
Quadratic	**< 0.0001**	**0.2895**	**0.9387**	**0.8464**	**Suggested**
Cubic	0.4421	0.2035	0.9438	0.0392	**Aliased**
Binary-strontium removal %					
Linear	< 0.0001	0.0843	0.6405	0.5802	
2FI	0.9231	0.056	0.5656	0.36	
Quadratic	**< 0.0001**	**0.657**	**0.9182**	**0.8224**	**Suggested**
Cubic	0.869	0.2799	0.8783	-0.7906	**Aliased**
Binary-barium removal %					
Linear	< 0.0001	0.0284	0.6007	0.5101	
2FI	0.932	0.0181	0.5151	0.1764	
Quadratic	**< 0.0001**	**0.2061**	**0.8826**	**0.697**	**Suggested**
Cubic	0.8017	0.0591	0.8393	-2.7664	**Aliased**

carbons (ACs) for barium (mass: 137.327 u) over strontium (87.62 u) can likely be attributed to their selectivity toward larger pollutants, as evidenced by the pore volume values (Table 3). This selectivity is also evident in the removal capacities of the same ACs for methylene blue, which has a molecular weight of 319.85 g/mol [31].

4. Conclusion

The removal of heavy metal contaminants from wastewater is a critical environmental concern especially concerning contaminants like strontium and barium, predominantly originating from oil and gas industries, which pose significant risks to human health and ecosystems due to their toxicity. Activated carbon has emerged as a promising adsorbent for the efficient removal of these pollutants; however, optimizing the adsorption processes is essential to maximize efficiency. This study explores waste-derived activated carbons and their potential for adsorbing strontium, barium, and binary contaminants. Employing response surface methodology (RSM), a statistical model has been developed to optimize parameters and predict pollutant removal percentages.

Following the activation of biosolids (KBS), cardboard (KCB) and mixed samples (KM) using potassium carbonate, the proximate and ultimate analysis reveals changes in their composition, including decreased moisture, increased ash, and reduced volatile and carbon content. The activated carbons exhibited enhanced surface area and pore volume, resulting in improved adsorption capacity. The use of potassium carbonate as an activation agent led to increased oxygen-containing functional groups on the surface.

The adsorption capacities of the waste-derived activated carbons were significantly higher than those reported in previous studies, demonstrating their effectiveness. The chemical processes and interactions between strontium, barium, binary pollutants, and activated carbon are influenced by various parameters, with temperature and dose significantly impacting adsorption efficiency.

Statistical analysis helps validate experimental results and aids in optimizing the process for maximum pollutant removal while considering practical constraints. Additionally, the preference of activated carbons for barium over strontium can be attributed to their selectivity towards larger pollutants. The statistical model developed based on RSM accurately predicted the removal percentages of the pollutants with less than 3 % error compared to experimental results. The optimization results suggest scenarios with high desirability for maximum pollutant removal at 40 ℃ temperatures, a dose of 0.3 g and pH of 5.5.

Overall, this study provides insights into the adsorption properties and optimization of waste-derived activated carbons for the efficient removal of strontium, barium, and binary contaminants. By understanding the factors that influence the adsorption process and utilizing the response surface methodology, we aim to contribute to the development of effective and sustainable wastewater treatment strategies in a double pronged approach to environmental sustainability.

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CRediT authorship contribution statement

Shifa Zuhara: Conceptualization, Methodology, Formal analysis, Writing. **Gordon McKay:** Supervision, Reviewing and Editing.

Table 7
Prediction equations of all responses.

Pollutant	Predicted equation
Strontium	Removal % = −221.556 + 49.789 * A + 3.78948 * B + 410.685 * C + 0.701335 * D + 0.32325 * AB + −8.14444 * AC + −0.07346 * AD + −0.0545333 * BC + 0.00189 * BD + −0.266222 * CD + −4.46023 * A^2 + −0.0858565 * B^2 + −400.902 * C^2 + −0.00191237 * D^2
Barium	Removal % = −205.411 + 42.0848 * A + 4.61583 * B + 487.233 * C + 0.475264 * D + −0.21325 * AB + −15.6807 * AC + −0.0116642 * AD + −0.752089 * BC + −0.0044778 * BD + −0.367333 * CD + −2.67412 * A^2 + −0.0372044 * B^2 + −415.666 * C^2 + −0.00121852 * D^2
Binary - strontium	Removal % = −236.372 + 47.8565 * A + 4.90701 * B + 302.064 * C + 0.726556 * D + 0.0127263 * AB + −3.55617 * AC + −0.0868597 * AD + 2.52769 * BC + −0.0010275 * BD + −0.117456 * CD + −3.63313 * A^2 + −0.0751959 * B^2 + −414.003 * C^2 + −0.000993211 * D^2
Binary – barium	Removal % = −194.948 + 34.751 * A + 5.2699 * B + 392.195 * C + 0.420241 * D + −0.168362 * AB + −15.0522 * AC + −0.00554 * AD + 1.523 * BC + −0.0112085 * BD + 0.1088 * CD + −2.1217 * A^2 + −0.0556353 * B^2 + −433.507 * C^2 + −0.000236913 * D^2

*A-pH; B-temperature; C-dose; D-cardboard

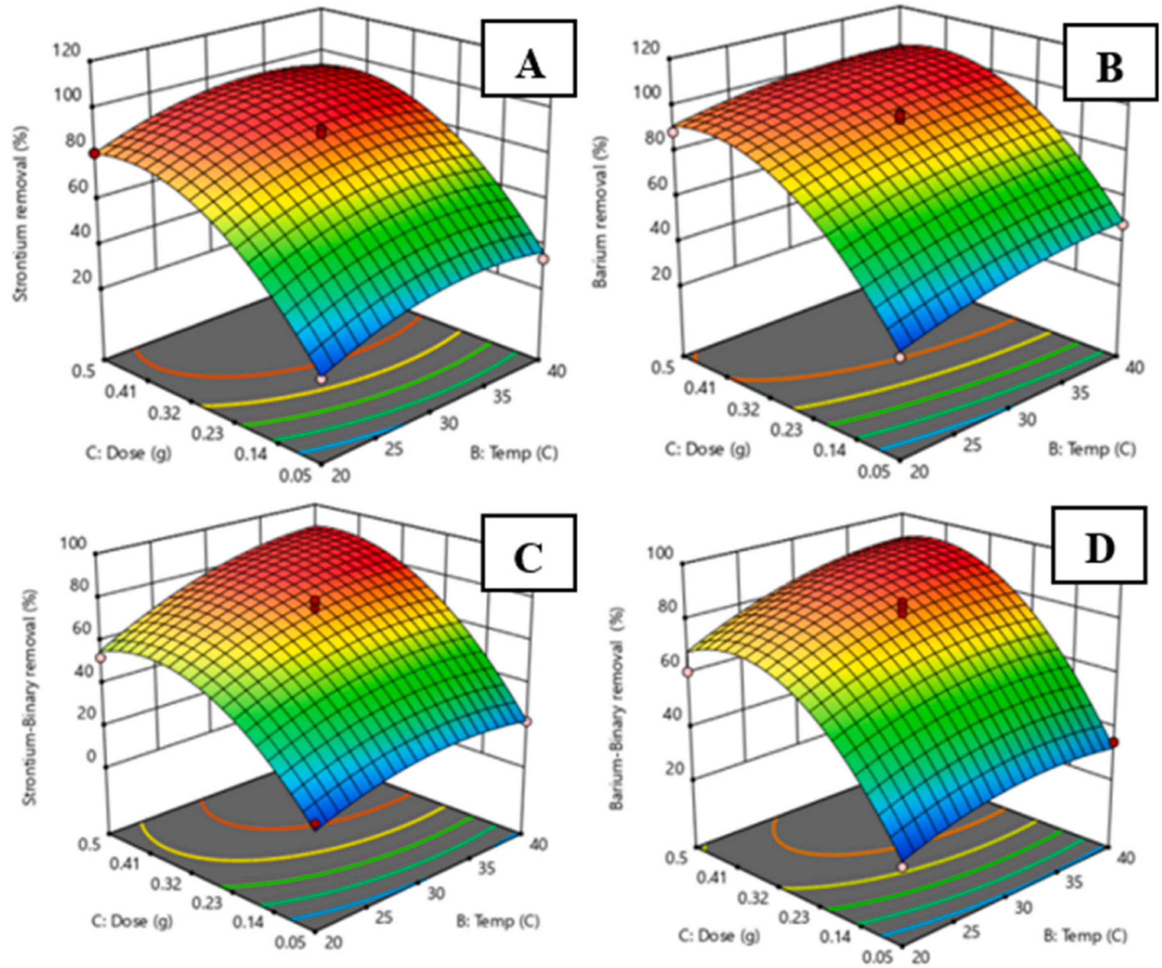

Fig. 8. Three-dimensional response surface plots (A) Strontium (B) Barium (C) Binary- strontium (D) Binary-barium.

Table 8
Validation of predicted and optimized results.

Removal (%)/ Adsorption capacity (mg/g)		Strontium		Barium		Binary- Strontium		Binary-Barium	
Sample	**Operating conditions**	**Predicted**	**Actual**	**Predicted**	**Actual**	**Predicted**	**Actual**	**Predicted**	**Actual**
Optimizing factors 1–3									
CB % = 0 (KBS)	pH: 6	84.94	81.94	94.91	90.33	77.77	75.21	86.73	82.73
CB % =100 (KCB)	Temperature: 40 °C	88.13	86.20	96.33	92.92	79.71	77.73	84.85	85.69
CB %= 50 (KM)	Dose: 0.3 g	90.19	88.14	98.44	93.54	79.72	77.77	86.14	86.15
Maximized removal %									
CB% = 67.56	pH: 6	92.35	88.03	102.05	93.00	84.42	78.01	89.45	86.02
CB% = 14.94	Temperature: 40 °C	93.76	88.45	104.65	93.90	83.04	77.21	90.18	86.78
	Dose: 0.3 g								

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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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jece.2024.112836.

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