



Na-alginate coated waste banana-derived biochar composite for heavy metals removal and parametric optimization using RSM-CCD model

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Abstract

Wastewater treatment is a key research focus due to the ever-increasing water pollution, while the findings on novel material combinations are becoming challenging. The present research aims to develop alginate-modified biochar from the waste banana stems biochar and investigate parametric optimization using the response surface methodology model. Analytical characterization showed the coating of sodium alginate on the surface of biochar (Scanning electron microscope), and the presence of OH, COOH, and C=O functional groups can aid high metal adsorption (Fourier transform Infrared spectroscopy). Parametric optimization was studied (pH, contact time, and adsorbent dose) with an experimental design using CCD-RSM, the variation in ANOVA with a *p*-value (< 0.05) proved RSM model fit with obtained maximum removal efficiency at the optimum parametric condition as pH-6, contact time 60 min, and 0.5 g alginate combined biochar composite for multi-metals aqueous environment. Kinetics and isotherms studies best fit with (PSO) and the Freundlich. The removal efficiency of mixed metals from industrial wastewater was achieved as 87.75% for Cd, 90.11% Co, 82.99% Cr, and 90.60% Ni. The study implied that the parametric optimization through response surface methodology and alginate-modified biochar could be a potential approach for the treatment of heavy metal-contaminated wastewater.

Keywords Adsorption · Wastewater treatment · Alginate bio-composite · Heavy metals removal · RSM-CCD · Parametric optimization · Analytical characterization

Introduction

The availability of fresh water for drinking and domestic applications is one of society's most fundamental needs (Niju et al., 2023). Water pollution occurs due to the presence of one or more chemical substances that alter the

characteristics of pure water. Wastewater is released from various sources such as industrial wastes, urban development sewage, chemicals, fertilizers, mining activities, radioactive waste, and energy use (Crini and Lichtfouse 2019). According to a research study conducted by the United Nations Environment Program (UNEP) and the United Nations World Water Assessment Program (UNWWAP), 70% of industrial effluents and 80% of pollutants are discharged into surface and underground waters without prior treatment

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(Shaheen et al. 2019). According to the World Health Organization (WHO), water pollution has affected approximately 250 million individuals globally (Hussain et al. 2023). A significant volume of contaminated water from industries (metal plating, mineral extraction, tanning, textile, and pesticide production) comprises heavy metals released into freshwater (Jing et al. 2013). These non-degradable toxic metals become part of water bodies and harm the lungs, vascular system, central nervous system, bladder, esophagus, stomach, and skin even at small concentrations (Lee et al. 2019).

In the previous decades, the elimination of toxic substances from contaminated water got attention from researchers and the industrial sector; for this reason, numerous methods have been investigated, such as carbon-based adsorption, precipitation, electrochemistry, ion exchange, membrane filtration, evaporation, oxidation, flocculation, solvent extraction, biodegradation, and phytoremediation. It is challenging to standardize one particular method because each treatment has its advantages in terms of cost, efficiency, feasibility, and environmental impact according to the complexity of industrial effluents (Crini and Lichtfouse 2019; Hussain et al. 2023; Jing et al. 2013; Zhang et al. 2019) including some of the disadvantages like energy consumption, some by-product release, and discharge of dangerous particles into the environment (Singh et al. 2018). Adsorption is the ultimate wastewater treatment approach due to various characteristics such as low cost, ease of use, eco-friendliness, and homogeneity. The adsorption process can remove both soluble and insoluble contaminants in multi-contaminated and the presence of complex matrix in wastewater (Ali et al. 2012).

On the other side, the development of novel and efficient materials that play a key role in wastewater treatment is the aim of many researchers, and work is getting more attention day by day to enhance circularity and sustainability for effective material development and water reuse (Abdelbasir et al. 2020). According to the United Nations Environment Program, nearly 11.2 billion tons of solid waste are generated annually, and more than 90 percent of waste is dumped openly (UNEP 2020) (Sharma et al. 2023).

Food waste, including leftovers, is a worldwide issue that causes economic, environmental, and, most importantly, health concerns. However, sustainable management of food waste can enhance the circular economy and reduce the environmental impact (Yuan et al. 2023). Upcycling of widely available bio-waste can be utilized as a non-renewable source for the production of carbon material for water treatment as an emerging paradigm (Sousa et al. 2020). The most popular and cost-effective method for the adsorption process is the conversion of bio-waste into biochar through the pyrolysis method (Flores-Trujillo et al. 2021; Thines et al. 2017). Biochar is an activated carbon compound that can be synthesized by thermochemical treatment of waste

in the absence of oxygen (Xiang et al. 2020). Biochar comprises prominent characteristics, including a high cation exchange value, high carbon content, and excellent surface area, the presence of carboxylic, hydroxide, amino, ethyl, esters, and carbonyl functional groups that play a dominant role in the removal of water contaminants (Shakoor et al. 2021). Some studies have already reported on the development of solid adsorbents from fruit peels for the removal of heavy metals such as chromium and lead. Cadmium, Zinc, Copper, and Arsenic (Abd-Talib et al. 2020; Lin et al. 2017; Wattanakornsiri et al. 2022) while some disadvantages have limited the direct application of biochar as an adsorbent, such as lower recovery of biochar, small particle size, single composition, and less adsorption efficiency compared to modified composite (Wang et al. 2022).

The modification of biochar with biopolymers significantly enhanced the adsorption efficiency of the material for effective wastewater treatment, and developed more powerful and novel materials (Aftab et al. 2020; Hussain et al. 2023; Pavithra et al. 2021) With recent advancements, biopolymer modified composites have attracted the attention of researchers for wastewater treatment due to their high functional groups, biodegradability, high surface area, cost, and environment-friendly (Abdelhamid and Mathew 2021; Aftab et al. 2020; Ahmed et al. 2016; Alabaraoye et al. 2018). Alginate is composed of a linear straight chain and two corrosive mannuronic and guluronic groups and is usually used to remove contaminants in the form of calcium alginate (Fouda et al. 2021). Alginate as a biopolymer for adsorption study is preferable because of its biodegradability, easy availability, and low cost compared to chitosan, and high hydroxyl group, which increases the versatility of the composite for the removal of heavy metals (Esmat et al. 2017).

Many studies have previously been reported for the optimization of parametric conditions (pH, contact time, and adsorbent concentration) using a one-factor constant but these efforts are not very reliable for the industrial process referred to as one factor at a time (OFAT). For multi-metal contaminated wastewater required urgency for optimization of parametric condition using statistical tools to limit the time consumption and labour demand (Afolabi et al. 2021).

Due to the ever-increasing water pollution, the research on wastewater treatment has enhanced the scientific effort to solve this issue, while the findings of cost and environmentally-friendly processes and innovative materials from a pool of available sources are becoming challenging day by day. According to our best knowledge, the combination of alginate-modified banana stem biochar for multi-metal contaminated water with the CCD-RSM model has never been investigated before. The present research work aims to synthesize alginate-coated biochar composite, identify the optimal parametric conditions for efficient mixed metal



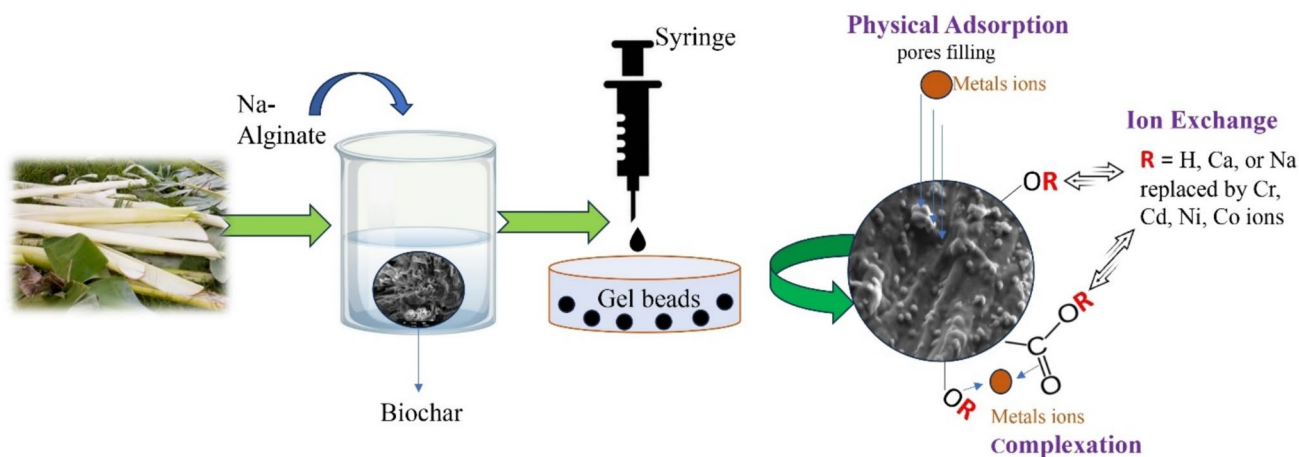


Fig. 1 Graphical illustration of the work

removal, and investigate the potential of alginate-modified composites on industrial wastewater, leveraging statistical tools to promote environmental sustainability and support a circular economy through the development of low-cost materials for enhanced water security.

Materials and methods

Chemicals and reagents

All the chemicals and reagents were of analytical grade (E. Merck, Darmstadt, Germany (Sigma-Aldrich) (Buch's, Switzerland)). The reagents and chemicals used in this work mainly are NaOH, HCl 37%, CaCl_2 , HNO_3 , and sodium alginate.

Synthesis of banana stem biochar

The overall synthesis route of the material is illustrated in Fig. 1. The waste banana stems were collected from the local market (Jhang Bazar, Faisalabad, Punjab-Pakistan). After collection, the stems were washed with tap water to remove impurities and then dried at 60 °C. To reduce the volume of dried stems were ground into powder using a conventional grinder to obtain the powder. The pyrolysis process was utilized to prepare the biochar of banana stems using an already-established method (Gokila et al. 2017). In detail, the 120 g of dried banana stem powder was pyrolyzed at 500 °C for 30 min with heating rates of 10 °C per minute using a muffle furnace. After thermal treatment, the obtained black powder was taken out and washed with distilled water

to remove impurities and neutralize the pH. Finally, the washed powder was dried in a drying oven at 60 °C and packed in a polythene bag to avoid moisture.

Synthesis of alginate combined biochar composite

Alginate combined biochar material was synthesized by following the previously established method (Ravulapalli and Ravindhranath 2018) Sodium-alginate 2 g was added to 100 ml deionized water and heated to 30–35 °C with continuous stirring (300 rpm) until a gel-like solution was obtained. Then 4 g of banana stem biochar was poured into sodium-alginate solution and stirred for 30 min to prepare a thick, sticky, and homogeneous black colour solution. Then, the homogeneous black material was filled in a 50 ml syringe and poured into 2%-calcium chloride (CaCl_2) solution dropwise to prepare beads and left overnight. After filtration, the synthesized beads were washed with deionized water twice and placed in the oven at 60 °C for drying. Finally, the dried granular beads are packed in tight polythene bags and placed in the desiccator.

Characterization

Synthesized biochar and alginate-biochar composite beads were characterized by Fourier Transfer Infrared (FTIR-Bruker) to check the different functional groups of the prepared composite material. Scanning Electron Microscopy (SEM) was used to examine the surface texture of the synthesized composite using (Tabletop SEM Cube Series, Em Crafts). Brunauer–Emmett–Teller (BET) analysis was performed using nitrogen adsorption/desorption for the

Table 1 Design of experiment for selected parameters and removal percentage of metals

Trial	pH	time (min)	Material (g)	% Removal (Cd)	% Removal (Cr)	% Removal (Ni)	%Removal (Co)
1	6.00000	60.0000	0.500000	87.484	83.924	82.196	93.461
2	6.00000	60.0000	0.500000	80.196	89.740	83.039	92.758
3	8.00000	80.0000	0.700000	90.546	78.160	90.341	87.188
4	8.00000	40.0000	0.700000	88.232	92.209	85.342	83.825
5	2.63641	60.0000	0.500000	79.779	89.781	69.466	90.090
6	4.00000	40.0000	0.700000	86.044	90.683	76.932	86.580
7	6.00000	60.0000	1.63641	82.575	80.174	63.201	84.426
8	4.00000	40.0000	0.300000	84.728	80.534	66.867	86.251
9	6.00000	60.0000	0.500000	86.097	88.160	76.818	92.746
10	9.36359	60.0000	0.500000	96.971	80.446	82.270	90.275
11	4.00000	80.0000	0.300000	84.346	86.692	70.518	86.406
12	6.00000	60.0000	0.500000	84.841	89.306	69.943	89.025
13	6.00000	60.0000	0.500000	82.435	83.840	70.524	87.602
14	6.00000	60.0000	0.500000	86.838	87.017	80.691	85.641
15	8.00000	80.0000	0.300000	80.827	86.043	80.001	84.833
16	6.00000	60.0000	0.836359	87.241	80.946	79.768	86.353
17	4.00000	80.0000	0.700000	83.717	74.777	80.388	85.245
18	6.00000	26.3641	0.500000	82.900	77.502	80.736	84.404
19	6.00000	93.6359	0.500000	84.865	89.678	61.012	88.526
20	8.00000	40.0000	0.300000	71.061	97.004	60.551	96.291

assessment of textural properties at 77 K using a Tristar II Plus Micromeritics instrument.

Adsorption experiments for parametric optimization

The adsorption experiment for the removal of metals from multi-metals containing water using the alginate combined biochar composites was accomplished by using the RSM model with central composite design CCD for three different parameters included e.g., pH ranges (2,4,6,8, and 10), adsorbent dose (0.1, 0.5, 0.7, and 0.9 g), and contact time (20, 40, 60, 80, and 120 min) Table 1. The samples were filtered before the adsorption experiment to remove undissolved contaminants. Firstly, 50 ml of metal-contaminated sample was taken in a 250 ml conical flask with the addition of the desired amount of adsorbent. The pH of the samples was adjusted using 0.1 M NaOH (sodium hydroxide) and 0.1 M HCl (hydrochloric acid) solution. The conical flasks were fixed into the incubator orbital shaker and fixed at 150 rpm for the desired period to initiate the water treatment procedure at room temperature. Finally, at the end of each adsorption experiment the samples were filtered to remove the adsorbent dose samples were digested by using 5 ml of HCl to characterize by ICP-AES (induced coupled plasma, Teledyne Leema Labs, Prodigy-7). The

removal efficiency of alginate combined biochar adsorbent was calculated by using the following formula.

$$\text{Removal\%} = \left(C_i - \frac{C_f}{C_f} \right) \times 100 \quad (1)$$

where C_i is the starting concentration of a metal ion in an aqueous solution While the C_f is the final amount of metal ions in the solution.

Results and discussion

Characterization

The functionality of the banana stem biochar and the biochar-alginate composite

Fourier transform infrared spectroscopy (FTIR) was used to identify the presence of functional groups in the composite materials. The FTIR analysis of banana stem biochar and alginate combined biochar was performed at 4000–600 cm^{-1} . The FTIR spectra of banana stem biochar are presented in Fig. 2a, which shows a broad peak for –OH between 3250 and 3350 cm^{-1} . The –OH stretching at 3400 cm^{-1} reflected the presence of the cellulosic compound in banana stem biochar that is liable for the attachment of metal ions (Castro et al. 2011). Moreover, FTIR

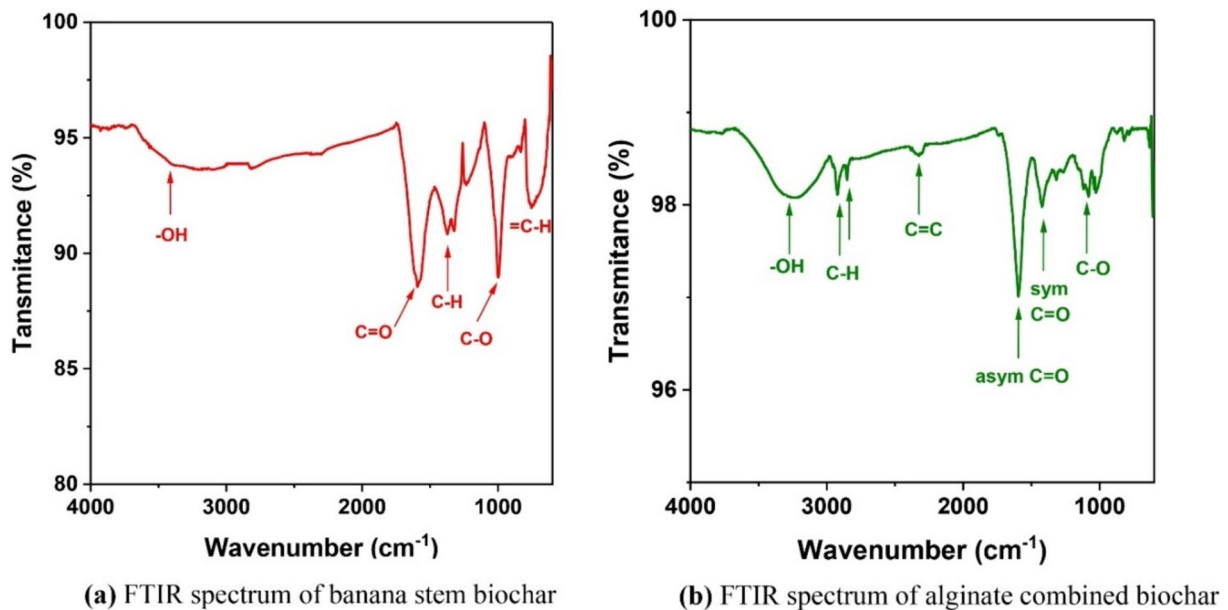


Fig. 2 FTIR analysis of biochar and alginate modified biochar composite

spectra proved the existence of carbonyl C=O stretching at 1720 cm^{-1} , and C–O stretching vibration at 1050 cm^{-1} .

The FTIR spectrum of alginate combined biochar composite showed a sharp peak at 2950 cm^{-1} (symmetric) and 2840 cm^{-1} (asymmetric) which proved the stretching of the C–H bond Fig. 2b. A strong peak was observed at $1580\text{--}1520\text{ cm}^{-1}$ for the presence of the C=O group which confirmed the presence of the –COOH functional group Fig. 2b. The peak observed at 1450 cm^{-1} was assigned to the asymmetric C=O group as shown in Fig. 2b aliphatic C–H stretching vibrations were seen around $2920\text{--}2850\text{ cm}^{-1}$ (Negroiu et al. 2021). Signals at 3374 cm^{-1} , which were linked with the stretching mode of hydroxyl groups, were found in both samples (Witek-Krowiak et al. 2014). In the literature, the bands at 2920 and 2951 cm^{-1} were assigned to C–H stretching that probably occurred due to Ca interaction with the polymeric chain (Lentz et al. 2022). The bands at 1704 or 1612 cm^{-1} indicated the C=O (C–O) symmetric stretching of carboxyl groups, demonstrating the presence of functional groups that include acidic oxygen and may be advantageous for the adsorption of metals (Wang et al. 2015). Due to the extensive presence of oxygen-containing functional groups –OH, –COONa, etc. on the alginate-modified biochar composite which showed hydrophilic characteristics that enhanced metal removal (Wang and Lu 2023) (Fig. 2b).

Surface morphology of banana stem biochar and alginate modified composite

Scanning electron microscopy (SEM) analysis was performed to examine the surface morphology of synthesized banana stem biochar and modified biochar with alginate at different magnifications. The SEM images of banana stem biochar present the open cell structure with a rough burst-like cavity which reveals the porous surface Fig. 3a–b. These open pores were generated during the rapid evaporation of organic compounds of banana stem powder that are responsible for the effective interaction of heavy metals on these active cavities. The biochar made from banana stems and leaf cavities contained comparatively smooth inner walls, creating a surface area for adsorption (Muhammad Bilal Shakoor, 2020).

The SEM images of the alginate combined biochar composite clearly showed the white coating of alginate on the biochar cavity. The white surface in the SEM images is due to an alginate coating on the biochar that is a highly permeable coating as well as tiny pores, which enhanced the active sites for the heavy metal adsorption process Fig. 3c–d. Similar grain morphology textures for alginate-modified composite were reported in the literature, which were visible in the prepared samples (Patel et al. 2021). Meikap and his colleagues prepared alginate



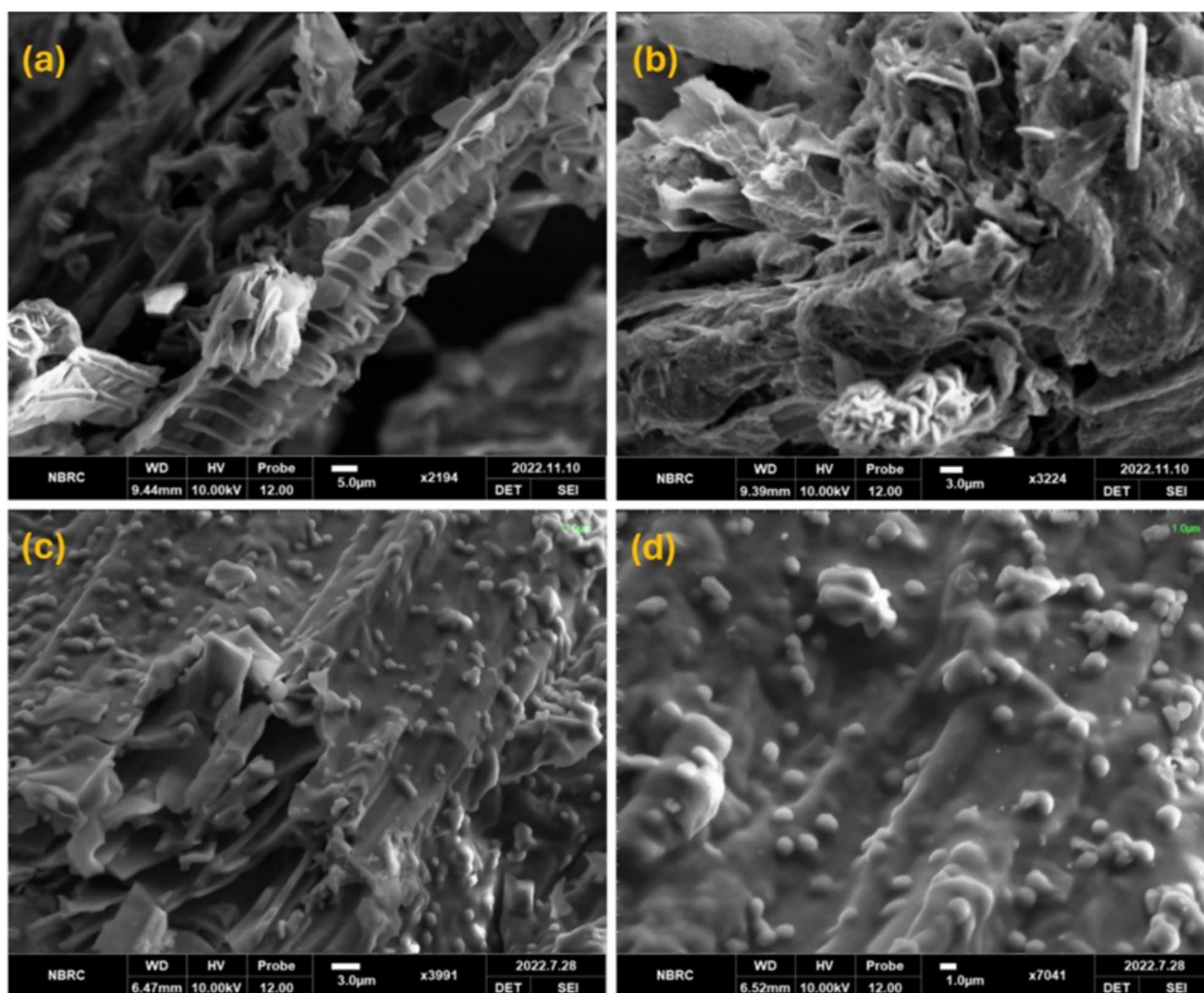


Fig. 3 a–b SEM images of Biochar, c–d SEM images of alginate modified biochar composite

mixed biochar for toxic metal ions elimination, and the surface morphology was a rough and porous structure that was changed due to the physical and chemical connection of metal ions (Subrata Biswas 2019). So the SEM results concluded the successful coating of alginate on porous biochar cavities for adsorption as well as highly active functional sites for the interaction of heavy metals present in the wastewater.

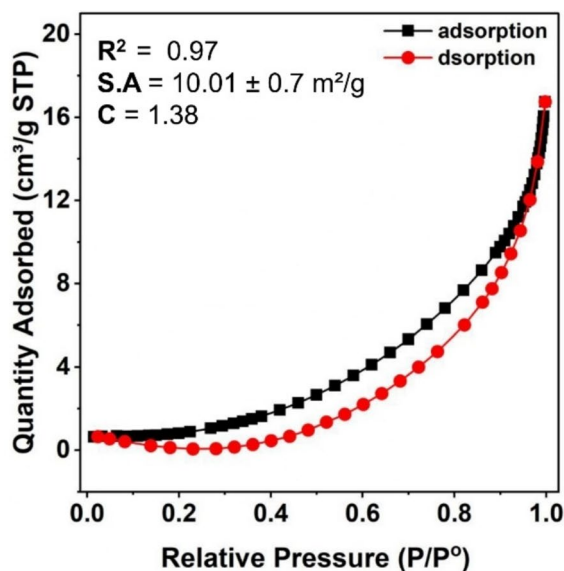
BET analysis of banana stem biochar and alginate modified composite

The surface textural property was evaluated using BET analysis, which was performed using N_2 adsorption/desorption at 77 K (the samples were degassed overnight at 373 K to remove residual moisture before analysis). The BET specific surface areas were calculated from adsorption data in the relative pressure (P/P_0). BET analysis revealed

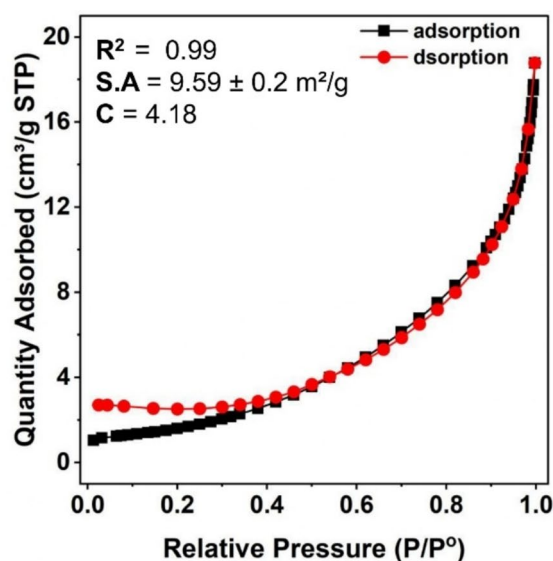
significant structural and functional modifications imparted by alginate coating on biochar. Figure 4 shows that the surface area of alginate-coated biochar slightly decreased compared to uncoated biochar, suggesting partial pore or surface coverage by the polymer matrix. On the other hand, the BET constant (C) increased markedly from 1.38 to 4.18, demonstrating stronger interactions between the adsorbate and the alginate-modified surface, which is likely due to the introduction of oxygen-containing functional groups (e.g., hydroxyl and carboxyl) on the surface. Regardless of surface area, the alginate combined biochar exhibits a substantially improved BET model fit ($R^2 = 0.99$), reflecting a more uniform and possibly stabilized surface structure.

RSM model significance for metal adsorption

The operation of the adsorption process is based on the proper optimization of some parameters such as pH,



(a) BET analysis of banana stem biochar



(b) BET analysis of alginate combined biochar

Fig. 4 BET analysis of biochar and alginate-modified biochar composite

adsorbent dose, and contact time. For such a purpose, some efforts are reported in the literature using conventional methods to optimize the parametric condition, but these cause high time consumption and labour-intensive process. In this regard, a new statistical model has been implemented for parametric optimization. Response surface methodology (RSM) is a statistical model based on three common types of design (Box–Behnken design (BBD), central composite design (CCD), and Doehlert design) that helps in reducing experiment time, reaction cost, and variability with upgraded reaction output (Zhang et al. 2016). According to the literature, Central Composite Design (CCD) is commonly used for optimizing parametric conditions, as it effectively predicts both linear and quadratic interaction effects of selected factors (Karimifard and Alavi Moghaddam 2018).

The experiment includes three factors: pH, time (minutes), and material (g). A popular response surface design called Central Composite Design (CCD) presented by Box and Wilson (1951) was chosen to run this experiment. This design has the most desirable statistical properties that can estimate a second-order model with satisfactory answers to the questions usually raised in the mind of experimenters, like individual effects of the variables on the response, their quadratic effects, and interaction effects, etc.

The second-order response surface model for three-factor experiment is given as:

$$y(x) = \theta_0 + \sum_{i=1}^3 \theta_i x_i + \sum_{i=1}^3 \theta_{ii} x_{ii} + \sum_{i=1}^2 \sum_{j=2}^3 \theta_{ij} x_i x_j \quad (2)$$

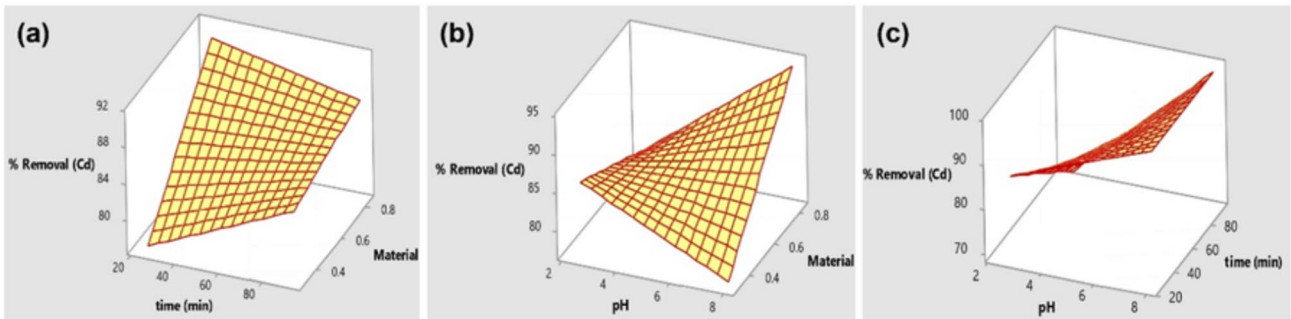
where $y(x)$ is the expected response, θ_0 represents the intercept (the quantity of response without involvement of three considered variables), $\theta(i)$ is the contribution of i th variable for the response, $\theta(ii)$ is the contribution of i th variable in the quadratic form, $\theta(ij)$ is the contribution of i th and j th variable as an interaction and x_i and x_j denote i th and j th variable.

The experiment comprises twenty trials following the CCD of three factors, Table 1. The observed responses are % removal of Cd, Cr, Ni, and Co. Table 1 obtained from twenty experiments, was investigated for multi-metal contaminated wastewater treatment using Na-alginate modified biochar composite, and results were interpreted with analysis of variance (ANOVA). Focusing on the obtained removal efficiency of alginate combined biochar composite for Cd, Ni, Co, and Cr it is observed that the satisfactory removal efficiency obtained in low acidic medium pH 6 in comparison to high acidic medium and alkaline conditions, while in terms of adsorbent dose, the significant results were obtained using 0.5 g with more repeated optimal contact time as 60 min Table 1. The removal efficiency of Cd and Cr in the RSM experimental design ranges from 71.061 to 88.232 and 74.777 to 97.004% respectively. The removal percentages of Ni and Co range from 60.551 to 90.341 and 83.825 to 96.291%, respectively. Due to the competition between metal ions and protons at highly acidic conditions, the removal efficiency is reduced during the adsorption of multiple metals in the same aqueous environment on alginate combined biochar material due to blockage at the active site of the composite (Chi et al. 2017).

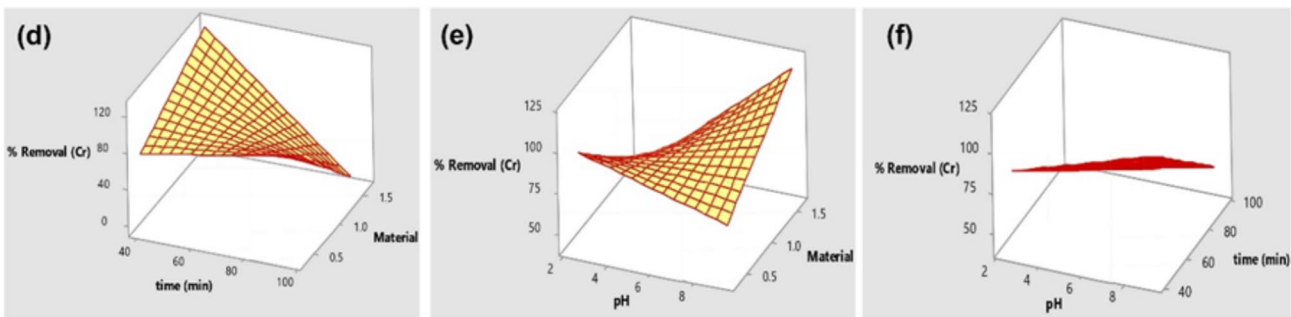
Among the twenty experiments designed by CCD, the obtained results showed variation in removal efficiency for each different metal using the same alginate combined biochar composite. These variations in the removal efficiency in a set of twenty experiments indicate the effect of each parameter in the presence of the other two parameters. The RSM model is considered the best fit if the p -value is lower than ($p < 0.05$), and the R^2 value should be close to

1 to be considered the overall model performance (Zhang et al. 2016). The resulting values obtained for the removal efficiency for twenty designed experiments were analyzed, indicating a p -value less than 0.05 which represents the best model fit with a significant quadratic effect. The 3-D graph describes the factor dependence in the presence of the remaining two factors in Figs. 5 and 6. The regression equation for the elimination of Cd, Cr, Ni, and Co is shown below with each model summary that shows the R^2 , R^2 (adj), and lack of fit p -value;

$$\begin{aligned} \%Removal(Cd) = & 106.0 - 6.94pH - 0.084time(min) \\ & - 16.0Material(g)0.0462pH * time(min) \\ & + 8.19pH * Material(g) - 0.294time(min) * Material(g) \end{aligned} \tag{3}$$

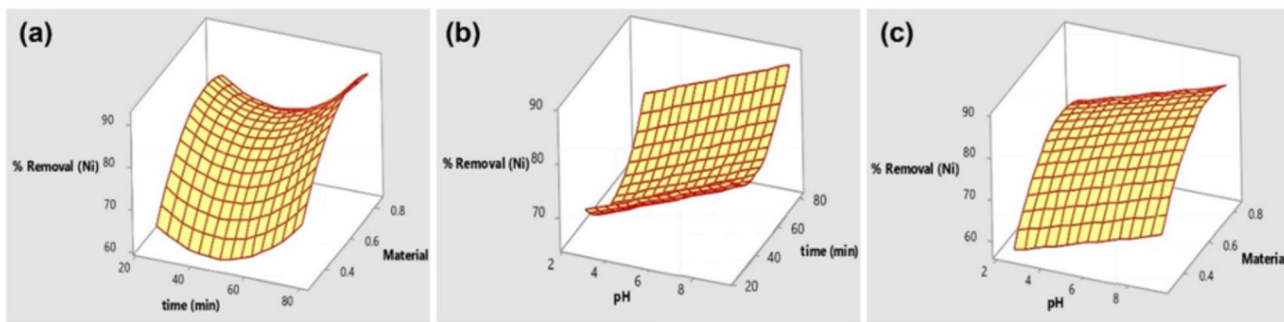


The effect of pH, contact time, and adsorbent dose on Cd metal adsorption (a) removal % of Cd vs adsorbent dose vs contact time hold pH 6 (b) removal % of Cd vs adsorbent dose vs pH holds value contact time 60 minutes (c) removal % of Cd vs contact time & pH holds adsorbent dose.

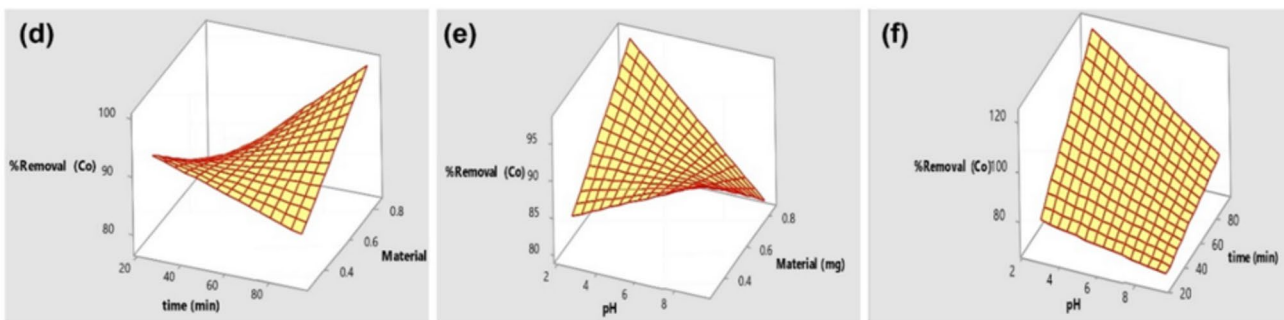


Effect of pH, contact time and dose for Cr metal adsorption (d) removal % of Cr vs adsorbent dose vs contact time hold pH 6 (e) removal % of Cr vs contact time & pH holds adsorbent dose (f) removal % of Cr vs adsorbent dose vs pH holds value contact time 60 minutes

Fig. 5 3D graphs of CCD-RSM analysis of Cd and Cr



Effect of pH, contact time and dose for Ni metal adsorption (a) removal % of Ni vs adsorbent dose vs contact time hold pH 6 (b) removal % of Ni vs contact time & pH holds adsorbent dose (c) removal % of Ni vs adsorbent dose vs pH holds value contact time 60 minutes



Effect of pH, contact time, and dose for Co metal adsorption (d) removal of Co vs adsorbent dose vs contact time hold pH 6 (e) removal of Co vs adsorbent dose vs pH holds value contact time 60 minutes (f) removal of Co vs contact time & pH holds adsorbent dose

Fig. 6 3D graphs of CCD-RSM analysis of Ni and Co

Model Summary

Coefficient of Determination: $R^2 = 77.60\%$,
 $R^2(\text{adj}) = 65.38\%$

Lack-of-fit: $p\text{-value} = 0.694$ (insignificant)

Lack-of-fit: $p\text{-value} = 0.387$ (insignificant)

$$\begin{aligned} \% \text{Removal}(\text{Ni}) = & 38.7 + 1.589pH - 1.070\text{time}(\text{min}) \\ & + 162.2\text{Material}(\text{g}) \cdot 0.01133\text{time}(\text{min}) * \text{time}(\text{min}) \\ & - 116.7\text{Material}(\text{g}) * \text{Material}(\text{g}) \end{aligned} \tag{5}$$

$$\% \text{Removal}(\text{Cr}) = 51.2 - 6.14pH + 1.340\text{time}(\text{min})$$

$$+ 62.6\text{Material}(\text{g}) - 0.0189pH * \text{time}(\text{min}) \tag{4}$$

$$+ 11.43pH * \text{Material}(\text{g}) - 2.270\text{time}(\text{min}) * \text{Material}(\text{g})$$

Model Summary

Coefficient of Determination: $R^2 = 81.48\%$,
 $R^2(\text{adj}) = 70.36\%$

Model Summary

Coefficient of Determination: $R^2 = 72.57\%$,
 $R^2(\text{adj}) = 58.86\%$

Lack-of-fit: $p\text{-value} = 0.852$ (insignificant)

$$\% \text{Removal}(\text{Co}) = 55.2 + 6.76pH + 0.344\text{time}(\text{min}) - 15.8\text{Material}(\text{mg})$$

$$- 0.00291\text{time}(\text{min}) * \text{time}(\text{min}) - 23.8\text{Material}(\text{mg}) * \text{Material}(\text{mg}) \tag{6}$$

$$- 0.0595pH * \text{time}(\text{min}) - 6.69pH * \text{Material}(\text{mg})$$

$$+ 0.795\text{time}(\text{min}) * \text{Material}(\text{mg})$$

Model Summary

Coefficient of Determination: $R^2 = 70.09\%$,
 $R^2(\text{adj}) = 43.50\%$

Lack-of-fit: $p\text{-value} = 0.911$ (insignificant).

Effect of pH on heavy metal adsorption

For heavy metal adsorption, pH has a key role in effective wastewater treatment because the optimal pH not only boosts the adsorption process efficiency but also helps to understand the behavior of each metal adsorption at a specific pH level (Vo et al. 2020). The pH optimization in the medium levels 5–7 is very crucial due to the adsorbent material nature and adsorbate heavy metal behavior to operate the wastewater treatment process effectively. Removal of heavy metals in highly acidic environments has a decreasing trend due to the protonation of the adsorption site, which lowers the active site of the alginate combined biochar composite. Similarly, at higher alkaline pH levels, the metals precipitated and reduced the role of the adsorbent used for wastewater treatment (Zhang et al. 2016). In the alkaline pH environment, the removal efficiency of Ni and Cd was noted to be lower 60.55% and 71.061% compared to slightly acidic pH-6 with a removal percentage of 82.19% and 87.48% achieved. The impact of competitive adsorption of various metals, time, and adsorbent dose of adsorbent was also responsible for the pH optimization (Xue et al. 2012). At low pH Cr (VI) exists as CrO_7^{-2} and CrO_4^{-2} and increased adsorption at low pH was caused by greater protonation, which occurred at pH 2.63 due to higher charge transfer at the surface of the adsorbent (Zhang et al. 2015). At lower acidic pH levels, hydrogen ions were eliminated, and the surface became active. Therefore, the adsorption of metal ions becomes attractive through electrostatic attraction. Similarly, in the literature, the optimal pH for Ni adsorption was noted in an acidic medium at pH 6 and the highest removal rate was 98.93% (Zamora-Ledezma et al. 2021). For the synthesized alginate combined biochar composite for the adsorption process, pH-6 was selected as the optimum pH level for the competitive adsorption of different heavy metals (Cr, Cd, Ni, Co) according to the satisfactory RSM model fit ($p < 0.05$) and obtained removal efficiency.

Effect of adsorbent dose

Generally, increasing the adsorbent dose leads to an increase in removal efficiency for the adsorption process, but until a certain amount is enough to obtain results. For this reason, the optimization of adsorbent material is required to make sustainable use of the desired material for the adsorption process (Zhang et al. 2016). In the present investigation, the set of designed twenty experiments provided satisfactory results 0.5 g

of alginate combined biochar composite is enough to achieve maximum removal efficiency in a multi-metal environment. RSM model results indicated the effect of adsorbent dose in the variation of pH and contact time, and ANOVA results $p < 0.05$ revealed the significant model fit. For each investigated metal under competitive adsorption the Cr, Cd, Ni, and Co the maximum efficiency of nearly 90% was achieved at 0.5 g among the set of twenty experiments under the influence of pH and contact time. So the obtained results recommended that 0.5 g of alginate biochar combined composite is enough to achieve maximum removal efficiency of 90% in a multi-metals competitive environment Table 1.

Effect of contact time

During the adsorption course, the contact time plays a significant role in the removal of heavy metals. Optimization of the contact time between the adsorbent and adsorbate is a key player to make the process time effective. A defined period is required for the adsorption of heavy metals on the surface of the used adsorbent until all the active sites are fully saturated (Ugwu et al. 2020). In the present study, the RSM model described the optimum contact time of 60 min for the multi-metal adsorption on synthesized alginate biochar composite material. Similar results for the contact time were reported for the mixed metal competitive environment on chitosan combined biochar composite (Hussain et al. 2023). The obtained results and model fit ($p < 0.05$) indicate that a contact time of one hour is sufficient to achieve maximum removal efficiency. The higher removal efficiency was reported for the Cr, Cd, Ni, and Co at the contact time of 60 min under slightly acidic conditions pH 6, with the use of 0.5 g of adsorbent.

Adsorption isotherm

Adsorption isotherm studies provide useful insight into understanding the adsorption behaviour. For this reason, Langmuir isotherm (Eq. 7) and Freundlich isotherm (Eq. 8) were studied to understand the interaction of adsorbate and adsorbent, the surface property of adsorbent, which helps to understand the mechanism for heavy metal removal.

$$\frac{c_e}{q_e} = \frac{1}{bQ_{\max}} + \frac{c_e}{Q_{\max}} \quad (7)$$

where C_e (mg/L) is the equilibrium concentration of adsorbate, amount of adsorbate (mg/g), Q_{\max} is the maximum adsorption capacity (mg/g), and b (L/mg) is the Langmuir constant.

Fig. 7 a Langmuir b Freundlich adsorption isotherm and c pseudo-first-order d pseudo-second-order model fitting of alginate combined biochar

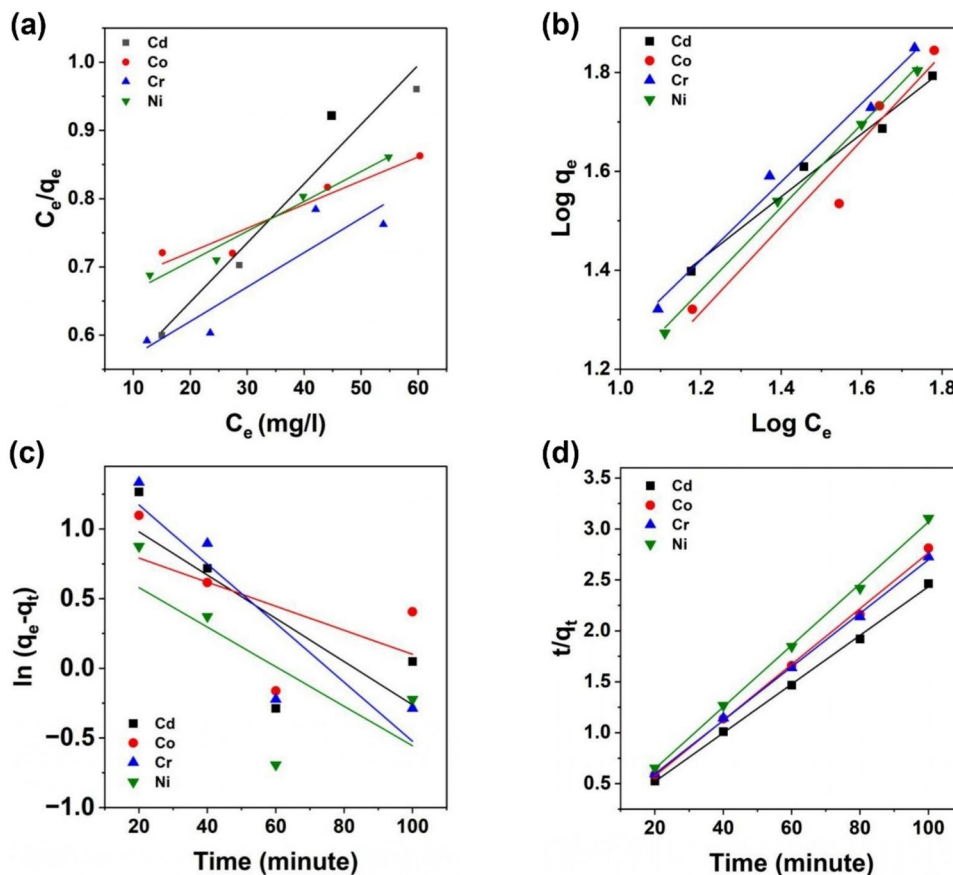


Table 2 Isotherm and kinetic parameters for the removal of Cd, Co, Cr and Ni

Parameters	Cd	Co	Cr	Ni
Langmuir				
R ²	0.94	0.92	0.84	0.97
K _L (L/mg)	0.0041	0.0022	0.0026	0.0027
R ^L	1.26	1.14	1.17	1.17
q _{max} (mg/g)	115.22	286.98	198.34	228.35
Freundlich				
R ²	0.97	0.91	0.97	0.99
1/n	0.6352	0.8698	0.7925	0.8411
K _f	4.565	1.867	2.947	2.237
PFO				
R ²	0.58	0.31	0.79	0.50
q _e (mg/g)	3.63	2.62	4.93	2.37
K ₁	-7.76E ⁻⁴	-2.16E ⁻⁴	-3.53E ⁻⁴	-1.77E ⁻⁴
PSO				
R ²	0.99	0.99	0.99	0.99
q _e (mg/g)	41.78	36.53	38.06	33.04
K ₂	0.013	0.026	0.009	0.021

$$\log q_e = \log k_f + \frac{1}{n} \log c_e \tag{8}$$

q_e is the amount of adsorbate (mg/g), C_e is the equilibrium concentration of adsorbate (mg/L), K_f is the Freundlich’s constant, and 1/n is the adsorption intensity constant (favorable (0.1 < 1/n > 1 or unfavorable 1/n > 2).

The equilibrium adsorption data of Langmuir and the Freundlich isotherm model fit for (Cd, Co, Cr, and Ni) are shown in Fig. 7a–b, which exhibits a strong correlation R², see Table 2. The results indicate the involvement of both monolayer and multilayer adsorption processes. The Langmuir isotherms show excellent adsorption capacity (Q_{max}: Co > Ni > Cr > Cd), which suggests a strong interaction of the adsorbate with the uniform functional groups of the alginate coating. The Freundlich model, which accounts for surface heterogeneity, presents an even better fit overall (R² > 0.97 for Cd, Co, and Ni), and for Cr (R² = 0.97) compared to Langmuir (R² = 0.84) indicating the heterogeneous surface adsorption on energetically diverse sites. These observation confirms the dominant Freundlich model fit, and the obtained results trend aligns with the principles of the Freundlich model. Furthermore, the Freundlich constant K_f and the adsorption intensity 1/n < 1 confirm favorable adsorption. The high values of Q_{max} and K_f indicate the

adsorbent's strong potential for practical application in treating complex industrial effluents containing multiple heavy metals.

To assess the adsorption behavior of Cd, Co, Cr, and Ni onto sodium alginate-coated biochar, the kinetic study is evaluated utilizing two versatile kinetic models, the pseudo-first-order (PFO) and pseudo-second-order (PSO).

Pseudo 1st order

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (9)$$

where, q_t corresponds to the adsorption capacity (mg/g) at time t while K_1 (min^{-1}) is the equilibrium rate constant.

pseudo 2nd order

$$\frac{t}{q_e} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad (10)$$

where K_2 ($\text{g mg}^{-1} \text{min}^{-1}$) is the equilibrium rate constant and q_e is the adsorption capacity (mg/g).

The outcomes of both investigated models help to understand whether the adsorption follows a physisorption or chemisorption mechanism, which involves the electron sharing or exchange between adsorbent and metal ions. The Fig. 7c–d shows that the PSO model presented an excellent model fit for all tested metal ions, with R^2 values exceeding 0.99 for all cases, see Table 2. Compared to the PFO model, which exhibits much lower correlation coefficients (R^2 ranging from 0.31 to 0.79), indicating poor agreement. These findings suggest the adsorption process is primarily governed by chemisorption that involves valence forces or electron exchange between the metal ions and the functional groups of alginate present on the composite material, which governs the chemically controlled mechanism likely

influenced by the complexation and ion-exchange capabilities of the composite.

The excellent fit of the PSO compared to PFO further supports the isotherm studies where the Freundlich isotherm is dominant, which reinforces the notion that adsorption onto alginate-coated biochar occurs on a heterogeneous surface via chemisorption. The obtained results present alignment between the Freundlich model and the pseudo-second-order kinetics, indicating multilayer adsorption likely influenced by the complex surface chemistry of the composite adsorbent. During the adsorption process, the removal mechanism of heavy metals greatly depends on the surface charge of the adsorbent, and pH has a significant role in this context. In previous studies, under highly acidic conditions, the removal efficiency was achieved at very low levels due to the highly protonated adsorbent surface. In the higher alkaline environment, the metal ions are precipitated, reducing the actual role of the adsorbent, which challenges the separation of separated ions (Ugwu et al. 2020). Similar precipitation effects were also reported in the present study for the removal of Cd and Ni at higher alkaline environments but the precipitation mechanism was not considered in the count due to CCD-RSM optimum results showing most of higher removal efficiency in slightly acidic conditions that lead to reduction in hydronium ion formation and more heavy metal attachment to alginate combined biochar composite. The present investigation suggested the ion exchange and complexation mechanism for the investigated metals on alginate combined biochar composite due to a slightly acidic environment and the presence of the lone pair of oxygen atoms from the hydroxyl group present on the carbon chain of alginate polymer Fig. 8a. Gendy and his colleague reported the ion exchange mechanism is dominant

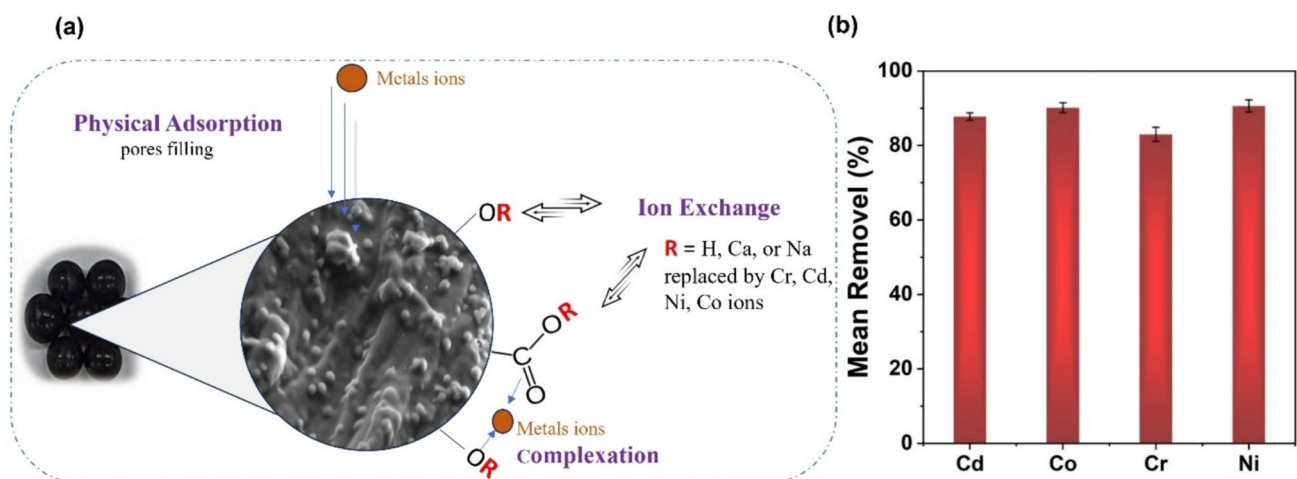


Fig. 8 a Adsorption mechanism of heavy metals removal on alginate-modified biochar composite b Removal efficiency of metal removal from wastewater

Table 3 Comparison of the removal efficiency of various adsorbents used for the removal of heavy metals

Adsorbent	Removal % (Cd)	Removal % (Co)	Removal % (Cr)	Removal % (Ni)	References
Schist/alginate composite	56.99	–	–	51.3	Allahkarami et al. (2023)
Jute/Polyacrylic Acid (PAA) Gel	79.3	–	96.2	–	Zhou et al. (2018)
Polyamine-functionalised silica	70	–	–	–	Althoman et al. (2010)
Natural clay	–	85.5	–	84	Es-Sahbany et al. (2021)
Modified-chitosan	–	26.6–53.48	–	–	Negm et al. (2015)
Succinic anhydride modified apple pomace	70	–	–	–	Chand et al. (2014)
Posidonia oceanica (biomass)	85	–	–	88	Boulaiche et al. (2019)
Lumbago zeylanica	–	–	80	61	Roy et al. (2021)
Sulphurised activated carbon (SAC)	–	81	–	–	Krishnan et al. (2008)
Surfactant-modified alumina	–	–	–	64	Khobragade and Pal (2015)
AC-Cucumis Melo peel	72.4	–	81.62	81.62	Manjuladevi et al. (2017)
Chitosan/magnetite composite	–	–	–	75.5	Tran et al. (2010)
HNT-BC@Alginate	55–92.5	–	–	50.6–80.85	Hassan et al. (2021)
Alginate combine biochar	87.75	90.11	82.99	90.60	Present study

in low acidic pH due to the presence of the lone pair of oxygen atoms from the hydroxyl group (Gendy et al. 2021). Another study recently reported on the chitosan composite has reported the heavy metal adsorption mechanism due to the excessive presence of hydroxyl group on the polymer chain and similar pH-6 optimization using the RSM model (Hussain et al. 2023) proved that the alginate combined biochar composite has greater potential to provide high removal efficiency due to highly functional surface.

Adsorption experiment of industrial wastewater

The treatment of real industrial wastewater presents certain challenges for heavy metal removal due to its complex matrix and multi-metal environment that requires the adsorbent to comprise high functionality that offers enough active sites for heavy metal binding. To understand the behaviour of the synthesized alginate composite was applied on real industrial wastewater (collected from the tannery industry and electroplating) using the CCD-RSM provided optimum condition (pH-6, contact time 60 min, and adsorbent dose 0.5 g). The industrial wastewater from different sources (tannery, electroplating, and battery) was collected, the experiment was performed three times, and the results are demonstrated in Fig. 8b with error bars. The removal efficiencies achieved were 87.75% for Cd, 90.11% for Co, 82.99% for

Cr, and 90.60% for Ni. The detailed comparison of the use of various adsorbents for heavy metal removal percentage is given in Table 3. These results highlight the strong metal-binding potential of the alginate combined composite, which facilitates selective and efficient metal uptake under realistic wastewater conditions. This emphasizes the ability of alginate-coated biochar as a sustainable and effective adsorbent for industrial wastewater remediation.

Conclusion

Due to ever-increasing water pollution various types of technologies have been invented for effective wastewater treatment, among the other available techniques (coagulation, precipitation, reverse osmosis, thermal water treatment), the adsorption process is still preferable in terms of low cost and eco-friendly nature with greater efficiency. The development of cost-effective and sustainable materials is another challenge for effective wastewater treatment towards the Sustainable Development Goals. In the present research work, the biochar of banana stems was modified with sodium alginate, and the removal efficiency of heavy metals was investigated with the RSM model for parametric optimization. The RSM model showed the best fit with $p < 0.05$, and the optimum parametric conditions are a low

acidic environment pH-6, with 0.5 g of alginate combined biochar composite with an adsorption course of 60 min. The composite investigated industrial wastewater and the removal efficiency for Cd, Co, Cr, and Ni was achieved as 87.75%, 90.11%, 82.99%, and 90.60%. Therefore, based on the findings of this study, alginate-modified biochar has been successfully developed for the removal of multi-metal-contaminated wastewater. Furthermore, the development of biopolymer-modified biochar will open a new era for the development of sustainable materials for wastewater treatment to enable circular economy practices.

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Data availability All data analyzed during this research work is included in this article.

Declarations

Conflict of interest There is no conflict of interest between authors regarding the publication of the present study.

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