



Research Article

Efficient Cr⁶⁺ Removal with Activated Bio-Sorbent: Process Optimization

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Abstract

This study investigated the biosorption of Cr⁶⁺ from aqueous solution using a gelling derived activated bio-sorbent, with process conditions optimized through a Box–Behnken response surface design. The exhausted sorbent was characterized by SEM–EDX and FTIR to elucidate changes in surface morphology, elemental composition, and functional groups involved in chromium binding. A systematic series of experiments was performed to evaluate the main and interactive effects of pH, sorbent dosage, and contact time at a fixed initial Cr⁶⁺ concentration and ambient temperature on removal efficiency. Under statistically optimized conditions at neutral pH, the activated bio-sorbent exhibited a maximum uptake capacity of 92.34 mg g⁻¹, underscoring its strong affinity for hexavalent chromium and its promise for practical wastewater treatment. The Box–Behnken design provided a robust quantitative framework for model development, significance testing, and prediction of process behavior across the investigated operating window. Analysis of variance yielded an F-value of 10.67 and a p-value of 0.0025, confirming that the quadratic model was statistically significant and suitable for describing Cr⁶⁺ biosorption performance. Overall, the findings demonstrate the feasibility of employing this low-cost, gelling-based bio-sorbent as an efficient alternative to conventional adsorbents for chromium remediation. Future work should focus on regeneration and reuse, sorption–desorption cycling, and pilot-scale implementation, as well as integration with existing treatment trains to enhance process sustainability and support broader deployment in industrial effluent management systems. Attention to kinetic and equilibrium behavior, potential competing ions, and real effluent matrices will further clarify operational limits and guide translation from laboratory conditions to field practice successfully.

Keywords

Optimization, Cr⁶⁺ Ion, Box-Behnken Design, Characterization

1. Introduction

The advancement of contemporary civilization has significantly augmented the presence of pollutants in aquatic systems. These contaminants encompass harmful chemicals, non-degradable substances, greenhouse gases, chlorofluorocarbons

(CFCs), and various toxic metal ions. Numerous scientists have devised strategies to mitigate the emissions of these pollutants at their source. Chromium ions, extensively utilized in various

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industrial processes, are discharged into water bodies in substantial quantities. In polluted water, chromium ions manifest as both Cr(III) and Cr(VI) species. At pH levels below 7, $\text{Cr}_2\text{O}_7^{2-}$ is the predominant species in wastewater, whereas CrO_4^{2-} is more prevalent at pH levels above 6.5 [1-3]. Prolonged exposure to Cr ions can lead to severe health issues in humans. In the context of drinking water purity, the United States Environmental Protection Agency (USEPA) has established a precise limit, ensuring that the concentration of hexavalent chromium ions remains below the critical threshold of 0.5 ppm, thereby safeguarding public health with meticulous care [4, 5]. The biosorption of Cr^{6+} ions using various techniques in chromium contaminated solutions has been well documented. However, these techniques often face challenges, such as high operational costs, limited pH range, and incomplete removal of toxic metal ions, expensive reagents, and high energy requirements. Researchers have developed environmentally friendly adsorbents from plant biomass, including carbohydrate polymers, cellulose, and hemicellulose, all of which are closely associated with lignin and organic chemicals [6, 7]. Mucilage, a gel-like substance, is employed as an ideal bio-sorbent precursor due to its biodegradability, high tolerance, absence of harmful effects on living organisms, and low preparation costs. The utilization of a novel bio-sorbent derived from *Euphorbia resinifera* stem mucilage for the biosorption of Cr^{6+} ions from water has not been previously reported. This plant is recognized for its anti-inflammatory, drought-resistant, antiseptic, succulent, shrubby, and antibiotic properties. The utilization of *Euphorbia resinifera* mucilage-based activated bio-composites for the removal of Cr^{6+} ions represents a significant advancement in bio-adsorbent technology [8, 9]. This innovation is characterized by the unique integration of natural polymeric mucilage with activated composite materials. Unlike previously documented bio-adsorbents, this novel bio-composite leverages the high binding affinity and functional groups inherent in *Euphorbia resinifera* mucilage, thereby enhancing both the adsorption capacity and selectivity for Cr^{6+} ions. Additionally, the activation process markedly increases the surface area and porosity, facilitating improved ion diffusion and interaction. This comprehensive approach offers superior efficiency and sustainability, providing a cost-effective and environmentally benign alternative to conventional adsorbents, effectively addressing limitations such as limited adsorption capacity and regeneration difficulties observed in earlier materials. Despite extensive research on bio-sorbents for Cr^{6+} ion removal, a notable gap exists in exploring mucilage-based bio-composites, particularly those derived from *Euphorbia resinifera* mucilage. Unlike conventional bio-sorbents, mucilage-based bio-composites offer several advantages, including enhanced biocompatibility, renewable and abundant natural sources, and unique physicochemical properties such as high viscosity and gel-forming ability, which improve adsorption efficiency and stability [9, 10]. The incorporation of *Euphorbia resinifera* mucilage into activated bio-composites can provide superior surface functional groups and structural integrity, facilitating higher removal capacities and

better regeneration potential under mild conditions. This approach addresses the limitations of traditional bio-sorbents, such as lower mechanical strength and limited reusability, thereby presenting a promising, sustainable alternative for efficient Cr^{6+} biosorption. Focusing on these mucilage-based composites could advance the development of eco-friendly, cost-effective solutions with improved performance metrics for heavy metal remediation in aqueous environments. Environmentally, it provides a sustainable and biodegradable alternative to conventional synthetic adsorbents, reducing reliance on non-renewable materials and minimizing secondary pollution associated with chemical treatments. The plant-based mucilage is naturally abundant and renewable, promoting eco-friendly waste management practices. Practically, this bio-sorbent demonstrates high efficiency in adsorbing diverse contaminants from complex wastewater matrices, including heavy metals and organic pollutants, which enhance treatment performance. Its ease of extraction and low production cost make it economically viable for large-scale applications [11]. Various researchers have employed response surface methodology optimization techniques to determine process parameters. The Box–Behnken design (BBD) is a systematic and efficient method for exploring the relationship between multiple input factors and key output responses. It has been widely applied by researchers to analyze the interaction effects of factors on responses, understand response behaviour as a function of these factors, and optimize responses. This study primarily aimed to utilize the Box–Behnken Design (BBD) model to refine the process parameters for the accurate forecasting of Cr^{6+} ion biosorption from aqueous solutions [12-14]. The predictive accuracy of the model was validated statistically using R^2 , RMSE, and F-test values to assess the model fitness.

2. Materials and Methods

2.1. Mucilage- Based Bio-composite Preparation and Characterization of Samples

The pads of *Euphorbia resinifera* (Order: Malpighiales, Family: Euphorbiaceae) were sourced from the Rourkela forest division in Sundergarh District, Odisha. Initially, these pads underwent a process of washing, drying, and weighing. Subsequently, they were longitudinally divided into two sections. The white fleshy parenchyma cells within the pads were carefully extracted using a spoon and then processed through a potato press to rupture the mucilaginous cells, thereby releasing the mucilage. This mucilage was left to dry naturally for a duration of 48 hours. After this drying period, the mucilage was subjected to oven drying to transform it into a powdered form. Figure 1 illustrates a schematic representation of the synthesizing a mucilage-based bio-composite, utilizing $\text{C}_6\text{H}_9\text{NaO}_7$ with CaCl_2 as a dopant. The physicochemical pa-

rameters of the gelling based activated bio-composites are represented in Table 1. The bio-composite prepared from *Euphorbia resinifera*, utilizing sodium alginate ($C_6H_9NaO_7$) with $CaCl_2$ as a dopant, exhibits distinct chemical composition and structural properties driven by crosslinking and modification mechanisms. Sodium alginate acts as a chelating agent, facilitating the coordination of Ca^{2+} ions from $CaCl_2$ with functional groups present in the *Euphorbia resinifera* matrix, such as hydroxyl and carboxyl groups. This interaction promotes the formation of ionic crosslinks between the polymer chains, enhancing the structural integrity and stability of the bio-composite. The Ca^{2+} ions serve as crosslinking bridges, reducing

polymer chain mobility and resulting in a denser network structure. Such crosslinking modifies the physicochemical properties by increasing mechanical strength and thermal stability, while potentially influencing porosity and adsorption capacity. The structural modifications can be further characterized by spectroscopic techniques (e.g., FTIR) to confirm shifts in characteristic peaks corresponding to these interactions. Overall, the incorporation of $CaCl_2$ dopant through sodium alginate-mediated crosslinking optimizes the bio-composite framework for enhanced performance in applications such as biosorption [15].

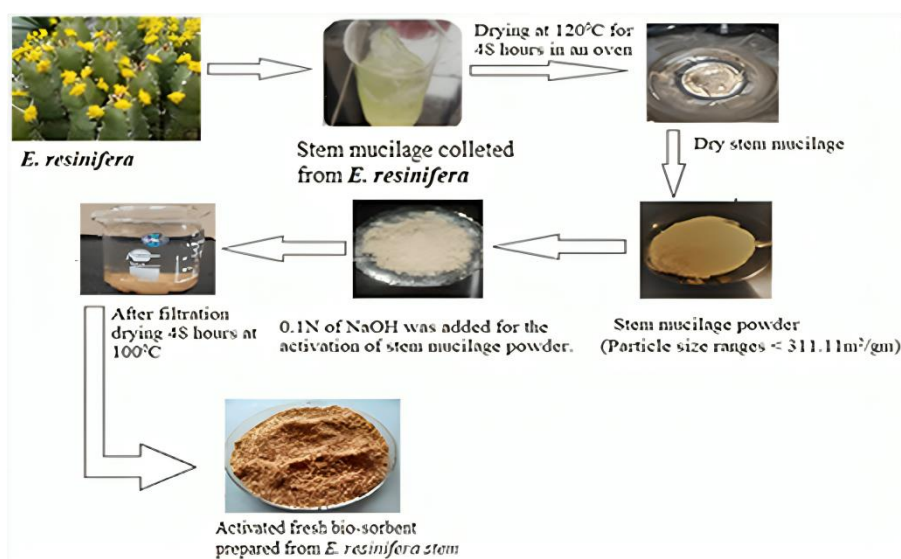


Figure 1. Schematic diagram of novel activated bio- composite preparation.

Table 1. Schematic diagram of novel activated bio- composite preparation.

Parameters	Units	Results	Parameters
Particle size	μm	100 to 312	Particle size
pH		7	pH
Moisture contained	%	4.25	Moisture contained
Conductivity	$\mu S\ cm^{-1}$	15.33	Conductivity
Specific gravity		0.36	Specific gravity
Porosity	%	87.23	Porosity
Bulk density	g/mL^{-1}	0.75	Bulk density
Ion exchange capacity	meq./g	0.81	Ion exchange capacity
Water soluble matter	%	2.14	Water soluble matter
Volatile matter	%	27	Volatile matter
Surface area	m^2/g	< 311.11	Surface area
Parameters	Units	Results	Parameters

2.2. Preparation of Standards and Reagents

Analytical-grade chemicals and double-distilled water (Milli-Q Millipore, 18.2 MΩ·cm conductivity) were used throughout the experiments. A 1000 mg/L Cr⁶⁺ stock solution was prepared by dissolving 2.828 g of anhydrous potassium dichromate (K₂Cr₂O₇) in 1000 mL of deionized water with 1.5 mL of 1 M HNO₃. Working Cr⁶⁺ solutions (1.0–10 mg/L) were prepared by diluting the stock following standard water analysis methods. Solution pH was adjusted using 1 M HNO₃ and 1 M NaOH. Fresh bio-composites were synthesized by mixing *Euphorbia resinifera* stem mucilage powder (5 g/L) with C₆H₇O₆Na (5%) and doping with CaCl₂ (0.1 mol/L).

2.3. Optimization Perceptiveness Using Box-Behnken Design (BBD)

The BBD (Box-Behnken Design) predicts the outcomes of

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 \quad (1)$$

The BBD (Box-Behnken Design) predicts the outcomes of the dependent variables analyzed without requiring a complete three-level factorial design. This design acknowledges the Y signifies the Cr (VI) ion adsorption process in an aqueous solution, which is affected by various parameters over distinct operational periods. Where β is the regression coefficient obtained through the statistical analysis of the experimental data using the least-squares method. The variable x represents the concentration of the coded independent variables, which include pH, activated bio-composite dosage, and experimental time. The credibility of the model and the statistical significance of the regression coefficients were determined using an analysis of variance (ANOVA). The experimental results for the dependent variables were analyzed with the help of Design-Expert version 22.6.0 software (Design Expert for Windows, Stat Soft, Inc, USA) [16, 17].

3. Results and Discussions

3.1. Characterization of Mucilage Based Activated Bio-composite Residuals

SEM-EDS images reveal a detailed view of the bio-composite material's surface before and after exposure to a 10-ppm chromium solution during the adsorption process. In the untreated bio-composite sample shown in Figure 2a, the surface appears rough and porous, with many visible holes and uneven areas. These pores and voids are important because they represent spaces where adsorption can occur. After treatment with chromium ions, as shown in Figure 2b, the surface morphology changes significantly. The previously rough and porous surface becomes smoother and forms a distinctive wavy, white pattern. This visible change indicates that chromium ions have

the dependent variables analyzed without requiring a complete three-level factorial design. This design acknowledges the expansion of the observed equation used to characterize the behavior of the dependent variables, which are evaluated under various independent variables (operational conditions) within the specified ranges. Seventeen experimental runs were conducted using the BBD matrix model, and the average results were used for the data analysis. A quadratic function equation (1) was employed to adjust the experimental range of the selected process variables, with their units and nomenclature being 'A' for pH, 'B' for activated bio-composite dosage (g/L), and 'C' for experimental time (min): Range (-1, 0, +1) and levels of different variables with coded [(pH: 3.5, 7.0, 9.5); (activated bio-composite dosage/ (g/L): 0.5, 5.75, 11); and (experimental time (min): 20, 70, 120)]. Different measurements will be obtained for each dependent variable studied (y).

interacted with and attached to the bio-composite surface, effectively covering and modifying it. The transformation in surface texture suggests a strong chemical and physical interaction between the adsorbate (chromium ions) and the adsorbent (bio-composite). Additionally, the energy dispersive spectrum (EDS) analysis for both samples, also presented in Figure 2a and 2b, confirms that the elemental composition of the bio-composite surface changes after adsorption. These spectra show the presence of Cr⁶⁺ ions on the treated bio-composite, which supports the conclusion that Cr⁶⁺ ions have been successfully adsorbed onto the material. Overall, the SEM-EDS analysis demonstrates how the morphology and surface chemistry of the bio-composite are altered through the adsorption process, providing important insights into the mechanism by which chromium ions are captured and retained by the bio-composite. The changes in the after-treated bio-composite image are due to the interaction between the adsorbate ions and the upper part of the adsorbent [18, 19]. Energy dispersive X-ray spectroscopy (EDS) analysis confirm the presence of Cr⁶⁺ ions on the adsorbent surface following the adsorption process to provide robust and direct evidence supporting the proposed adsorption mechanism. By accurately detecting and measuring the elemental composition of the adsorbent material, particularly focusing on chromium signals, EDS analysis serves as a critical tool to verify that Cr⁶⁺ ions have been effectively captured and immobilized by the adsorbent. This quantitative confirmation is essential not only for validating the occurrence of adsorption but also for elucidating the nature and extent of the interaction between chromium species and the adsorbent surface. Through detailed elemental mapping and concentration analysis, EDS can reveal spatial distribution patterns of chromium, which further contributes to understanding the adsorption sites and mechanisms involved. Such comprehensive data strengthen the correlation between surface chemistry

modifications of the adsorbent and its adsorption capacity, thereby providing insights that are vital for optimizing the adsorbent design and performance in practical environmental remediation applications. Moreover, this analysis supports the reproducibility and reliability of the adsorption process by offering measurable, scientific proof of chromium uptake, which is indispensable for advancing both fundamental research and applied technologies in heavy metal removal. The energy-dispersive spectra (EDS) of the untreated and treated bio-composites are shown in Figure 2(a) and (b), respectively. The SEM-EDS results were further validated using FTIR (Fourier-transform infrared spectroscopy). FTIR spectroscopy was employed to identify the different functional groups attached to the mucilage-based bio-composite. FTIR spectral analysis revealed the key functional groups involved in Cr^{6+} ion binding in the bio-composite. The shifts in the peak positions upon Cr^{6+} ions loading indicate specific interactions between the metal ions and functional groups. The shift of the broad $-\text{OH}$ stretching band from 3448.94 cm^{-1} to 3450.21 cm^{-1} suggests the coordination of hydroxyl groups with Cr^{6+} ions. The $\text{C}=\text{O}$ stretching peak moving from 1655.83 cm^{-1} to 1648.97 cm^{-1} indicates the complexation of carboxylic groups with Cr^{6+} ions. The shift from 1407.37 to 1412.83 cm^{-1} corresponds to nitrogen interactions, likely via $\text{N}-\text{H}$ groups. The $\text{C}-\text{N}$ stretching vibration peak shifted from 1074.06 cm^{-1} to 1118.07 cm^{-1} , indicating amino group involvement in chromium binding. The $\text{O}-\text{C}-\text{O}$ scissoring vibration peak changed from 889.44 cm^{-1} to 849.69 cm^{-1} , reflecting interactions with

polysaccharide components [15, 18]. Notably, key functional groups, such as hydroxyl ($-\text{OH}$), carboxyl ($-\text{COOH}$), and amine ($-\text{NH}_2$) groups, demonstrate significant spectral changes following exposure to Cr^{6+} ions, which directly indicates their active participation in the adsorption process. For example, the broadening and shifting of the $-\text{OH}$ stretching vibration band suggest the formation of hydrogen bonds or coordination complexes between hydroxyl groups and chromium species, which facilitates the anchoring of Cr^{6+} ions onto the adsorbent surface. Additionally, modifications observed in the $\text{C}=\text{O}$ stretching region reflect complexation or electrostatic interactions between carboxyl groups and Cr^{6+} ions, highlighting the role of these groups in stabilizing the adsorbed Cr^{6+} ions. The amine groups also exhibit notable spectral alterations, implying their involvement through either direct coordination or electrostatic attraction mechanisms. These combined spectral modifications confirm that the adsorption mechanism is predominantly governed by surface complexation and ion exchange processes, wherein Cr^{6+} species interact strongly with the functional groups present on the adsorbent surface. This enhanced interaction not only increases the affinity of the adsorbent for Cr^{6+} ions but also improves its overall adsorption capacity, making it highly effective for Cr^{6+} ion removal [18, 19]. These spectral changes collectively confirm that Cr^{6+} ions coordinate with the hydroxyl, carboxyl, and amino groups on the mucilage-based activated bio-composite surface, altering the vibrational environment of these functional groups as shown in Figure 3.

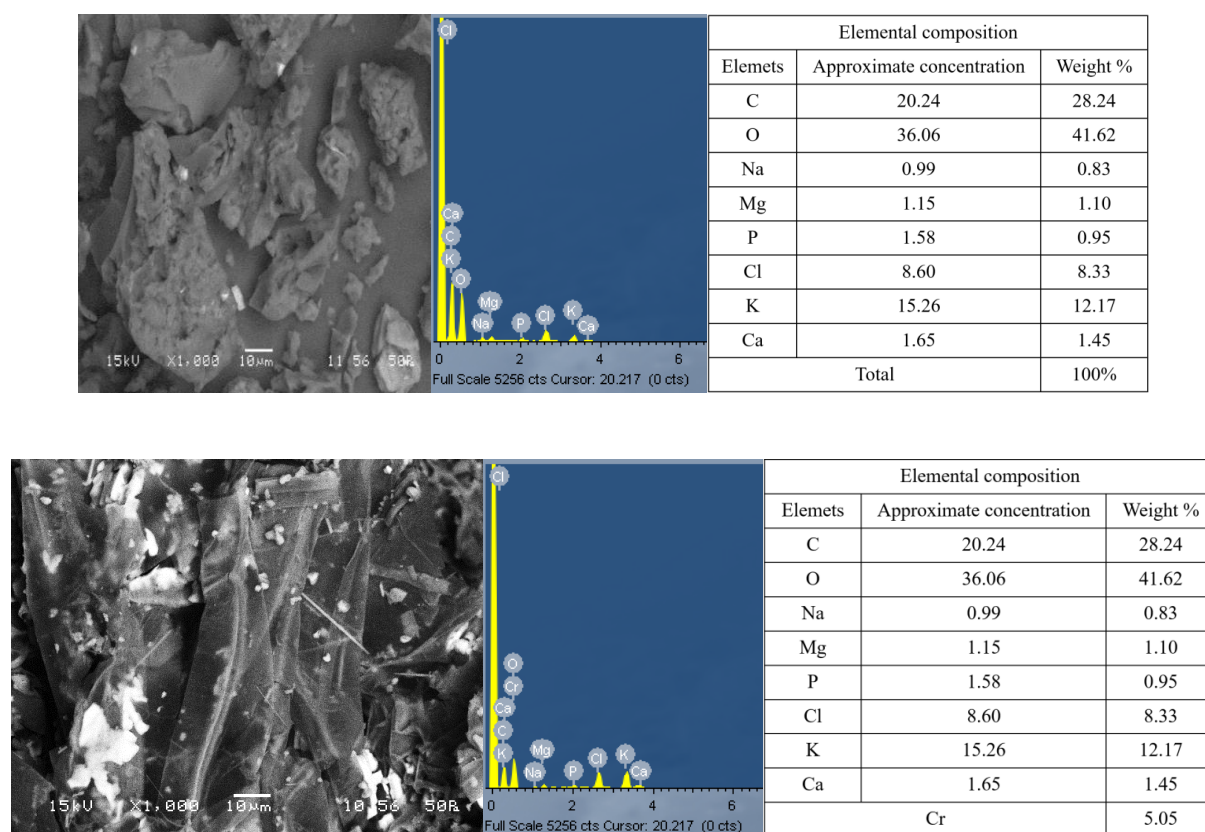


Figure 2. SEM-EDS diagrams of bio-composite (a) without and (b) with 10 mg/L the removal of Cr^{6+} ion.

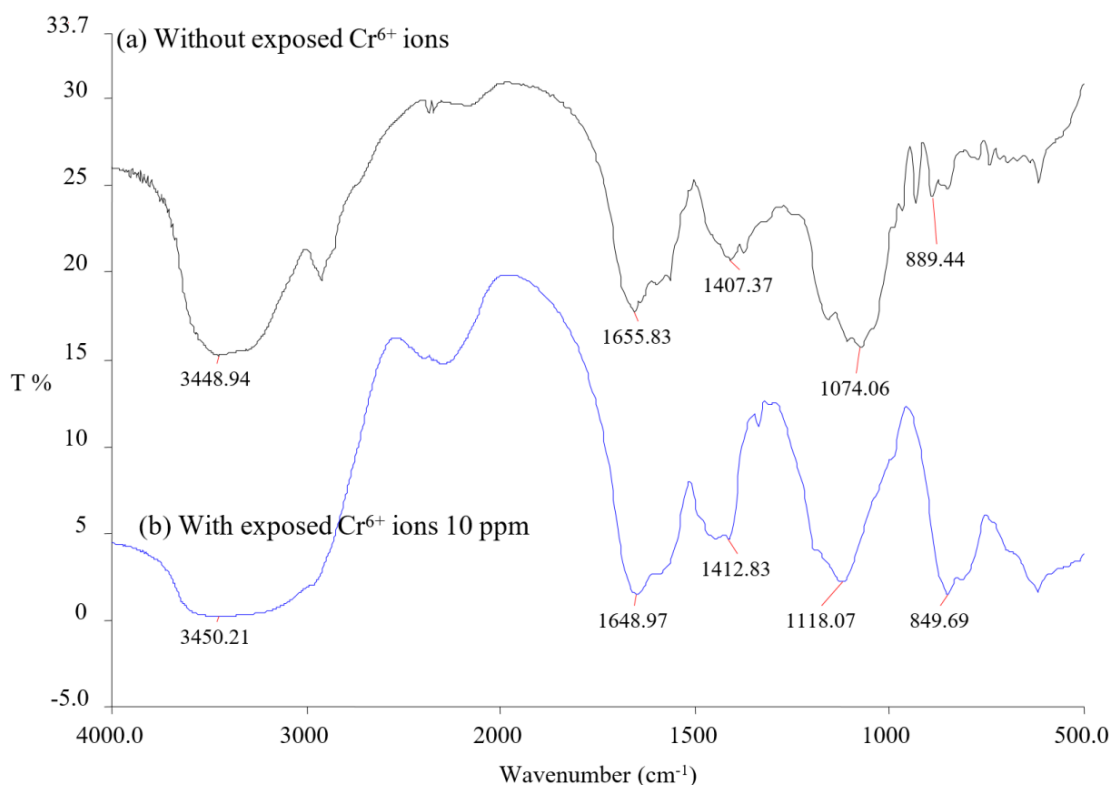


Figure 3. FTIR spectra of the treated neutralized activated bio-composite (a) without exposed and (b) Exposed with 10 mg/L of Cr^{6+} solutions.

3.2. The Integrated Effects of Different Process Parameters through Bio-sorption Studies

In the present experimental examination, the integrated effects of different process parameters were accomplished using the Box-Behnken design through bio-sorption analysis, and response surface methodology diagram, as shown in Figure 4. The gelling-based activated bio-composite dosage and pH were the most significant experimental parameters affecting the removal efficiency of the bio-sorption process. The integrated effects of bio-composite dosage (1-10 g/L) and pH (3–9) on the removal of Cr^{6+} ions using bio-composite materials were considered at a constant concentration of 10 mg/L Cr solution, an experimental duration of 70 min, and a temperature of 30°C.

3.2.1. The Integrated Effects of pH and Activated Bio-composite Dosage

Optimization of a two-dimensional contour plot and three-dimensional surface response graphical presentation (Figure 4a) as a function of two independent variables, pH (3 to 9) and

mucilage-based activated bio-composite dosage (1 to 10 g/L). The contour plot and three-dimensional surface response figure clearly revealed that the removal of Cr^{6+} ions from the aqueous medium increased with increasing pH (7) and mucilage based activated bio-composite dosage (7 g/L); thereafter, with increasing pH and, the bio-sorbent ions decreased [20, 21]. The maximum removal of Cr^{6+} ions was 88.76% at pH 7 with a bio-composite dosage of 7 g/L, Cr^{6+} ions concentration of 10 mg/l, exposure time of 70 min, and temperature of 30 °C, occurring when more negative species ($\text{Cr}_2\text{O}_7^{2-}$, CrO_4^{2-}) were available in an aqueous medium. At low pH (3 to 7), there is a strong attraction between the negative species of the test solution and the positively charged surface of the bio-composite materials. The experimental results clearly revealed that as the activated bio-composite mass increased from 1 to 7 g/L, the removal percentage of Cr^{6+} ions also increased. After the bio-composite dosage of 7 g/L, the removal efficiency did not significantly change because partition possessions may have formed on the surface, defending the active sites of the bio-sorbents. Therefore, a bio-composite dosage of 7 g/L and pH 7 were used for further experiments [11]. The removal efficiency with the pH of various bio-sorbent is described in Table 2.

Table 2. Comparison of adsorption capacity of bio-composite for Cr^{6+} ion with that of different adsorbents.

Biosorbents	Analysis medium	pH	Cr^{6+} (mg/g)	References
<i>Eichornia crassipes</i>	Aqueous solution	4.5	36.34	Giri et al. 2012
Cassava root husk derived biochar	Industry waste water	6.28	2.35-5.24	Tho et al. 2021
Pea-nut shell biochar	Aqueous solution	2.0	2.475	Kumar et al. 2022
Anion exchange resin	Simulated waste water	4.0	294.11	Leonard et al. 2023
Mucilage based bio-composite	Aqueous solution	7	80	Present study

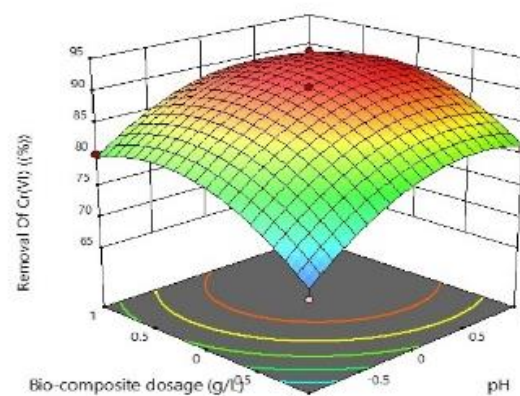
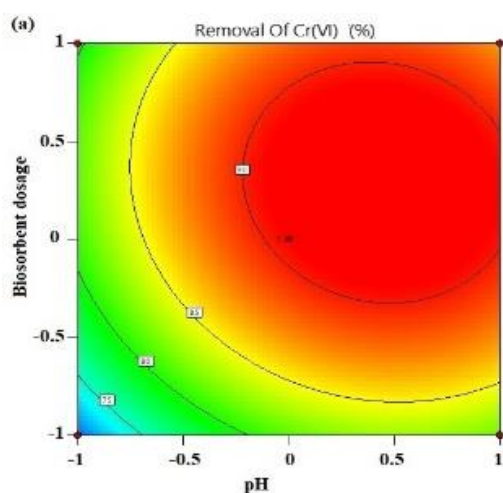
3.2.2. The Integrated Effects of Activated Bio-composite Dosage and Contact Time

In the present investigation, the optimum removal percentage (Figure 4b) of Cr^{6+} ions was at pH 7, bio-sorbent concentration of 10 mg/l, and a constant room temperature using two independent variables, such as activated bio-composite dosage and experimental time. The optimization of the process parameters of a two-dimensional contour plot and three-dimensional response surface graphical diagram is presented in Figure 4b. The maximum bio-sorption of Cr^{6+} ions was observed at a bio-composite dosage of 7 g/L with respect to the equilibrium time after 70 min. The results of the bio-sorption of Cr^{6+} ions in the first 45 min of the treatments because the Cr^{6+} ions needed to diffuse on the surface of the bio-sorbent materials by an intraparticle diffusion process, and significantly hydrolyzed ions were released at a slower rate. This suggests the feasible monolayer formation of Cr^{6+} ions on the surface of the bio-composite [23, 24].

3.2.3. The Integrated Effects of Contact Time and pH

pH (3 to 9) and experimental time (20-120 minutes) are the

most important parameters influencing the removal of bio-sorbent ions from aqueous medium. In the present research work shows the optimization of a two-dimensional contour plot and three-dimensional surface response graphical presentation (Figure 4c) as a purpose of two independent variables that is pH and experimental times. Apart from the figure that more than 75% above removal of bio-sorbent ions take place within the first 45 minutes and equilibrium maintain slowly come after 70 minutes. The rate of removal of the bio-sorbent ions is more firstly because of each one of the bio-composite sites are empty and the bio-sorbent concentration gradient is very high. The results from the Figure 4b clearly indicate the bio-sorption efficiency is elevate along the raises pH ranges from 3 to 7 then decline extended up to pH 9. In an acidic medium, experiments convey that the surface of the bio-composite is the high insertion of a proton to an atom, molecule, or ion. Then, the results indicated a strong attraction pursuit between CrO_7^{2-} and the cations surface of the bio-composite. In an alkaline medium (pH 7-9) treatments shows the bio-sorption of Cr^{6+} ions decreases because of material-formed anions by fascinating OH^- groups on the surface of the bio-composite [18, 23, 24]. Figure 5 shows the details bio-sorption mechanisms of bio-sorbent ions with the surface of bio-composite [7].



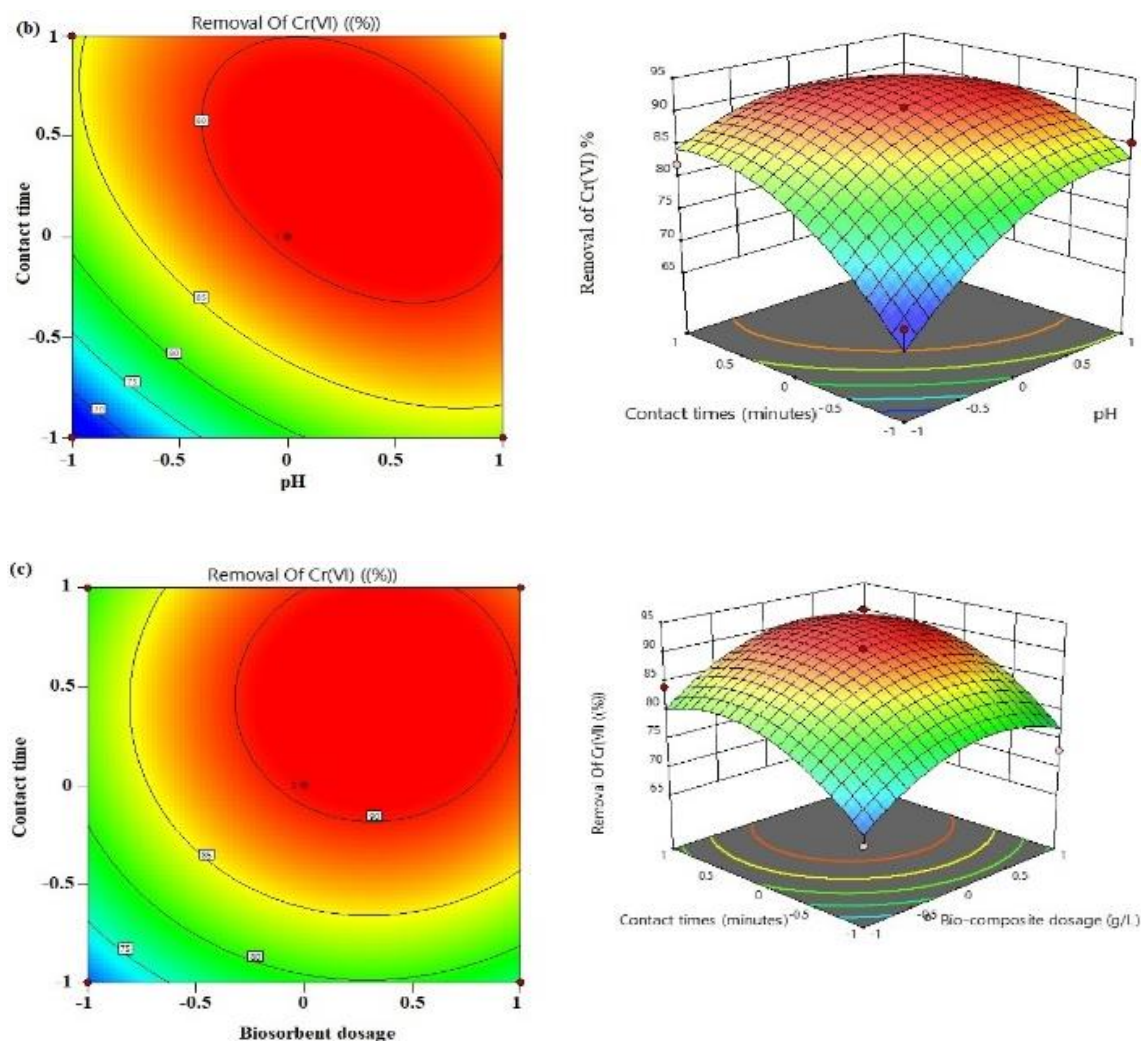


Figure 4. A contoured and response surface plots showing the integrated effects of (a) pH and Bio-sorbent dosages, (b) pH and contact time and (c) contact time and bio-sorbent dosage on the adsorption of Cr^{6+} ions with a constant initial concentration of 10 mg/L and room temperature $30 \pm 2^\circ\text{C}$.

3.3. Possible Mechanism of Cr^{6+} Ions with Treated Activated Bio-sorbent

The mechanism of Cr^{6+} ion adhesion on the surface of bio-composite bio-sorbents is the most important segment of the bio-sorption process. To understand the characteristics of the mucilage-based bio-composite, this supports the development of a new bio-sorbent for future applications. In the current study, the bio-sorption of Cr^{6+} ions from an aqueous medium by a mucilage-based bio-composite was a mechanism of the ion-exchange reaction. The $\text{pH} > 7$ of the acidic medium showed that (i) the bio-sorbent surface sites were positively charged centre (nitrogen, OH) and hence the negatively

charged $\text{Cr}_2\text{O}_7^{2-}$ in the test solutions was removed by an electrostatic interaction process, and (ii) an ion-exchange reaction occurred between the positively charged bio-sorbent centre sites and, $\text{Cr}_2\text{O}_7^{2-}$ in the test solution. At $\text{pH} < 7$ of the test solution, Cr^{6+} ions bio-sorption constant there was no change perhaps the surface of the treated bio-sorbent enhanced and more negatively charged by reason of the electrostatic repulsion force formed between the same charges. During hydration, the activated bio-sorbent surface sites complete their coordination shells by interacting with the available hydroxyl (OH) groups. Within varying pH ranges, these surface-active hydroxyl groups can either bind or release H^+ ions, while the surface maintains a positive charge as a result of the reaction. Figure 5 graphically representation of the bio-sorption mechanism of bio-sorbent ions with bio-composite mass in acidic and alkaline mediums [24-26].

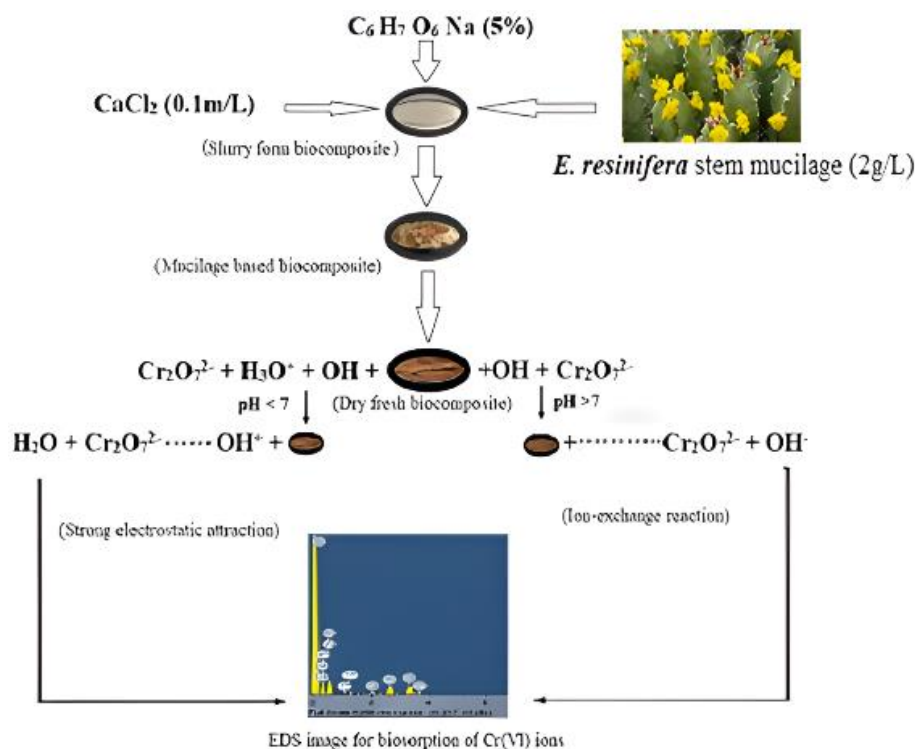


Figure 5. Adsorption mechanism of Cr^{6+} ion with activated bio-composite materials.

3.4. An Optimization Approach for Bio-sorption Efficiency Using Box-Behnken Design (BBD)

The quadratic model equation adhere the response surface methodology activities to the prepared independent parameters as proceed with the design expert Stat-Ease version 22.0.6 [20].

The equation uses the actual factor levels in their original units to predict the response, as demonstrated in Eq. (2).

$$\begin{aligned} \text{The removal (Y \%)} = & +90.62000 + 4.65375 (\text{pH}) + 3.66375 \\ & (\text{activated bio-composite dosage}) + 5.33750 (\text{Contact time}) - \\ & 0.790000 (\text{pH} * \text{Bio-composite dosage}) - 4.17250 (\text{pH} * \text{Con-} \\ & \text{tact time}) + 0.357500 (\text{Bio-composite dosage} * \text{Contact time}) \\ & - 5.12500 (\text{pH})^2 - 5.72500 (\text{Bio-composite})^2 - 5.99250 (\text{Con-} \\ & \text{tact time})^2. \quad (2) \end{aligned}$$

A total of 17 experimental runs were conducted using the Box–Behnken design matrix model (Table 3), with concurrent data analysis performed to evaluate the results. The combined effects of the parameters were assessed using this design approach, as illustrated in Figures 4a, 4b, and 4c. Statistical evaluation of the quadratic surface model and regression coefficients was carried out using analysis of variance (ANOVA) to

determine the significance and suitability of each variable's impact on the response, with the results presented at a defined confidence level in Table 4. The quadratic model demonstrated statistical significance, indicated by an F-value of 10.67 and a p-value of 0.0025. Individual factors—A (pH), B (activated bio-composite dosage), and C (contact time)—were also significant, with respective F-values of 16.20, 10.04, and 21.30, and p-values of 0.0050, 0.0157, and 0.0024 (Table 3). Interaction effects between parameters AB, AC, and BC showed moderate influence on the response, with F-values of 0.2334, 6.51, and 0.0478 and p-values of 0.6438, 0.0380, and 0.8332, respectively. Quadratic terms A2, B2, and C2 were statistically significant, reinforcing the robustness of the model (Table 4). Residual analysis confirmed the normality and consistency of the model, with a normal probability plot approximating a straight line (Figure 6a) and a strong correlation between actual and predicted responses ($R^2 = 0.9421$, Figure 6b). Additional residual plots (Figures 6c and 6d) supported model adequacy, with a predicted R^2 of 0.9012 and an adjusted R^2 of 0.9201. The regression model's overall significance was further validated by an R^2 value of 0.9610 and a low coefficient of variance (C.V. = 1.96), indicating high accuracy and reliability of the experimental data. These comprehensive analyses confirm the effectiveness of the Box–Behnken design in optimizing the experimental parameters with promising predictive capability [20, 22, 24].

Table 3. Box-Behnken design for three independent variables used present research work along with the observed response.

Source	Sum of squares	Df	Mean square	F-value	P-value	Remarks
Model	1027.75	9	114.19	10.67	0.0025	Significant
A-pH	173.26	1	173.26	16.20	0.0050	Significant
B-Biocomposite dosages	107.38	1	107.38	10.04	0.0157	Significant
C-Contact time	227.91	1	227.91	21.30	0.0024	Significant
AB	2.50	1	2.50	0.2334	0.6438	Non-significant
AC	69.64	1	69.64	6.51	0.0380	Significant
BC	0.5112	1	0.5112	0.0478	0.8332	Non-significant
A ²	110.59	1	110.59	10.34	0.0148	Significant
B ²	138.00	1	138.00	12.90	0.0088	Significant
C ²	151.20	1	151.20	14.13	0.0071	Significant
Residual	74.89	7	10.70			
Lack of Fit	74.89	3	24.96			
Pure Error	0.0000	4	0.0000			
Total	1102.64	16				

Table 4. Analysis of variance for the response surface quadric models for the adsorption of Cr⁶⁺ ions by bio-composite.

Run No.	Coded Values			Actual Values			Actual Cr ⁶⁺ removal %	Predicted Cr ⁶⁺ removal %	Residual values	Leverage
	A (pH)	B (Activated Bio-composite Dosage, mg/L)	C (Contact Time, minutes)	A (pH)	B (Activated Bio-composite Dosage, mg/L)	C (Contact Time, minutes)				
1	-1	0	+1	3.5	5.75	120	82.00	85.32	-3.32	0.750
2	-1	-1	0	3.5	0.5	70	69.15	70.66	-1.51	0.750
3	0	-1	-1	6.5	0.5	20	68.45	70.26	-1.81	0.750
4	0	+1	-1	6.5	11	20	73.00	76.87	-3.87	0.750
5	-1	0	+1	3.5	5.75	120	82.00	84.36	-2.36	0.750
6	0	0	0	6.5	5.75	70	90.62	90.62	0.0000	0.200
7	0	0	0	6.5	5.75	70	90.62	90.62	0.0000	0.200
8	0	-1	+1	6.5	0.5	120	84.09	80.22	3.87	0.750
9	+1	+1	0	9.5	11	70	88.81	87.30	1.51	0.750
10	-1	+1	0	3.5	11	70	80.12	79.57	0.5500	0.750
11	0	0	0	6.5	5.75	70	90.62	90.62	0.0000	0.200
12	-1	0	-1	3.5	5.75	20	68.66	65.34	3.32	0.750
13	+1	0	-1	9.5	5.75	20	85.35	82.99	2.36	0.750
14	0	+1	+1	6.5	11	120	90.07	88.26	1.81	0.750
15	0	0	0	6.5	5.75	70	90.62	90.62	0.0000	0.200
16	0	0	0	6.5	5.75	70	90.62	90.62	0.0000	0.200

Run No.	Coded Values			Actual Values			Actual Cr^{6+} removal %	Predicted Cr^{6+} removal %	Residual values	Leverage
	A (pH)	B (Activated Bio-composite Dosage, mg/L)	C (Contact Time, minutes)	A (pH)	B (Activated Bio-composite Dosage, mg/L)	C (Contact Time, minutes)				
17	-1	-1	0	3.5	0.5	70	81.00	81.55	-0.5500	0.750

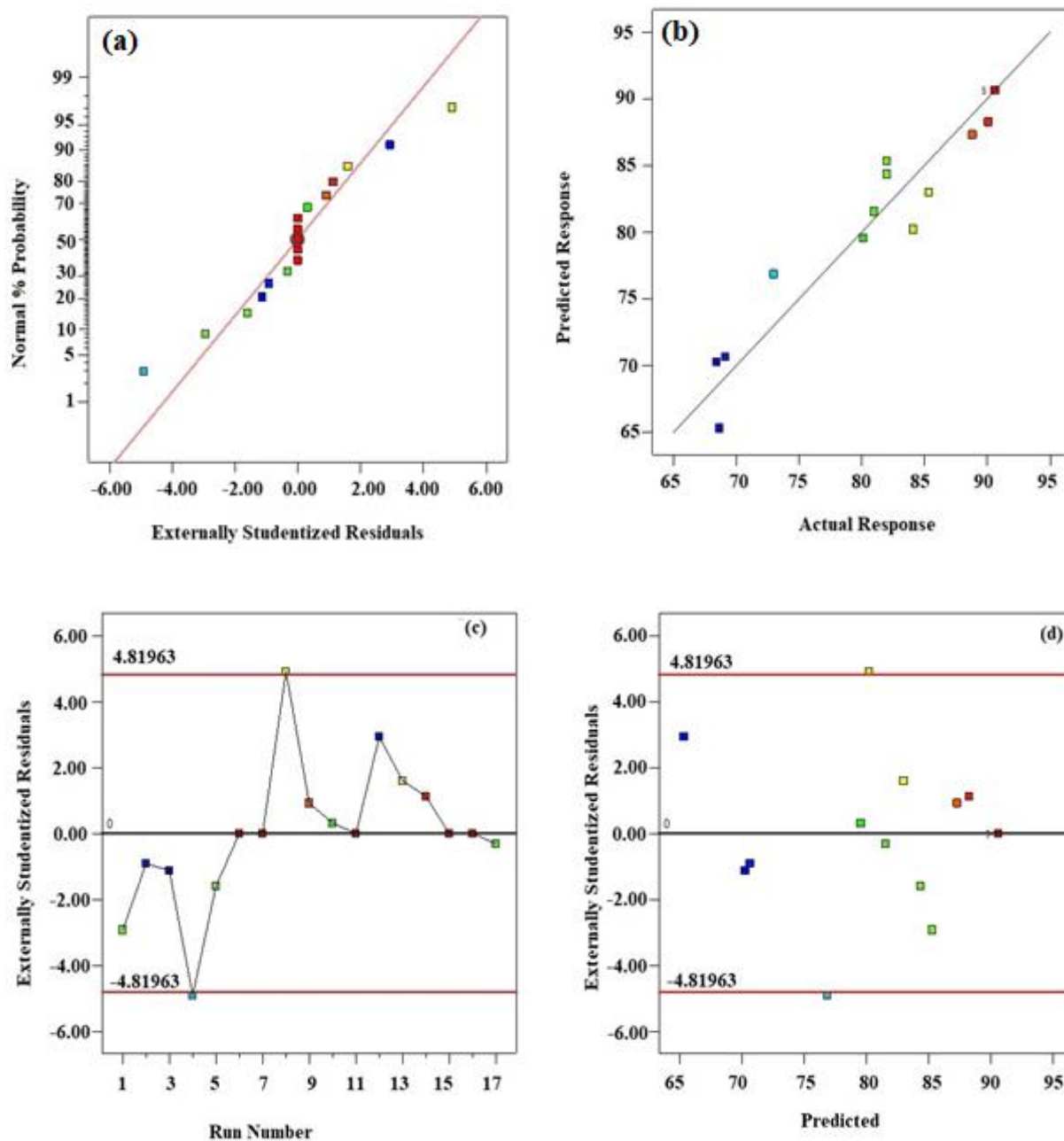


Figure 6. (a) Normal% probability versus residual error, (b) Predicted response versus actual response, (c) Residual response versus run, (d) Residual response versus predicted response.

4. Conclusions

The optimization of key process parameters influencing Cr^{6+} ion removal efficiency was conducted employing the Box-Behnken design. The combined effects of the three process variables were analyzed using response surface methodology (RSM) diagrams. The removal process is significantly influenced by the adsorbate concentration and flow rate. The use of the Box-Behnken design allowed for a clear understanding of how different factors interacted, making the process easier to scale or refine. It demonstrated superior performance in capturing Cr^{6+} ions from aqueous solutions via column biosorption. The quadratic regression model (RSM) is highly reliable, featuring a correlation coefficient (R^2) of 0.9610 and a low coefficient of variance (CV) of 1.96%. The p-value of 0.0025 and an F-value of 10.67 confirm that the results are statistically significant and that the model is precise. The low CV indicates that the removal process was highly reproducible and consistent. The regeneration and reusability of mucilage have not been sufficiently explored, potentially limiting its practical application in continuous treatment systems. The scalability of this bio-sorbent for industrial wastewater treatment also requires further investigation, including cost-effectiveness and long-term stability. Future research should focus on optimizing the extraction and modification of mucilage to enhance its adsorption performance, evaluating its behavior in real wastewater matrices, and developing regeneration protocols to improve sustainability. Integrating this bio-sorbent with other treatment methods could also be explored to achieve higher removal efficiencies and broader applicability.

Abbreviations

BBD	Box-Behnken Design
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive Spectroscopy
FTIR	Fourier-Transform Infrared Spectroscopy
RSM	Response Surface Methodology
ppm	Parts Per Million

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Author Contributions

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Prativa Kar: Formal Analysis, Software, Supervision, Investigation, Visualization, Validation, Writing – review & editing

Anil Kumar Giri: Software, Supervision, Investigation, Visualization, Validation

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Data Availability Statement

The data supporting the outcome of this research work has been reported in this manuscript.

Conflicts of 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.

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