

Research Article

Assessment of chromium removal efficiency from chromium-contaminated water using ball-milled *Eichhornia crassipes* biochar

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Abstract

Hexavalent chromium {Cr (VI)} contamination in water sources poses a significant threat to human health and the environment due to its toxicity and carcinogenicity. In recent years, biochar-based adsorbents have gained attention for their potential to effectively remove heavy metals from water. The present study focuses on the synthesis and application of ball-milled nano-*Eichhornia* (water hyacinth) biochar (BMNEBC) as an innovative adsorbent for the removal of Cr (VI) from aqueous solutions. In this research, *Eichhornia* plant leaves were utilized as the precursor for biochar production through a pyrolysis process. The produced biochar was subjected to physical modification (ball milling) to obtain nano-sized particles, thereby increasing the surface area and porosity of the material. Batch adsorption experiments were conducted to evaluate the Cr(VI) removal efficiency of BMBNBC under various operating conditions, including initial Cr(VI) concentration (10 to 70 mg), adsorbent dosage (25 to 125mg), contact time (0 to 180 minutes), and pH (3 to 12). The study revealed that the optimal conditions were identified as pH 6, an adsorbent dosage of 75 mg, and a contact time of 4 hours, resulting in an impressive Cr (VI) removal capacity of 256.3 mg/g (91%). These findings provide valuable insights for developing effective strategies to remove chromium from contaminated water samples.

Keywords: Adsorption and water treatment, ball-milled nano-*Eichhornia* biochar, Cr (VI) removal, Hexavalent chromium, Human health

INTRODUCTION

Due to the variable speciation, carcinogenicity, and high mobility of chromium (Cr), environmental water pollution has garnered considerable interest (Dhal *et al.*, 2013). The two main oxidation states of Chromium are Cr(III) and Cr(VI), with Cr(VI) having greater solubility and toxicity than Cr(III). Therefore, changing the speciation of Cr by triggering the redox reaction (i.e., reduction and oxidation) using electroactive materials becomes a potential tactic (Springthorpe *et al.*, 2019). The removal of Cr (VI) from wastewater has been a significant challenge, and various techniques have been explored in recent years to address this issue. These techniques include electrodialysis, chemical pre-

cipitation, ion exchange, membrane technology, biological methods, and adsorption processes (Juve *et al.*, 2022). Among these techniques, the adsorption method has garnered considerable attention due to its advantages of simplicity in operation and cost-effectiveness (Kokab *et al.*, 2021; Rashid *et al.*, 2021). The adsorption method utilizes adsorbent materials that can attract and bind Cr (VI) ions from wastewater, thereby facilitating their removal. Traditional carbon-based adsorbents, including activated carbon, biochar, graphene, and carbon nanotubes, have demonstrated potential for treating Cr(VI) pollution. However, their adsorption efficiency still needs improvement to achieve more effective and efficient removal of Cr (VI) from wastewater (Gopinath *et al.*, 2021; Gul *et al.*,

2021). The adsorption capacity and selectivity of these materials may be limited, or they may suffer from issues such as poor regeneration or high cost. Given the limitations of existing adsorbent materials, there is a pressing need to explore and develop new adsorbent materials that can complement or overcome the shortcomings of traditional adsorbents (Crini *et al.*, 2018). These new materials are expected to exhibit improved adsorption efficiency, higher selectivity for Cr(VI) ions, better regeneration capabilities, and potentially lower costs. These efforts aim to provide more effective and sustainable solutions for removing Cr (VI) from wastewater, ultimately contributing to environmental protection and public health.

Due to its widespread availability, plentiful production, ease of use, and affordability, biochar has regained popularity as an adsorbent. Corn straw (Zhang *et al.*, 2011), water hyacinth (Masto *et al.*, 2013), rice husk (Asadi *et al.*, 2021; Singh *et al.*, 2018), and municipal sludge (Goldan *et al.*, 2022) are a few examples of solid biomass waste products that may be used as raw materials to produce biochar. Biochar's wide availability stems from the diverse range of biomass waste materials that can be used as feedstock. The vast quantities of these waste materials generated globally offer an abundant supply for biochar production (Zhang *et al.*, 2016). The production process of biochar, which involves the thermal decomposition of biomass under controlled conditions, is relatively straightforward. This simplicity makes large-scale biochar production easier, reducing complexity and costs. Biochar production utilizes waste materials that are often readily available and inexpensive as raw materials (Kokab *et al.*, 2021). Additionally, the simple production process contributes to lower manufacturing costs. These factors make biochar a cost-effective adsorbent material for wastewater treatment. The diverse range of biomass waste materials offers flexibility and adaptability to local resources (Amalina *et al.*, 2022). Furthermore, biochar production from waste materials promotes sustainable waste management practices.

Eichhornia crassipes, commonly known as the water hyacinth (WH), is a South American native and a type of invasive weed found worldwide Ilo *et al.*, 2020). Due to eutrophication of the water, this weed has overgrown and poses a major threat to agriculture, human health, and the ecosystem. The water hyacinth, or *Eichhornia crassipes*, is an invasive weed species that has severely harmed the environment due to its exceptional fertility and rapid growth rate (Mengesha, n.d.). However, water hyacinths are widely used as resources for multiple purposes. For example, it is used as animal feed or as organic substrates for agricultural production.. Utilizing this abundant biomass for biochar production serves as a sustainable approach to managing invasive growth while producing a valuable adsorbent material. Eich-

hornia-biochar has garnered significant attention for its ability to effectively remove Cr from wastewater (Dhinesh *et al.*, 2024). Studies have demonstrated its high adsorption capacity and affinity for Cr (VI) ions, often outperforming biochar derived from other sources. This superior performance can be attributed to the unique physicochemical properties of Eichhornia biochar, such as its high surface area, porosity, and the presence of specific functional groups. The production of Eichhornia-biochar is a relatively simple and low-cost process, as it utilizes a waste biomass as the feedstock (P. Singh *et al.*, 2023). This aligns with the principles of circular economy and sustainable waste management. Additionally, the use of Eichhornia-biochar for wastewater treatment contributes to the overall reduction of environmental pollution caused by the uncontrolled growth of this invasive plant. Eichhornia-biochar can be further modified or functionalized to enhance its adsorption performance, selectivity, and regeneration capabilities. Its unique properties may provide advantages or synergies with certain modification techniques compared to biochars derived from other sources (Dhinesh *et al.*, 2024).

The use of heavily metal-contaminated water severely affects human health issues and leads to mortality. Therefore, scientists are currently concentrating on sustainable methods to eliminate heavy metals from water bodies. Nowadays, biochar provides a low-cost, renewable, and sustainable solution for remediation of heavy metals in contaminated water bodies. It is well known that biochar has potential efficacy in removing heavy metals from water through adsorption mechanisms. The present study focused on the preparation of ball-milled biochar from WH and to evaluate the effectiveness of ball-milled WH biochar in removing Cr from contaminated water.

MATERIALS AND METHODS

Collection of plant samples

The taxonomically identified *Eichhornia crassipes* plants were collected from the Kousika River (Tirupur), Tamil Nadu, India. A herbarium of *E. crassipes* was prepared for morphological studies and authenticated by a plant botanist. The herbarium specimen was deposited at the JJ College of Arts and Science, Pudukkottai, Tamil Nadu, India. The collected plant material was allowed to dry in the shade for further processing.

Production and modification of biochar

For biochar preparation, the dried WH plant material was ground into a fine powder. The fine powder from the WH plant was taken for the pyrolysis process. For pyrolysis, powdered WH plant material was kept in a muffle furnace at 300°C for 2 hrs. Then the nano-sizing the biochar was done by using a ball miller. For ball

milling, a 1:100 ratio was used, i.e., 100 balls were added for every 1 g of biochar. Stainless steel balls were used for ball milling (approximately 76.4g) with a residence time of 10 hours (RSBM-1/060-718, Hindustan Scientific). The nano-sized biochar was used for further studies.

Assay for detecting chromium

Two ml of Cr contaminated water sample (100mg of potassium dichromate mixed with 1L water as a standard) was acidified by using 3 ml of 6N H₂SO₄, 1 ml of 1M of H₃PO₄. Followed by 2 ml of DPC (1,5-diphenyl carbazide) (0.25%) (EPA methods 7196A). The solution was mixed thoroughly and kept for full-colour development for 5 min. After the development of colour, the solution was transferred to a cuvette and the absorbance was measured at 540 nm using UV-spectrophotometer (Jasco V-730). The distilled water used as a blank solution. Absorbance readings of samples were corrected by subtracting the absorbance of a blank carried through the method. From the corrected absorbance, the amount of Cr was determined from the calibration curve previously described by Balasubramanian and Pugalenthi (1999).

Batch experiment

Adsorption isotherms were applied to explain the equilibrium adsorption characteristics. For this study two different experiments were conducted. The isotherm experiment was made to determine the equilibrium concentration, and the kinetics experiment sought to understand the influence of time on the adsorption process. Briefly, to study on these experiments were conducted using varying concentrations of Cr (10-70mg/L) and fixed concentrations of biochar (75mg) under different contact time. After experimentation, the Cr remaining in the solution was estimated by the PC assay (Lace *et al.*, 2019).

Table 1. Parameters of the coded equation values of Adsorption = A+B+C-AB+BC+A2-B2-C2

Removal of chromium	-19671.56246
pH	-132.61837
Dosage Biochar	4.66E+05
Contact time	858.31523
pH Dosage Biochar	1143.33333
pH Contact time	18.68333
Dosage Biochar Contact time	-7593.33333
pH	-2.14677
Dosage Biochar	-2.76E+06
Contact time	-51.32043

Experimental design and optimization

Concentration of adsorbent

Five different concentrations of adsorbent, varying from 25 to 125 mg, were tested against a Cr concentration of 50 mg/L, each flask contain 100ml solution. The entire reaction was carried out in an orbital shaker at 150 rpm for 2 hrs. At the end of the experiment, the chromium in the remaining solution was estimated using DPC method (Anupam *et al.*, 2011).

Influence of pH

A solution containing Cr (50 mg/L) and a fixed concentration of biochar (adsorbent - 75 mg) was tested against varying pH levels ranging from 3 to 12. The experiment was carried out in an orbital shaker at 150 rpm for 2 hours. At the end of the experiment, the chromium was estimated using DPC method (Sanchez-Hachair and Hofmann, 2018).

Contact time

The concentration of Cr and the biochar was kept constant, as previously stated. The time was set as 0 hrs, 2 hrs, 4 hrs, 6 hrs, 8 hrs, 10 hrs, and 12 hrs. After every 2 hrs. the solution was taken out and the remaining chromium in the solution was estimated using the DPC method (Anupam *et al.*, 2011).

Response surface methodology

A statistical method called Response Surface Methodology (RSM) is used to model and optimize the relationship between a set of predictor factors and one or more response variables. It is frequently used to streamline procedures, raise the calibre of output, and cut expenses in engineering, chemistry, and other scientific disciplines. RSM entails a set of tests was carried out at certain locations within the design space to mathematically model the observed responses. The response was then predicted using the model, which was also used to determine the best collection of predictor variables to utilize in order to obtain the intended answer (Myers *et al.*, 2016). This experiment was designed using Design expert software version 11 (Stat Ease). One of the most widely used RSM procedures is CCD, which is a sequence of tests carried out at specific locations in the design space with the aim of fitting a quadratic model to the observed responses. RSM is a potent instrument for process optimization and creating new goods. It enables scientists and engineers to methodically investigate the link between predictor factors and response variables, and to determine which set of predictor variables is most effective in achieving the desired outcome.

Response Surface Methodology is a significant method for increasing productivity, lowering costs, and enhancing product quality in various scientific and tech-

nical applications (Khuri & Cornell, 2018). Three factors have been taken for optimization pH, dosage, and the contact time. Three levels have been established for each factor in the pH unit, with the lower level being 4 and the higher level being 6. For contact time, the lower limit was 3 hrs, the middle and higher limits were 4 hrs. For dosage concentration, the lower limit was 75 mg, and the higher limit was 85 mg. These values were coded as -1 for lower, 0 for middle, +1 for higher. The following is the coded output generated by the software: After completion of the experiment, the Cr in the remaining solution was estimated using the DPC assay and calculated, and the result was fed into the Response (mg/g). The quadratic function was fitted to correlate the relationship between the variables and the response, determining the optimal point. The coded equation is given below and the parameters of the coded equation, values of Adsorption = $A+B+C-AB+BC+A^2-B^2-C^2$, are mentioned in Table 1.

Statistical analysis

All the experiments were conducted in triplicated to avoid handling errors. The Statistical data was assessed using Microsoft Excel for constructing graphs and tables.

RESULTS AND DISCUSSION

Batch experimentation

The results of Cr adsorption studied using a batch adsorption process where in an isotherm study, the adsorbent at a concentration of 75mg was added to different Cr concentrations ranging from (10-70mg/L) for 24 hours, showed that the equilibrium concentration was achieved at the Cr concentration of (50mg/L) after which the Cr uptake was found to decline. The highest Cr absorption was observed at 50 mg/L, followed by 60 and 70 mg/L. The lowest Cr absorption was achieved at 10 mg/L, followed by 20 mg/L. The detailed results of this are depicted in Table 2. These results showed that the adsorbent at a concentration of 75 mg was highly effective for removing a high level of Cr from water

Table 2. Batch Experimentation-Adsorption Isotherm

Initial concentration	Final concentration	$q_e = (C_i - C_e) \cdot V / W$
10	0.38	8.41
20	4.78	12.15
30	12	14
40	16.57	17.62
50	21.39	21.07
60	35	18.66
70	45	18.66

samples. The adsorption isotherms are crucial for understanding how biochar (a porous charcoal-like material) removes Cr from contaminated water and for determining its uptake efficiency.

Furthermore, another batch adsorption study assessed the adsorption of 75 mg of biochar against a fixed concentration of Cr (50 mg/L) at different time intervals, ranging from 0 to 180 minutes, to determine the kinetic model. The results indicated that a contact time of 90 minutes promoted the maximum removal of Cr (198.4 ng/g), followed by 60 minutes (158.9 ng/g). Therefore, this result revealed that contact time is a crucial parameter for removing a higher level of Cr from water samples.

Various parameters influencing the adsorption of Cr (VI)

Influence of adsorbent concentration

The adsorbent dosage is a crucial parameter for enhancing the efficiency of Cr (VI) adsorption (Akram *et al.*, 2017). Therefore, optimization of the adsorbent dosage is a crucial step for removing Cr from water samples. In this study, the influence of adsorbent concentration was investigated using a varied range of adsorbent (WH biochar) concentrations, from 25 mg to 125 mg. The results revealed that the adsorption was maximum at an adsorbent (WH biochar) concentration of 75 mg/L, and the removal of Cr was found to be 241 mg/g, followed by 25, 50 mg/L, respectively (Table 4). Therefore, this result indicates that the optimal concentration of adsorbent significantly influences the higher level of Cr removal from the test sample. Similarly, many researchers have successfully determined the optimum concentration of biochar for enhanced Cr absorption in biological water samples (Tytlak *et al.*, 2015).

Influence of contact time

Many parameters control the Cr adsorption process in biological samples. The contact time is one of the critical parameters that significantly influences the adsorption of the adsorbate by the adsorbent (Jain *et al.*, 2010). In this study, the influence of contact time on Cr removal was investigated from 0 to 12 hours, using Cr at a concentration of 50 mg/L and biochar at a dosage of 75 mg (Table 3). The results indicated that there was an increased removal of 147 mg/g during the 4th hour, after which the adsorption declined (Fig. 1). However, there was again a hike in adsorption around the 8th hour. Fig. 1 shows the effect of contact time on the removal of Cr (VI) from the test sample. The 4th hour was chosen as the assessment level in RSM because it represents the optimal balance between practical contact time and removal efficiency for industrial applications, even though maximum removal occurs at the 8th hour - the additional 4 hours of contact time likely provides only marginal improvement that doesn't justify the in-

Table 3. Batch adsorption experimentation – Kinetic model

Hour	Cr removal (mg/g)
0 th	96.3
30 min.	102.7
60 min.	158.9
90 minutes	198.4
120 minutes	153.5
150 minutes	120.9
180 minutes	106.5

Table 4. Influence of adsorbent concentration

Sl. No.	Adsorbent (mg/L)	Adsorbate (mg/g)
1	25	187
2	50	184
3	75	213
4	100	179
5	125	165

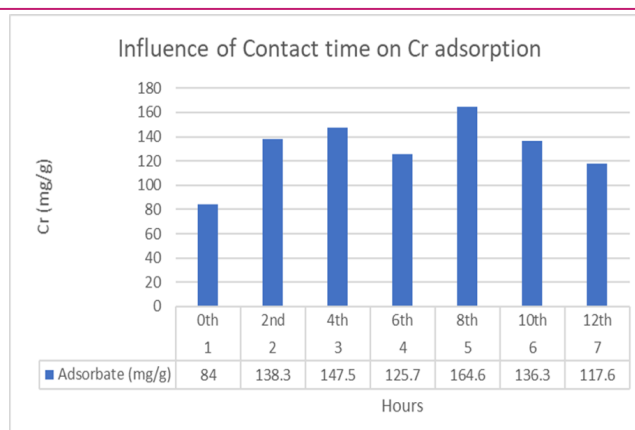
creased processing time and costs. Therefore, the 4th hour was considered as the assessment level in RSM. Recently, Masuku *et al.* (2023) reported that a contact time of 20-80 min results in a steady increase in the uptake of Cr (VI) by the zinc-doped nickel ferrite pinecone biochar (Zn-NiF@PBC) used as an adsorbent. These similar findings are supported in the present study.

Influence of pH

In this study, the pH of the solution at different levels, ranging from 3 to 12, was assessed as an optimization parameter using a fixed rate of 75 mg of biochar and a Cr concentration of 50 mg/L. This result revealed that the pH significantly influenced the absorption of Cr in a test solution. The highest Cr removal was observed at pH 5, followed by pH 6, pH 7, and pH 8, respectively (Table 5). Similarly, Mitrakas *et al.* (2011) reported that complete Cr (VI) removal (40 mg L⁻¹) was achieved from a water sample at pH levels of 1–2. It indicates that the optimum pH of the biological sample is crucial for removing Cr at higher levels (Mitrakas *et al.*, 2011). Moreover, the present results revealed that the adsorption amount of Cr was reduced at higher concentrations of pH. Hence pH 5 was considered for RSM experiment.

Response Surface Methodology: 3-Level Factorial

A three-level factorial design, one of the most popular response surface methodologies, was used in the experiment's design and execution. The process begins with statistically planned experiments, after which the coefficients and outcomes of the resulting mathematical model are calculated. The design and evaluation of

**Fig. 1.** Influence of contact time on chromium absorption

experiments were conducted using Design Expert 11 (Stat Ease) software version 23.1.1.0. The formula ($N = 2k + 2k + n_0$) in the CCD was used to determine the number of tests necessary when the number of parameters is given. Where N is the total number of experiments, n_0 is the number of trials carried out at the parameter centres and k is the number of variables. This study consisted of 32 experiments in total, with the centre points being performed six times. Table 6 presents the actual values and responses of the tested parameters in the 3-level factorial. The pH (A) varied between 5 and 6 units; the adsorbent dosage (B) varied between 75 mg and 100 mg; and the contact time (C) varied between 3 and 4 hours, as indicated in Table 6. The response variables were the adsorption (mg/g). From the results, it was inferred that run 15, with a pH of 6, a dosage of 75 mg, and a contact time of 4 hours, achieved effective removal of chromium up to 256.3 mg/g (Table 6). The removal efficiency was only increased by a minimal fold. However, further experiments are needed to validate these results.

However, this model is found to be significant, as indicated by the ANOVA for the Quadratic model (Table 7). The model with a p-value of 0.001 was found to be significant, and the lack of fit was deemed “not significant,” thereby supporting the viability of this model. This model can be effectively utilized for optimizing the adsorp-

Table 5. Influence of pH on Cr adsorption

Sl. No.	pH	Adsorbate (mg/g)
1	3	155.8
2	4	189.3
3	5	216.7
4	6	190.6
5	7	186.4
6	8	172.5
7	9	158.9
8	10	132.4
9	11	129.5
10	12	119.7

Table 6. Response Surface Methodology: 3 Level Factorial

STD	RUNS	Factor 1: A pH	Factor 2: B Dosage biochar	Factor 3: C con- tact time	Response: Adsorption
32	1	5	0.08	3.5	228.5
31	2	5	0.08	3.5	230.6
20	3	5	0.075	4	148.6
13	4	4	0.08	3.5	233.5
24	5	6	0.08	4	226.5
1	6	4	0.075	3	146.8
27	7	6	0.085	4	142.6
18	8	6	0.085	3.5	199.8
14	9	5	0.08	3.5	256.3
25	10	4	0.085	4	98.6
22	11	4	0.08	4	210.3
17	12	5	0.085	3.5	186.7
11	13	5	0.075	3.5	140.8
4	14	4	0.08	3	241.3
10	15	4	0.075	3.5	145.7
21	16	6	0.075	4	156.8
19	17	4	0.075	4	149.6
16	18	4	0.085	3.5	184.7
6	19	6	0.08	3	196.9
15	20	6	0.08	3.5	248.4
29	21	5	0.08	3.5	232.1
5	22	5	0.08	3	231.5
30	23	5	0.08	3.5	229.9
26	24	5	0.085	4	160.9
2	25	5	0.075	3	121.4
7	26	4	0.085	3	189.6
12	27	6	0.075	3.5	142.9
3	28	6	0.075	3	139.7
9	29	6	0.085	3	196.4
8	30	5	0.085	3	196.8
23	31	5	0.08	4	231.7
28	32	5	0.08	3.5	235.2

STD: Standard Order (original run number)

Response: Removal
Current transform: No Transform
Current Lambda: 1.5
Recommended transform: No Transform
Lambda: 1.5

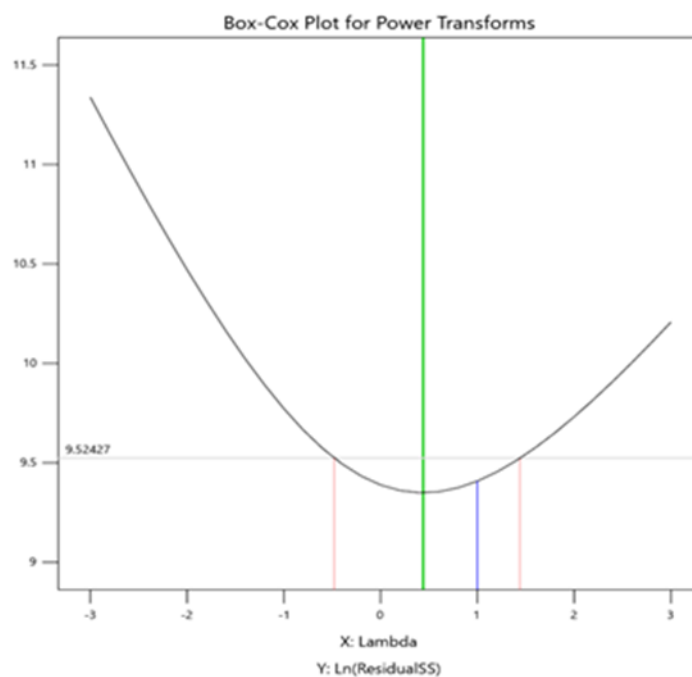


Fig. 2. Box-Cox plot for chromium removal efficiency using biochar at various parameter

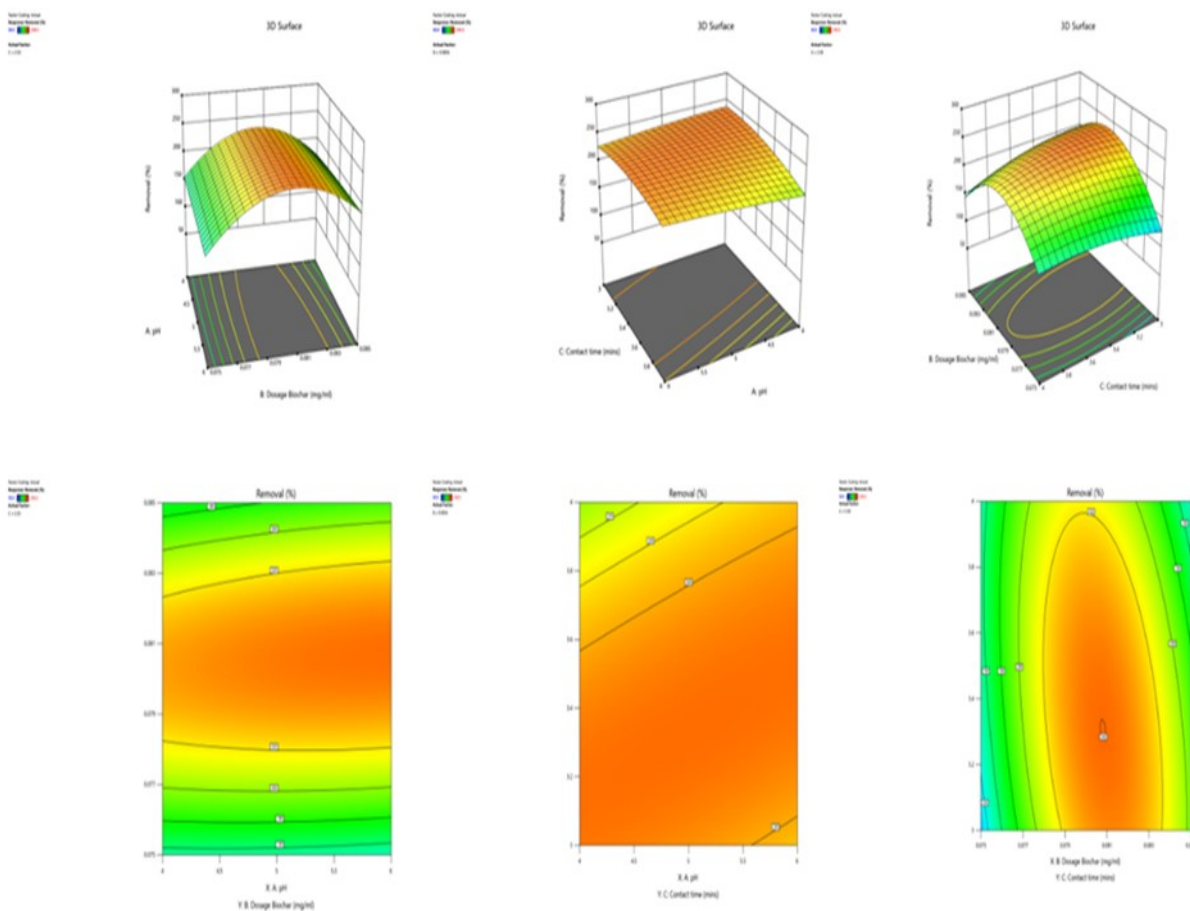
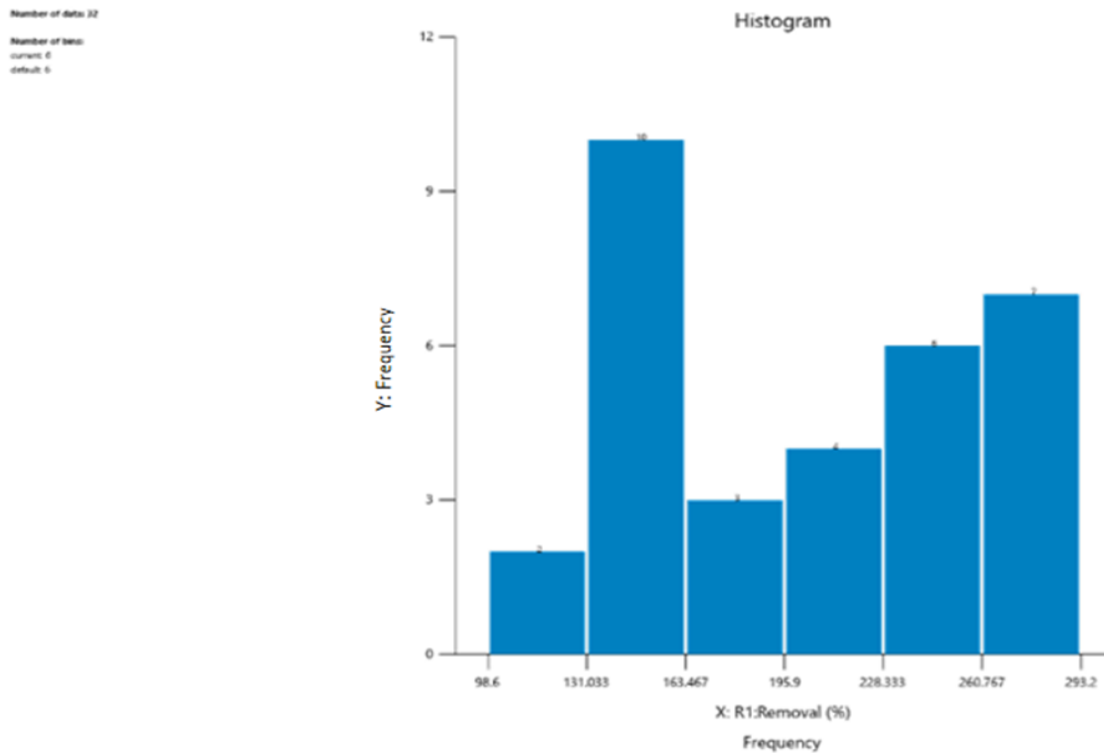


Fig. 3. Interactions of A- PH, B-Dosage and C-Contact time

Table 7. Analysis of Variance (ANOVA) for the quadratic model

Model	53692.46	9	5965.83	30.88	< 0.0001	significant
A-pH	138.33	1	138.33	0.7159	0.4066	
B-Dosage Biochar	3866.14	1	3866.14	20.01	0.0002	
C-Contact time	1009.5	1	1009.5	5.22	0.0323	
AB	392.16	1	392.16	2.03	0.1683	
AC	1047.2	1	1047.2	5.42	0.0295	
BC	4324.4	1	4324.4	22.38	0.0001	
A ²	32.97	1	32.97	0.1706	0.6836	
B ²	34155.07	1	34155.07	176.76	< 0.0001	
C ²	1177.61	1	1177.61	6.09	0.0218	
Residual	4250.95	22	193.23			
Lack of Fit	3702.32	17	217.78	1.98	0.2307	not significant
Pure Error	548.63	5	109.73			
Cor Total	57943.41	31				

Cor Total: Corrected Total Sum of Squares


Fig. 4. Histogram for chromium removal efficiency using biochar at various parameters

tion of Cr from contaminated water. Moreover, the box-cox plot (Fig. 2) also showed that the better power transforms had occurred with this model, and the lambda value lay between the red lines and closer to the optimal lines, indicating that the model exhibited the actual values were in close congruence with the predicted values.

The interactions of the various factors were depicted through counterplots and 3D plots (Fig. 3). The interaction plot was found to be very close to the optimized lane, indicating significant interactions. Simi-

larly, the interactions of pH and contact time are indicated in Fig. 4. The plots were in the nearest to optimal region, indicating their supportive interactions in achieving an efficient adsorption phenomenon. The interactions between the dosage and concentrations are depicted in Fig. 5. The area of coverage is improved, and the plots were positioned optimally. The umbrella-shaped 3D Fig. 3 indicates good interaction; however, the interactions are located away from the significant optimal areas. However, further optimization and validation of this parameter may

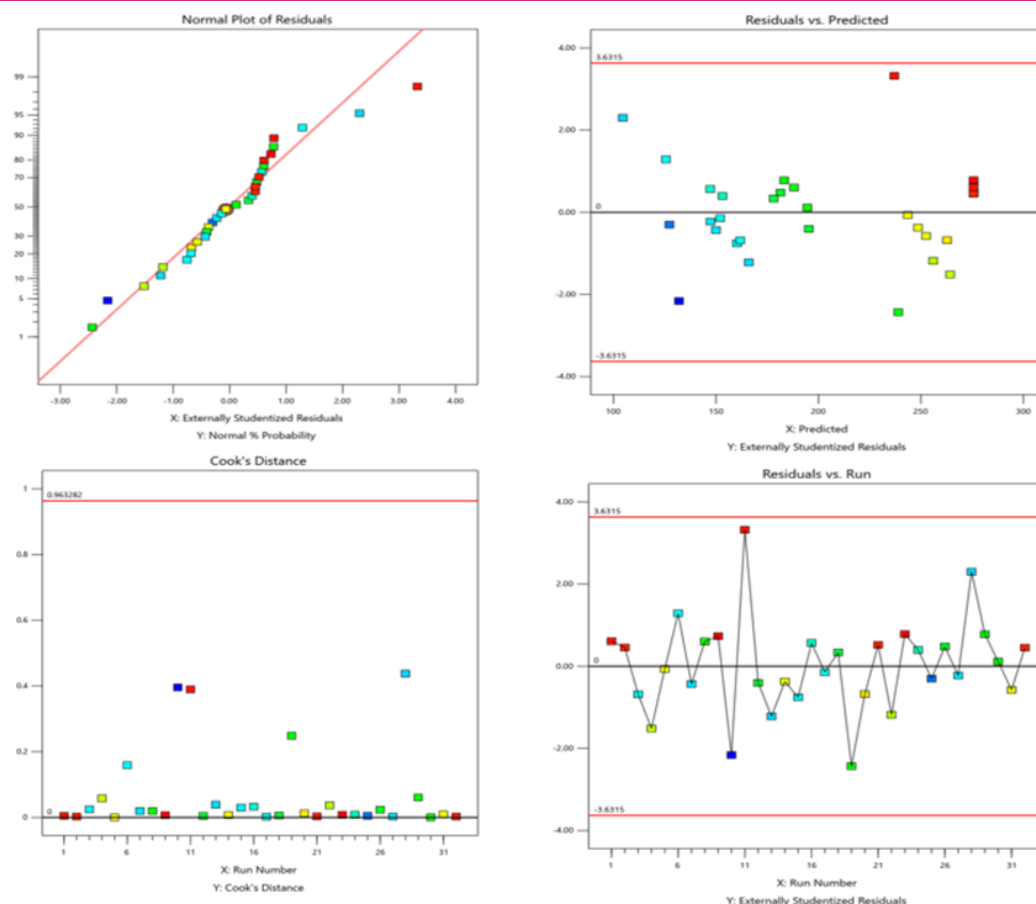


Fig. 5. Diagnostic plots for model residuals (A) Normal probability plot of residuals, (B) Residuals vs. predicted values, (C) Cook's distance plot. (D) Residuals vs. run order

help achieve the maximum removal of Cr from contaminated water.

Conclusion

Biochar is one of the promising materials for removing heavy metals from contaminated soil. The present study investigated the behaviour of adsorbents in removing Cr(VI) from chromium-contaminated water. The batch adsorption experiments in this study revealed that the Cr (VI)contaminated water removal efficiency was significantly ($p < 0.05$) influenced by initial concentration, adsorbent dosage, contact time, and pH. Optimal conditions were identified as pH 6, an adsorbent dosage of 75 mg, and a contact time of 4 hours, resulting in an impressive Cr (VI) removal capacity of 256.3 mg/g. These findings provide a practical framework for designing effective Cr(VI) removal systems for industrial wastewater and groundwater remediation, contributing to improved water quality and environmental health in contaminated areas.

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Conflict of interest

The authors declare that they have no conflict of interest.

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