Research Square

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Enhanced Chromium removal from water using ball milled nano-biochar derived from Eichhornia crassipes (Water hyacinth)

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Research Article

Keywords: Hexavalent chromium, Cr (VI) removal, ball-milled nano-Eichhornia biochar, adsorption, and water treatment

Posted Date: November 21st, 2024

DOI: https://doi.org/10.21203/rs.3.rs-5312229/v1

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Additional Declarations: No competing interests reported.

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. This 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, adsorbent dosage, contact time, and pH. The equilibrium adsorption data were fitted to different isotherm and kinetic models to analyze the adsorption mechanism and kinetics. The results indicated that BMBNBC exhibited a high affinity for Cr (VI) adsorption, with a maximum adsorption capacity of 293.2 mg/optimal operating conditions determined to achieve maximum removal efficiency, and the adsorption process was found to follow the Langmuir isotherm and pseudo-second-order kinetic model, suggesting monolayer adsorption and chemisorption as the predominant mechanisms.

1. Introduction

Due to the variable speciation, carcinogenicity, and high mobility of chromium (Cr), environmental water pollution has attracted a lot of interest (Dhal et al., 2013). The two main oxidation states of Cr are Cr (VI) and Cr (III), 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 precipitation, 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 involves the use of adsorbent materials that can attract and bind the Cr (VI) ions from the wastewater, facilitating their removal. Traditional carbon-based adsorbents, such as activated carbon, biochar, graphene, and carbon nanotubes, have shown 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 should 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 the removal of Cr (VI) from wastewater, ultimately contributing to the protection of the environment and public health.

Because of its widespread availability, plentiful manufacturing, ease of use, and affordability, biochar has acquired popularity as an adsorbent once again. Corn straw (G. Zhang et al., 2011), water hyacinth (Masto et al., 2013), rice husk (Asadi et al., 2021; C. 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 (D. Zhang et al., 2016). The production process of biochar, involving the thermal decomposition of biomass under controlled conditions, is relatively simple. 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, often known as water hyacinth (WH), is a South American native and a type of damaging weed that is found around the world (Ilo et al., 2020). Due to water eutrophication, this weed has overgrown and is posing a major threat to agriculture, human health, and the ecosystem. The water hyacinth, or *Eichhornia crassipes*, is an invasive weed species that has seriously harmed the environment because of its exceptional fertility and pace of growth (Mengesha, n.d.). It hasn't, however, been utilised as a resource yet. Utilizing this abundant biomass for biochar production serves as a sustainable approach to manage the invasive growth while producing a valuable adsorbent material. Eichhornia-biochar has gained significant attention for its ability to effectively remove Cr (VI) 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 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). In our study focussed WH is an aquatic plant that has proven effective in removing heavy metals like cadmium (Cd), lead (Pb), Cr, mercury (Hg), nickel (Ni), and copper (Cu) from contaminated water bodies. It achieves this through adsorption onto its fibrous roots, absorption into the plant tissues, and accumulation of high concentrations of heavy metals, particularly in the roots and rhizomes. Factors like pH, temperature, nutrient availability, and metal concentration affect its uptake efficiency. Phytoremediation techniques include rhizofiltration using the roots, phytoextraction by harvesting the entire plant, and use in constructed wetlands. While offering advantages like low cost, eco-friendliness, and efficiency, limitations include slow process, potential biomagnification, invasiveness, and need for proper disposal of contaminated biomass through methods like composting, incineration, or phytomining.

2. Materials and methods

2.1. Collection of plant sample

The taxonomically identified *Eichhornia crassipes* plants were collected from the Kousika River (Tirupur), Tamil Nadu, India. The collected plant material was allowed to shade dry for further process.

2.2. Production and modification of biochar

For biochar preparation, the dried WH plant material was powdered. The fined powder of WH plant was taken for pyrolysis process. For pyrolysis, powdered WH plant material kept in muffle furnace at 300°C for 2 hrs. Then the nano-sizing the biochar by using ball miller. For ball milling 1:100 ratios have been used i.e. in 1g of biochar 100 balls need to add. Stainless steel balls were used for ball milling (76.4g approx.) and the residence time is 10 hrs. (RSBM-1/060–718.Hindustan scientific). The nano-sized biochar used for further studies.

2.3. Assay for detecting chromium

2 ml of sample (containing Cr) is acidified by using 3 ml of 6N H_2SO_4,1 ml of 1M of H_3PO_4 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 blank solution. Absorbance readings of samples were corrected by subtracting 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) (Balasubramanian & Pugalenthi, 1999). The details of calibration curve were presented in Fig. 1 and Table 1.

Table 1
Standard chromium assay using DPC

Concentration (µg)	OD
0	0
10	0.1757
20	0.3254
30	0.5324
40	0.7566
50	0.9666

2.4. Batch experiment

Adsorption isotherms were applied to explain the equilibrium adsorption characteristics. For this study two different experiments were conducted. The isotherm experiment to know about the equilibrium concentration and the kinetics to know about the influence of time for the adsorption to carryover. Briefly, to study on these experiments were conducted using varying concentrations of Cr (10-70mg/L) and fixed concentration of biochar (75mg) under different contact time. After experimentations, the Cr remained in the solution were estimated by DPC assay (Lace et al., 2019).

2.5. Experimental design and optimization

2.5.1. Concentration of adsorbent

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

2.5.2. Influence of pH

Solution contain Cr (50 mg/L) and fixed concentration of biochar (adsorbent − 75 mg) was tested against the varying pH ranged from 3 to 12. Experiment carried out in orbital shaker at 150 rpm for 2 hours. At the end of the experiment the chromium was estimated using DPC method (Sanchez-Hachair & Hofmann, 2018).

2.5.3. Contact time

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

2.6. Response surface methodology

A statistical method called Response Surface Methodology (RSM) is used to model and optimise 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 carried out at certain locations within the design space with the aim of mathematically modelling the observed responses. The response is then predicted using the model, which is also used to determine the best collection of predictor variables to utilise in order to get the intended answer (Myers et al., 2016). This experiment was designed using Design expert software version 11 (Stat Ease). One of the most used RSM procedures is CCD, which is a sequence of tests carried out at particular locations in the design space with the aim of fitting a quadratic model to the observed answers. RSM is a potent instrument for process optimisation and creating new goods. It enables scientists and engineers to methodically investigate the link between predictor factors and response variables and to decide which set of predictor variables is most effective for getting the desired answer. RSM is a significant method for increasing productivity, lowering costs, and raising product quality in a variety of scientific and technical applications (Khuri & Cornell, 2018). Three factors have been taken for optimization pH, dosage, and the contact time. Three levels have been taken for each factor for PH unit the lower is 4, higher is 6. For contact time the lower is 3 hrs, middle and higher is 4 hrs. for dosage concentration the lower is 75mg, higher 85mg. These values have been 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 has been estimated using DPC assay and was calculated and fed in the Response (mg/g). The quadratic function was fitted to correlate the relationship between variables and response for determining the optimal point. The coded equation is followed by, Adsorption = A+*B+*C-*AB+*BC+*A2-*B2-*C2: the parameters values are mentioned in Table 2.

Table 2
Parameters of coded equation

Removal	**-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
pH²	-2.14677
Dosage Biochar²	-2.76E + 06
Contact time²	-51.32043

2.7. Statistical analysis:

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

3. Results and discussions

3.1. Adsorption of Cr

Hexavalent Cr in a known concentration (50mg/L) is made in the water for adsorption experimentations and used unless and otherwise stated. The standard calibration curves of Cr (10–50 µg) were used and assayed spectrophotometrically using DPC method. From the calibration curves (Table. 1 and Fig. 1), we obtained the slope value (0.0194) and intercept (0.025). These calibrated values were used completely throughout the experimentation in measuring the unknown Cr in all reactions.

3.2. Batch experimentation

The Cr adsorption was studied using a batch adsorption process wherein in isotherm study the adsorbent at a concentration of 75mg was added to different Cr concentrations ranging from (10-70mg/L) for 24hrs. The results showed that the equilibrium concentration was achieved at the Cr concentration of (50mg/L) after which the Cr uptake found to decline. The highest Cr absorption was observed at 50mg/L and it is followed by 60 and 70 mg/L. The detailed results of this are depicted in Table 3. These results showed that the adsorbent at a concentration of 75mg very effective for removal of high level of Cr in water samples.

Table 3
Batch Experimentation-Adsorption Isotherm

Initial concentration	Final concentration	qe = ci-ce*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

Table 4
Batch adsorption experimentation – Kinetic model

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

Further another batch adsorption study assessed the adsorption of 75mg of biochar against the fixed concentration of Cr (50mg/L) for different time intervals from 0th minute to 180 minutes for Kinetic model. The results indicated that the contact time of 90 minutes promoted maximum removal of Cr (198.4 ng/g) and it is followed by 60 minutes (158.9 mg/g). Therefore, this result revealed that the contact time is an essential parameter for removing the higher level of Cr in water sample.

3.3. Various parameters influencing the adsorption of Cr (VI)

3.3.1. Influence of Adsorbent concentration

The adsorbent dosage is one of the important parameters for improving the efficiency of Cr (VI) adsorption (Akram et al., 2017). Therefore, optimization of adsorbent dosage is very crucial steps for removal Cr in water samples. In this study, the influences of adsorbent concentration were studied with the varied range of adsorbent (WH biochar) concentration from 25 mg -125 mg was analysed. The results revealed that the adsorption was maximum at adsorbent (WH biochar) concentration of 75 mg/L and the removal of Cr was found to be 241mg/g and it is followed by 25, 50 mg/L respectively (Table 5). Therefore, this result indicate that the optimal concentration of adsorbent is significantly influence the higher level of Cr removal from the test sample. Similarly, many researchers are successfully determined the optimum concentration of biochar for better absorption of Cr in biological water sample (Tytłak et al., 2015).

Table 5
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

Table 6
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

3.3.2. Influence of contact time

Many parameters control the Cr adsorption process in biological samples. The contact time is one of the critical parameter that significantly influence the adsorbate adsorption by the adsorbent (Jain et al., 2010). In this study, the influence of contact time in Cr removal was studied from 0th to 12th hour using Cr at 50mg/L and biochar at a dosage of 75mg. The results indicated that there was increased removal of 147mg/g during 4th hour after which the adsorption was declined (Fig. 2). But again, there found a hike in adsorption around 8th hour. The Fig. 2 showed the effect of contact time can influence the removal of Cr (VI) from test sample. Therefore, the 4th hour is considering as the assessment level in RSM. Recently,(Masuku et al., 2023) has been reported that the contact time of 20–80 min, a steady increase in the uptake of Cr (VI) by the adsorbent is observed. These findings are supported in our present study.

4. Influence of pH

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

Response surface methodology: 3 Level Factorial

Three-Level Factorial, which is one of the most popular response surface methodologies, was used in the experiment 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 experiments were carried out using the file version 23.1.1.0 of Design Expert 11 (Stat Ease) software. The formula ($N = 2k + 2\ k + n0$) in the CCD was used to determine how many tests are necessary when the number of parameters. Where N is the total number of experiments, n0 is the number of trials carried out at the parameter centres and k is the number of variables. This study had 32 experiments in all, with the centre points being performed six times. Table 7 provides the actual values and response 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 7. The response variables were the adsorption (mg/g). From the results, it was inferred that run 15 with the factor pH 6, dosage 75 mg and contact time 4 hrs gave the effective removal of chromium up to 256.3mg/g (Table. 7). The removal efficiency was only increased minimum fold. But further experiments need to be conducted to validate these results.

Table 7
Response Surface Methodology: 3 Level Factorial

STD	RUNS	Factor 1: A pH	Factor 2: B Dosage biochar	Factor 3: C contact 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

STD	RUNS	Factor 1: A pH	Factor 2: B Dosage biochar	Factor 3: C contact time	Response: Adsorption
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

Table 8
Anova for 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□	32.97	1	32.97	0.1706	0.6836	
B□	34155.07	1	34155.07	176.76	< 0.0001	
C□	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				

However, this model is found to be significant as from the Anova for Quadratic model (Table.8). The model with 0.001 was found to be significant and the lack of fit as “not significant” the viability of this model. This model can well be utilized for optimizing the adsorption of Cr from contaminated water. Moreover, the box – cox plot (Fig. 3) also showed the better power transforms had happened with this model and the lambda value lied in between the red lines and nearer to the optimal lines, denoting the model exhibited our actual values were in close congruence with the predicted values.

The interactions of the various factors were depicted through counter plots and 3D plots (Fig. 4). The interaction plot was found very nearer to the optimized lane indicating their significant interactions. Similarly, the interactions of pH and contact time is indicated in Figure.5. The plots were in the nearest to optimal region indicating their supportive interactions to achieve the efficient adsorption phenomenon. The interactions between the dosage and concentrations are depicted in figure. 6. The area of coverage is better, and the plots were in optimal positions. The umbrella shaped 3D Fig. 4 indicates the good interaction; however, the interactions are away from the significant optimal areas. However, further optimization and validation of this parameter may help to achieve the maximum removal of Cr from the contaminated water.

Conclusion

this study's batch adsorption experiments revealed that Cr (VI) removal efficiency is significantly 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 valuable insights for developing effective strategies to remove chromium from contaminated water samples, addressing a critical environmental and public health concern. The optimized parameters offer a practical framework for designing Cr (VI) removal systems in various settings, from industrial wastewater treatment to groundwater remediation. This research not only presents a robust solution for Cr (VI) removal but also contributes to the broader field of water treatment and environmental remediation, potentially leading to improved water quality in affected areas.

Declarations

Acknowledgement

We sincerely thank JJ college of arts and science, Pudukkottai, Tamil Nadu for providing the research facilities and support.

Authors Contribution

Mohanadevi conceptualized and wrote the manuscript. Dhanabalan critically revised the manuscript for publication.

Funding

There is no found provided

Conflicts of interests: The authors declare no conflict of interest.

Consent for publication

All authors have agreed to the publication

Data availability

Data will be made available on request.

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Figures

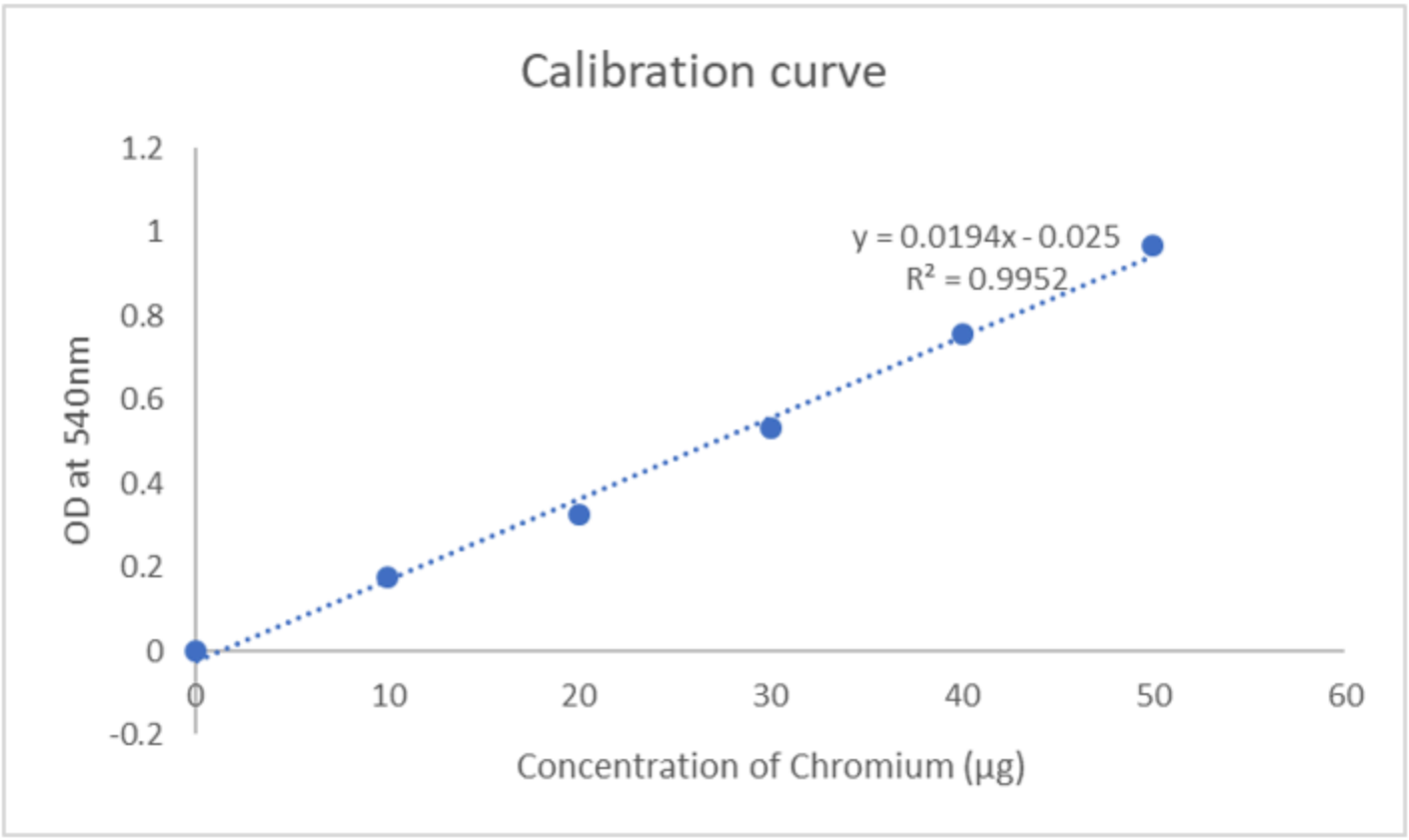


Figure 1

Calibration curve

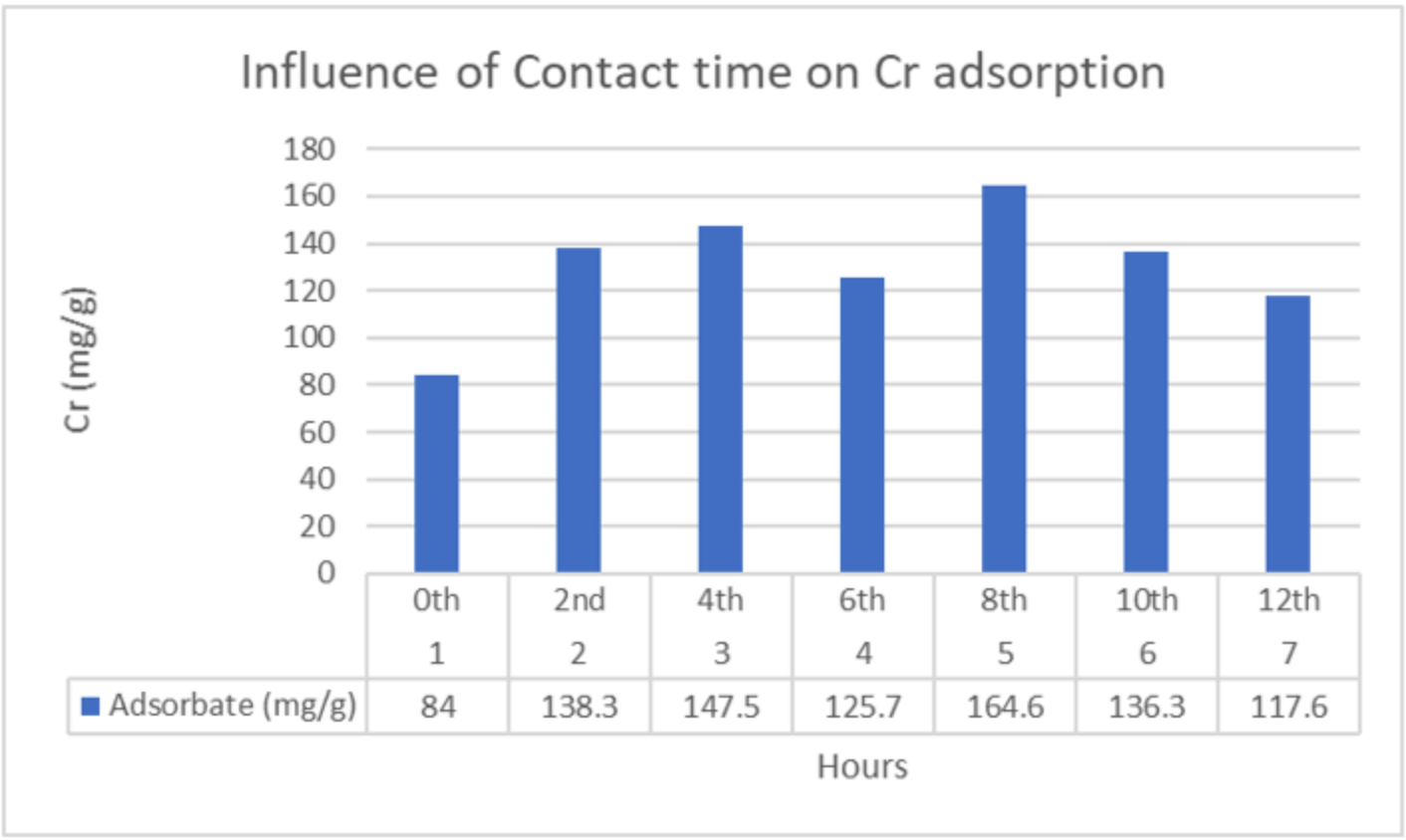


Figure 2

Influence of contact time

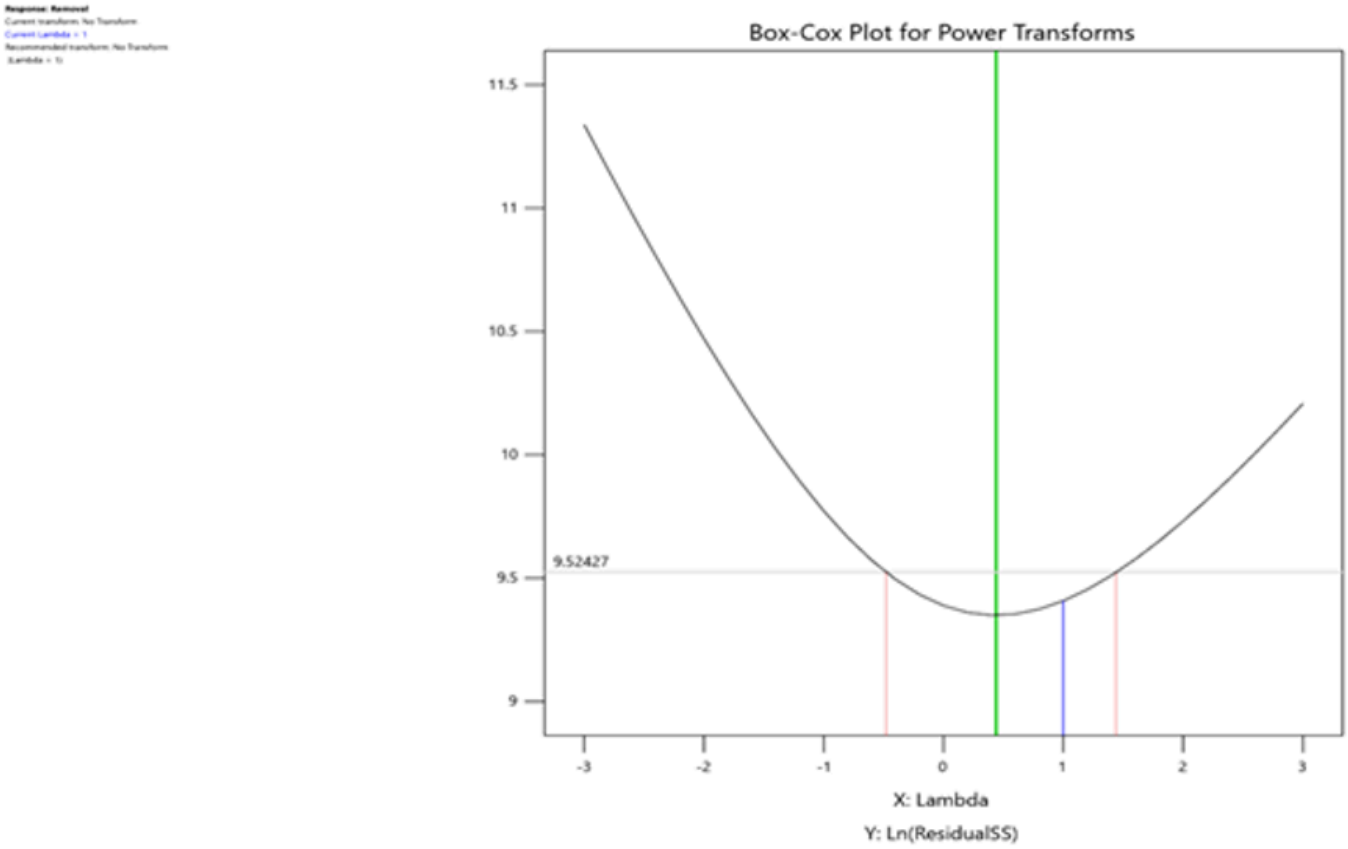


Figure 3

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

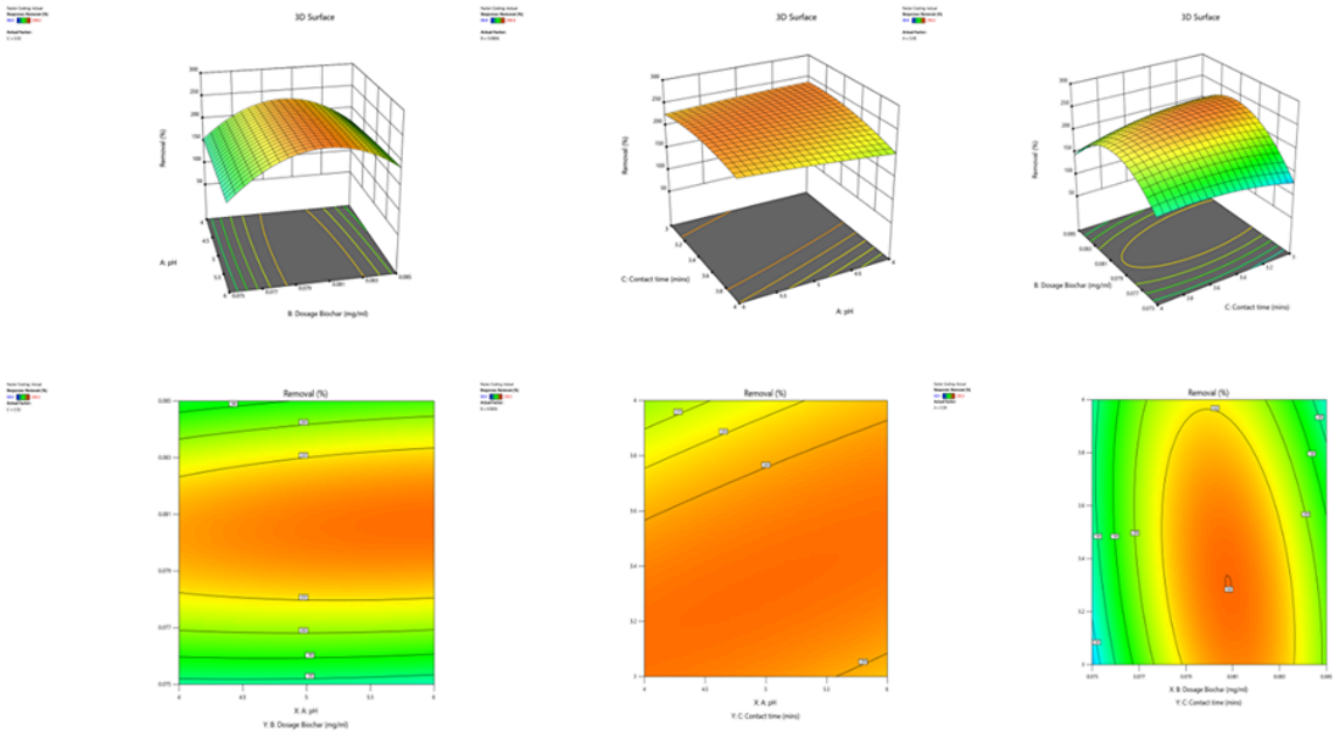


Figure 4

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

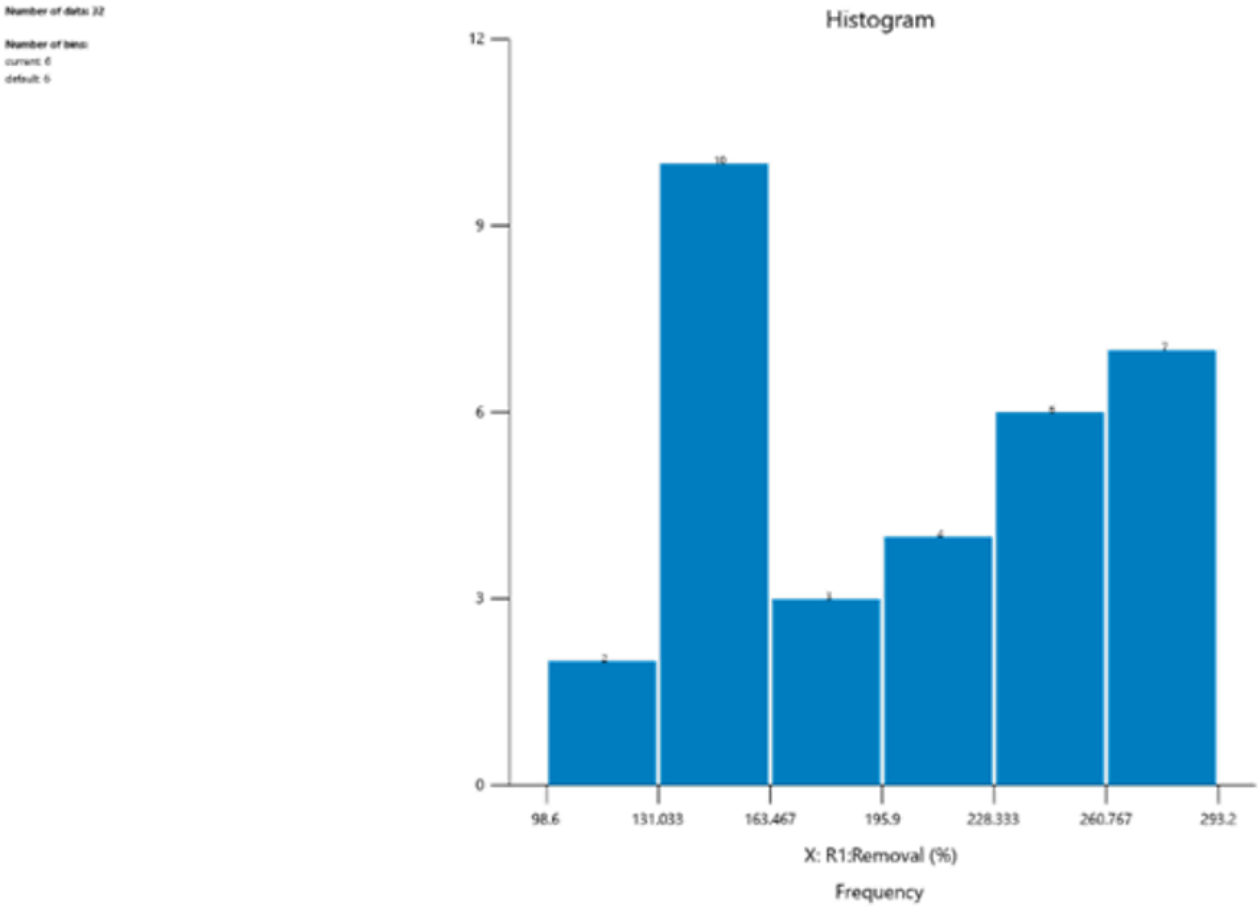


Figure 5

Histogram for chromium removal efficiency using biochar at various parameter.

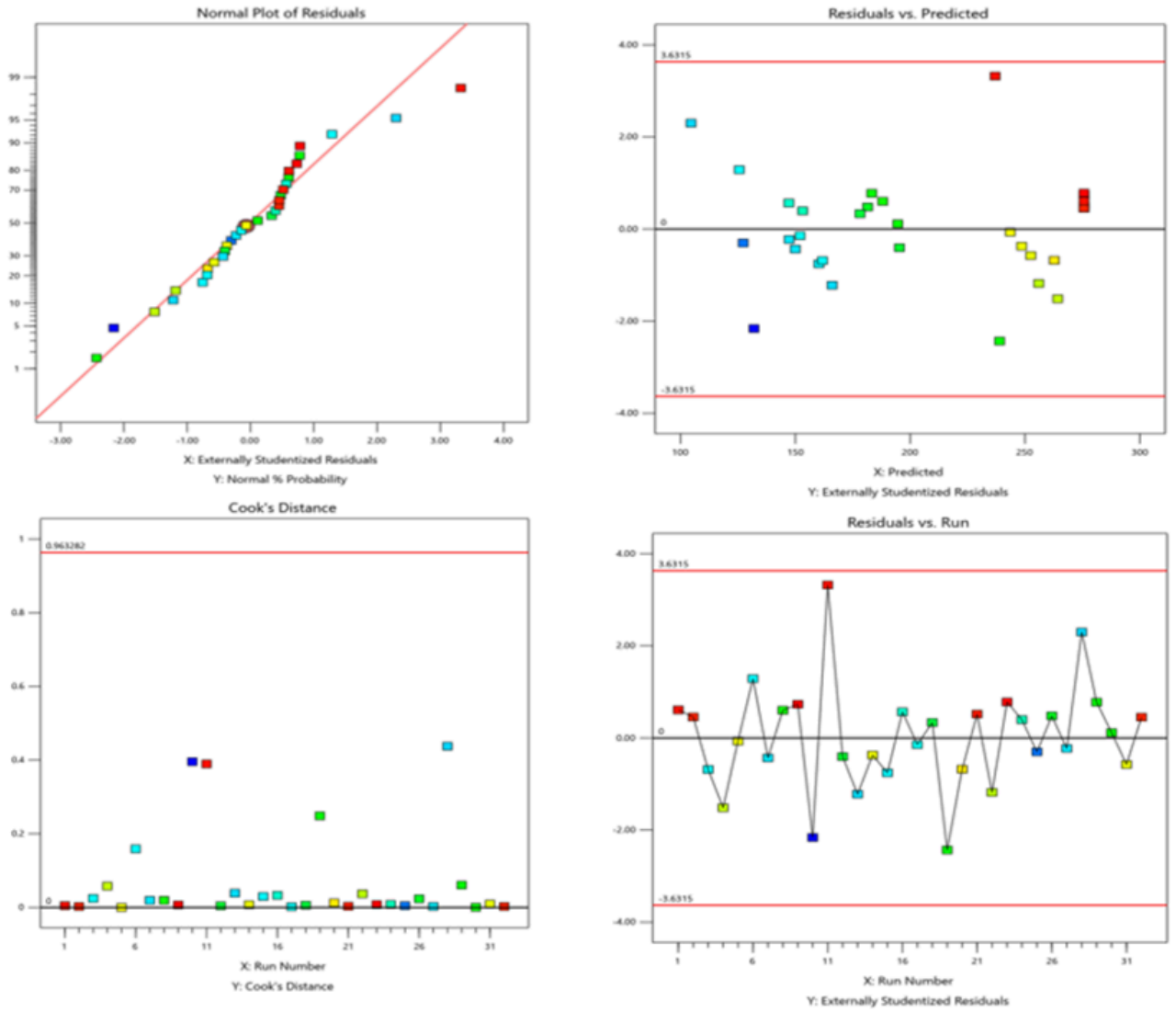


Figure 6

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.