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Research Article

Exclusion of Chromium(VI) Ion in Grueling Activated Carbon Fabricated from *Manilkara zapota* Tree Wood by Adsorption: Optimization by Response Surface Methodology

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The current paper makes obvious the elimination of chromium(VI) ion, from wastewater via adsorption technique with activated carbon generated from *Manilkara zapota* tree (MZTWAC). Preliminarily MZTWAC has undergone characterization studies which uncovered the suitability of MZTWAC to expel chromium(VI) from aqueous solution. Batch adsorption experimentation was premeditated with the competence of central composite design (CCD) and it was executed. Response surface methodology (RSM) was the key optimization software to appraise the adsorptive chattels of MZTWAC engaged in removing chromium(VI) ion in aqueous solution which explored the interactions flanked between four expounding variables explicitly initial concentration of chromium(VI) ion, pH of the solution, MZTWAC dose and time of exposure, and contact time. The response variable that was concentrated in the study was adsorption capacity. It was deduced a polynomial in quadratic equation was documented amid the adsorption capacity and variables influencing the adsorption with $R^2 = 0.9792$ which was projected as the best suit for the adsorption process. ANOVA that is expanded as analysis of variance judged the connotation of adsorption process variables. 0.2 g of MZTWAC dosage has removed 87.629% chromium(VI) from aqueous solution. The enhancement of adsorption process reclined on the attainment of maximum adsorption capacity which further depends on the optimization of variables under consideration. This criterion was accomplished by the desirability function optimizing the process variables.

1. Introduction

Rapid increase in population and modern innovative technology and its progression guide environment towards the polluted scenario reported during the history of the last few decades [1]. Natural resources are extensively used and exploited nowadays abruptly deteriorating the atmosphere and thereby the lives on the earth in turn. The metals like chromium, copper, lead, and arsenic are naturally occurring objects whose usage increases randomly in many industrial operations. These metals are universally accepted as heavy metals which hold a 5 g/cm^3 specific density more than that of water. Though these metals are inherent to the functioning of the physiological activities for the humans, its addition more than that required adversely distresses the surroundings and livelihood of the organisms [2]. Ejection of effluents domestically and industrially has provoked in superior altitudes of heavy metal attentiveness in resources of water [3]. Amid different metal ions, chromium(VI) is the most frequently used and present in the surroundings and very perilous to the ecosystem. Exposure to chromium(VI) and health hazards go hand in hand with each other. Health issues like abdominal cancer [4, 5] and skin problems are inferred due to exposure towards chromium(VI) [6, 7]. The chromium(VI) concentration necessity is to be below 0.1 mg/L [8, 9] which is prescribed according to USEPA (U.S. Environment Protection Agency) Bureau of Indian Standards (BIS) that is highlighted in El-Bestawy et al. in the year 2013 and by Sharma in the year 2009. Environmental pollution is indebted to chromium mainly manifested in many industries that manufacture leather products, manufacturing electronic devices, and industries that engage in operations making inherent use of metals [10, 11]. Every researcher is inquisitive in their research to exclude the toxic metal from wastewater by customary treatment practices such as precipitation of chemicals [12, 13], coagulation, ion exchange [14], adsorption [15, 16], solvent extraction [17], foam flotation, and electrodialysis [18]. Conversely, adsorption process is the proficient, cost-effective, and green technique distinctively in removing heavy metal ions from wastewater. Many adsorbents were utilized so far in extracting chromium from the polluted water. One among them is groundnut shell powder reported in the year 2019 by Jonas et al. Commercial activated carbon is in usage worldwide for the deletion of heavy metals from the effluent expelled from industries. Since the expenditure of its use is in elevated side, researchers are striving to uncover economical sources for preparing the adsorbents. As a consequence, the main extent of the current paper is meant at uncovering a novel adsorbent. *Manilkara zapota* tree [19] which is commonly known as sapodilla, a fruit-producing tree which is profusely grown in Indian nation, may possibly be employed as an alternating adsorbent. Existing study gave attention to synthesis of derivative, an activated carbon from *Manilkara zapota* tree wood treated with citric acid [19] to treat chromium(VI) ion-polluted water batch adsorption mode. Wood pieces of *Manilkara zapota* tree wood [19, 20] were gathered. Optimum conditions for process variables, for instance, initial concentration of chromium(VI), pH, contact time of the experiment, and MZTWAC (adsorbent) dosage, were tested through a rotatable central composite design under response surface methodology with support of software version 7, namely, Design Expert.

2. Materials and Methods

Activated carbon was prepared from *Manilkara zapota* tree wood. The process remained encrypted in the authors' original article [20]. To get to know the presence of functional groups that favour chromium(VI) ion adsorption and to have an idea on the surface morphology, Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) analysis were performed.

2.1. Experimentation. Optimize the process variables of the least experiments by employing the response surface methodology (RSM) technique. The main intent here is in the direction of upgrading the response on which the chosen process variables have much impact. It too computes the association amid the chosen inputs and the response. A set of advanced design of experiment (DoE) techniques is said to be a response surface design that helps to understand better and to optimize the response. The design procedure [21] for RSM follows four stages as listed below.

(i) Experimental design for satisfactory and trustworthy extent of the response by central composite design

(ii) Emergence of the second-order mathematical response model by means of the best fit

(iii) Establishing the optimal solution for investigational variables producing a maximum response

(iv) Two- and three-dimensional (3D) charts are used to represent the collaborative impacts of process factors

The requisite experimental figures are reliant on the elected design in central composite design [22, 23]. CCD gives ample facts and figures as a multilevel factorial which entails considerably littler experiments more than a complete factorial that are satisfactory to pronounce the majority of steady-state course responses. The factors (variables) investigated were initial concentration, pH, MZTWAC (adsorbent), and interaction time, with adsorption capacity as response. The objective of this current study is to see how the factors interact, and a four-factor second-order polynomial equation was employed in the determination of those statistics. Batch adsorption process is affianced in adsorbing chromium(VI) ion from wastewater. As a result, the process design is more solidified. Table 1 lists the variables and investigational values utilized in the rotatable CCD.

As a result, the design of the process has become more defined. In a summary, the variables and experimental settings utilized in the rotatable central composite design are listed in Table 1 (CCD).

2.2. Batch Adsorption Studies. To build up a 1000 mg/L concentration of chromium(VI) stock solution, dissolve 2.89 g of $K_2Cr_2O_7$ in 1 L of distilled water [24, 25]. Dilution with distilled water is used to create any remaining solutions. Batch adsorption mode experiments were carried out for the specified time duration at a constant rpm using a shaker (manufactured by Remi). Batch adsorption investigations were conducted with chains of conical flasks containing 100 mL chromium(VI) solution. At ambient conditions, the reaction of concentration, pH, adsorbent dosage, and contact time were investigated, culminating in filtration using Whatman filter paper no. 41 [16], and filtrate was stacked to determine the equilibrium concentration of chromium(VI) with the support of a UV/Vis spectrophotometer.

Table 1: Chosen process variables.

Variable	Unit	Low value	High value
Initial chromium(IV) ion concentration (*A*)	mg/L	40	60
pH (*B*)	Not applicable	4	6
Adsorbent dose (*C*)	g	0.2	0.4
Contact time (*D*)	min	60	80

The proportion of chromium(VI) removed was calculated using the equation below [26].

$$\%\text{removal of chromium(VI) ion by adsorption} = \frac{(C_0 - C_t)}{C_0} \times 100, \quad (1)$$

where C_0 denotes the initial chromium(VI) content in mg/L and C_t denotes the chromium(VI) equilibrium concentration in milligrams per litre. The following equation [23, 27] was utilized to compute adsorption capacity of the adsorbent:

$$q_e = \frac{(C_0 - C_t)W}{V}, \quad (2)$$

where q_e represents the quantity of chromium(VI) adsorbed in mg/g, V denotes the solution volume represents in L, and W denotes the adsorbent weight in grams.

3. Results and Discussion

3.1. Properties of MZTWAC. The SEM exhibited in Figure 1 MZTWAC reveals the availability of irregular and porous surface that facilitates the adsorption process to take place [20]. From Figure 2 [19], it is visible that MZTWAC possessed broad peaks, -OH stretching from hydroxyl to phenolic groups [28–30], C-H stretching of aliphatic acids, C-O stretching of alcohols [28, 29], and C-O stretching of carboxylic acid [7] and alcohols.

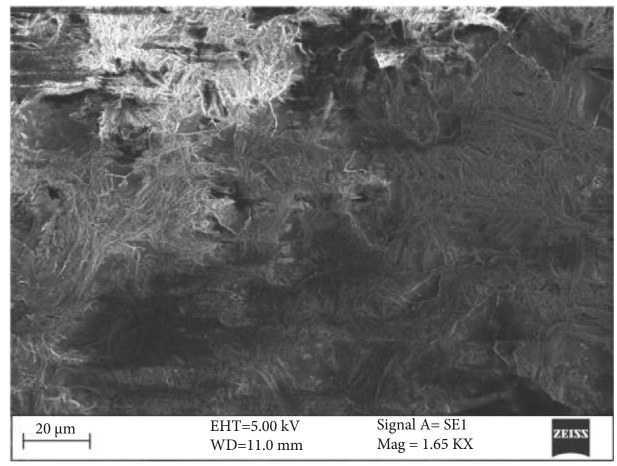

Figure 1: SEM image of MZTWAC (source: [19]).

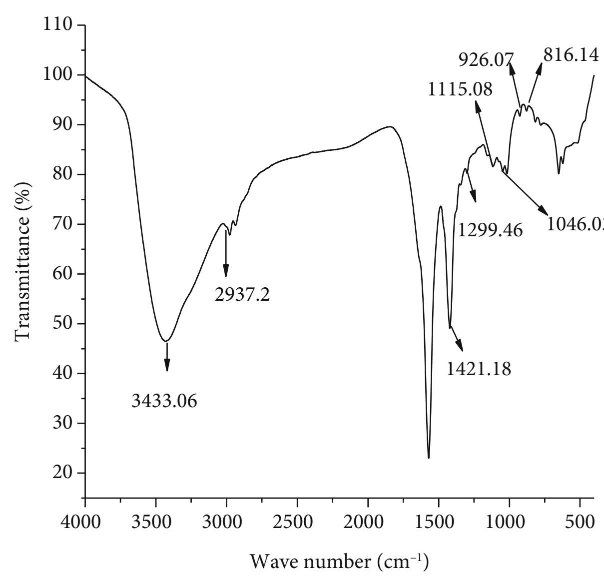

Figure 2: FTIR range of MZTWAC (source: [19]).

3.2. Experimental Design and Model Development

3.2.1. Mathematical Model Development and Design of Experiments. The mathematical-statistical quadratic model was assessed by analysis of variance (ANOVA). Critical parameters taken into account have been experimented for their optimized values in adsorption which were figured using the models that may fit and the same were validated. The current experimental work hired fourteen experiment A, B, C, D, AB, AC, AD, BC, BD, CD, A^2, B^2, C^2, and D^2, where A is the beginning concentration, B is the pH, C is the adsorbent dosage, and D is the contact period. At this point, the Design Expert has a variety of modeling options for fitting the response, including linear, two-factor interaction (2FI), quadratic, and cubic polynomials, and the evaluation was done to confirm the best model fit based on the R^2 value obtained from sequential model sum of squares. Table 2 is in charge of compiling a model evaluation summary. The programme selected the optimal model for the research project. When compared to other models, the quadratic model was suggested, while the cubic model was aliased; thus, it must not be used.

3.3. Validation of Quadratic Model. Multiple regression analysis was applied to figure out the coefficients of quadratic model that was intended to fit the results. The noteworthy connotation of autonomous process parameters was studied with 29 batch experiments. From Table 3, the model's F value of 1292.46 entailed that the model is

Table 2: Summary data for various models.

Source	Std. dev.	R^2	Adj. R_2	Pred. R^2	PRESS	Results
Linear	2.69	0.836784	0.805696	0.732741	248.4826	
2FI	3.01	0.853407	0.755678	0.574057	396.0193	
Quadratic	0.26	0.999211	0.998206	0.991994	7.44358	Suggested
Cubic	0.16	0.999856	0.999281			Aliased

Table 3: ANOVA table.

Source	SS	df	MS	F value	p value Prob > F	Inference
Model	928.97	12	77.41	1292.46	<0.0001	Significant
A	104.38	1	104.38	1742.61	<0.0001	
B	4.39	1	4.39	73.35	<0.0001	
C	392.60	1	392.60	6554.58	<0.0001	
D	6.48	1	6.48	108.23	<0.0001	
AB	1.25	1	1.25	20.87	0.0005	
AC	8.87	1	8.87	148.05	<0.0001	
AD	3.12	1	3.12	52.01	<0.0001	
BD	1.71	1	1.71	28.51	0.0001	
CD	1.27	1	1.27	21.19	0.0005	
A^2	0.36	1	0.36	6.08	0.0284	
B^2	1.02	1	1.02	17.11	0.0012	
C^2	126.41	1	126.41	2110.47	<0.0001	
Residual	0.78	13	0.06			
Lack of fit	0.64	8	0.08	3.0	0.1197	Not significant
Pure error	0.13	5	0.03			
Cor. total	929.75	25				

significant. There is only 0.01% chance that an F value is large, and this could occur due to noise. Values of "Prob > F" less than 0.0500 indicate the model terms are significant. Here, A, B, C, D, AB, AC, AD, BD, CD, A^2, B^2, and C^2 are significant model terms. The rest of the source term values, which are larger than 0.1000 signpost, are not noteworthy and are deliberated as eliminating terms. The "lack-of-fit F value" entails the lack of fit is not significant.

In addition, the ANOVA in Table 4 is supplemented by a variety of indications provided by the Design Expert. Low experimental variance is shown by the standard deviation of 0.247295. The 0.9955 "Pred. R^2" and the 0.9984 "Adj. R^2 " have an excellent correlation, with a difference of less than 0.2. The tolerable precision of 163.9238 indicates that the signal is viable. The signal-to-noise ratio is measured by "Adeq. precision," and a number greater than 4 is preferable. In summary, the R^2 statistics are excellent since they are near to 1, implying that the regression model offers a substantial connection amid the observed and anticipated values. As a result, this model may be utilized to traverse the design.

The second-order polynomial equation is derived based on RSM results. Equation (3) shows how a particular component and the interaction between the four independent process parameters impacted the adsorption process [22].

$$\begin{aligned}\text{Adsorption Capacity } Y = &\ 12.200 \\ &+2.895 \ *A \\ &-0.598 \ *B \\ &-5.219 \ *C \\ &+0.630 \ *D \\ &\ 0.241 \ *A*B \\ &-0.752 \ *A*C \\ &+0.621 \ *A*D \\ &-0.490 \ *B*D \\ &-0.417 \ *C*D \\ &+0.282 \ *A^2 \\ &-0.199 \ *B^2 \\ &+2.709 \ *C^2.\end{aligned} \tag{3}$$

Table 4: Post-ANOVA statistics.

Std. dev.	0.247295	R^2	0.99919668
Mean	14.91756	Adj. R^2	0.99845516
C.V. %	1.657743	Pred. R^2	0.99554983
PRESS	4.40417	Adeq. precision	163.923897

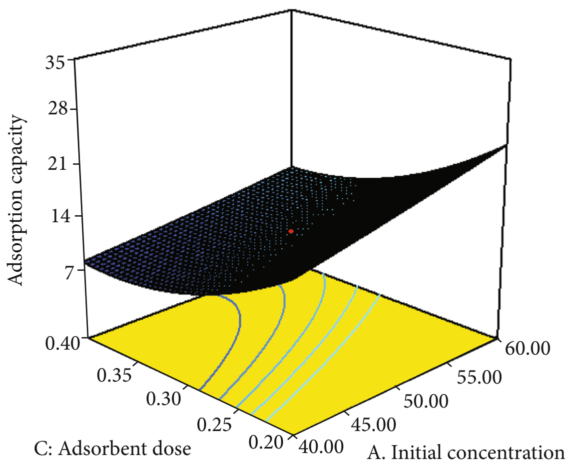

Figure 3: Upshot of interface connecting pH and the initial concentration of chromium(VI).

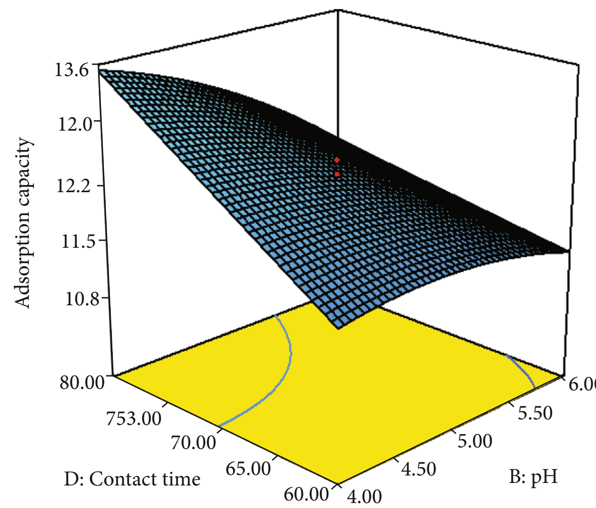

Figure 4: Upshot of interface connecting MZTWAC (adsorbent dose) and the initial concentration of chromium(VI).

Equation (3), when used with coded variables, may predict the response for different degrees of each element. Obviously, the process variables' higher levels are entered as +1, and the variables' low degrees are coded as -1. When looking at the factor coefficients, the coded condition is expedient for detecting overall influence of variables. Negative and positive coefficients show how considerable metal particles are ejected in a negative or positive way. An optimistic consequence of a factor indicates that response improves as the factor level increases, whereas a negative effect indicates that the reaction does not improve as the factor level increases. As the circumstances show, starting concentration and contact duration have a beneficial impact on chromium(VI) retention, but pH and adsorbent percentage have a negative impact. As a result, as IC increased, so did adsorption ability. In any instance, when the pH and adsorbent fraction increased, the adsorption rate lessened. The relapse equation is graphically shown in the 3D response surface plots shown below (Figures 3–5). The adsorption limit widened as the underlying fixation was amplified from 40 mg/L to 60 mg/L and the pH declined from 6 to 4, as shown in Figure 6. The addition of the adsorption limit was suggested by the high initial fixing of initial concentration and the low pH values. The adsorption limit grew in lockstep with the experiment, even if not as dramatically (Figure 4). Similarly, a large adsorbent dosage did not support the adsorbent's adsorption limit, as shown in Figure 4. Low pH and long contact duration, as seen in Figure 5, resulted in a remarkable adsorption limit for the MZTWAC.

3.4. Capability of Model. Analytic plots, with predicted versus definite and normal probability plot that has studentized residuals, are used to assess the adequacy of the numerical model and to analyse the relapsing model's sufficiency. Figure 6 depicts the association amid the actual and projected adsorption limits. The graph demonstrates proclivities in straight relapse fit, and the model effectively describes the trial run under consideration. Furthermore, it establishes a strong link between the data gathered through testing and the predictions made by the regression model meant for the chromium adsorption process (VI).

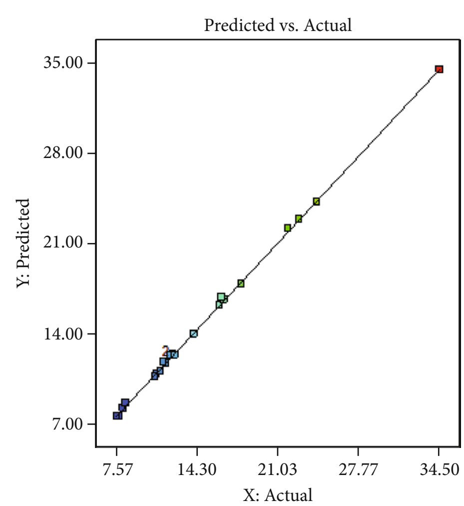

Figure 5: Upshot of interface connecting pH and the contact time.

The normal probability plot (Figure 7) is also regarded as the most appropriate graphical method for verifying and

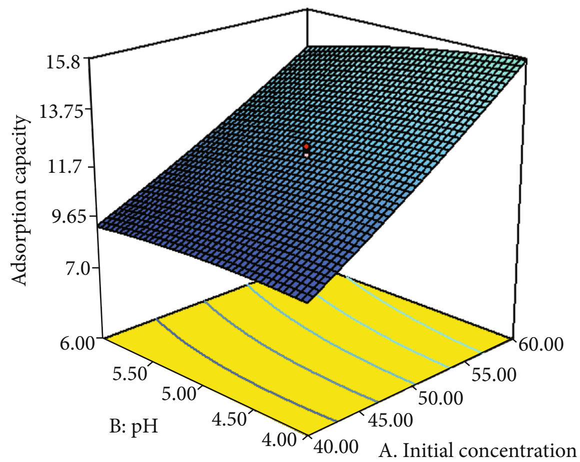

Figure 6: Interface connecting the actual and anticipated figures of adsorption capacity.

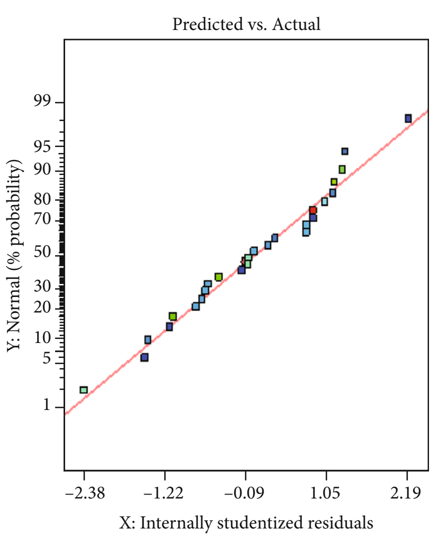

Figure 7: Normal probability design.

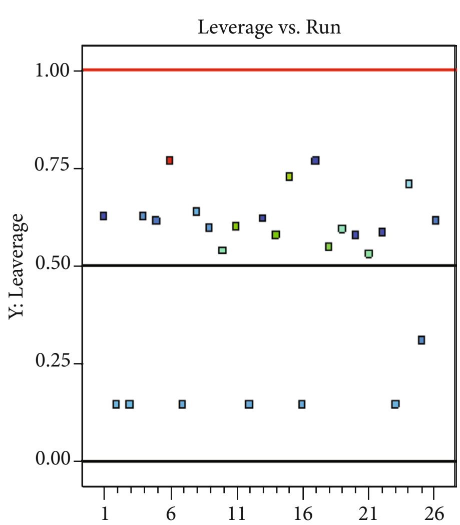

Figure 8: Leverage vs. run.

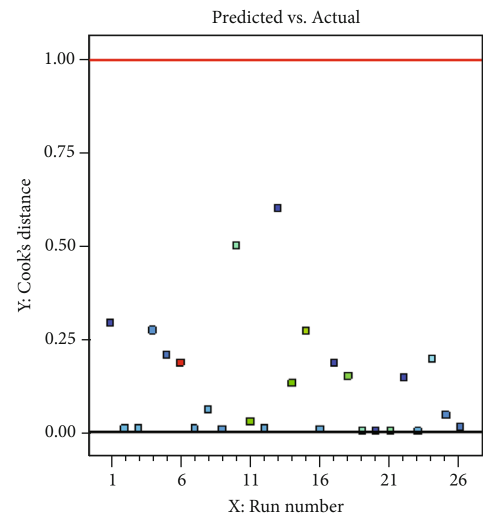

Figure 9: Cook's distance.

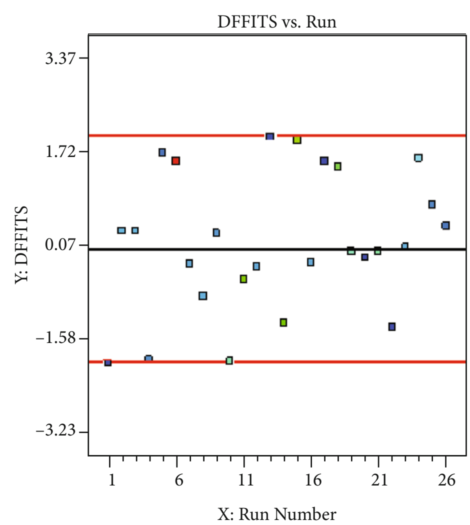

Figure 10: DEFFITS vs. run number.

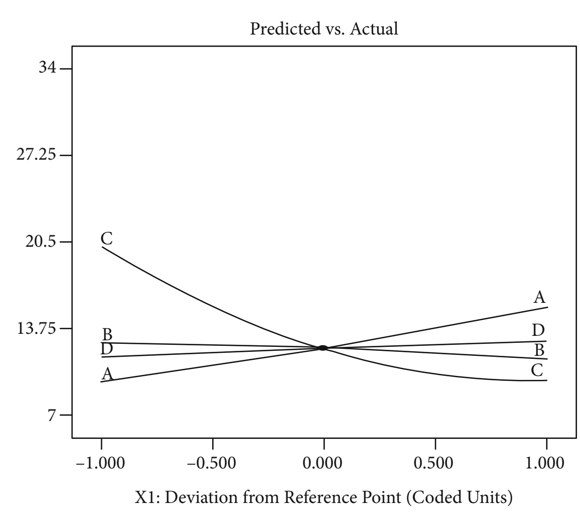

Figure 11: Perturbation plot.

Table 5: Numerical optimization report showing optimized predicted adsorption capacity and calculated %removal of chromium(VI).

Adsorbent	Adsorbate	Process variables: Initial concentration (mg/L)	pH	Adsorbent dose (g)	Contact time (min)	Predicted adsorption capacity (mg/g)	Desirability value	%removal (calculated)
MZTWAC	Chromium(VI)	59.98	4.08	0.2	80	26.279	0.694	87.629

evaluating normality of the residual. The aforementioned depicts residual behaviour that tracks a normal distribution and is straight, i.e., dispersal of residuals in a regular manner with minimal divergences. It is discovered that the model is suitable meant for the evacuation of chromium(VI) during the adsorption method when MZTWAC is used.

3.5. Influence Plots. The model's residuals are roughly parallel to the straight line. Although they appear to follow a normal probability distribution, the residuals explicit minimal deviation from normalcy. To examine the impact of outliers, it is never needed that the mistakes be genuinely regularly distributed in general terms. In this examination, Design Expert is used to create several graphical representations of the residuals. Estimates of the leverage (influence), Cook's distance, and DFFITS plots are requested to examine the regression model's exactness to discover the statistical features of the experimental model. Figure 8 shows that the influence statistics were all in the range of 0 to 1 [31]. There is no information which emphasizes that there is an unreasonably negative impact on the model fit because the effects of all run values in the current evaluation are less than 1. Figure 8 shows that the influence values were all within the range of 0 to 1 [31]. There are no information foci that have an unreasonably negative impact on the model fit because the effects of all run values in the current evaluation are less than 1.

Additionally, Cook's distance (Figure 9) estimates the impact of erasing a certain observation and is used to govern the outlier in the data (data). It must fall between the ranges of +1 and −1. Apart from DFFITS plots, Figure 10 assesses the influence of observed values on its projected value, and its run is in the centre of the typical farthest that reaches +2 to −2.

3.6. Outcome of Independent Variables. Tsai and Wu [32] used a case-weight perturbation plan in 1992 to get an elective nearby impact indicator that considers the Jacobian perturbation consequences. However, in this study, a perturbation diagram (Figure 11) was plotted to consider the relationship between four independent factors, pH, adsorbent dose, contact time, and initial concentration, in the adsorption procedure aimed at the removal of chromium(VI), as well as their contrasting impact on the response.

The response acquired by operation Design Expert is assessed by altering only one variable while keeping the other components constant. Although the starting concentration (A) line goes upward on the opposite side, the perilous slant down of adsorbent dosage (C) and pH (B) reveals that adsorption capacity is subtle to adsorbent dose

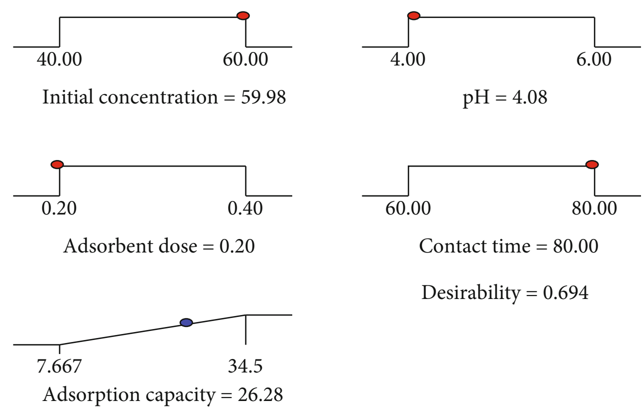

Figure 12: RAMP plot for adsorption of chromium(VI) by MZTWAC.

(C) followed by pH (B) for expulsion. It shows that adsorption capability for removing chromium(VI) increases with increasing chromium(VI) concentration (A), but declines with growing adsorbent dosage (C) and pH (B).

Initial concentration (A) and adsorbent dosage (C) were additionally profound to the adsorption process than changing pH (B) and contact duration, according to the perturbation plot (D). Nevertheless, the perturbation plot reveals that pH, adsorbent dosage, contact duration, and beginning concentration all have a significant influence on adsorption capacity in the chromium(VI) expulsion proficiency.

3.7. Optimization of the Process Variable. Exploratory data is used to ensure the most accurate estimates of variables for chromium(VI) expulsion by MZTWAC from the model. The optimization of multiple response models is done using the desirability function. It is filled out as target work and used to create a custom mix of process parameters ahead of time. For each element and reaction, Design Expert provides five alternative objectives: maximize, reduce, target, in range, and set to a precise number. In this current study, the desired target for adsorption capacity was set to 1 maximum and "in the range" for four independent variables such as starting concentration, pH, contact duration, and adsorbent dosage.

The analysis of desirability predicted that the desirability for the response function selected is furnished in detail in Table 5. The RAMP plot (Figure 12) depicts that 26.279 mg/g is an optimized response while the independent variables are 59.98 mg/L (initial concentration), 4.08 (pH), 0.2 g (adsorbent dosage), and 80 minutes at maximum desirability.

4. Concluding Remarks

The high match was established among the experimental value and predicted value of the adsorption capacity in the optimization procedure of chromium(VI) adsorption by MZTWAC which recommended that the model chosen was an excellent fit. Auxiliary inference was that the optimized outcome acknowledged from the plots of RAMP that the activated carbon fabricated from *Manilkara zapota* tree is the best choice in removing chromium(VI) ion from aqueous solution and it is economically worthy too. The inclusion of central composite design in the experimental study reduced the experimentation work, and the optimum results were arrived for the chosen process parameters. The response surface methodology conferred a clear picture between the process parameter interactions through the three-dimensional images. Many adsorbents such as aluminum–lanthanum mixed oxyhydroxide (ALMOH), chitosan/aluminum–lanthanum mixed oxyhydroxide (CSALMOH), ionic solid impregnated phosphate chitosan, and PAN–CNT/TiO_2–NH_2 have been involved in the removal of chromium ion with the adsorption capacity of 49.8, 78.9, 266.67, and 714.27 mg/g, respectively. The more water pollution, the more there is a want of a new adsorbent to treat polluted water to make safe water either to be disposed of or be reused. In this context, MZTWAC is best suited which costs low and is efficient as well.

Data Availability

The data used to support the findings of this study are included within the article. Should further data or information be required, these are available from the corresponding author upon request.

Disclosure

The study was performed as a part of the employment of Mettu University, Ethiopia.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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