



Research Article

## Response Surface Methodology for the Optimization of Cadmium Ion Adsorption using Activated Carbon

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### ABSTRACT

Adsorption refers to the process of chemical species adhering to the surface of particles. Activated carbon, also known as activated charcoal, is a type of carbon widely utilized for purifying water and air by filtering out various contaminants. One of the transition metals, Cadmium (Cd), situated in the d-block and 12th group of the periodic table, possesses toxic and poisonous properties. Adsorbent material was prepared using standard method. The adsorbate was prepared by dissolving cadmium nitrate in distilled water. Different volume of the solution was added adsorbate and later filtered and atomic adsorption Spectroscopy (AAS) was carried out. The potential of Activated carbon for removal of cadmium from contaminated water, it was found that highest efficiency in terms of percentage removal was achieved at 50 ppm initial metal ion concentration of Cadmium. The highest percentage removal was 98.5% confidence level. Therefore, the hypothesis that Activated carbon adsorbents are effective in removal of cadmium (II) ion from contaminated water at different experimental conditions is upheld

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## INTRODUCTION

In recent years, there has been a significant focus on extensively investigating methods to eliminate hazardous heavy metal ions from wastewater. The urgency stems from the detrimental impact these ions have on animal and plant life, alongside their contribution to various environmental issues. Several methods have been used to extract these metal ions from industrial waste streams and soil compositions over the years (Fu & Wang, 2011; Tripathi *et al.*, 2019). Low-cost adsorbents were employed to extract heavy metals from polluted solutions, along with other techniques. While modified carbon substrate adsorption shows promise in eliminating various trace elements from wastewater and solutions, its relatively higher cost hampers its widespread adoption for large-scale applications. In recent years, effective removal of large volumes of Cd(II) ions from wastewater using low-cost adsorbents has been proposed as a viable solution to some of these challenges and researched (Ahmed & Ahmaruzzaman, 2016; Uddin, 2017). The degree of success in removing these ions from water differs from one metal to another, and depends on prevailing experimental conditions such as temperature, concentration and contact time (Ali, 2012). Water contamination is a worldwide challenge that is caused by dyes, pathogens, turbidity, oil and its derivatives and heavy metals from industries and agricultural effluents (El-Gaayda *et al.*, 2021). Heavy metals in particular persist in the environment indefinitely and cause dangers to public health, with lead being the most common heavy metals (Mahurpawar, 2015; Mohammed *et al.*, 2011). Cadmium poisoning causes kidney diseases, fragile bones, cancer, stomach cramps, diarrhoea, shortness of breath (Jaishankar *et al.*, 2014).

Many methods have been employed in the cleaning of water for human consumption. These include chlorination to kill pathogens and reverse osmosis, electrochemical methods, chemical precipitation, biological processes, ion exchange, floatation and membrane processes to remove heavy metals from water (Anis *et al.*, 2019; Manikandan *et al.*, 2022). Some of these methods, however, produce toxic sludge that poses disposal challenges and are relatively expensive (Marsh *et al.*, 2007). Therefore, there is need for continued research into cheaper ways or technologies for water treatment. Adsorption is one of the alternatives for removing heavy metal ions from water (Lim & Aris, 2014).

In this research, we explore the extraction of cadmium ions from liquid solutions with different experimental parameters such as contact time, initial metal ion concentration, and adsorbent dosage.

## METHODOLOGY

### Preparation of Adsorbate Solution

Preparation of 1000 mL (L) 0.1M HNO<sub>3</sub>

Using the dilution formular:

$$C_1V_1 = C_2V_2$$

Where C<sub>1</sub> = Initial concentration of the acid.

C<sub>2</sub> = Final concentration of the acid. V<sub>1</sub> =

Initial volume of the acid. V<sub>2</sub> = Final volume of the acid.

C<sub>1</sub> = 15M HNO<sub>3</sub>; V<sub>1</sub> = ?; V<sub>2</sub> = 1000ml HNO<sub>3</sub>;

C<sub>2</sub> = 0.1M HNO<sub>3</sub>

By substitution: 15MV<sub>1</sub> = 0.1M x 1000ml

$$\Rightarrow V_1 = \left( \frac{0.1M \times 1000ml}{15} \right) = 6.7ml$$

First, 6.7 ml of 15M HNO<sub>3</sub> was carefully added to 500 mL of distilled water. The mixture was thoroughly shaken until well-mixed and then brought to a total volume of 1000 mL by adding de-ionized water. This resulted in an expected concentration of 0.1M for the acid solution.

Next, the stock solution of 1000 ppm (Cd) was prepared using the following procedure

- i. Molar mass of  $Cd(NO_3)_2 \cdot 4H_2O = 308 \text{ g/mol}$
- ii. Relative atomic mass of cadmium  $Cd = 112 \text{ g/mol}$   
 By definition:  
 Mass Cd = Molar mass of Cd  
 $(NO_3)_2 \cdot 4H_2O$   
 Relative atomic mass of Cd = 308 g/mol  
 Since only 100 mL of 1000ppm (Cd) is required, 0.275 g was dissolved in 100 mL of 0.1 M  $HNO_3$ .

**Preparation of an intermediate stock solution of 100 ppm**

Using dilution formular:  $C_1V_1 = C_2V_2$   
 (Dilution formular)  
 Where  $C_1 = 1000 \text{ ppm}$ ;  $V_1 = ?$ ;  $C_2 = 100 \text{ ppm}$ ;  
 $V_2 = 100 \text{ mL}$   
 By substitution:  $1000 \text{ ppm} \times V_1 = 100 \text{ ppm} \times 100 \text{ mL}$   
 $V_1 = (100 \text{ ppm} \times 100\text{mL}) / 1000 \text{ ppm} = 10 \text{ mL}$

10 mL of 1000 ppm (Cd) was added in an empty conical flask and make it to the mark 100 mL, using 0.1 M  $HNO_3$  solution after which 50 ppm and 150 ppm were prepared with the used of dilution formular.

**Preparation of Adsorbent Material**

Activated charcoal used for this study was available in Maiduguri region, Borno, Nigeria. Neem Tree was obtained from 505 Housing Estate. The tree was sun dried for 24 h then burnt, then grounded and sieved and obtained as activated charcoal adsorbent.

**Experimentations**

For the adsorption process a series of central composite design of experiments were ran and presented in Table 1.

After the adsorption, the samples were filtered. The filtrate was analysed for metal ion concentration using Atomic Absorption Spectroscopy (AAS). Experiment was performed to find the effect of Contact time, Adsorbent mass and the initial metal ion concentration on Adsorption of Cd ion.

**RESULTS**

**Table 1:** Factorial design experimental result on percentage removal on cadmium on activated charcoal

Std	Run	Factor 1 A: Adsorbent dose (g)	Factor 2 B: Time (hrs)	Factor 3 C: Concentration (ppm)	Response Percentage Removal (%)
11	1	5	1	100	98.8
4	2	8	8	50	96.6
10	3	5	8	150	98.2
13	4	5	4.5	50	97.5
12	5	5	8	100	98.75
5	6	2	4.5	150	99.16
1	7	2	1	50	100
3	8	2	8	50	100
6	9	8	4.5	150	99.27
8	10	8	4.5	100	99.05
9	11	5	1	150	99.03
7	12	2	4.5	100	99.25
2	13	8	1	50	100

For optimization of the variables, the CCD under RSM was applied and 13 runs of

experiments were designed. A quadratic model was used and the multiple regression

analysis was performed, the R (%percentage removal) response and the test variables were calculated.

**Table 2:** ANOVA for Reduced Quadratic model

Source Model	Sum of Squares	Df	Mean Square	F-value	p-value	
	8.50	5	1.70	3.65	0.0607	not significant
A-Adsorbent dose	1.52	1	1.52	3.26	0.1138	
B-Time	2.29	1	2.29	4.91	0.0623	
C-Concentration	0.2486	1	0.2486	0.5330	0.4891	
AB	2.89	1	2.89	6.20	0.0417	
A <sup>2</sup>	1.78	1	1.78	3.81	0.0919	
<b>Residual</b>	3.27	7	0.4665			
<b>Cor Total</b>	11.77	12				

Factor coding is **coded**.

Sum of squares is **Type III - Partial**

The Model F-value of 3.65 indicates that there is a 6.07% probability that such a large F-value could arise solely from noise or random fluctuations. Model terms with P-values below 0.0500 are considered significant, indicating that they have a notable impact on the model. In this particular scenario, term AB is found to be statistically significant. Conversely, P-

values greater than 0.1000 suggest that the model terms lack significance. When numerous model terms are found to be insignificant (excluding those necessary to maintain hierarchy), reducing the model by removing these terms could lead to improvements in its performance.

**Table 3:** Fit Statistics

<b>Std. Dev.</b>	0.6830	<b>R<sup>2</sup></b>	0.7225
<b>Mean</b>	98.89	<b>Adjusted R<sup>2</sup></b>	0.5244
<b>C.V. %</b>	0.6906	<b>Predicted R<sup>2</sup></b>	-0.4752
		<b>Adeq Precision</b>	5.9700

If the Predicted R<sup>2</sup> value is negative, it suggests that the overall mean might be a more effective predictor of your response

compared to the current model. In certain instances, employing a higher order model could lead to better predictions.

Adeq Precision assesses the signal to noise ratio, and a ratio exceeding 4 is considered favorable. With a ratio of 5.970, the signal is

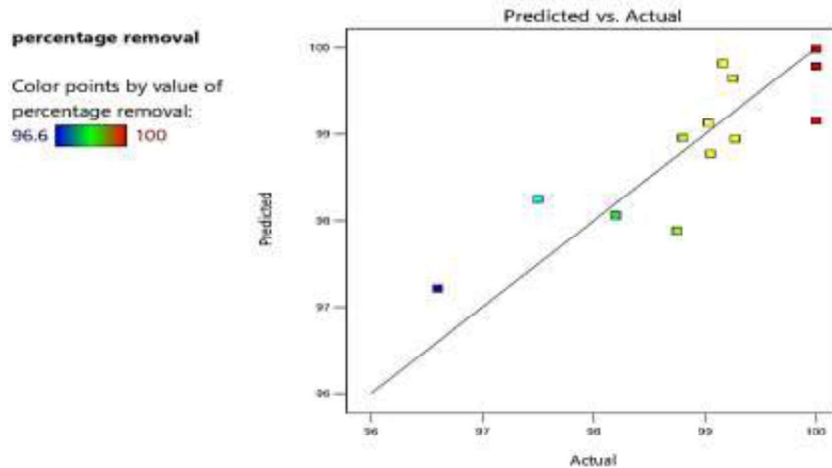
adequate. This model can effectively guide you through the design space.

**Table 4:** Final Equation in Terms of Actual Factors

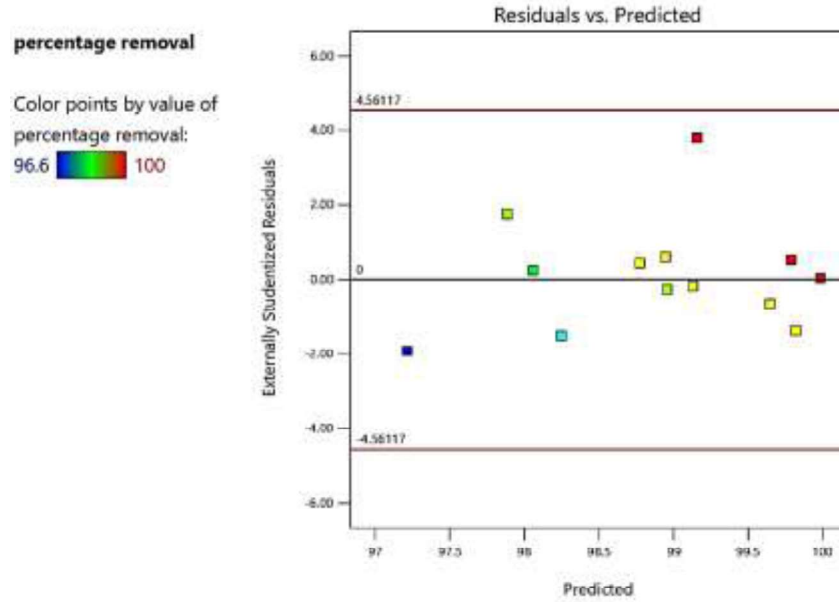
Percentage removal	Equal to
+99.85801	
-0.656834	Adsorbent dose
+0.251905	Time
+0.003461	Concentration
-0.080952	Adsorbent dose * Time
+0.087570	Adsorbent dose <sup>2</sup>

The equation, when expressed in terms of actual factors, allows for predictions about the response given specific levels of each factor. It is important to specify the levels in the original units for each factor. However, caution should be exercised when attempting

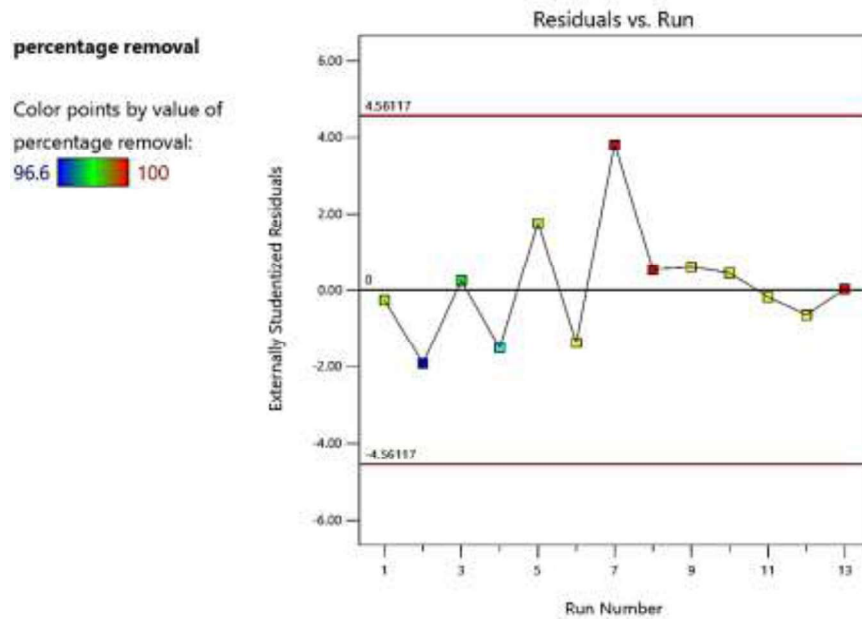
to determine the relative impact of each factor using this equation, as the coefficients are scaled to accommodate the units of each factor, and the intercept is not centered within the design space.



**Figure 1:** A plot of residuals versus predicted values of actual percentage removal %.



**Figure 2:** A plot of residuals versus predicted values of percentage removal %.



**Figure 3:** A plot of residuals versus run values of percentage removal %.

**Table 5:** Detailed results for the predicted, residual, leverage and outlier statistical data values

Run Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residuals	Externally Studentized Residuals	Cook's Distance	Influence on Fitted Value DFFITS	Standard Order
1	98.80	98.96	-0.1564	0.330	-0.280	-0.260	0.006	-0.183	11
2	96.60	97.22	-0.6152	0.693	-1.625	-1.907	0.993 <sup>(1)</sup>	-2.863 <sup>(1)</sup>	4
3	98.20	98.06	0.1405	0.402	0.266	0.248	0.008	0.203	10
4	97.50	98.25	-0.7483	0.373	-1.384	-1.504	0.190	-1.161	13
5	98.75	97.89	0.8636	0.330	1.545	1.761	0.196	1.236	12
6	99.16	99.82	-0.6588	0.438	-1.287	-1.364	0.215	-1.205	5
7	100.00	99.16	0.8423	0.693	2.225	3.807	1.861 <sup>(1)</sup>	5.717 <sup>(1)</sup>	1
8	100.00	99.79	0.2123	0.693	0.561	0.531	0.118	0.798	3
9	99.27	98.95	0.3237	0.438	0.632	0.603	0.052	0.532	6
10	99.05	98.77	0.2767	0.258	0.470	0.442	0.013	0.261	8
11	99.03	99.13	-0.0995	0.402	-0.188	-0.175	0.004	-0.143	9
12	99.25	99.65	-0.3958	0.258	-0.673	-0.644	0.026	-0.379	7
13	100.00	99.99	0.0148	0.693	0.039	0.036	0.001	0.054	2

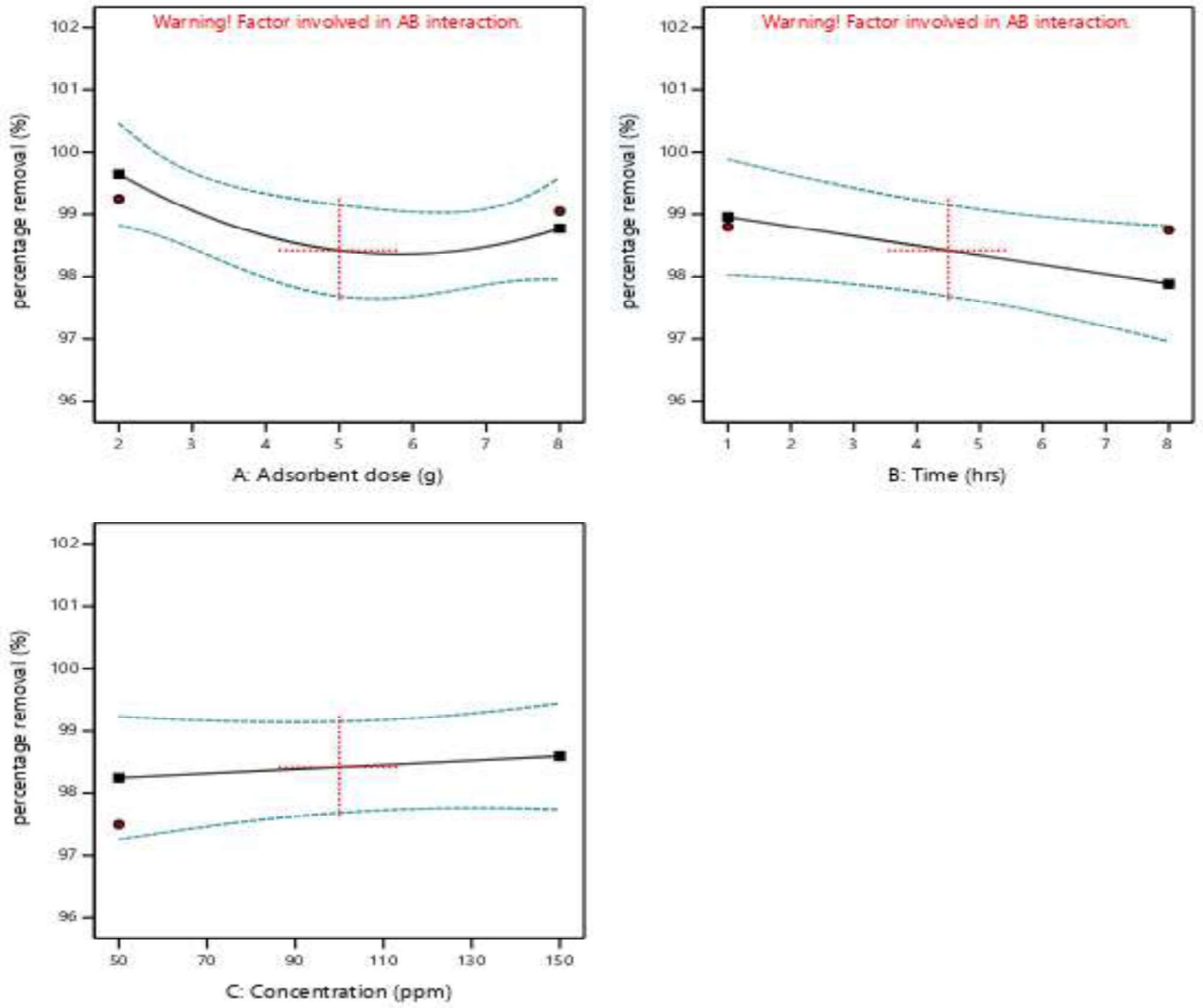
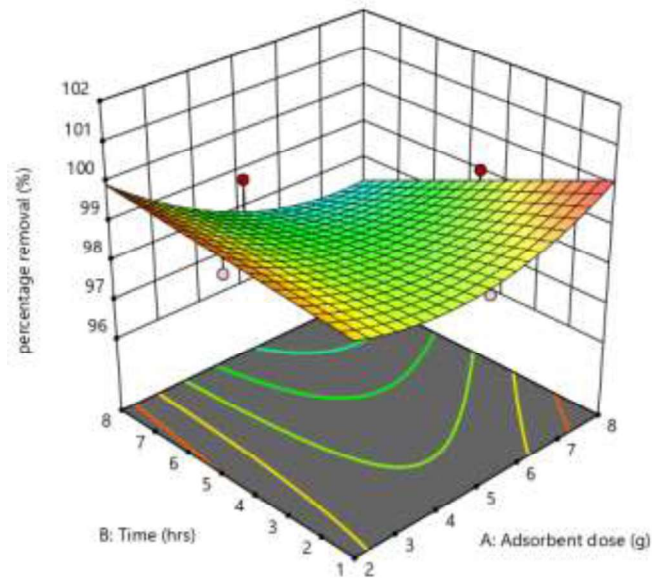
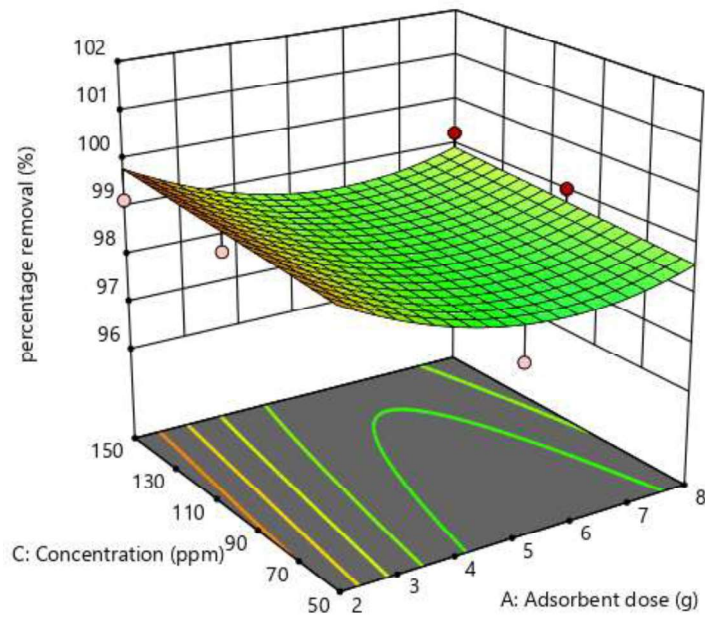


Figure 4: 2D plots of percentage removed % against the process variables.

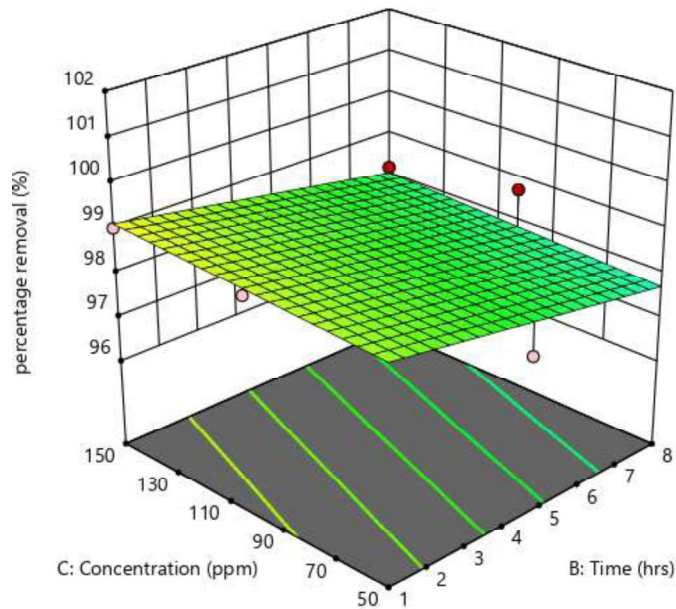




**Figure 5:** 3D surface plots of percentage removed (%) response adsorbent dose (g) by time (hrs)



**Figure 6:** 3D surface plots of percentage removed (%) response concentration (ppm) by adsorbent dose.



**Figure7:** 3D surface plots of percentage removed (%) response concentration (ppm) by time (hrs)

## DISCUSSION

### a) Model evaluation

To confirm the model fitness, the analysis of variance (ANOVA) was used, which its results are presented in Table 2. The  $R$  (%) response regression was statistically significant at  $F$ -value of 36.5 and  $p$ -value of 0.0607. The  $p$ -value for quadratic model was obtained higher than 0.0607, indicating the significance of its regression at 95% confidence interval. Therefore, the model suitably explained the relationship between variables and response. The relationship between actual and predicted values of  $R$  (%) response is illustrated in Figures 1 to 6. The actual values were original measurements of percentage removal % calculated by

$$\% \text{ Removed} = \frac{A-X}{A} \times 100$$

Where  $A$ = Total amount of concentration of metal solution prepared.

$X$ = Total amount of concentration of metal ion absorbed by adsorbent.

And the predicted values were generated by model design. The findings suggest that sugarcane bagasse can be used as raw material

for synthesizing adsorbent materials for removal of cadmium from contaminated water.

The levels and the ranges of the studied process parameters (A- Adsorbent dosage, B- Time, C- Concentration) affecting the removal employed in the experiment are given in Table 2. The observed and predicted % of Cd removal by Charcoal were Presented in Table 4. The results were also analysed using software MINITAB 18 along with main effect, interaction of different factors were determined.

After estimating the main effect, the interacting factors affecting the removal of Cd were also determined by performing the Analysis of Variance (ANOVA). The main and interaction effects of each factor having  $p$ -value 0.0607 are considered not significant.

## b) Effect of process variables

### Effect of contact time

Figure 5 illustrates the 3D surface response depicting the relationship between adsorbent dose and contact time uptake for the selected adsorbents. The investigation involved conducting separate experimental runs with varying contact times ranging from 1 to 8 hours. As anticipated, the quantity of ions adsorbed into the adsorbent augmented with time, reaching a saturation point beyond which no further adsorption occurred. This equilibrium state indicates that the amount of ions desorbing from the adsorbent is dynamically balanced with the amount being absorbed by the adsorbent. The 3D plots shows that increasing the contact time from 1–8hrs decreases the percentage removal from 97.5 to 100% as it shows in figure 7 that the %percentage removed significantly decreases during the period of 1– 8 hrs by 2.5%, showing that the adsorbent is very weak.

### Effect of Adsorbent dosage

To investigate the interaction of Adsorbent dosage with Cd concentration on the *R* (%) Response, 3D surface plots are displayed in Figure 6. The results showed that increasing the Adsorbent dosage lead to an increase in Percentage removal %. The percentage removal % showed an increase from 99.03 to 99.27 as a result of increasing the Adsorbent dosage from 2 g to 8g. Increasing the percentage removal % is related to an increase in number on the Adsorbent surface for complexation, which results in more Cd binding attached to the surface Adsorbent.

### Effect of Cd ion concentration

For evaluating the interaction of Cd ion concentration with Time (hrs) on the *R* (%)

response, 3D surface plots are encapsulated in Figure 7. Increasing the concentration from 150 to 100 ppm caused a reduction in percentage removal %from 99.03 to 98.75%. This phenomenon occurs because, at low concentrations of metal ions in the solution, the adsorbent's available surface area relative to the total metal ions is high. This high surface area facilitates effective interactions between all metal ions and the adsorbent, leading to their successful removal from the solution. Consequently, the removal efficiency increases. In contrast, when the concentration of metal ions is high, the ratio of active sites on the adsorbent's surface to the total metal ions in the solution decreases. As a result, not all metal ions can effectively interact with the adsorbent and be removed from the solution. Consequently, the percentage removal decreases.

## CONCLUSION

The potential of Activated charcoal for removal of cadmium for contaminated water, it was found that highest efficiency in terms of percentage removal was achieved at 50 ppm initial metal ion concentration of Cadmium. The, highest percentage % removal was 100% respectively at 95% confidence level with p-values 0.0607 not significant. Therefore, the hypothesis that Activated charcoal adsorbents are effective in removal of Cd from contaminated water at different experimental conditions is upheld. The adsorbent dosage that showed highest removal was 2, 2 and 8 g. Highest removal occurred at 1, 8 and 1 hr contact time respectively.

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