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Modelling and Optimization of Lead Adsorption onto Sugarcane Bagasse Activated Carbon

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ABSTRACT

Modeling and optimization of heavy metals adsorption onto activated carbon produced from locally available agricultural residues are important because they save time and resources for laboratory experiments. In this work, sugarcane bagasse was collected from Gaskiya Road in Zaria, Nigeria and carbonized at three different temperatures, 300, 325 and 350 °C and then impregnated with 1 M H_3PO_4 at the ratio of 1:2 (1 g of sugarcane bagasse to 2 ml of H_3PO_4). The modeling and optimization were carried using response surface methodology (RSM) out at the lower and upper input variables of carbonization temperatures (300 – 350 °C); initial lead concentrations of (10 - 60 mg/l) and adsorbent dosages of (0.2 - 1.0 g) with corresponding responses of: removal efficiency and adsorption capacity. The optimum operating conditions obtained were carbonization temperature of 350 °C, initial lead concentration of 60 mg/l and adsorbent dosage of 1 g with the corresponding optimum removal efficiency of 100% and adsorption capacity of 5.9895 mg/g. Similarly, the validated results revealed the percentage errors for the removal efficiency and adsorption capacity to be 0.415% and 0.241%, respectively. The study determined that the statistical models developed were good in predicting the responses and the optimum conditions obtained were valid.

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1. INTRODUCTION

Nigeria has been estimated to generate about 1.4 million tons of sugarcane across the country with Kaduna State producing about 13% of the sugarcane production (FAO, 2013; Adamu, 2018). The amount of sugarcane generated across the country yield high volume of sugarcane bagasse, being the residue left after the removal of sugar from the sugarcane (Adamu and Ahmadu, 2015). However, some of the sugarcane bagasse produced across the country are either improperly disposed or sometimes subjected to burning, thereby causing environmental pollution, hence, the need to convert them to a more beneficial product that could be used in the removal of some physical and chemical pollutants from wastewater (Adamu, 2018).

Heavy metals are metallic elements that are toxic even at low concentrations and known to have densities of greater than 5 g/cm^3. Lead is a heavy metal that is bluish-white and comparatively soft. The exposure of humans and animals to lead (Pb) causes some deleterious effects which include headache, renal defects, eye problem, brain damage and could ultimately lead to death (Jan *et al.,* 2015). Therefore, the removal of lead from wastewater is very vital way of mitigating risks it poses to human and environmental health.

There are various methods of removing heavy metals in wastewater. They include ion-exchange, membrane filtration and reverse osmosis (Adamu, 2018). These methods have demerits such as high costs, difficulty in operation and release of residues that needs further treatment. The removal of heavy metals from wastewater onto activated carbon is easy to operate and effective in terms of effluent quality (Ekpete *et al.,* 2017; Adamu, 2018). Nonetheless, the cost for production as well as regeneration of conventional activated carbon has necessitated the use of cheap agricultural by- product as adsorbent precursor materials for use in treatment wastewater. It is on this note that researches have been embarked upon to explore the application of different agricultural by-products for the production of activated carbons (Mohammad 2015). In addition, agricultural wastes are readily available and can be regenerated for further use (Pareira and Voorwald, 2011; Gupta and Mote, 2014).

Moreover, the application of agricultural wastes in the adsorption of pollutants from wastewater involve series of experimental procedures which are often tedious and time consuming. It is therefore important to incorporate modelling and optimization technique in order help in easing or reducing the experimental loads there by saving time, energy and resources (Mohammad, 2015). This research therefore, focused on the application of the sugarcane bagasse activated carbon for the adsorption lead from wastewater as well as modelling and optimization of the lead adsorption.

2. MATERIALS AND METHODS

2.1. Materials

The materials used are shown in Table 1.

Table 1: Equipment, glassware and reagents

Equipment	Glassware	Reagents
Weighing balance (Mettler, P160N, oven, electric furnace (Naberthem), pH meter (Hanna instrument), Magnetic stirrer, centrifuge machine and atomic absorption spectroscope (AA 6800, Shimadzu)	50, 100 and 1000 ml measuring cylinders, 10 ml conical flask and 25 ml pipette	0.1 M sodium hydroxide (NaOH) solution, 0.1M hydrochloric acid (HCl) solution, 1 M phosphoric acid (H_3PO_4), 100 mg/l stock solution of lead

2.2. Methods

2.2.1. Preparation of stock solution of lead

Lead stock solution (100 mg/l) was prepared by dissolving 0.1599 g of lead (II) trioxonitrate (V) ($Pb(NO_3)_2$) in 10 ml of 0.1 M trioxonitrate (V) (HNO_3) acid in the 1000 ml conical flask. Thereafter, de-ionized water was used to top it up to 1000 ml mark. From the stock solution, the working solutions of 10, 35 and 60 mg/l of lead were produced by adding 90, 65 and 40 ml of de-ionized water to the 10, 35 and 60 ml of the stock solution.

2.2.2. Preparation of the sugarcane bagasse activated carbon

The sugarcane bagasse was collected from Gaskiya road, opposite Nuhu Bamalli Polytechnic Zaria, Nigeria. It was washed thoroughly with tap water, followed by distilled water, in order to remove the impurities, after which it was subjected to oven drying at 100 °C for 24 hours in accordance with Pareira *et al.* (2010) and Gaikwad and Mane (2013). It was then subjected to grinding, followed by sieving through 0.60 mm sieve size. Thereafter, it was carbonized at the temperatures of 300, 325 and 350 °C for 1 hour. The impregnation was carried with 1M H_3PO_4 at ratio of 1:2 (1 g of sugarcane bagasse carbon to 2 ml of H_3PO_4) after which the pH was adjusted to 7 using 0.1M NaOH solution, and then it was dried, cooled in a desiccator and stored in a container for use.

2.2.3. Design of experiment

The design of experiment was carried out using central composite design (CCD) of the Design Expert software (version 10). The input variables were: A (carbonization temperature, (°C)), B (initial concentration of lead, (mg/l)) and C (adsorbent dosage, (g)); while the responses were Y_1 (removal efficiency, (%) and Y_2 (adsorption capacity, (mg/g)) as shown in Table 2. The central composite design (CCD) was applied to obtain the correlation between the input variables and responses. The model with highest polynomial order, in which more of its terms were significant and free from being aliased was chosen (Cronje *et al.,* 2011; Mohammad 2015; Adamu 2018). Batch adsorption experiments were conducted based on the experimental design shown in Table 3 by adding 0.6 g of activated sugarcane bagasse carbonized at 325 °C to 35 mg/L of Pb solution in a 250 mL capacity flask. The setup was stirred (agitated) for 60 mins at 150 rpm. After which the solids were removed using centrifugation and the equilibrium Pb concentration was measured using AAS. Similar runs were conducted with adjusted variables according to the protocols depicted Table 3 and the corresponding responses were recorded accordingly.

$$Y_1 = \left(\frac{C_i - C_e}{C_i}\right) \times 100 \tag{1}$$

$$Y_2 = \left(\frac{C_i - C_e}{m}\right) V \tag{2}$$

Where C_i is the initial concentration of lead, C_e is the final concentration, m is the mass of the adsorbent and V is the volume of the lead solution.

Table 2: Input variables and responses

Input variables	Lower value	Upper value		
A (□)	300	350	responses	Y_1 (%)
B (mg/l)	10	60		
C (g)	0.2	1		Y_2 (mg/g)

3. RESULTS AND DISCUSSION

3.1. Experimental Results

Table 3 presents the design matrix of lead adsorption onto sugarcane bagasse activated carbon. The inputs variables were randomized by the design expert using Response Surface Methodology (RSM). The removal efficiencies and adsorption capacities obtained from the laboratory experiments were also indicated as Y_1 and Y_2.

Table 3: Design matrix for the input variables and responses

Run	Input variables			Responses	
	A (°C)	B (mg/l)	C (g)	Y_1 (%)	Y_2 (mg/g)
1	325	35	0.6	99.842	5.824
2	350	60	0.2	99.887	29.967
3	300	35	0.6	98.142	5.725
4	300	10	1.0	92.443	0.924
5	300	60	0.2	99.328	29.798
6	325	10	0.6	99.729	1.662
7	300	60	1.0	98.286	5.897
8	325	35	0.6	99.842	5.824
9	325	35	0.2	99.920	17.480
10	325	60	0.6	99.979	9.998
11	325	35	0.6	99.842	5.824
12	350	35	0.6	99.619	5.811
13	350	60	1.0	99.585	5.975
14	325	35	1.0	99.878	3.496
15	325	35	0.6	99.842	5.824
16	350	10	0.2	99.969	4.998
17	325	35	0.6	99.842	5.824
18	300	10	0.2	98.572	4.929
19	325	35	0.6	99.842	5.824
20	350	10	1.0	98.113	0.981

3.2. Generation of Model Equations

In Table 4, the quadratic model has the highest R^2 value of 0.9032 and adjusted R^2 value of 0.8161 with the lowest standard deviation of 0.73. In addition, it was free from being aliased and was therefore suggested by the software. Table 5 also indicates that the quadratic model had highest R^2 value of 1.0000 and adjusted R^2 value of 0.9999 and the lowest standard deviation of: 0.73. It was free from being aliased and was therefore suggested. Furthermore, the predicted R^2 value of 0.9995 was in agreement with the adjusted R^2. According to the adjusted R^2 values of 0.8161 and 0.9999 in Table 4 and 5, it can be inferred that 81.61% and 99.99% of the changes in the removal efficiencies and adsorption capacities, respectively could be attributed to the carbonization temperature, initial lead concentration and adsorbent dosage. The model equations generated for Y_1 and Y_2 are shown in Equations (3) and (4), respectively.

Table 4: Model summary statistics for removal efficiency (Y_I)

Source	Standard deviation	R^2	Adjusted R^2
Linear	1.33	0.4814	0.3842
2FI	1.12	0.7012	0.5632
Quadratic	0.73	0.9032	0.8161
Cubic	0.18	0.9964	0.9885

Table 5: Model summary statistics for adsorption capacity (Y_2)

Source	Standard deviation	R^2	Adjusted R^2
Linear	0.17	0.9705	0.9650
2FI	0.19	0.9706	0.9571
Quadratic	0.0077	1.0000	0.9999
Cubic	0.0019	1.0000	1.0000

$$Y_1 = -116.17728 + 1.29632\mathrm{A} + 0.34795\mathrm{B} - 24.19566\mathrm{C} - 0.00104180\mathrm{AB} + 0.062662 + 0.083012\mathrm{BC} - 0.00193207\mathrm{A}^2 - 0.000374473B^2 - 1.18153C^2 \quad (3)$$

$$\mathrm{Y}_2 = -0.51384 + 0.013139\mathrm{A} + 0.079461\mathrm{B} - 4.46971\mathrm{C} + 0.000010972\mathrm{AB} + 0.000662184\mathrm{AC} + 0.000867886\mathrm{BC} - 0.0000195744\mathrm{A}^2 - 0.000574837\mathrm{B}^2 + 1.82347\mathrm{C}^2 \quad (4)$$

3.3. Statistical Analysis

The analysis of variance (ANOVA) was applied to determine the significance or otherwise of the model and each of the model term. The model terms that recorded P-values less than 0.05 were considered to be significant model terms in accordance with Esfandiar *et al.* (2014) and Azmi *et al.* (2015). In addition, the higher the recorded F-value, the more the influence of model term on the response (Garg *et al.*, 2009; Azmi *et al.*, 2015; Iji, 2016). In Table 6, the model for the removal efficiency (Y_1) had an F-value of 10.37 with a corresponding P-value of 0.0005, which implied that the model was statistically significant at the 95% confidence level.

Table 6: Analysis of variance of removal efficiency

Source	Sum of squares	df	Mean square	F value	P-value
Model	49.51	9	5.50	10.37	0.0005
A	10.82	1	10.82	20.39	0.0011
B	6.79	1	6.79	12.79	0.0050
C	8.78	1	8.78	16.55	0.0023
AB	3.39	1	3.39	6.39	0.0300
AC	3.14	1	3.14	5.92	0.0353
BC	5.51	1	5.51	10.39	0.0091
A^2	4.01	1	4.01	7.56	0.0205
B^2	0.15	1	0.15	0.28	0.6058
C^2	0.098	1	0.098	0.19	0.6761
Residual	5.31	10	0.53		
Lack of fit	5.31	5	1.06		
Pure error	0.000	5	0.000		

Table 7: Analysis of variance of adsorption capacity

Source	Sum of squares	df	Mean square	F value	P-value
Model	15.26	9	1.70	28598.03	< 0.0001
A	1.15×10^{-3}	1	1.15×10^{-3}	19.42	0.0013
B	8.18	1	8.18	1.38×10^{5}	< 0.0001
C	6.63	1	6.63	1.11×10^{5}	< 0.0001
AB	3.76×10^{-4}	1	3.76×10^{-4}	6.34	0.0305
AC	3.51×10^{-4}	1	3.51×10^{-4}	5.92	0.0353
BC	6.03×10^{-4}	1	6.03×10^{-4}	10.16	0.0097
A^2	4.12×10^{-4}	1	4.12×10^{-4}	6.94	0.0250
B^2	0.35	1	0.35	5986.00	< 0.0001
C^2	0.23	1	0.23	3947.53	< 0.0001
Residual	5.93×10^{-4}	10	5.93×10^{-5}		
Lack of fit	5.93×10^{-4}	5	1.19×10^{-4}		
Pure error	0.00	5	0.00		

The significant model terms were: A, B, C, AB, AC, BC and A^2 while the insignificant model terms were B^2 and C^2. However, the model term that had the most significant effect on the removal efficiency was A with F-value of 20.39 and the effect of the model terms on the response were in the order: $A>C>B>BC>A^2>AB>AC$. Table 7 depicts the ANOVA for adsorption capacity. It indicated that the model had F-value of 28598.03 with a P-value of <0.0001, which signified that the model was statistically significant and all the model terms were also statistically significant. Furthermore, the model term that had the most significant effect was B having F-value of: 1.379E+005, followed by C with F-value of: 1.12×10^5 and the effect of the model terms were in the order: $B> C> B^2 > C^2 >A>BC> A^2 >AB>AC$. When the insignificant model terms were removed, Equations (3) and (4) became (5) and (6) respectively.

$$\begin{aligned} Y_1 = {} & -116.17728 + 1.29632\mathrm{A} + 0.34795\mathrm{B} - 24.19566\mathrm{C} - 0.00104180\mathrm{AB} \\ & + 0.062662\mathrm{AC} + 0.083012\mathrm{BC} - 0.00193207\mathrm{A}^2 \end{aligned} \tag{5}$$

$$\begin{aligned} \mathrm{Y}_2 = {} & -0.51384 + 0.013139\mathrm{A} + 0.079461\mathrm{B} - 4.46971\mathrm{C} - 0.000010972\mathrm{AB} \\ & + 0.000662184\mathrm{AC} + 0.000867886\mathrm{BC} - 0.0000195744\mathrm{A}^2 \\ & - 0.000574837\mathrm{B}^2 + 1.82347\mathrm{C}^2 \end{aligned} \tag{6}$$

3.4. Comparison of the Experimental and Predicted Values

Table 8 shows the actual and predicted values obtained for the adsorption capacity and removal efficiency with their corresponding residuals (errors). The Table shows that the predicted values for the adsorption capacity and removal efficiency were in good agreement. In addition, the mean absolute errors (MAE) for the prediction of the adsorption capacity and removal efficiency were: 0.05 and 0.40, respectively.

Table 8: Actual-predicted values and residuals

	Input variables			Response 1			Response 2		
Run	A (°C)	B (mg/l)	C (g)	Exp. Y_1 (%)	Pred. Y_1 (%)	Residual	Exp. Y_2 (mg/g)	Pred. Y_2 (mg/g)	Residual
1	325	35	0.6	99.84	99.94	0.10	5.82	5.81	0.01
2	350	60	0.2	99.89	99.00	0.89	29.97	29.67	0.30
3	300	35	0.6	98.14	97.69	0.45	5.73	5.70	0.03
4	300	10	1.0	92.44	93.40	0.96	0.92	0.93	0.01
5	300	60	0.2	99.33	99.48	0.15	29.80	29.96	0.17
6	325	10	0.6	99.73	98.88	0.85	1.66	1.65	0.01
7	300	60	1.0	98.29	98.01	0.28	5.90	5.87	0.03
8	325	35	0.6	99.84	99.94	0.10	5.82	5.81	0.01
9	325	35	0.2	99.92	100.69	0.77	17.48	17.64	0.16
10	325	60	0.6	99.98	100.53	0.55	10.00	10.07	0.08
11	325	35	0.6	99.84	99.94	0.10	5.82	5.81	0.01
12	350	35	0.6	99.62	99.77	0.15	5.81	5.81	0.00
13	350	60	1.0	99.59	100.00	0.41	5.98	5.99	0.01
14	325	35	1.0	99.88	98.81	1.07	3.50	3.46	0.04
15	325	35	0.6	99.84	99.94	0.10	5.82	5.81	0.01
16	350	10	0.2	99.97	100.32	0.35	5.00	5.00	0.00
17	325	35	0.6	99.84	99.94	0.10	5.82	5.81	0.01
18	300	10	0.2	98.57	98.19	0.38	4.93	4.90	0.03
19	325	35	0.6	99.84	99.94	0.10	5.82	5.81	0.01
20	350	10	1.0	98.11	98.04	0.07	0.98	0.980	0.00
				MAE		0.40			0.05

3.5. Combined Effects of the Input Factors on the Responses

Figures 1 and 2 depict the joint effects of the carbonization temperature and initial lead concentration on the removal efficiency and adsorption capacity, respectively. Figure 1 indicates that the carbonization temperature and initial concentration had effect on the removal efficiency. This is because, increase in both carbonization temperature and initial concentration, resulted to increase in the removal efficiency, and the joint effects happened to be more at higher values of the two input factors. The increase in removal efficiency due to increase in initial concentration could be attributed to multilayer adsorption; while increase in the carbonization temperature provided more pores that could adsorbed lead from the solution. This observation was reported by Itodo and Itodo (2010); Adamu and Adie (2020a) and Adamu and Adie (2020b). In Figure 2, the initial lead concentration had more effect on the adsorption capacity than the carbonization temperature. In other words, increase in the initial concentration resulted to higher adsorption capacity than the carbonization temperature. In Figure 3 and 4, it could be observed that increase in the carbonization temperature resulted in an increase in both the removal efficiency and adsorption capacity due to provision of more pores for adsorption as was reported by Mohammed (2015). An increase in the adsorbent dosage resulted in a decrease in the removal efficiency due to attainment of optimum dosage. This observation agreed with those of Itodo and Itodo (2010) and Adamu *et al.* (2018). In Figures 5 and 6, it could be observed that increase in the adsorbent dosage resulted to decline in both the removal efficiency and adsorption capacity; while increase in the initial lead concentration resulted to both increase in the removal efficiency and adsorption capacity. This is because, at higher adsorbent dosages, the amount of lead in solution could not satisfy all the sorption sites, hence the decrease in adsorption capacity. This observation was reported by Adamu (2019); Adamu and Adie (2020a) and Adamu and Adie (2020b). However, the initial lead concentration had more effect than the adsorbent dosage on both adsorption capacity and removal efficiency.

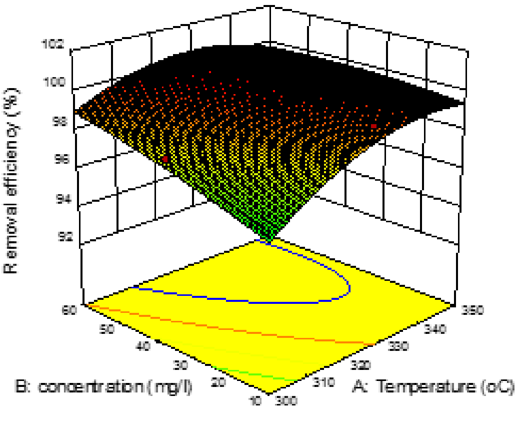

Figure 1: Combined effects of A and B on Y_1

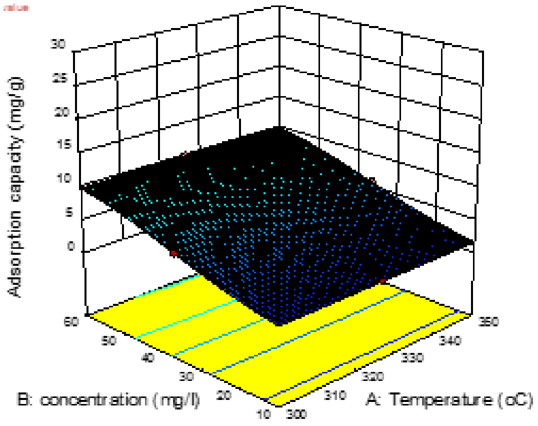

Figure 2: Combined effects of A and B on Y_2

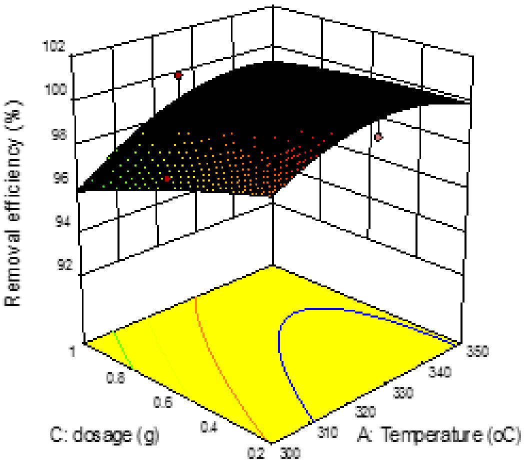

Figure 3: Combined effects of A and C on Y_1

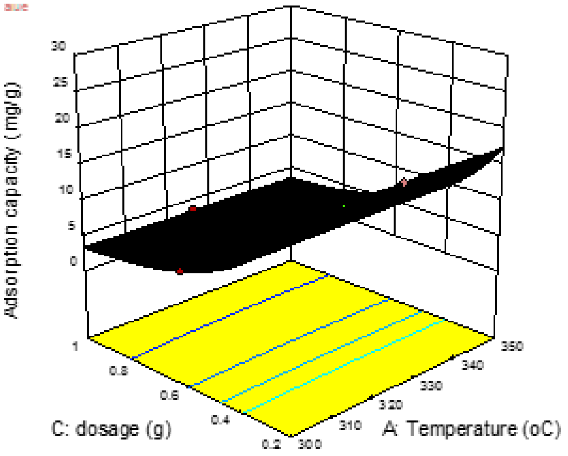

Figure 4: Combined effects of A and C on Y_2

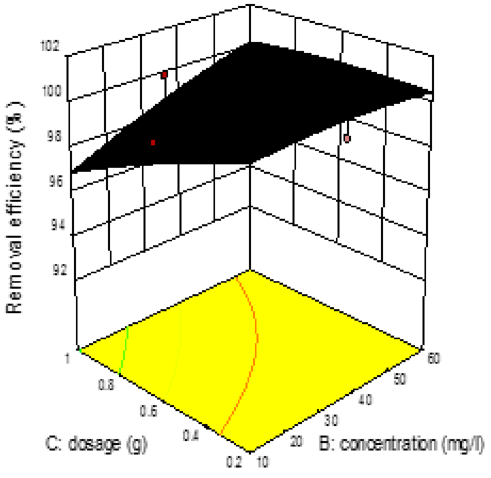

Figure 5: Combined effects of B and C on Y_1

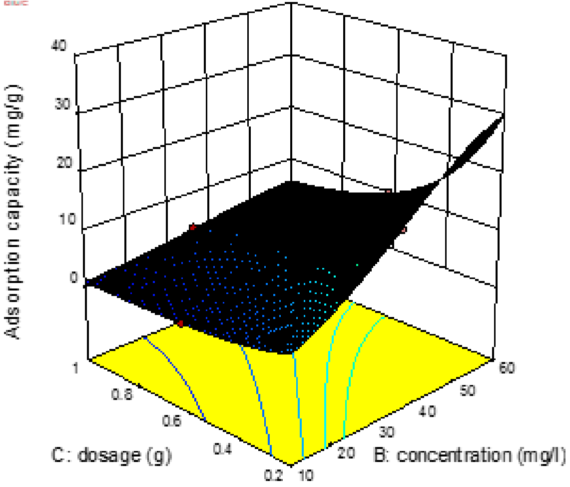

Figure 6: Combined effects of B and C on Y_2

3.6. Optimization of Lead Adsorption onto Sugarcane Bagasse Activated Carbon

The optimized removal efficiency of 100% and adsorption capacity of 5.9895 mg/g obtained are shown in Table 9. It could be observed that, the optimum removal efficiency and the adsorption capacity were obtained at the adsorption conditions of carbonization temperature of 350 °C, initial lead concentration of 60 mg/l and adsorbent dosage of 1 g.

Table 9: Optimum removal efficiency and adsorption capacity

A (°C)	B (mg/l)	C (g)	Y_1 (%)	Y_2 (mg/g)
350	60	1	100	5.9895

3.7. Validation of the Optimized Removal Efficiency and Adsorption Capacity

Table 10 shows the comparison between the predicted and experimental values of the optimized removal efficiency and adsorption capacity. From the Table, it could be observed that the predicted and experimental adsorption capacities and the removal efficiencies were in good agreement. Furthermore, the percentage errors for the adsorption capacity and removal efficiency recorded were 0.241 and 0.415 %, respectively. This signified that the model was reliable in predicting the responses and optimum conditions generated were valid.

Table 10: Validation of the optimum adsorption conditions

Input variables			Predicted response		Experimental response		% Error	
A (°C)	B (mg/l)	C (g)	Y_2 (mg/g)	Y_1 (%)	Y_2 (mg/g)	Y_1 (%)	Y_2	Y_1.
350	60	1	5.9895	100	5.9751	99.585	0.241	0.415

4. CONCLUSION

Modeling and optimization of lead adsorption from wastewater onto sugarcane bagasse activated carbon was successfully carried out using the input variables of carbonization temperature, initial lead concentration and adsorbent dosage with removal efficiency and adsorption capacity as the responses. The optimum removal efficiency of 100% and adsorption capacity of 5.9895 mg/g, respectively were recorded at the adsorption conditions of carbonization temperature of 350 °C, initial lead concentration of 60 mg/l and adsorbent dosage of 1.0 g. In addition, the validated results revealed that the percentage errors for the removal efficiency and adsorption capacity to be 0.415 and 0.241% respectively. This implied that the statistical models are reliable in predicting the responses and optimized removal efficiency and adsorption capacity were valid. Therefore, the model can be used to optimize the use of resources as well as save time for adsorption systems that are operated under similar conditions presented in this study.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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