OPTIMIZATION OF SORPTION OF LEAD IONS FROM LAKE GERIO WATER USING ACTIVATED SUGARCANE BAGASSE

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ABSTRACT

Lead ions above required limits are observed to have effect on living things and the aquatic environment. Sorbents from petrochemicals are expensive and not environmentally friendly. There is the need to investigate the optimal parameters for sorption of lead ions from lake Gerio water or the aquatic environment using low cost and environmentally friendly sorbent from agricultural waste materials such as sugarcane bagasse that is readily available in the region. Sorption of lead ions from Lake Gerio water in a batch process system using activated sugarcane bagasse was investigated. The factors affecting sorption considered for the investigation include contact time, sorbent dosage and temperature. Two responses were used which were the lead uptake q and percentage removal X. The sorbent was characterized using Fourier transform infrared spectroscopy and Scanning electron microscope. The maximum uptake of lead ions was evaluated to be 0.0094 mg/g at contact time of 30 min, temperature of 36°C and sorbent dosage of 2g. Whereas the maximum percentage removal of lead ions was recorded to be 92.45% at contact time range of 90 min, temperature of 37.5°C and sorbent dosage of 6 g in the batch process system. From the Response surface methodology, the model for uptake of Lead ions was significant with P-values of < 0.0001 and higher F-value of 73.43 was recorded. Also, the model for percentage removal was significant with P-values of < 0.0201 and higher F-values of 7.14 signifying better fitness of model for uptake and percentage removal of lead ions by the activated sugarcane bagasse.

Keywords: Lead, Sorption, Activated sugarcane bagasse, Optimization

1 INTRODUCTION

Lake Gerio is in the capital of Adamawa state known as Jimeta -Yola in the Semi-Arid, drylands climate of north-eastern Nigeria and the photograph is indicated in Plate 1. Lake Gerio is located at latitude 9°18’29’’N and longitude 12°26’55’’E [1], very close to River Benue in Jimeta-Yola [2]. The activities occurring in the lake can also affect the river because when the lake is filled or flooded during raining season the water is channeled into the river and vice versa [2, 3]. These activities end up polluting the water in the lake and river which eventually affect the inhabitant of the area. Lake Gerio is used for irrigation, rearing of animals, aquatic life and washing by the inhabitants of Jimeta-Yola especially the Jambutu resident [3, 4].

Plate 1. Pictorial view of Lake Gerio

Lead is a highly toxic heavy metal that can enter water sources through various pathways, including industrial discharges, corrosion of plumbing systems, and atmospheric deposition. The permissible level of lead in drinking water set by the World Health Organization (WHO) and many national regulatory bodies is 10 micrograms per liter (µg/L) [5]. The ecological consequences of the presence of lead in aquatic environments can disrupt the delicate balance of ecosystems. High concentrations of these heavy metals can be toxic to aquatic organisms, such as fish, invertebrates, and algae. Lead exposure can lead to impaired reproduction, growth abnormalities, and even death in aquatic species [6]. In general, Lead ion causes anemia, brain, gastrointestinal, liver and kidney damage, loss of appetite and mental retardation in children [7]. Sugarcane bagasse is a readily available, inexpensive raw material that can be used in the manufacturing of activated carbon locally [8]. Sugarcane bagasse is a waste product in abundance that litters the environment. There is need for low-cost and environmentally friendly adsorbent for remediation of lead ions from lake, river and surface water in general. The effect of optimal parametric values such as contact time, temperature and sorbent dosage on the sorption of the lead ions called for investigation. Sorption involves sorbent (activated sugarcane bagasse) and solvent (Lake Gerio water) containing dissolved species to be sorbed known as sorbate (lead ions) [9]. The conventional linear optimization involves changing a parameter and fixing other variant parameters constant which is more commonly known as one-variable-at- a time as adapted by Luka *et al.*, [10]. Response surface methodology (RSM) is one of the modern techniques used for optimization of sorption parameters to remediate any metal ions from aqueous environment [2, 9]. RSM uses several variables by operating the regression statistical analysis on the independent variables to obtain the optimum overall factor surface. RSM involves the collection of mathematical and statistical techniques useful for analyzing the effects of several independent variables. RSM can also aid in evaluating the interactive effect of process variables and building a mathematical model that accurately describes the overall process [2, 11, 12]. Through the representation of the surface model, responses based on the combined variables can be determined [2, 12]. The aim of this research is to investigate the optimization of sorption of lead ions from Lake Gerio water using activated sugarcane bagasse as sorbent in a batch system. This was achieved using a full-factorial central composite design (CCD) method of RSM statistical analysis with a probability of significance 5% ($\alpha = 0.05$) with the aid of Design Expert software [2].

2 MATERIALS AND METHODS

2.1 Formation of activated carbon from sugarcane bagasse

The sugarcane bagasse was obtained from Savannah sugar company in Numan. It was washed with tap water and then distilled water to remove dirty particles to purify it, after which it was oven (WHL-25A) dried at 110°C for 24 h. Then carbonized at a temperature of 350°C for 1 h 30 min. It was then mixed with 0.5M HNO_3 at the ratio of 1:1 (1 ml of HNO_3 to 1 g of sugarcane bagasse). Finally, it was washed

with distilled water, filtered, and dried for 24 h. It was then crushed and sieved to powdered form due to the nature of the bagasse and since a smaller particle size means more particles and a larger external surface area [13]. The sugarcane bagasse before and after carbonization are presented in Plate 2.

(a) (b)

Plate 2. (a) Sugarcane bagasse sample, (b) Activated sugarcane bagasse.

2.2 Characterization of the activated sugarcane bagasse

The Characterization of activated sugarcane bagasse was done before and after sorption of lead ions to test for the suitability of the sorbent. PHENOM Prox Netherland Scanning electron microscope (SEM) gives information on surface morphology of the sorbent. while PERKIN ELMER SPECTRUM VERSION 10.4.3 Fourier transform infrared spectroscopy (FTIR) gives information on functional groups presence and chemical composition of the sorbent [10].

2.3 Batch Process System Optimization

Batch process system that was used in this study are Erlenmeyer conical flasks of 250 ml [10]. Twenty (20) flasks were incubated in a rotary shaker type at 150 rpm to obtain final concentration for each sorbate at a given process parameter. The concentration of each metal ions was evaluated using Standard Method for Examination of Water and Wastewater as adapted by Luka *et al.* [10]. The mixtures of Lake Gerio water and activated sugarcane bagasse were decanted and the residual concentration of each solution after sorption was determined using ACUSYS 211 – AAS for each lead ions in the final solution. Lake Gerio water initial mean metal concentration for lead ions was recorded as 0.53 mg/l. The uptake value at equilibrium was calculated using equation (1) [10, 14, 15, 16].

$$q = \frac{V(C_i - C_f)}{m} \tag{1}$$

Where, q is the uptake of metal (mg/g), C_i and C_f are the initial and equilibrium metal concentrations in the water (mg/l) respectively; V is the volume of solution (l) and m is the mass of sorbent (g). The removal percentage (X) of the metal ion by the sorbent was achieved using equation (2) [16, 17].

$$X(\%) = \frac{C_i - C_f}{C_i} \times 100 \tag{2}$$

Optimization of Batch Process Systems experiment design was carried out using Design Expert 7.0. The RSM ascertain the number of experiments for the optimization. A full-factorial central composite design (CCD) was adapted [2] involving the three independent variables comprising of contact time, biosorbent dosage and temperature as presented in Table 1. The range of values chosen for each independent parameter was based on information from literature [2].

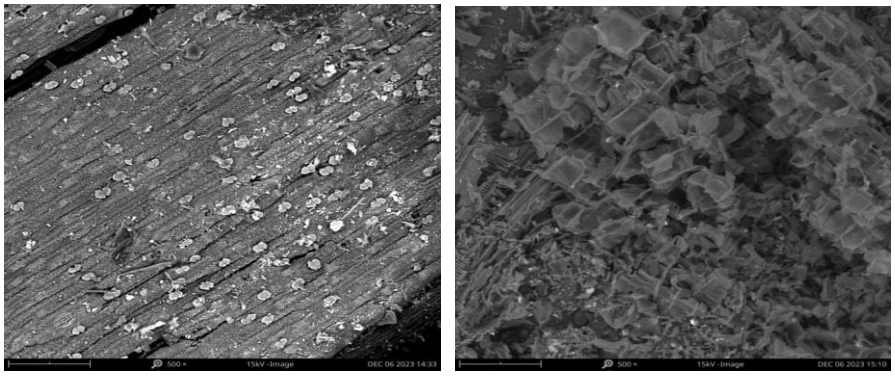
Table 1. Sorption variables and Levels in RSM Design

Variables	Symbols	-1	0	+1
Contact Time (min)	A	60	90	150
Temperature (°C)	B	36	37.5	50
Sorbent Dosage (g)	C	2	6	10

3 RESULTS AND DISCUSSION

3.1 Characterization of Biosorbent

The SEM images results for activated sugarcane bagasse before and after sorption of lead ions are presented in Plate 3.



(a) (b)

Plate 3. SEM images at 500 X magnification for activated sugarcane bagasse before (a) and after (b) sorption

SEM images are shown of the sugarcane bagasse in Plates 3a and 3b before the metal uptake and after the metal uptake respectively. The magnification power of both images was set to 500 X magnification. They both also have a rough and irregular shape. It is observed that after sorption as in Plate 3b the pores have become more larger because of the metal uptake [2, 8, 10]. The FTIR results for sorption before and after are presented Figures 1and 2, respectively.

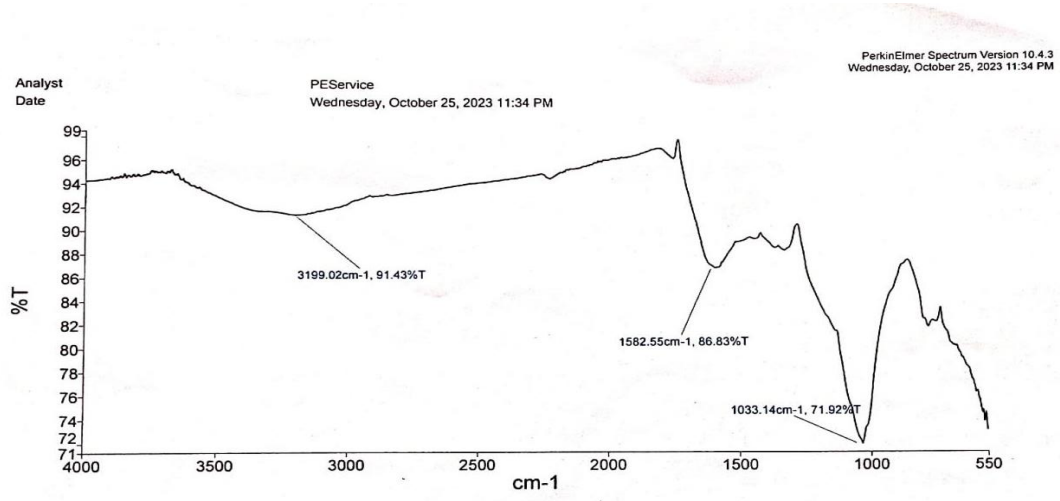


Figure 1. FTIR spectrum for activated sugarcane bagasse before sorption

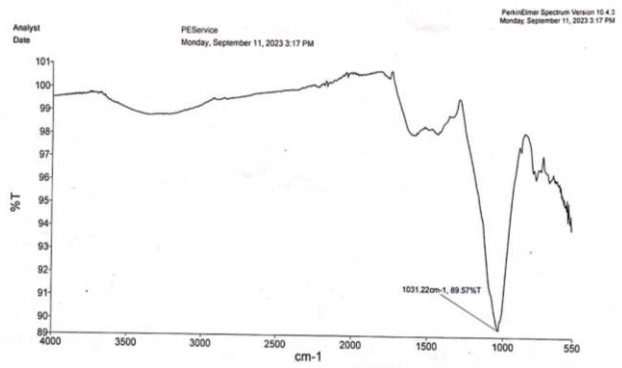


Figure 2. FTIR spectrum for activated sugarcane bagasse after sorption

The peaks in the FTIR spectrum at 3199.02 cm^{-1}, 1582.55 cm^{-1}, and 1033.14 cm^{-1} (Figure 1) correspond to specific vibrational frequencies of functional groups in the sample. The peak at 3199.02 cm^{-1} is indicative of the stretching vibration of O-H bonds, suggesting the presence of alcohol or phenol groups. The peak at 1582.55 cm^{-1} is likely associated with C=C stretching vibrations, indicating the presence of aromatic compounds. The peak at 1033.14 cm^{-1} may be attributed to C-O stretching, suggesting the presence of ethers or alcohols. In spectrum after sorption (Figure 2) a notable change of a peak at 1031.22. cm^{-1} which is a slight variation from 1033.14 cm^{-1}. The slight variations recorded in the FTIR peaks of the samples before and after the sorption indicates that there was sorption of the lead ions on the surface of the sugarcane bagasse activated carbon [8, 10].

3.2 Process Optimization

The results for batch process system optimization of lead ions onto activated sugarcane bagasse for the three variables (contact time (A), temperature (B), and sorbent dosage (C)) that can affect sorption considered in this investigation with their responses, uptake of metal ions by the sorbent (q) and percentage removal of sorbate from Lake Gerio water (X) were evaluated for each run and are presented in Tables 2 - 6 and Figures 3- 6. The total number of experimental runs performed were 20, with 8 factorial point runs, 6 axial point runs, and 6 center point runs as replicate which helps to reduce experimental error [2, 12]. The results for experimental design for optimization of sorption of lead ions is displayed in Table 2.

Table 2. Experimental Design for optimization of sorption

Run	Factor 1 A	Factor 2 B	Factor 3 C	Response 1 Q (mg/g)	Response 2 X (%)	Q predicted	X predicted
1	30	50	2	0.002	18.86	0.002	18.86
2	90	37.5	6	0.003	86.79	0.0028	78.95
3	150	37.5	6	0.0011	32.08	0.0011	32.08
4	90	37.5	6	0.0032	92.45	0.0028	78.95
5	150	36	2	0.0074	69.81	0.0074	69.81
6	90	36	6	0.0016	47.17	0.0016	47.17
7	150	50	2	0.0058	54.71	0.0058	54.71
8	90	37.5	2	0.003	28.3	0.003	28.3
9	30	36	2	0.0094	88.68	0.0094	33.68
10	90	37.5	6	0.0027	77.38	0.0028	78.95
11	30	36	10	0.0014	64.15	0.0014	64.15
12	90	37.5	10	0.0012	56.6	0.0012	56.6
13	30	36	10	0.0017	81.13	0.0017	81.13
14	90	37.5	6	0.0027	77.35	0.0028	78.95
15	150	50	10	0.0011	50.94	0.0011	50.94
16	150	36	10	0.0011	50.94	0.0011	50.94
17	90	37.5	6	0.0024	69.92	0.0028	78.95
18	90	37.5	6	0.0025	69.81	0.0028	78.95
19	90	50	6	0.0025	69.81	0.0025	69.81
20	30	37.5	6	0.001	28.3	0.001	28.3

The results for model selection for uptake of metal ions by the sorbent (q) and percentage removal of sorbate from Lake Gerio water (X) are presented in Tables 3 and 4, respectively.

Table 3. Model selection for q

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	
Linear	0.0123	0.0002	0.3874	0.0136	
2FI	0.3709	0.0002	0.4026	-1.1892	
Quadratic	0.2072	0.0002	0.4976	-1.4045	Aliased
Cubic	0.0002		0.9816		Suggested

Table 4. Model selection for X

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2
Linear	0.5323	0.0168	-0.0392	-0.3946
2FI	0.2747	0.0175	0.0410	-0.2450
Quadratic	0.2318	0.0190	0.1727	-0.8590 Aliased
Cubic	0.0190		0.8190	Suggested

Regarding Tables 3 and 4, the Design expert software suggested a cubic model for both uptake of lead ions by the activated sugarcane bagasse as sorbent (q) and for percentage removal of lead ions from Lake Gerio water (X). Table 5 shows the results for analysis of variance (ANOVA) for uptake of lead ions by the sorbent (q).

Table 5. ANOVA for q

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0001	14	6.682E-06	73.43	< 0.0001	significant
A-Time	9.572E-06	1	9.572E-06	105.18	0.0002	
B-Temperature	9.373E-09	1	9.373E-09	0.1030	0.7612	
C-Dosage	0.0000	1	0.0000	119.71	0.0001	
AB	9.122E-06	1	9.122E-06	100.24	0.0002	
AC	0.0000	1	0.0000	125.87	< 0.0001	
BC	0.0000	1	0.0000	110.37	0.0001	
A^2	4.641E-07	1	4.641E-07	5.10	0.0735	
B^2	1.138E-06	1	1.138E-06	12.51	0.0166	
C^2	6.338E-07	1	6.338E-07	6.96	0.0460	
ABC	4.904E-06	1	4.904E-06	53.89	0.0007	
A^2B	2.143E-06	1	2.143E-06	23.55	0.0047	
A^2C	0.0000	1	0.0000	112.84	0.0001	
AB^2	9.692E-06	1	9.692E-06	106.50	0.0001	
AC^2	0.0000	0				
B^2C	0.0000	1	0.0000	119.86	0.0001	
BC^2	0.0000	0				
A^3	0.0000	0				
B^3	0.0000	0				
C^3	0.0000	0				
Pure Error	4.550E-07	5	9.100E-08			
Cor Total	0.0001	19				
Std. Dev.	0.0003	R^2	0.9952			
Mean	0.0028	Adjusted R^2	0.9816			
C.V. %	10.62	Predicted R^2	NA(1)			
		Adeq Precision	32.1535			

Table 5 shows some statistical analysis (ANOVA) for uptake of Lead ions *q* by the sorbent. Luka *et al.*, [2] and Luka *et al.*, [19] state that the P-values less than 0.0500 indicate model terms are significant. The results show among the main three variables, that the temperature (B) does not have a significant

influence on the uptake of the lead ions, having P-value of 0.7612. Another variable not significant is the quadratic variable (A^2) with P-valve of 0.0735. The F-value depicts the strength of the effect of each parameter to response. In addition, higher F-value expressed stronger influence of variable on response [12,18,19]. Table 5 shows that significant level and higher F-values was recorded for the two main variables A (Contact time) and C (sorbent dosage). This implies a very strong influence on the lead ion uptake from Lake Gerio water. The model was found to be significant at F-value of 73.43 and P-value of < 0.0001 [2,19]. Table 6 depicts the results for analysis of variance (ANOVA) for percentage removal of lead ions from Lake Gerio water (X).

Table 6. ANOVA for X

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	8274.37	14	591.03	7.14	0.0201	significant
A-Time	2085.26	1	2085.26	25.20	0.0040	
B-Temperature	0.3805	1	0.3805	0.0046	0.9486	
C-Dosage	3221.04	1	3221.04	38.92	0.0015	
AB	1040.65	1	1040.65	12.57	0.0165	
AC	4430.24	1	4430.24	53.53	0.0007	
BC	3107.57	1	3107.57	37.55	0.0017	
A^2	515.71	1	515.71	6.23	0.0547	
B^2	883.46	1	883.46	10.67	0.0223	
C^2	1998.38	1	1998.38	24.15	0.0044	
ABC	2417.50	1	2417.50	29.21	0.0029	
A^2B	1532.66	1	1532.66	18.52	0.0077	
A^2C	3139.55	1	3139.55	37.93	0.0016	
AB^2	2218.25	1	2218.25	26.80	0.0035	
AC^2	0.0000	0				
B^2C	3174.29	1	3174.29	38.35	0.0016	
BC^2	0.0000	0				
A^3	0.0000	0				
B^3	0.0000	0				
C^3	0.0000	0				
Pure Error	413.82	5	82.76			
Cor Total	8688.19	19				
Std. Dev.	9.10	R^2	0.9524			
Mean	58.01	Adjusted R^2	0.8190			
C.V. %	15.68	Predicted R^2	NA(1)			
		Adeq Precision	7.9036			

Similarly, Table 6 shows the ANOVA for the percentage removal of lead ions *X* by the activated sugarcane bagasse as sorbent. It is also observed that the temperature (B) does not have a significant influence on the percentage removal of the lead ions, having P-value of 0.9486. Also, the quadratic variable (A^2) has shown to be insignificant which is like that of q in Table 5. The two main variables A (time) and C (dosage) have a very strong influence on the lead ion uptake from Lake Gerio water with a significant level and higher F-values as shown in the table. The model was found to be significant at F-value of 7.14 and P-value of < 0.0201. From the fit summary in Tables 3 and 4 for q and X respectively the quadratic model was aliased. Cubic function regression models developed in terms of coded factors for the uptake and percentage removal of lead by the sorbent were optimized based on the response factors (q and X) the two independent variables. The uptake of the lead ions investigated by the biosorbent are given in Equations 3 and 4 for *q* and *X* yields, respectively.

$$q = +0.0048 +0.0123A + 0.0001B + 0.1310C + 0.0014AB - 0.0031AC - 0.0240BC - 0.0005A^2 - 0.0024B^2 - 0.0007C^2 + 0.0020ABC + 0.0013A^2B + 0.0726A^2C - 0.0129AB^2 + 0.0000AC^2 - 0.1808B^2C + 0.0000BC^2 + 0.0000A^3 + 0.0000B^3 + 0.0000C^3 \quad (3)$$

$$X = + 135.62 + 181.76A + 0.3353B + 2252.58C + 14.76AB - 61.35AC - 422.32BC - 16.67A^2 - 66.14B^2 - 36.50C^2 + 44.84ABC + 34.77A^2B + 1268.93A^2C - 195.10AB^2 + 0.0000AC^2 - 3084.56B^2C + 0.0000BC^2 + 0.0000A^3 + 0.0000B^3 + 0.0000C^3 \quad (4)$$

Figures 3 and 4 show the plot of predicted versus actual for uptake of lead ions by the sorbent (q) and percentage removal of lead ions *X* by the sorbent, respectively.

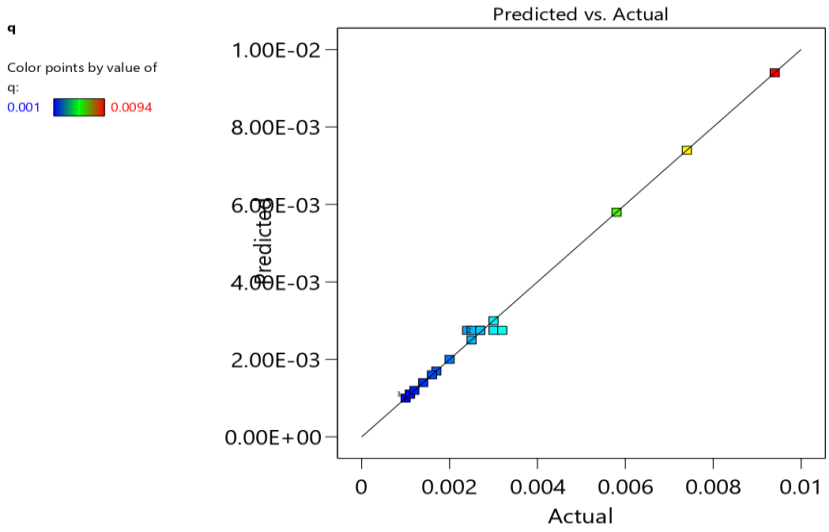


Figure 3. q predicted vs actual values.

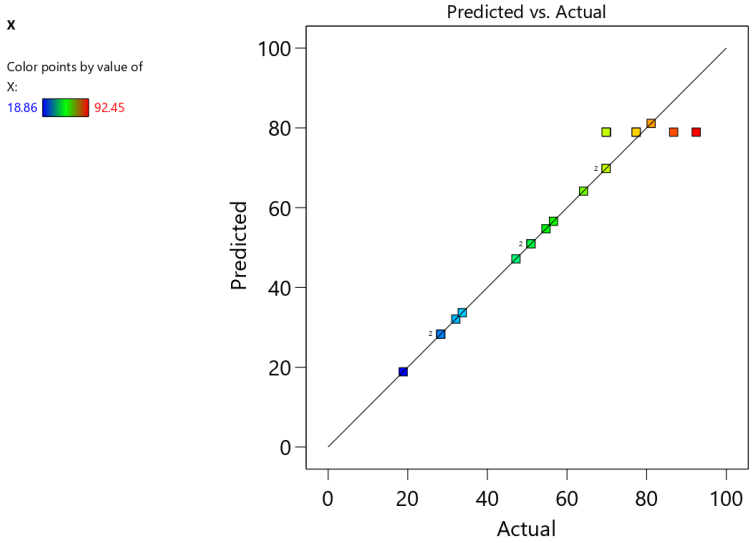


Figure 4. X predicted vs actual values.

Figures 3 and 4 present the findings of the correlation between the predicted and actual q and X. The actual indicated by the dots closely aligned with the predicted dots indicated by the diagonal line. This implies that there is a very high correlation between the predicted result of the model and the data. Therefore, the model will be able to predict q (the uptake of Pb) and X (percentage removal with accuracy) with high accuracy [19, 20]. Figures 5 and 6 show interaction of variables and their effects on response which is normally investigated with the aid of three-dimension plots [2, 12].

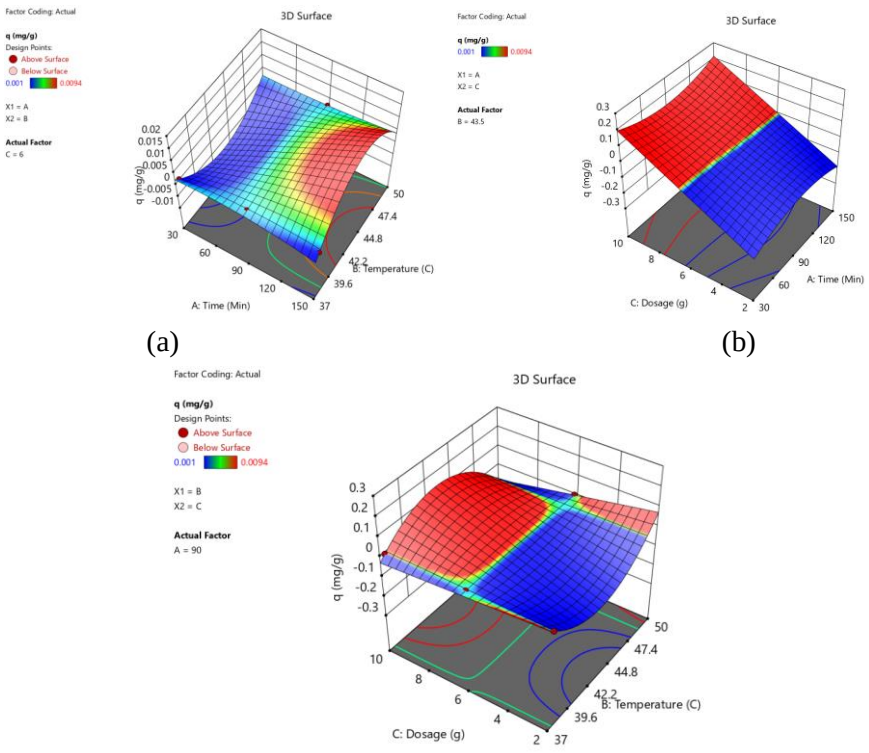


Figure 5. Combined response plot of q versus A-B, A-C, and C-B

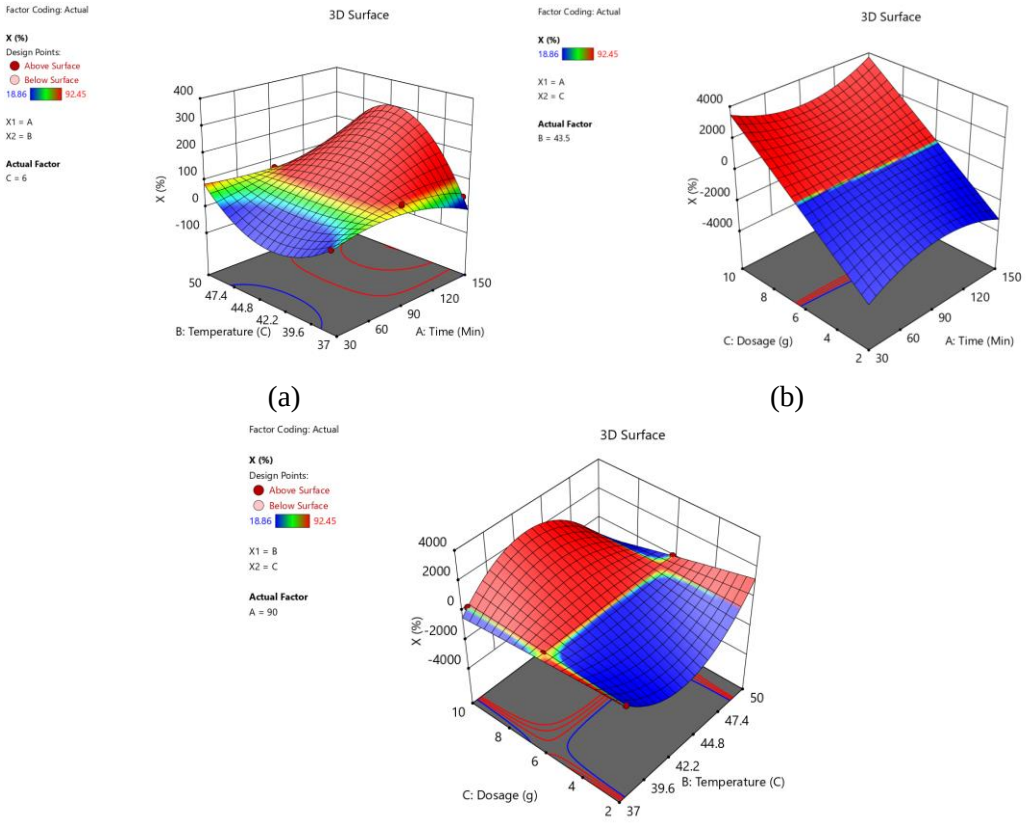


Figure 6. Combined response plot of X versus A-B, A-C, and B-C

From Figure 5 Lead uptake q increases with increase in biosorbent dosage and increase in temperature up to a certain point then begins to decrease, also slightly decrease with time after a certain point. Similarly, in Figure 6 the percentage removal X increases with increase in temperature till about a certain point and begins to decrease, like wise with time. Lastly X increases with increase in dosage.

4 CONCLUSIONS

The following conclusions were deduced from the study: The SEM showed a visual change in the pore structure of the activated sugarcane bagasse confirms the uptake of lead ions onto the activated sugarcane bagasse. The FTIR displayed some change in peaks recorded before and after sorption which also confirmed for binding of lead ions onto the activated sugarcane bagasse. The contact time and sorbent dosage were shown to have more significant effect on the sorption of the lead ions given by q and X responses. The maximum uptake and percentage removal by activated sugarcane bagasse sorbent was recorded as 0.0074 mg/g and 92.45% for lead ions, respectively.

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