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Optimization Of The Adsorption Process In Landfill Barrier Using Response Surface Methodology (Rsm).

Asogwa E. O., Adie D. B., Ibrahim F. B., Amadi A. A., Saidu, M., Adesiji A. R., Mangey J.A
[1,4,5,6] *Civil Engineering Department, Federal University Of Technology Minna, Nigeria.*
[2,3,7] *Water Resources And Environmental Engineering Department, Ahmadu Bello University Zaria, Nigeria.*

Abstract

Response Surface Methodology was used to examine the percentage of heavy metal removal from a sanitary landfill site utilising a lateritic soil-geopolymer composite. By Using the model, the lateritic soil geopolymer composite's adsorption potential was enhanced. The applicability of the employed model to predict the adsorption state is supported and confirmed by the good agreement between the observed and anticipated values of the removal efficiency. The applied models showed that all three of the components examined had an impact on the removal of heavy metals from sanitary landfill liners, but that the effects of dosage and contact time were more pronounced and had a substantial impact on the removal % of heavy metals. With dosage and contact time proving to be the most relevant of the three independent variables, the ANOVA findings show that the model parameters are significant. After refining the replies, the following settings proved ideal: dosage of 10g, contact time of 48 hours, and temperature of 50 °C. These yielded percentage removals of lead, zinc, and copper of 97.88%, 94.36%, and 99.48%, sequentially.

Keywords: *Heavy Metals, Geopolymer, Landfill, Barrier, Optimization, Lateritic-soil, Response Surface Methodology (RSM)*

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I. Introduction

The majority of the hazardous pollutants found in leachate from sanitary landfills include organic waste, ammoniacal nitrogen ($N–NH_3$), and heavy metals, all of which are harmful to the environment and public health [1-3]. In addition to having a negative impact on soil surface and groundwater [8–10], incorrect disposal of leachate that has not been appropriately treated can also have a negative impact on population health and quality of life [11]. Therefore, for the purpose of public health safety and environmental sustainability, sanitary landfill barriers should be installed in landfill sites. These barriers are crucial for preserving both underground water, surface water and consequently the environment in general. A barrier that will not only house the waste but plays vital role in contaminant adsorption should be encouraged, hence a synthesized eco-friendly geopolymer was adopted and mixed in proportion with lateritic soil for the barrier development. Optimising Contaminant adsorption in the lateritic soil- geopolymer composite developed was carried out using response surface methodology (RSM) to improve the adsorption efficacy. The model performance demonstrated a significant level of contaminant adsorption and therefore should be applied to enhance heavy metal removal efficiency in lateritic soil geopolymer composite landfill barrier system (Table 3, 4 and 5) [12, 13].

II. Materials And Methods

Materials

- Leachates Sample
- Soil Sample
- Geopolymer
- Sieves of Different Sizes
- Incubator
- Distilled water
- Tap water

Chemicals

Both sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) of analytical grade were obtained from Central Research Laboratory Ilorin and used without additional purification. All of the solutions were made using deionized distilled water. All glassware was washed with HNO_3 and then rinsed with double distilled water.

Methods

Preparation of metakaolin based geo-polymer and lateritic soil geo-polymer Composites

Figure 1.0 below shows the stages involved in the synthesis of the geo-polymer sample. Initially, Na_2SiO3 powder and sodium hydroxide NaOH (12 M) were dissolved at a mass ratio of 2.5 to create the activator solution. After stirring the mixture for fifteen minutes at room temperature. Metakaolin and activator solution are combined in a mixer with continuous stirring at room temperature for 15 minutes in order to achieve adequate homogeneity. This is the second step in the elaboration process. After that, to get the appropriate workability of the geopolymer paste, distilled water will be added at a water/metakaolin ratio of 0.34. After the mixture is put into a cylindrical mould, it will be treated for 24 hours at 60°C. In order to characterise and examine the adsorption tests, the matrix was lastly crushed, sieved, and kept in a desiccator with particle sizes less than 200 μm. According to a sieve analysis, 92% of the air-dried material passes through the BS No. 200 sieve. For a geopolymer amendment of 0, 5 and 10%, 16g of lateritic soil geopolymer composite was employed as the adsorbent.

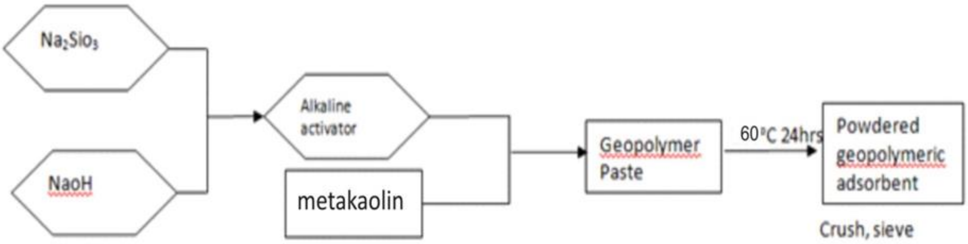


Figure. 1: Preparation process of metakaolin-based Geopolymer

Three independent aspects were taken into consideration while using the RSM to optimise the adsorption of heavy metal ions: dosage, temperature, and contact duration. Design Expert Version 11 statistical software was used to conduct the analysis. Equation 1 describes how the uncoded independent variables from the Box Behnken design (BBD) were used to create the second-order polynomial equation.

$Y= \beta_0 + \beta_1A + \beta_2B + \beta_3C + \beta_{12}AB + \beta_{13}AC + \beta_{23}BC + \beta_{11}A^2 + \beta_{22}B^2 + \beta_{33}C^2$ …………. (**1**)

where A is the dosage, B is the contact time, and C is the temperature. Additionally, β1, β2, and β3 are linear coefficients; β11, β22, and β33 are interaction coefficients; and Y is the expected response (%). Finally, β0 is the intercept coefficient. The experimental design points utilised in the three-variable interaction trials are described in Table 2.

Table 1 displays the BBD design along with the RSM experiment results for the adsorption investigation, which produced twenty runs depending on how the three variables interacted. The cubic and quadratic models were fitted to the experimental data. To explain how dosage, temperature, and contact time affect Pb, Zn, and Cu ion adsorption, the quadratic model was selected. Equations 2 through 4 govern the generated quadratic model of the metal ion adsorption processes in terms of coded components, and ANOVA was used to assess the statistical analysis of the mathematical models.

Utilising the regression coefficient (R^2), Fisher test values, and lack of fit, one might assess the statistical analysis derived from mathematical models through ANOVA. The results of the quadratic model fitting using ANOVA analysis are shown in Table 3,4 and 5 and the produced mathematical model's capacity is indicated by the low p-values (less than 0.0500) and Fisher values. In addition, the model was tested with predicted versus real plots, as shown in Figures. 3(c), 4(c), and 5(c).

III. Result And Discussion

X- ray Fluorescence (XRF) or Oxide Composition of Kaolin Metakaolin and Geopolymer

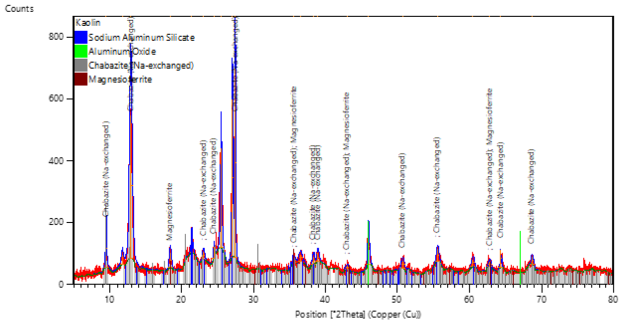
The high percentage of Aluminum Oxide and silicon Oxide found in raw kaolin, makes it a good precursor for geopolymerization. The values of the silicon to aluminum ratio and the loss in ignition are good indicators that the formed geopolymer is of high quality which will enhance the durability of the sanitary landfill liner and improve contaminant adsorption. The formed geopolymer have porous structure that is both linked and open, with a negatively charged surface, which are all important for adsorption operations.

Table 1: Oxide Composition of Kaolin, Metakaolin and Geopolymer

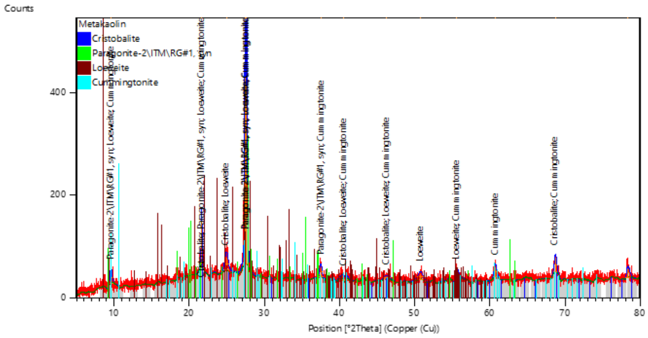
Oxides (Wt %)	Kaolin	Metakaolin	Geopolymer
Fe_2O_3	3.12	2.45	1.69
Al_2O_3	27.8	20.08	13.87
SiO_2	40.06	38.7	31.79
Cao	3.07	2.43	1.75
SO_3	5.11	4,29	3.64
MgO	15.02	8.97	5.99
K_2O	1.98	1.94	1.18
Na_2O	10.82	15.06	1.23
Loss in Ignition	7.91	8.13	0.896
SiO_2/AlO_3	1.44	1.92	2.29

X-Ray Diffraction (XRD) of Kaolin, Metakaolin and Geopolymer

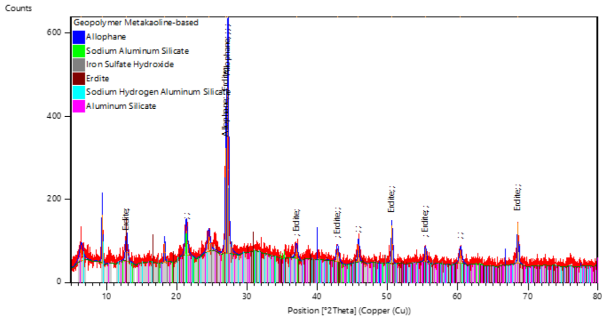
The result of the XRD showed the raw kaolin clay is rich in quartz and kaolinite minerals. There is disappearance of peaks associated to quartz and kaolinites after calcination, which is due to dihydroxylation of water molecules that exist in the quartz and kaolinite minerals in the metakaolinite by heat treatment. This peak disappearance as the kaolin metamorphose to geopolymer is accompanied by reduction in crystallinity and increase in amorphousity which translates to increase in adsorption and mechanical strength of the geopolymer.



(a) XRD of Kaolin



(b) XRD of Metakaolin



(c) XRD of Geopolymer

Figure 2.0: X-ray diffraction (XRD) of Kaolin Metakaolin and Geopolymer

Table 2: The actual and predicted values of metal ions removal

Run	Dosage (g)	Time (hr)	Temp (°C)	Actual Pb (%)	Predicted Pb (%)	Actual Zn (%)	Predicted Zn (%)	Actual Cu (%)	Predicted Cu (%)
1	5	24	50	85	85.5	81.19	84.19	93.81	89.59
2	0	24	30	67.65	68.35	80.1	78.2	79.29	78.6
3	0	0	50	62.88	61.86	68.2	68.25	80.8	80.61
4	5	24	50	78.7	85.5	80.51	84.19	81.34	89.59
5	5	24	50	77.32	85.5	79.05	84.19	83.81	89.59
6	5	0	30	70.3	70.62	72.54	74.39	85.09	85.97
7	5	24	50	90.1	85.5	91.36	84.19	93.4	89.59
8	10	24	70	90.86	90.16	78.66	80.56	81.39	82.08
9	10	0	50	73	74.94	76.71	74.53	82.93	81.39
10	10	24	30	93.76	91.51	89.49	89.83	88.87	89.53
11	5	24	50	93.17	85.5	90.85	84.19	94.1	89.59
12	5	48	30	90.58	91.81	92.81	92.52	89.95	89.11
13	0	24	70	68.56	70.81	72.44	72.1	76.69	76.03
14	10	48	50	96.86	97.88	94.41	94.36	99.29	99.48
15	5	24	50	83.81	85.5	80.56	84.19	95.05	89.59
16	5	24	50	85.46	85.5	83	84.19	84.1	89.59
17	5	0	70	78.83	77.6	68.48	68.77	72.88	73.72
18	5	24	50	90.46	85.5	87	84.19	91.1	89.59
19	5	48	70	86.26	85.94	84.61	82.77	92.22	91.34
20	0	48	50	70.39	68.45	78.37	80.55	81.75	83.28

Lead percentage removal

One of the most crucial and vital processes in the sanitary landfill system is lead removal. For the sake of environmental sustainability, a sanitary landfill's lead concentration must be reduced as much as possible. In light of this, research was done on the effects of the three independent design variables: dosage, time, and temperature. To describe the link between the three independent variables and the dependent response (Lead), the best-fitting quadratic model was created. Equation 2 represents the quadratic model.

$$Pb = 85.63 + 10.63A + 7.39B + 0.2775C + 4.09AB - 0.9525AC - 3.21BC - 5.57A^2 - 4.28B^2 + 0.1425C^2 \ldots\ldots\ldots \text{(2)}$$

The analysis of variance (ANOVA) results for Lead (Pb) as response factor is shown in Table 3. The result depicted a successful fitting of experimental data to the quadratic model. The model F- value of 7.83 implies that the model is significant. All the model terms are significant but Dosage is found to be the most influential of all the variables in Lead reduction as can be seen in Table 3 where F-value for Dosage is 36.91 indicating a strong influence on Lead. A plot of actual experimental values against the predicted values is shown in figure 1c. It could be observed that the points representing the experimental values diverged a little from the regression line that represents the predicted values. Fig 3a represents the response surface interaction between Dosage and contact time while Figure 3b demonstrates the contour plot of Lead removal.

Table 3: Lead percentage removal using ANOVA for quadratic model

%Pb removal	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	1724.60	9	191.62	7.83	0.0017	Significant
A-Dosage	903.13	1	903.13	36.91	0.0001	Significant
B-Time	436.31	1	436.31	17.83	0.0018	Significant
C-Temp.	0.6160	1	0.6160	0.0252	0.8771	Not significant
AB	66.83	1	66.83	2.73	0.1294	Not significant
AC	3.63	1	3.63	0.1483	0.7082	Not significant
BC	41.28	1	41.28	1.69	0.2231	Not significant
A^2	141.70	1	141.70	5.79	0.0369	Significant
B^2	83.84	1	83.84	3.43	0.0939	Not significant
C^2	0.0928	1	0.0928	0.0038	0.9521	Not significant
Residual	244.70	10	24.47			
Lack of Fit	23.94	3	7.98	0.2531	0.8569	not significant

Key (Pb): F-value = Fisher value; degree of freedom, P = probability; $R^2 = 0.8757$, Adjusted $R^2 = 0.7639$ and Predicted $R^2 = 0.7091$

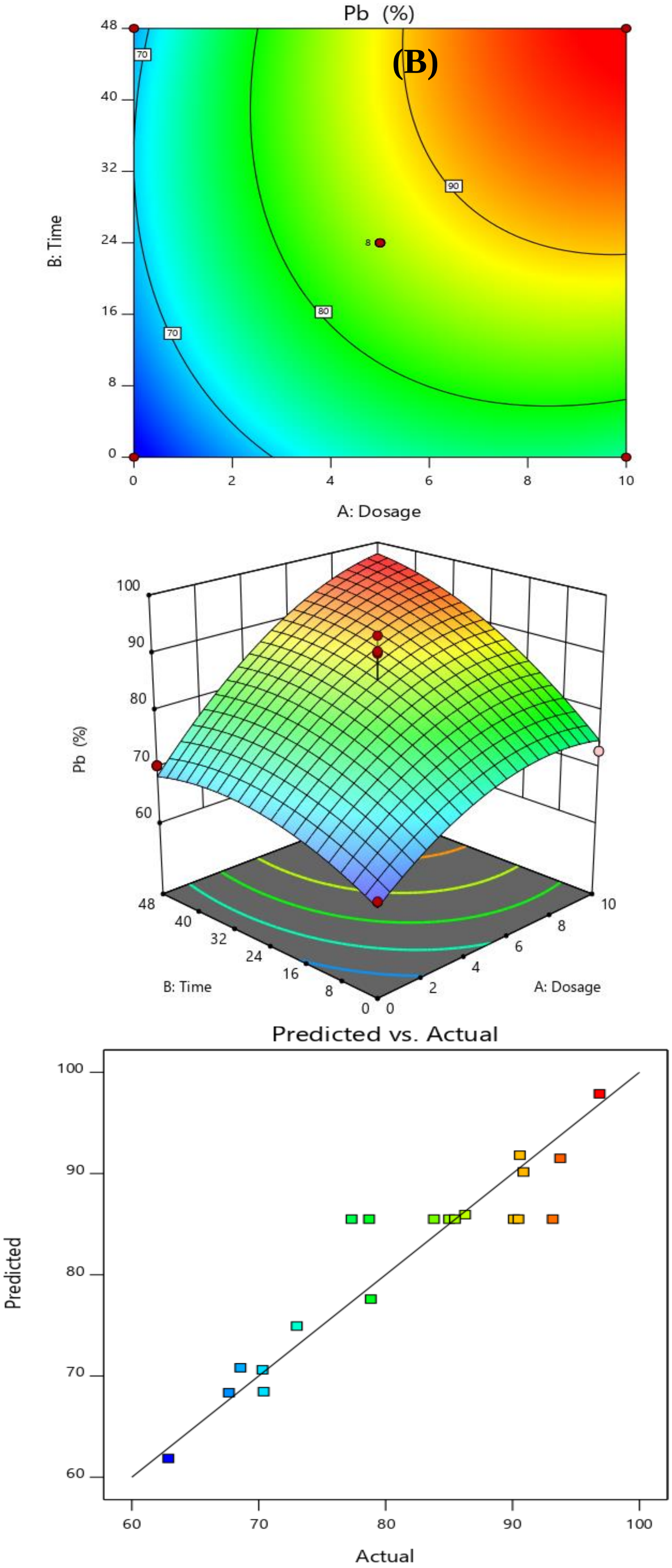


Figure 3: (a) Response surface interaction between contact time and dosage for the removal of Pb ion (b) Contour plot of Pb ion removal (c) Plot of predicted against actual values for the removal of Pb ion

Zinc Percentage Removal

Zinc reduction in a sanitary landfill site is another challenge that demands critical attention in attaining environmental sustainability. Environmental contamination of Zinc from the sanitary Landfill leachate has raised a concern over the years because of the resultant implication on the surface water, underground water and health of the populace.

It is important to investigate the influence of the three independent design variables on Zinc percentage removal. The model equation (a second order polynomial) from the statistical design using Box-Behnken method is shown in Equation (3). A quadratic model gave the best fitting of the experimental values and was used to express the relationship between the dependent and the independent variables.

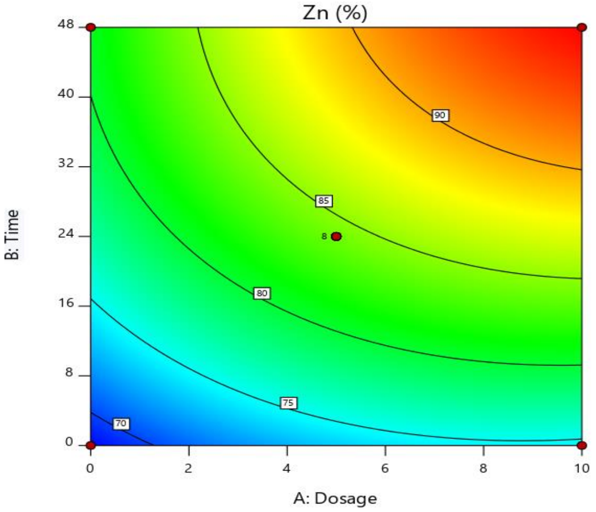
$$Zn = 84.19 + 5.02A + 8.03B - 3.846C + 1.88AB - 0.7925AC - 1.04BC - 2.10A^2 - 2.66B^2 - 1.92C^2 \quad \textbf{(3)}$$

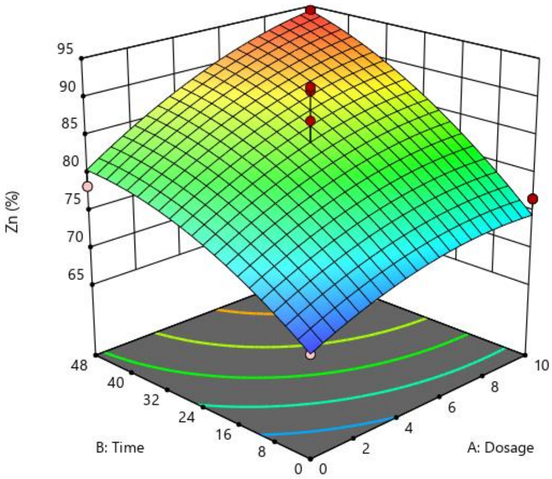
The ANOVA results presented in Table 3 demonstrates the significance of the quadratic model as depicted by the model F- value of 5.54. A model p-value of 0.0066 also indicate model terms are significant. The p-values of all other model coefficients, with the exception of temperature, are all significant. A correlation co-efficient of (R^2) of 0.8330 was achieved for the model. Figure 4c shows the comparison between the actual and predicted values of the experimental results while figure 4a and 4b represents the response surface interaction of dosage and contact time for zinc ion removal and contour plot of zinc ion removal respectively.

Table 4: Zinc percentage removal using ANOVA for quadric Model

% Zn removal	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	953.58	9	105.95	5.54	0.0066	Significant
A-Dosage	201.60	1	201.60	10.55	0.0088	Significant
B-Time	516.33	1	516.33	27.01	0.0004	Significant
C-Temp.	118.20	1	118.20	6.18	0.0322	Significant
AB	14.18	1	14.18	0.7416	0.4093	Not significant
AC	2.51	1	2.51	0.1314	0.7245	Not significant
BC	4.28	1	4.28	0.2242	0.6461	Not significant
A^2	20.21	1	20.21	1.06	0.3281	Not significant
B^2	32.47	1	32.47	1.70	0.2217	Not significant
C^2	16.76	1	16.76	0.8770	0.3711	Not significant
Residual	191.15	10	19.12			
Lack of Fit	23.94	3	7.98	0.3340	0.8015	not significant

Key (Zn): F-value = Fisher value; degree of freedom, P = probability; R^2 = 0.8330, Adjusted R^2 = 0.8027 and Predicted R^2 = 0.7746





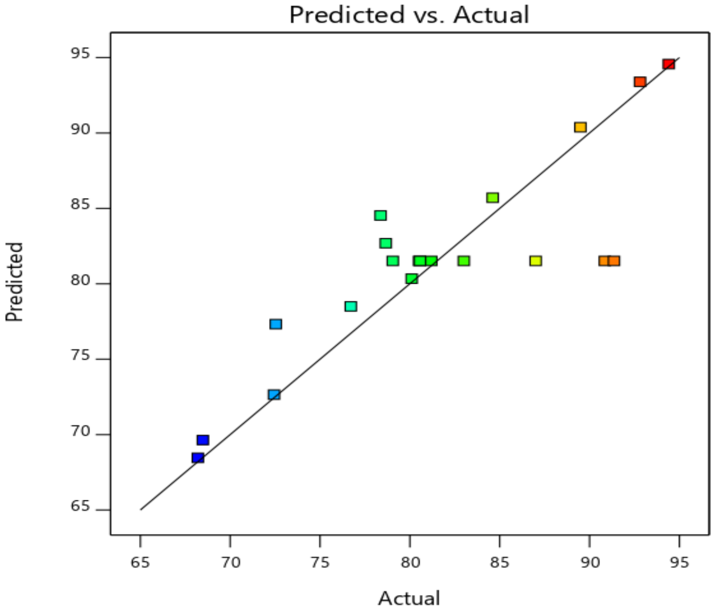


Figure 4. (a) Response surface interaction between contact time and dosage for the removal of Zn ion (b) Contour plot of Zn ion removal (c) Plot of predicted against actual values for the removal of Zn ion

Percentage Copper Removal

The efforts geared towards copper reduction in sanitary landfill leachate cannot be over emphasized because of the damaging effects of Zinc ions on the environment. The influence of the three independent variables; dosage, contact time and temperature on the copper removal was investigated and a model derived from the statistical analysis. The quadratic model gave the best fit and was used to describe the relationship the percentage copper rection as a response and the independent variables. The response equation is represented in equation 4 thus;

$Cu = 89.59 + 4.24A + 5.19B - 2.50C + 3.85AB - 1.22AC + 3.62BC - 3.44A^2 + 0.0394B^2 - 4.59C^2$ ……………………………………………………………………………. ……………… **(4)**

The ANOVA result shown in Table 4 shows all the model terms are significant, with model F-value of 3.49 and P-value of 0.0322. It also shows that the model terms are significant except temperature. Contact time and Dosage are the most influential and significant terms as can be seen from their F and P values. The R^2 is 0.7586. A plot of variation of contact time versus dosage as a response is presented in figure 5a and b while the graph of actual versus predicted response is presented in figure 5c.

Table 5: percentage removal of copper using ANOVA for quadratic model

% Cu removal	**Sum of Squares**	**Df**	**Mean Square**	**F-value**	**p-value**	
Model	709.97	9	78.89	3.49	0.0322	Significant
A-Dosage	144.08	1	144.08	6.38	0.0301	Significant
B-Time	215.39	1	215.39	9.53	0.0115	Significant
C-Temp.	50.10	1	50.10	2.22	0.1673	Not significant
AB	59.37	1	59.37	2.63	0.1361	Not significant
AC	5.95	1	5.95	0.2635	0.6189	Not significant
BC	52.42	1	52.42	2.32	0.1587	Not significant
A^2	53.96	1	53.96	2.39	0.1533	Not significant
B^2	0.0071	1	0.0071	0.0003	0.9862	Not significant
C^2	96.44	1	96.44	4.27	0.0657	Not significant
Residual	225.94	10	22.59			
Lack of fit	23.83	3	0.76	0.2432	0.8367	Not significant

Key (Cu): F-value = Fisher value; degree of freedom, P = probability; $R^2 = 0.7586$, Adjusted $R^2 = 0.7413$ and Predicted $R^2 = 0.7345$

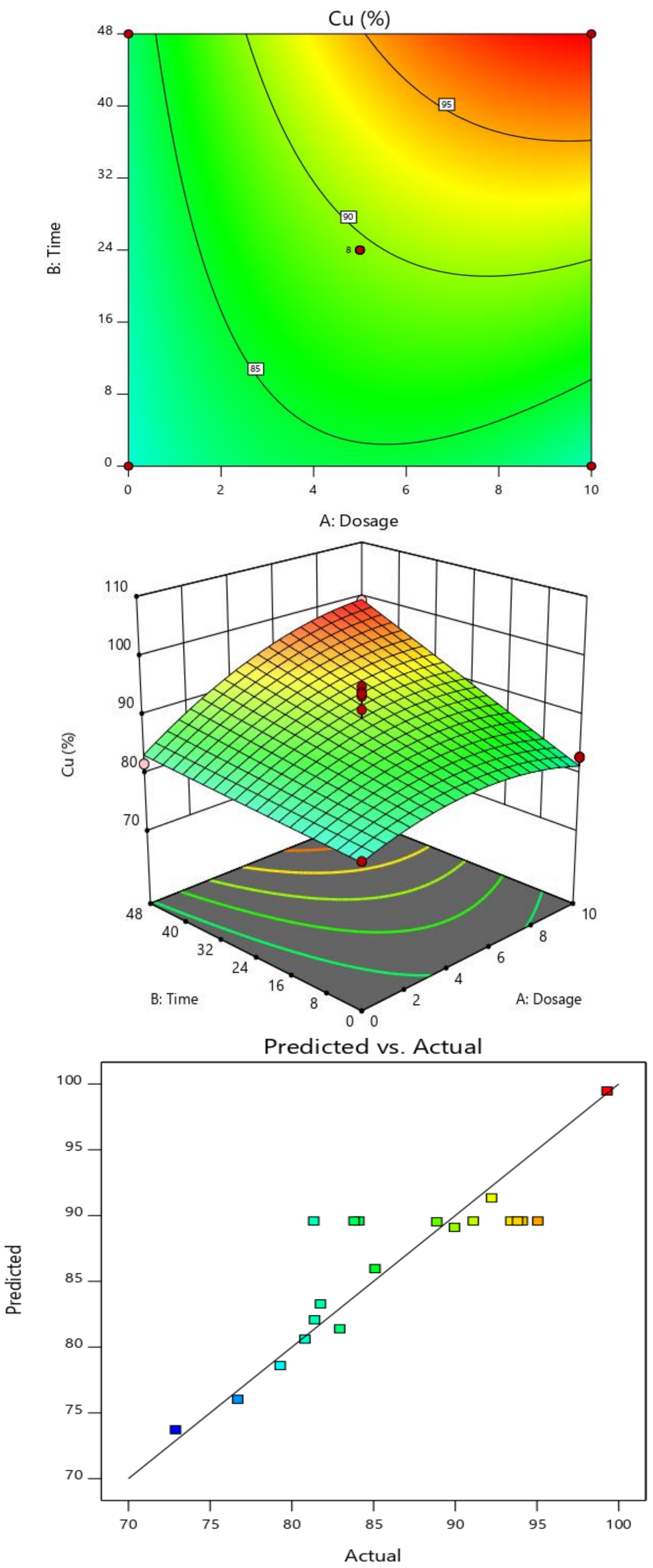


Figure 5. (a) Response surface interaction between contact time and dosage for the removal of Cu ion (b) Contour plot of Cu ion removal (c) Plot of predicted against actual values for the removal of Cu ion

Table 6: Optimal Pb, Zn and Cu Removal Condition generated from the Response

Dosage (%)	Time (hr)	Temp. (°C	Pb Removal (%)	Zn Removal (%)	Cu Removal (%)	Desirability
10	48	50	97.88	94.36	99.48	

Validation of the Model Using Experimental Results

Laboratory experiments were carried out considering the performed optimal conditions to confirm the optimization results. The Box Behnken Design predicted heavy metal removal to the tune of 2.77mg/l, 2.23mg/l and 3.12mg/l for Pb. Zn and Cu respectively. The result of the laboratory experiments is in tandem with the one obtained using Response Surface Methodology and therefore validates the results of the Optimization. Analysis of variance (ANOVA) was used to determine the level of significance of the model.

%Error = [(Actual – Predicted Value)/Actual Value]x100 ……………………. (5)

Table 7: Validation of Experimental results at optimum conditions

Optimum Condition	Pb Removal Efficiency (%)	Zn Removal Efficiency (%)	Cu Removal Efficiency (%)	Pbq(mg/g)	Znq(mg/g)	Cuq(mg/g)
Experimental Results	96.86%	94.41%	99.29%	2.196	2.228	1.684
Model Response	97.88%	94.36%	99.48%	2.219	2.227	1.687
Percentage Error	1.04%	0.05%	0.19%	1.05	0.04	0.77
Standard Deviation	±0.72	±0.03	±0.13	±0.02	±0.001	±0.002
RMSE	1.02	0.05	0.19	0.023	0.001	0.003
MSE	1.0404	0.0025	0.0361	0.0005	0.000001	0.000009
MAE	1.02	0.05	0.19	0.023	0.001	0.003

IV. Conclusion

The main focus of this work is the use of lateritic soil geopolymer composite for the adsorption of pollutants on sanitary landfill leachate. To maximise the response, an experimental design utilising Response Surface Methodology (RSM) was executed. Reducing the number of runs under one element at a time experiment was the goal of the experimental design utilising the Box Behnken approach in order to optimise the system and examine the influence of other parameters. The quadratic models were developed for each response factor, as shown by ANOVA, and they effectively suited the experimental data.

Out of the three operating parameters that were used—dosage, contact time, and temperature—it was discovered that dosage and contact time had the most influence and significance across the board. The implementation of the ideal conditions found after optimising the response shows that an increase in dosage and contact time results in a commensurate rise in the percentage reduction of the heavy metal in question. The percentage reduction of lead, zinc, and copper was 97.88%, 94.36%, and 99.48%, respectively, based on the following parameters: dosage (10g), contact time (48 hours), and temperature (50 °C).

The experimental data points lie close to the diagonal lines which confirms that there is a strong correlation between the predicted and adjusted R^2 values, indicating good relationships between predicted and experimental data and that the model is significant. Lead, Zinc and Copper demonstrated a correlation coefficient of 0.8757, 0.8330 and 0.7586 in the plot of actual values against the predicted values.

The response surface plots and curved contour lines (refer to Figures.2 through 4) show how various parameters interact and how effective they are at removing heavy metal ions. The figures illustrate the influence of dosage and contact time. The degree of metal ion removal efficiency rose with increasing dosage and contact time (maximum of 10 g and 48 hours, respectively). This discovery may be explained by the fact that the agitation of metal ions onto the adsorbent surface increases the metal ions' removal efficiency. More metal ion adsorption was also made possible by the adsorbent's increased number of active adsorption sites.

The model is substantial for all three response heavy metals. All of the model factors are significant, but Dosage is the most influential variable in Lead reduction, as shown in Table 2, with an F-value of 36.91 showing a high influence on Lead. Contact time is the most influential and significant term, as evidenced by their F values in % zinc and copper removal (27.01 and 9.53, respectively).

The results showed that the experimental data and the model's projected response were in good agreement, with percentage errors of 1.04%, 0.05%, and 0.19% for Pb, Zn, and Cu, respectively. As a result, the Response Surface Methodology is appropriate for maximising heavy metal percentage adsorption with a lateritic soil geopolymer composite adsorbent.

Conflict of interest

The author hereby declares that there are no conflicts of interest whatsoever regarding this research publication.

Data Availability Statement

The data for this journal publication is domiciled in an unpublished PhD research thesis in the Department of water Resources and Environmental Engineering Ahmadu Bello University, Zaria.

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