https://dx.doi.org/10.4314/dujopas.v8i4a.7
ISSN (Print): 2476-8316
ISSN (Online): 2635-3490
Dutse Journal of Pure and Applied Sciences (DUJOPAS), *Vol. 8 No. 4a December 2022*

Adsorption of Mn (II) Ions from Aqueous Solution: A 2^4 Factorial Design Approach

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Abstract
Adsorption involves the accrual of a substance near an interface in relation to its concentration in the bulk solution. The effects of interaction time, adsorbent dosage, solution concentration and pH of solution on adsorption of Mn^{2+} were studied using the 2^4 factorial experimental design. Optimum adsorption of Mn^{2+} was 95.97 % with a predicted response of 97.64 % and a residual value of -1.67 obtained at 150 mins of interaction time, 1.00 g adsorbent dosage, 10.00 mg/dm^3 solution concentration and pH 4.00. Estimated effects and coefficients of response with main and interaction effects of the four adsorption factors revealed that adsorbent dosage, concentration and pH of solution had P-values of 0.040, 0.000 and 0.001 respectively; and had significant effect on adsorption of Mn^{2+}. The interaction of adsorbent dosage with solution pH, adsorption time with adsorbent dosage also had significant influence on the removal of Mn^{2+} from aqueous solution. Analysis of variance used to validate the model showed that there is no lack of fit at 95 % confidence level. Correlation coefficient of the experiment and predicted values indicate a good performance of the model. Hence, factorial experimental design can be used to predict the extent of different adsorption conditions of the variables and optimum adsorption.

Keywords: Adsorption, aqueous solution, adsorbent, adsorbate, factorial design

INTRODUCTION
Adsorption is a physico-chemical process that involves the movement of solute (adsorbate) from the liquid phase to the adsorbent surface until no further net adsorption occur (Siti *et al.*, 2012). The process involves the accrual of a substance near an interface in relation to its concentration in the bulk solution. Adsorption also defined as the formation of a gaseous or liquid layer by molecules in a fluid phase on the surface of a solid by molecular attraction of Van der Waals type (Hu and Xu, 2020). Adsorption is majorly the result of surface energy and should not be misunderstood with absorption. Whilst adsorption is basically the attraction and/or bonding a surface, absorption involves a solute been taken across a membrane.

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Adsorption provides an effective way of removing pollutants (contaminants) from solution to very low levels (Mhemeed, 2018).

The one factor per time approach to adsorption experiments does not ascertain the relationship between all the experimental factors and the removal efficiency (output response); it requires lots of time and demands that several experiments be performed in order to identify the optimum condition for adsorption (Nkuzinna *et al*., 2014). Even though it is helpful in determining the predominant factors, it is not easy to detect the optimal value of the working factors (parameters) as interaction between them is not considered. A factorial design provides useful means to simultaneously determine the effects of two or more adsorption parameters. It is employed to minimize the number of experiments needed to be performed to attain optimal result. Factorial design is known to reduce times and the overall process cost as well as shows how the effect of a factor differs with the levels of other parameters (Giovanilton *et al*., 2011). It also minimizes the number of experiments needed to be performed to attain maximum optimization without affecting the reliability of the experiments (Geyikci and Buyukgungor 2013).

Factorial design is usually expressed as $2^{K,}$ where K represents the experimental factors. This design is employed in an experiment that involves several parameters, where it is essential to study the combined effect of various parameters on adsorption efficiency (response). The design indicates the parameters (K) each at only two (2) levels (minimum and maximum). The 2^K factorial design enables one to perform an analysis of variance and the fitting of responses (Meski *et al*., 2011). The response is believed to be linear over the range of the parameter levels selected because there are only two (2) levels for each parameter. For example, if four factors (K = 4) are used in the experiment, the 2^4 factorial design would require 16 runs either expressed in run or standard order. While Gurkan *et al*. (2021) applied the 2^3 factorial design in the removal of selected heavy metals from lead smelting slag using leachate solution, adsorbent type and dosage as factors, Alkhatibb *et al*. (2017) studied the application of 3^3 and 2^2 factorial designs in the adsorption of textile dye on commercial activated carbon. This study seeks to apply the 2^4 factorial design to enhance the removal of Mn^{2+} from aqueous solution onto adsorbent prepared from the calyx waste of gold coast bombax plant.

MATERIALS AND METHODS

Preparation of solution

Manganese stock solution (1000 mg/dm^3) was prepared by dissolving 3.608 g of manganese chloride ($MnCl_2.4H_2O$) in 50 cm^3 concentrated HCl; then transferred to 1000 cm^3 volumetric flask and volume of the solution was made to mark using distilled water. Solutions used for experiments were prepared by diluting appropriate volume of the stock solution. Solution pH was adjusted with 0.1 mol/dm^3 HCl or 0.1 mol/dm^3 NaOH.

Adsorbent and Adsorption

Activated carbon employed for the study was earlier prepared using the two step method to modify Calyx of Gold coast bombax and H_2SO_4 was used as activating reagent (Rahman *et al*., 2014). Interaction of the solutions with adsorbent was done using the batch adsorption method (Edet and Ifelebuegu, 2020).

Factorial Design for the Adsorption of Mn^{2+}

The experiment employed the 2^4 factorial design for the study of the removal of Mn^{2+} from aqueous solution onto the already prepared adsorbent. Factorial design reduces the number of experiments needed to be performed in order to achieve optimum result. Time (mins) of interaction of adsorbent with solution, adsorbent dosage (g), concentration (mg/dm^3) of solution and pH of the solution were the factors used to study the adsorption of Mn^{2+} from the solutions. Table 1 shows the low (-1) and high (+1) levels of variables for the adsorption of Mn^{2+}.

Table 1: Levels of variables for the adsorption of Mn^{2+} from aqueous solutions

Variables	Low level (-1)	High Level (+1)
Time of interaction (mins)	30.0	150.0
Adsorbent dosage (g)	0.2	1.0
Concentration (mg/dm^3)	10.0	50.0
pH of solution	4.0	8.0

Concentration of Mn^{2+} in the solutions was determined before and after interaction with the adsorbent using the AA320N Atomic Absorption Spectrophotometer. Adsorption efficiency (%) was determined using equation 1 (Shaba *et al.*, 2021):

$$\text{RE (\%)} = \frac{(Co - Cf)\text{x } 100}{Co} \quad (1)$$

Where: C_o is the concentration of Mn^{2+} before interaction with adsorbent; C_f is the concentration of Mn^{2+} after interaction with adsorbent.
Mathlab 2013A was used to determine (design) the number and order of runs required for the 2^4 factorial experiment for the adsorption factors and their 2-way interactions. It was also employed to determine the predicted adsorption efficiency (response) of Mn^{2+} adsorption in comparison to the experimental (actual) percent of adsorption and adsorption equations. Minitab 16 version 14.12.0 was employed to treat data obtained from the experiments in order to determine the estimated effects, main effects, P – and F – values in the analysis of variance and interaction plots for the responses.

RESULTS AND DISCUSSION

Experimental and Predicted Responses for Adsorption of Mn^{2+}

Actual and predicted values (responses) for the 2^4 (2 by 4) factorial experimental design for the removal of Mn^{2+} from aqueous solutions and their residuals (differences) as arranged in run order and influenced by contact time (mins), adsorbent dosage (g), concentration of solution (mg/dm^3) and pH of the medium (solution) are presented in Table 2. Temperature of the medium and rate of agitation per minute (rpm) were 28±2 ^{o}C and 200 rpm respectively. Row 7 had the highest actual response (experimental value). Percent actual removal efficiency (response) of 95.97 %, predicted response (theoretical value) of 97.64 % and residual value (difference) of -1.67 were obtained for the adsorption of Mn^{2+}. The 95.97 % obtained was lower than the 97.75 and 99.30 % reported for adsorption of Cr^{6+} and Cu^{2+} (Musah *et al.*, 2018; Turan and Ozgonenel, 2014) but slightly higher than 95.53 and 91.05 % obtained for adsorption of Cd^{2+} and Pb^{2+} (Musah *et al.*, 2016; Kalantari *et al.*, 2014). Low residual values (< 2.00) obtained for adsorption of Mn^{2+} are indications of a good agreement between the actual (experimental) and predicted values (responses) and hence a good fit of the model.

Table 2: Responses for the 2^4 factorial experiment for the removal efficiency (response) of Mn^{2+}

Run Order	Coded levels of variables				Removal efficiency		
	Time (mins)	Dosage (g)	Concentration (mg/dm^3)	pH	Actual	Predicted	Residual
1	-1	-1	-1	-1	82.62	83.90	-1.28
2	-1	-1	+1	+1	82.96	82.76	0.20
3	-1	-1	-1	+1	88.51	88.23	0.28
4	+1	+1	+1	-1	89.04	87.86	1.18
5	-1	+1	-1	+1	81.03	81.89	-0.86
6	-1	+1	+1	-1	81.16	82.54	-1.38
7	+1	+1	-1	-1	95.97	97.64	-1.67
8	-1	+1	+1	+1	76.72	76.34	0.38
9	+1	-1	+1	+1	71.35	71.74	-0.39
10	+1	+1	+1	+1	70.47	70.66	-0.19
11	+1	-1	-1	+1	79.67	79.76	-0.09
12	+1	-1	-1	-1	87.52	86.44	1.08
13	+1	-1	+1	-1	76.15	76.75	-0.60
14	+1	+1	-1	+1	79.44	78.77	0.67
15	-1	+1	-1	-1	91.62	89.76	1.86
16	-1	-1	+1	-1	77.57	76.77	0.80

Temperature = 28±02 ^oC; Agitation = 200 rpm

From the results in Table 2, high adsorption of Mn^{2+}occurred at low concentration (10.00 mol/dm^3) and pH (4.00), and when dosage is 1.00 g (upper dosage level). Adsorbent removed available cation from solution due to less competition for binding sites when concentration of solution is low (Al-Saad *et al.*, 2019; Santuraki and Muazu, 2015). When solution pH exceeds 4, deprotonation of carboxylic functional groups on the adsorbent surface occurs causing the adsorbent surface to become negatively charged thereby enhancing adsorption of cations; while adsorption is reduced when pH is lower than 3 due to increased number of protons on the surface of adsorbent making binding (adsorption) sites less available for attraction of cations (Abuoa *et al.,* 2019; Farghali *et al*., 2013).

Figure 1 present plots of actual versus predicted responses of Mn^{2+}. The figure indicated the values obtained were close to the straight line. This suggests the suitability of the model and is supported by equations 2 and 3. The closer the actual and predicted values are to the line, the lower the residual value (± 2.00) and the better the fit of the model.

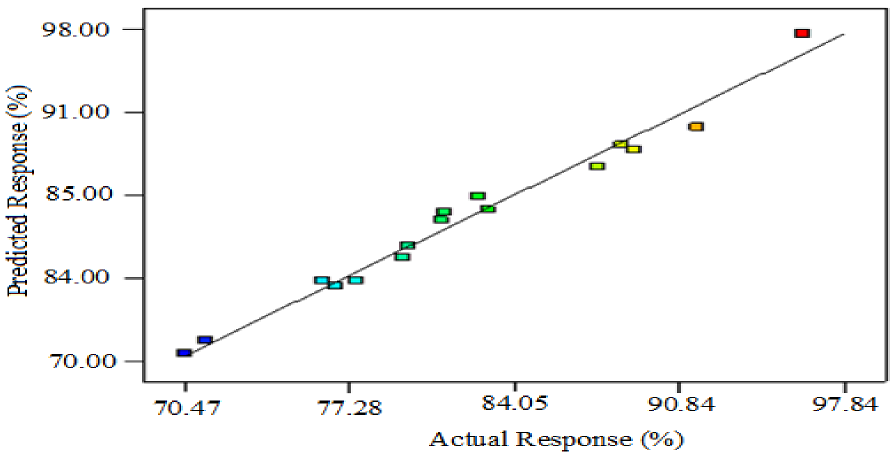

Figure 1: Predicted vs Actual Responses for Adsorption of Mn^{2+}

Davarnejad *et al*. (2015) also used similar plots of actual versus predicted values to determine the fitness of a model to experimental data.

Figure 2 present the plots of residuals versus experimental run order showing randomly scattered residuals that can also be used to validate the model. Residuals are the difference between experimental and predicted values (Turan and Ozgonenel, 2014).

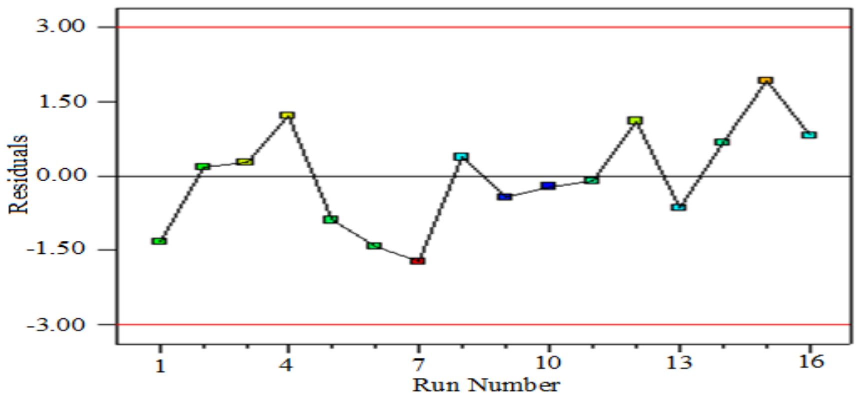

Figure 2: Residual vs Number of Run Response for Adsorption of Mn^{2+}

Residual values obtained for the sorption of Mn^{2+} were within ± 2.00 which are indications of a good fit of the model to experimental data. Residual values obtained are similar to the range of ±2.00 reported for adsorption Cd^{2+} (Musah *et al.*, 2020).

Main and Interaction Effects of Factors on Adsorption Efficiency

Figure 3 present result of main and interaction effects of adsorption factors for the sorption of Mn^{2+}. The factors were time of interaction (A), adsorbent dosage (B), concentration of solution (C) and pH of solution (D). From the normal plot of standardized effect in figure 3a, points that are closed to the line indicate factors that do not have significant influence on the adsorption of Mn^{2+} while points that are further away from the line indicate factors that have significant influence on adsorption. Adsorbent dosage, concentration and pH of solution are the significant main factors in the adsorption of Mn^{2+} as depicted in figure 3a. Interaction (*) of main factors that showed strong influence in the sorption of Mn^{2+} is adsorbent dosage*pH. Other interactions with low influence on the sorption of $Mn^{2+}$are interaction time*pH; and adsorbent dosage*time. Factors on the positive (right) side of the line (adsorbent dosage*time) had positive influence on the adsorption of Mn^{2+} while those on the negative (left) side of the line (interaction time*pH and adsorbent dosage*pH) had negative influence on the sorption process (Musah *et al.*, 2018; Tibet and Coruh, 2015).

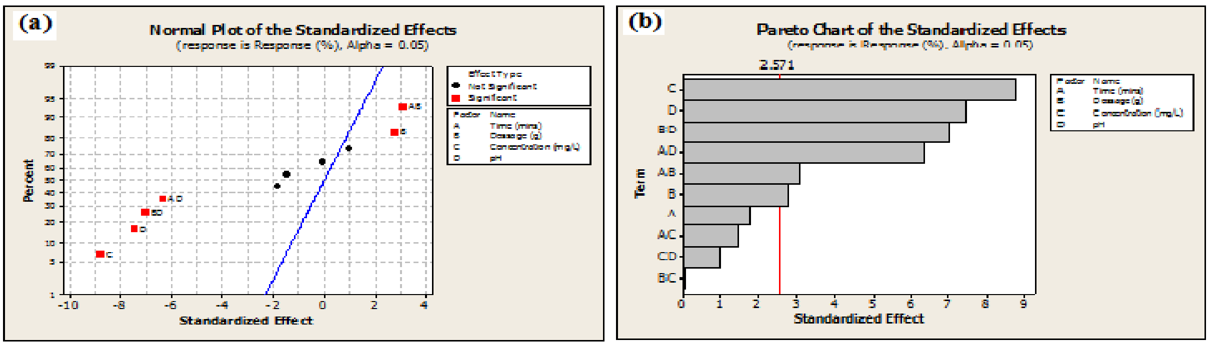

Figure 3: Statistical analysis of adsorption of Mn^{2+}

Figure 3b present the student's t-test as Pareto chart which was used to determine whether the calculated effects vividly differ from zero. At 95 % confidence level, the t-value is equals 2.571 (Al-Mamun *et al.*, 2011). The vertical line in figure 3b represent the minimum statistically significant effect level (Musah *et al.*, 2016) and values in the horizontal row represent the student's t-test value for each effect and are absolute (Turan and Ozgonenel, 2013). Factors whose values were ≥ 2.571 depicted those that had significant influence on the sorption of Mn^{2+}.

Figure 4a present the main effects plots for adsorption, and it give a visual understanding of the factors that influence sorption the most. The magnitude of adsorption factor is small if the slope is near to horizontal line (Tibet and Coruh, 2015). Time of interaction tends towards the horizontal line hence had a very weak effect on the sorption of Mn^{2+}. The strong effect of adsorbent dosage on the adsorption of Mn^{2+} is similar to effect reported for adsorption of Cu^{2+} (Turan and Ozgonenel, 2013). Figure 4b present the interaction plot for effects of factors on the sorption of Mn^{2+}. The plot depicted the impact that varying one adsorption factor had on other factor(s) during the adsorption process.

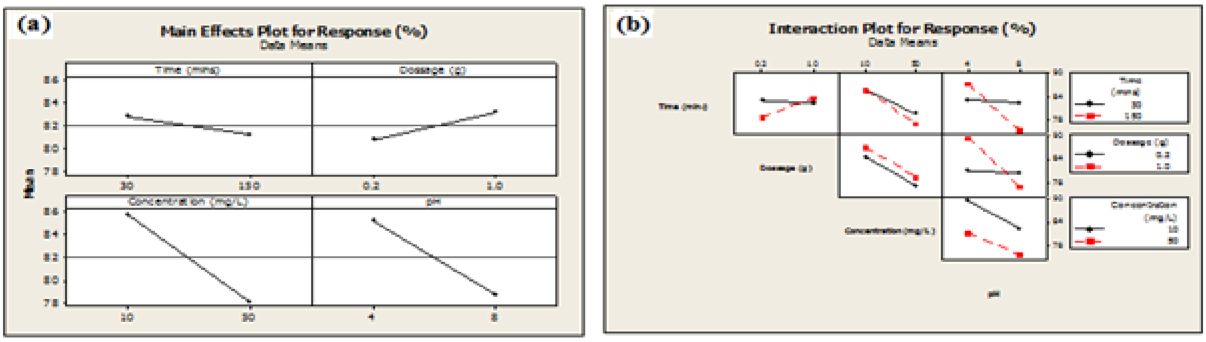

Figure 4: Main effects and interaction plots for responses

There is no interaction between two factors if the lines of the two factors run parallel to each other but if the lines are seen to appreciably cross each other, then the two factors are said to interact (Al-Saad *et al.*, 2019). The interaction of adsorbent dosage*pH had higher interaction effect than those of interaction time*pH and interaction time*adsorbent dosage.

Equations for Adsorption of Mn^{2+}

Regression model equation (equation 2) was employed to illustrate the behaviour of each adsorption factor relative to the adsorption of Mn^{2+}. To develop the model equation, 16 runs (see Table 2) were used for calibration; and percent removal of Mn^{2+} from the solution varied depending on time of interaction, adsorbent dosage, concentration of solution and pH of solution (main factors) and their interactions (2-way). When student's t-test result (presented as Pareto charts in the figure 3b and P-values in Table 3) were considered, some factors were found to be statistically significant ($P \leq 0.05$) while others were not ($P > 0.05$) on the responses (adsorption). Significant factors depict limiting factors such that any variation in their value will affect the removal of the ions from solution (Al-Mamun *et al.*, 2011). The model was recalculated on the bases of aforementioned to eliminate the non significant effects; the resultant model equation for adsorption Mn^{2+} is presented as a simplified equation in terms of significant factors only (equation 3).

Equation for the removal of Mn^{2+} from solution in terms of actual factors:

Adsorption efficiency (%) =

$$74.0500 + 0.10699 \times A + 20.92344 \times B - 0.020325 \times C + 2.42656 \times D + 0.055625 \times A \times B - 0.0005323 \times A \times C - 0.022917 \times A \times D - 0.002969 \times B \times C - 3.80917 \times B \times D + 0.010406 \times C \times D \quad \textbf{(2)}$$

Final equation in terms of significant factors for the removal of Mn^{2+}

Adsorption efficiency (%) =

$$+ 74.04500 + 20.92344 \times B - 0.20325 \times C. + 2.42656 \times D + 0.055625 \times A \times B - 0.022917 \times A \times D - 3.80937 \times B \times D \quad \textbf{(3)}$$

Where A = Time of interaction; B = Adsorbent dosage; C = Concentration of solution; D = pH of solution

A positive sign in front of a factor is an indication of a synergistic effect of the factor on sorption while a negative sign represent an opposed (antagonistic) effect of such factor (Chowdhury et al., 2012). Single variable coefficients of adsorbent dosage, contact time, concentration or pH of solution depict the effect of that factor on the adsorption of Mn^{2+}. Coefficients with two adsorption factors represent the interaction of those factors and their effects on the adsorption of Mn^{2+} (Banch *et al.*, 2019). Similar models of 2^4 and 2^3 factorial equations derived for the adsorption of Cr^{6+} and Pb^{2+} (Musah *et al.*, 2018; Tibet and Coruh, 2015).

Estimated Effects of Adsorption Factors

Main effect, interaction effect, coefficient of the model, standard deviation of every coefficient and probability of responses for the adsorption of Mn^{2+} are presented in Tables 3. Statistically significant main effects and interactions are considered to be those that have P - values lower than 0.05 ($P < 0.05$). When coefficients of main and interaction effects are positive, then the uptake of ion is favoured at high value (Musah *et al.*, 2016). Contrarily, there will be a reduction in percent removal of Mn^{2+} for high level of a factor if the effect is negative (Gaikwad *et al.*, 2010). Larger magnitude of t – values and smaller P - values indicate high significance of the coefficient (Al-Mamun *et al.*, 2011). When values of effect and coefficient in Table 3 are carefully observed, they reveal that factors which have t – values < ± 2.60 and P – values greater 0.05 ($P > 0.05$) are not significant in the adsorption of Mn^{2+}. From the Table, adsorbent dosage, concentration of solution and pH of solution showed significant effect on the removal of Mn^{2+} with P-values lower than 0.05 ($P < 0.05$).

Table 3: Estimated effects and coefficients for removal efficiency (response)

Term	Effect	Coef.	SE Coef.	T	P
Constant		81.987	0.433	189.32	0.000
Time (mins)	-1.573	-0.786	0.433	-1.82	0.129
Dosage (g)	2.388	1.194	0.433	2.76	0.040
Concentration (mg/dm^3)	-7.620	-3.810	0.433	-8.80	0.000
pH	-6.437	-3.219	0.433	-7.43	0.001
Time (mins) *Dosage (g)	2.670	1.335	0.433	3.08	0.027
Time (mins) *Concentration (mg/dm^3)	-1.277	-0.639	0.433	-1.47	0200
Time (mins)*pH	-5.500	-2.750	0.433	-6.35	0.001
Dosage (g) *Concentration (mg/dm^3)	-0.047	-0.024	0.433	-0.05	0.958
Dosage (g)*pH	-6.095	-3.048	0.433	-7.04	0.001
Concentration (mg/dm^3)*pH	0.832	0.416	0.433	0.96	0.381

R-Sq = 98.01 %   R-Sq(adj) = 94.02 %

The value R^2 (0.9801) obtained revealed good agreement of experimental and predicted adsorption efficiency (response). Adsorbent dosage indicate positive effect in the adsorption of Mn^{2+} with high positive t– value and low P– value indicating that adsorbent dosage is significant in the adsorption process. An increase in adsorbent dosage leads to a corresponding increase in adsorption. The negative t – values and negative effect values of concentration and pH suggest that an increase in these parameters lead to decrease in the removal of Mn^{2+}.

Analysis of Variance for Adsorption of Mn^{2+}

Table 4 present result of analysis of variance (ANOVA) for the adsorption of Mn^{2+}. ANOVA was used to reveal the effects of the main factors and their interaction on the adsorption of Mn^{2+}. From the results obtained (Table 4), it can be deduced that significant parameters and estimated adsorption efficiency are functions of the main effects which are time of interaction, adsorbent dosage, concentration and pH of solution and their two-way (2-way) interactions. Main effects that showed significant influence on adsorption with P-values less than 0.05 ($P < 0.05$) are dosage (0.040), concentration (0.000) and pH (0.001). Interactions of time with dosage (0.027), time with pH (0.001) and dosage with pH (0.001) showed some levels of significance in the adsorption of Mn^{2+}. Musah *et al.* (2018) reported similar trends in the biosorption of Cr^{6+} using factorial design experimental.

Table 4: Analysis of variance for removal efficiency (response) of adsorption of Mn^{2+}

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	4	430.715	430.715	107.697	35.89	0.001
Time (mins)	1	9.891	9.891	9.891	3.30	0.129
Dosage (g)	1	22.801	22.801	22.801	7.60	0.040
Concentration (mg/dm^3)	1	232.258	232.258	232.258	77.40	0.000
pH	1	165.766	165.766	165.766	5.24	0.001
2-Way Interactions	6	307.421	307.421	51.237	17.08	0.003
Time (mins)*Dosage (g)	1	28.516	28.516	28.516	9.50	0.027
Time (mins)*Concentration (mg/dm^3)	1	6.528	6.528	6.528	2.18	0.200
Time (mins)*pH	1	121.000	121.000	121.000	40.32	0.001
Dosage (g)*Concentration (mg/dm^3)	1	0.009	0.009	0.009	0.00	0.958
Dosage (g)*pH	1	148.596	148.596	148.596	49.52	0.001
Concentration (mg/dm^3)*pH	1	2.772	2.772	2.772	0.92	0.381
Residual Error	5	15.003	15.003	3.001		
Total	15	753.139				

F- values were used to verify the significance of the model. F-value is a statistical measure of how well a model describes the variation in the data about the mean (Gaikwad *et al*., 2010). When F-value is high ($F > 5.00$) and P-value is low ($p < 0.05$), the main effects and/or interactions with such values are significant (during adsorption) at 5 % level of significance. From Table 4, main effects showed high statistical significance with the F-value for concentration reaching 77.40. Values obtained indicated the model is in good prediction of the experimental result. Interaction of adsorbent dosage with pH also had high positive effect (F-value equals 49.52). This is because an increase in adsorbent dosage leads to a corresponding increased surface area and number adsorption sites available on the adsorbent (Farghali et al., 2013). Similar observation was reported on the adsorption of Cr^{6+} using factorial design experiment (Musah *et al*., 2018).

CONCLUSION

Factorial design for the experiment has proven to be a good technique for studying the effect of adsorption factors and their interactions on adsorption of Mn^{2+} from aqueous solution. The use of factorial design enables the identification of factors that influence adsorption and also, mathematically determines the significant factor(s) among others in a single experiment. Significant factors for the adsorption of Mn^{2+} in this study are adsorbent dosage, concentration and pH. Estimated effects and analysis of variance indicate that the model is fit for the adsorption of $Mn^{2+}$from solutions; therefore the application of factorial design in the removal of metal ions from solutions is recommended.

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