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Full Length Research Paper

Studies of interactive effects of some variables on removal of nickel from petroleum wastewater by factorial analysis

[+]Nwafulugo F. U., Adefila S. S.*, Olawale A. S.*

[+]Department of Chemical Engineering, Kaduna Polytechnic, Kaduna, Nigeria
*Department of Chemical Engineering, Ahmadu Bello University, Zaria, Nigeria.

[+]Corresponding authors e-mail: francaunoma@yahoo.com

Abstract

A factorial analysis using Mini Tab 14 software was carried out to study the interaction effects of the factors; zeolite 4A dose, contact time and initial concentration. This screening process helped in determining factors that has significant effects on the response among the three factors of interest. The result indicates that the three-way-interaction had the highest effect on the response and should be more carefully controlled during routine experiment. The p-values were less than 0.05 for the one way interaction (zeolites 4A dose), the two way interaction (zeolite 4A dose – contact time) and the three way interaction depicting a high interactive effect of the three factors on the response with the three-way interaction having the highest magnitude as aforementioned. The values of R^2= 98.90% R^2 (adj) = 97.94 % indicates that the model is significant to the process. This was further validated by the residual plots which proved the model to be a good replica of the experimental data.

Keywords: Zeolite 4A, Crudeoil, Petroleum wastewater, Sorption, Pollution, Heavy metals.

INTRODUCTION

Pollution of water, especially in communities close to crude oil deposits and refineries, has created worldwide concerns because of the associated heavy metals, inter-alia, released to the environment. These heavy metals according to Mudi (2010) cannot be diluted or degraded. They have high adverse effects on the environment - human and aquatic life (Isehunwa, 2011). This has attracted the attention of authorities concerned like National Environmental Standards and regulations enforcement agency(NESREA) and federal environmental protection agency (FEPA) resulting to tighter legislation being imposed on levels of pollutants discharged to the environment. Despite all the efforts of the refineries to curtail this, the inorganic pollutants were not sufficiently catered for. To curtail this pollution challenge, Babel and Kurniawan (2004) had earlier stated that, conventional treatment processes such as chemical precipitation, electrochemical removal etc. have significant disadvantages. Some of the disadvantages are incomplete removal, high-energy requirements, and production of toxic sludge (Eccles, 1999). According to Leung et al (2000), the search for low cost adsorbents has really intensified which could be of mineral origin or organic resins (Kurniawan et al, 2005, Aderemi 2004). According to El-Nafaty et al (2008), zeolites have been found to be one of the effective materials for removal of both organic and inorganic contaminants from the environment. Some researchers have also noted that zeolites' chemical inertness, modifications, unique properties and selectivity marked them as effective materials for solving environmental pollution. There are so many factors that could affect the adsorption of these heavy metals among which are adsorbent dose, contact time, pH, temperature, solubility, surface area and initial concentration.

It now became necessary to investigate the parameters that have higher effect on the response, then the interaction effects of the parameters on the response. The factorial analysis seems to be a useful tool for this process because it is simple and gives useful information on the screening processes and their magnitude (Montgomery, 2005).

Table1. Design of Experiment for Factorial Analysis

RunOrder	CenterPt	Blocks	ZD	CT	IC
1	1	1	1.5	3	30
2	1	1	1.5	3	30
3	1	1	0.5	3	100
4	1	1	0.5	24	30
5	1	1	1.5	24	100
6	1	1	0.5	3	100
7	1	1	1.5	24	100
8	1	1	0.5	24	30
9	1	1	1.5	3	100
10	1	1	0.5	24	100
11	1	1	1.5	24	30
12	1	1	0.5	3	30
13	1	1	0.5	3	30
14	1	1	0.5	24	100
15	1	1	1.5	24	30
16	1	1	1.5	3	100

Factorial Analysis

Batch adsorption laboratory experiment by one factor at time has been found to be expensive and time consuming, therefore the modeling process by factorial design could not only be very helpful in understanding of the process mechanism but also in winding down the costs of application by reducing the costs of scaling-up the process from laboratory to industrial scale. Design of experiment (DOE) has become one of the most popular statistical techniques since the 1990s (Hsien et al, 2006). The main advantage of experimental design as aforementioned is that it can cover larger area of experimental statistics and obtain unambiguous results at minimum expense. The general factorial design is a standard technique and widely used for studying a random response to a set of K possible factors. Main and interaction effects can be easily evaluated. The main effect refers to the effect caused by the change factor while the interaction effect of one factor is dependent on the value of another factor. The experimental design is obtained as; No of runs = 2^K. Where 2 means two level factorial (higher and lower level). K means the number of factors under investigation (Montgomery, 2005).

MATERIALS AND METHODS

Atomic absorption spectrometer (AAS) model AA240FS, Fast Sequential absorption spectrometer (VARIAN) from multi user laboratory at chemistry department of Ahmadu Bello University was used for the research. Zeolite 4A was procured from UOP, a Honeywell group of companies New Jersey, USA. While the petroleum wastewater was collected from the dewatering tanks in the tank farm of Kaduna refinery and petrochemical company ((KRPC) by a composite sampling method. The adsorption/ion exchange process were carried out by weighing accurate quantities of zeolite 4A dose mixed with 50ml of petroleum wastewater at an initial concentration specified in the design matrix in Table 1 at pH of 4 as established from literature and at a contact time of 3 to 24 hours as also specified in the design matrix to ensure that complete ion exchange has been achieved over the shorter period of time. The slurry was filtered and the solution digested and analyzed for metal ion content. The cation content in the filtrate was determined by atomic absorption spectrometer (AAS). A total of 16 runs were carried out as presented in Table 1 depicting a two-level design with three factors having 2^3 (or 8) runs, a design with all possible combinations, called general factorial design was adopted with 2 replicates giving a total of 16 experimental runs, meaning that each run could be repeated for precision sake (3 factors, 2 levels and 2 replicates).

RESULTS AND DISCUSSION

Table 2 indicates that nickel is a measurable heavy metal in the petroleum wastewater sample. The heavy metal profile in Table 2 indicates the efficiency of nickel removal and was selected as the response of the experimental design. It is a known fact that a good standard by which to evaluate a model is to look at p-values (Hsienet al, 2006). If all terms have p-values less than the level appropriate for the experiment, it is certain to have a good model. In this research, $\alpha = 0.05$ was chosen and the p-value for each term in the model were less than 0.05 except for zeolite 4A dose (ZD) and zeolite 4A-contact time interaction (ZD*CT) as depicted in Table 3

Table 2. The Design Parameters and the Percentage Nickel Removal

ZD	CT	IC	Ni uptake %
1.5	3	30	84.6535
1.5	3	30	84.6535
0.5	3	100	79.3069
0.5	24	30	94.5545
1.5	24	100	81.0891
0.5	3	100	79.3069
1.5	24	100	81.0891
0.5	24	30	94.5545
1.5	3	100	80.198
0.5	24	100	72.9703
1.5	24	30	77.5248
0.5	3	30	76.6337
0.5	3	30	76.6337
0.5	24	100	72.9703
1.5	24	30	77.5248
1.5	3	100	80.198

Table 3: Estimated Effects and Coefficients for Ni uptake % (coded units)

Term Constant	P
ZD	0.181
CT	0.001
IC	0.005
ZD*CT	0.686
ZD*IC	0.000
CT*IC	0.001
ZD*CT*IC	0.000

S = 0.945143 R-Sq = 98.90% R-Sq(adj) = 97.94%

Table 4Analysis of Variance for Ni uptake % (coded units)

Source	DF	P
Main Effects	3	0.001
2-Way Interactions	3	0.000
3-Way Interactions	1	0.000

Legend;
DF = Degree of freedom
P = P-value= less than 5% for a model fit.

indicating a model that is good for further exploration and validation. This model is considerably simpler and fits the data. The residual error only increased by a small amount. Table 4 shows one – way, two – way and three – way interaction effects indicating all the p- values less than 0.05 which are appropriate for the process. A model equation 1 was generated and the appropriate coefficients substituted from Table 5 which gave rise to equation 2. The equation was refined using the p-values less than 0.05 in Table 3 which led to equation 3 which is the refined developed model for the process.

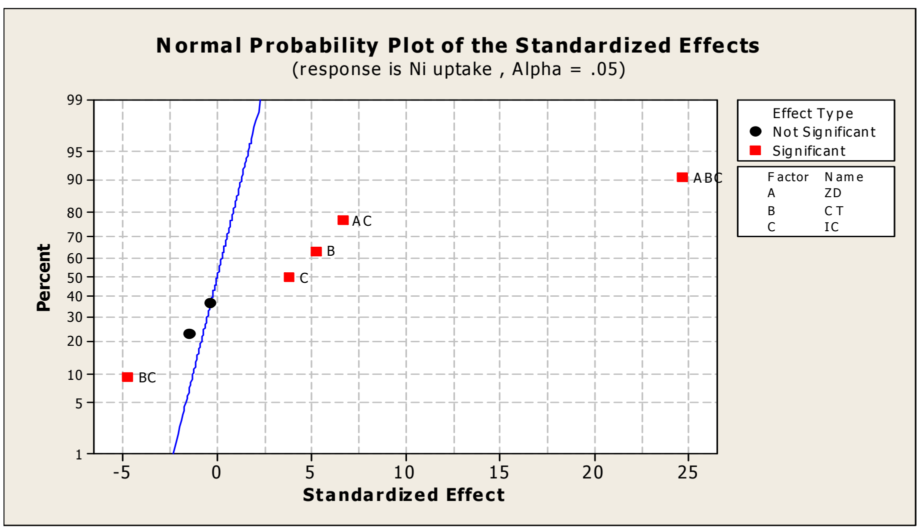
The model was further validated by using the residual plots. The fitted values are the results predicted by the model which were randomly distributed depicting a good

Table 5. Estimated Coefficients for Ni uptake % using data in uncoded units

Term	Coefficient
Constant	56.3917
ZD	21.5609
CT	2.39796
IC	0.405638
ZD*CT	-2.08089
ZD*IC	-0.338452
CT*IC	-0.0347882
ZD*CT*IC	0.0317882

Legend;
ZD =Zeolite Dose =x1
CT = Contact Time = x2
IC = Initial Concentration = x3

Figure1. Normal Probability plot of the standardized effects



Legend;
ZD =Zeolite Dose =x1
CT = Contact Time = x2
IC = Initial Concentration = x3

model as shown in Figures 5a, b, c, d. The residuals plots are the actual response data minus the predicted response data(Figure 5). Active effects are effects that are significant or important as depicted in Figure 3. In the normal plot of the effects, points that do not fit the line well usually signal active effects. Active effects are larger and further from the fitted line than inactive effects. Inactive effects tend to be smaller and centered on zero, the mean of all the effects. The normal probability plot in Figure 1 labels effects that are lower than the level. Figure 1shows that all other terms in the model are significant except the ZD and ZD*CT.A Pareto chart of the effects shown in Figure 2 is another useful tool that was used to determine which effects are active. The Pareto chart uses the same α as the normal plot to determine the significance of effects, only terms that cross the significant line are significant to the model. Thus all terms in the model except ZD and ZD*CT are significant (α = 0.05).Figure 3 shows that the contact time (CT) has a bigger main effect than initial concentration of

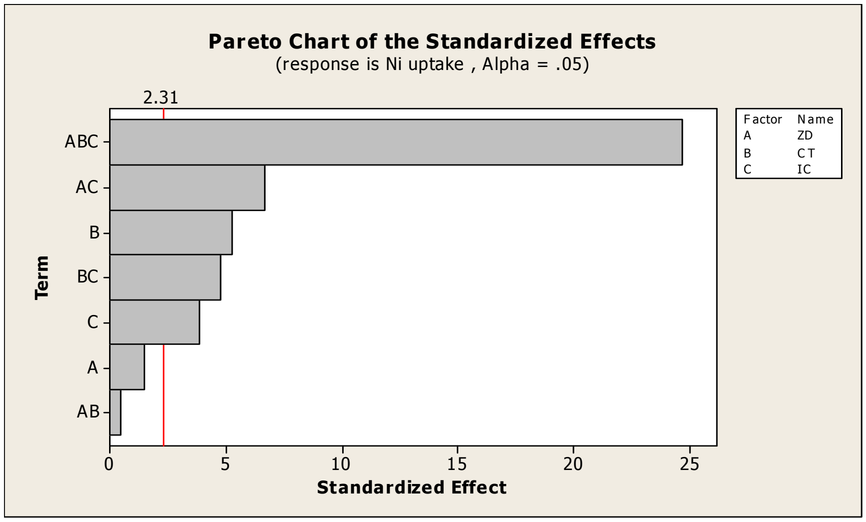


Figure 2: The pareto chart of the standardized effect

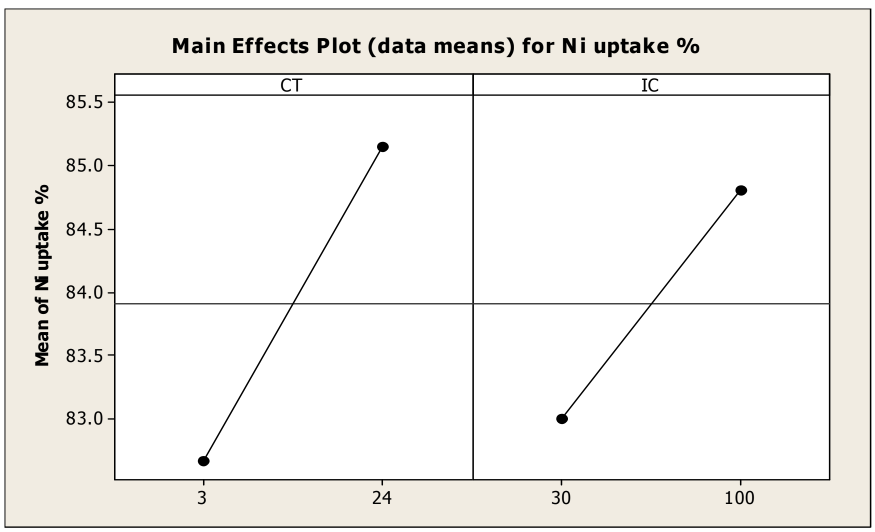


Figure 3. Main effect plot of Nickel uptake

the adsorbate (IC). That is, the line connecting the mean responses for contact time of 3hours, the contact time of 24hours has a steeper slope than the line connecting the mean responses at the low and high settings of initial concentration of the adsorbate. Although the type of contact time appears to affect the nickel adsorbed more than initial concentration of the adsorbate, it is very important to look at the interaction. An interaction can magnify or cancel out a main effect as was the case in this process.

To calculate main effects, Minitab subtracts the mean response at the low or first level of the factor from the mean response at the high or second level of the factor. The Table 6 summarizes the findings: Model equations 1, 2, and 3 were generated for the process from Table 5 which were further refined based on the P values less than 0.05 as indicated in Table 3,depicting a high significant model for the process.

An interaction plot (Fig 3) shows the impact that changing the settings of one factor has on another factor. Because an interaction can magnify or diminish main effects, evaluating interactions is extremely important. The significant interaction between contact time and initial concentration of the adsorbate shows up as two lines with sharply differing slopes. The nickel adsorbed for IC (Initial concentration) of 100 are greater than nickel adsorbed for IC of 30 at both 3 and 24 hours of CT (Contact time). However, the interaction plot in Figure 4

Table 6. Summary of the findings:

Factor	Size of Effect	Interpretation
Contact Time	+2.4686	runs at 24 hours of contact time had higher nickel adsorbed than runs at 3 hours atmosphere of pressure
IC	+1.7876	runs that used IC of 30 mg/L had higher nickel than runs that used IC of 100 mg/L

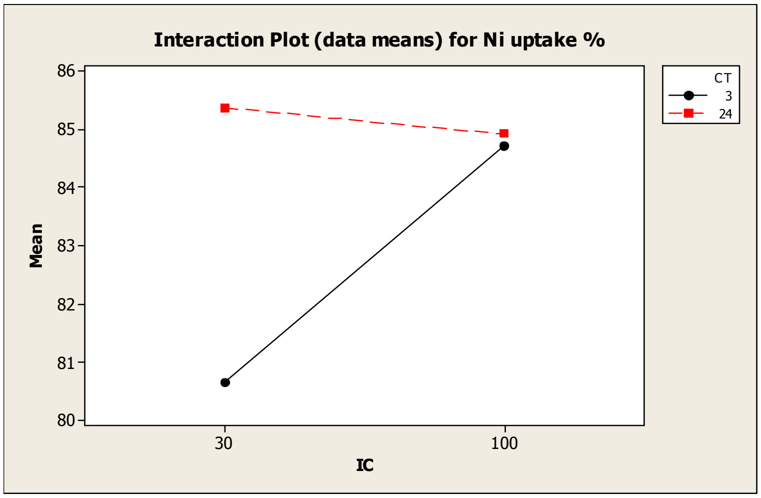


Figure 4 The Interaction plot for Nickel uptake in petroleum wastewater

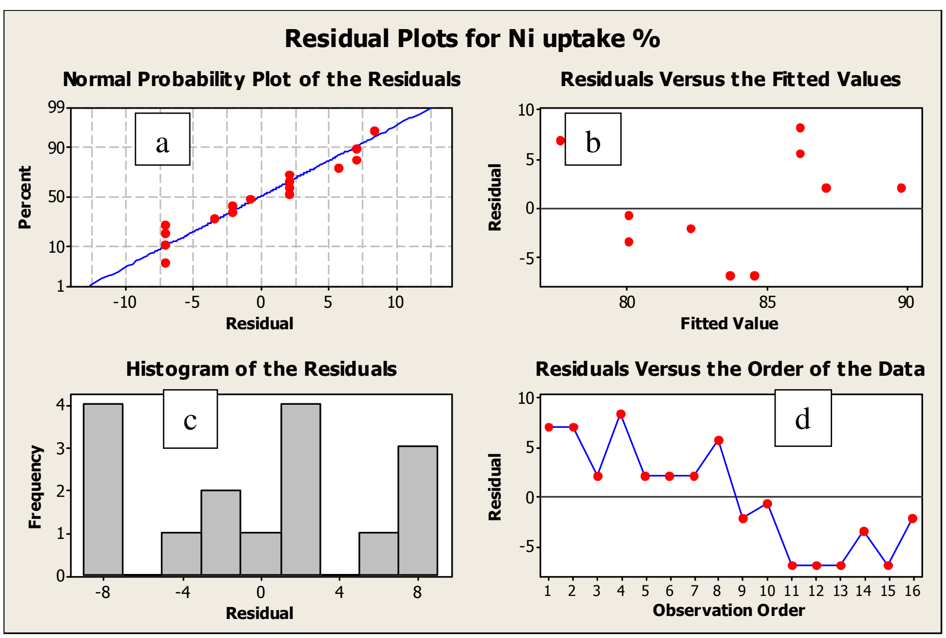


Figure 5a, b, c, d. The Residual Plots for Nickel uptake

shows that the difference in nickel adsorbed between runs using initial concentration of 100ml and 30ml petroleum wastewater (PW) at contact time of 24 hours is much greater than the difference in nickel adsorbed

between runs using IC of 100ml PW and of 30ml PW at contact time of 3 hours. In order to get the highest nickel adsorbed for this experiment, results obtained suggest that the initial concentration (IC) of 100ml should be used while the contact time should be adjusted to 24 hours.
The general model is presented below;

Model: $\mathbf{Ni_{Adsorbed} = a_0 + a_1 \cdot x1 + a_2 \cdot x2 + a_3 \cdot x3 + a_4 \cdot x1 \cdot x2 + a_5 \cdot x1 \cdot x3 \cdot + a_6 \cdot x2 \cdot x3 + a_7 \cdot x1 \cdot x2 \cdot x3} \quad (1)$

Model: $\mathbf{Ni_{Adsorbed} = 56.3917 + 21.5609 \cdot x1 + 2.39796 \cdot x2 + 0.405638 \cdot x3 + 2.08089 \cdot x1 \cdot x2 + 0.338452 \cdot x1 \cdot x3 - 0.0347882 \cdot x2 \cdot x3 + 0.0317236 \cdot x1 \cdot x2 \cdot x3} \quad (2)$

The refined model becomes:

$$\mathbf{Ni_{Adsorbed} = 56.3917 + 2.39796 \cdot x2 + 0.405638 \cdot x3 - 0.338452 \cdot x1 \cdot x3 - 0.0347882 \cdot x2 \cdot x3 + 0.0317236 \cdot x1 \cdot x2 \cdot x3} \quad (3)$$

CONCLUSION

The factorial analysis using Minitab 14 softwareis a useful tool for the screening process.It indicates that all the interactiveeffects of the factors on the response were highly significant but the 3-way-interaction had the highest magnitude. All terms in the model are significant except ZD and ZD*CT. The R-Sq and R-adjusted depicts that the model equation is a good representation of the experimental data. The residual plot showed randomly distributed points indicating a good model.

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