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Kinetics and Isotherm Studies on Divalent Lead Ions Adsorption by Zeolite Solution

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Abstract: **Today, synthetic zeolites are used commercially more often than natural zeolites due to the purity of crystalline products and the uniformity of particle sizes. In view of the increasing rate of environmental pollution resulting from lead containing industrial effluents, The work reports the utilization of synthetic zeolite as an adsorbent for the removal of lead (II) ions from stimulated wastewater, and the significance of this study is to remediate industrial effluent so that the harmful effect of lead to man, aquatic life microorganisms and the environment will be reduced. The effect of sorption time, temperature,heavy metal concentration and pH, on the adsorption process were investigated using batch method. Langmuir, Freundlich, and Temkin isotherms were used to describe the adsorption isotherms and their constants evaluated. The equilibrium data was found to fit the Langmuir and Freundlich models. Kinetic models such as First-order, pseudo first-order, second-order and Elovich models were used to fit the experimental data. The result indicated that pseudo second-order model best describes the kinetics of the adsorption process.**

Keywords: **Adsorption, Di lead ion, Zeolite.**

1. INTRODUCTION

The progressive increase of industrial technology results in the continuous increase in environmental pollution. Industries discharge different types of heavy metal waste into the environment at an unprecedented and at a constant increasing rate [20]. These heavy metal wastes may be discharged into the streams, rivers, and lakes and the continuous enrichment of these waters with these metal wastes beyond the healthy levels may cause poisoning, leading to various sicknesses [2].

Consequently the remediation of polluted industrial wastewater before they are released into the environment remains a topic of global concern [11]. Moreover, the contamination of ground water is today a major concern and the management of water is today a major concern in the management of water resources [23]. A number of materials can be used as adsorbents, such as activated carbon, aluminosilicate (clay materials), natural and synthetic zeolites. Adsorption by clays and modified clays had been and alternative solution for water and wastewater treatment [8][14]. The sorption of kaolin is particularly extractive, in the case of A-NMC; it is a local product, therefore cheap and easily accessible source of aluminosilicate. However, its adsorption capacity can greatly be improved via conversion into zeolite using thermal and chemical treatment [6]. Zeolites are hydrated aluminosilicate formed under hydrothermal conditions. It can be found naturally, or can be synthesized. Synthetic zeolites were first utilized commercially as molecular sieve adsorbent [6]. They are preferably synthesized from solution of sodium silicate and sodium aluminates. Zeolites can also be synthesized from a variety of raw materials; natural and synthetic glasses, aluminosilicate gels, and clay materials, example kaolin [25]. It has been found that the starting material and conditions for preparation have influence on the resulting types and amount of zeolites to be obtained [17]. Based on available materials and publications, it could be concluded that due to their unique properties, zeolites have a great potentials as effective sorbent material for large number of water treatment applications such as water softening ammonia removal (from municipal sewage, animal farms, fertilizer factory waste water, fish breeding pond, swimming pools), removal of heavy metals (from natural water acid mines drainages, industrial wastewater), phosphates removal of dissolved organic compounds and dyes, oil spillage treatment and many others [3]. Ions of heavy metals like copper, nickel, zinc, cadmium, lead, chromium and mercury have a significant impact on the environment, since they are often detected in industrial wastewater [26]. Removal of ions can be accomplished by a variety of techniques, in which adsorption method is currently considered very suitable for water treatment because of its simplicity and cost effectiveness and zeolites is one of the adsorbent for adsorption of the heavy metal [31]. The presence of heavy metals in industrial wastewater as a result of many manufacturing process is known to cause detrimental effects on human health and environment [18]. Removing these heavy metals, demand high energy or advanced operational requirements, a number of convenctional technologies such as coagulation, precipitation, extraction, biosorption and adsorption have been considered for treatment of contaminated wastewater [16]. Among these methods, adsorption is found to be very suitable for wastewater treatment because of its simplicity and cost effectiveness [24], commercial activated carbon is regarded as the most effective material for controlling the metal ions load. However, due to its high cost and 10%-15% lost during regeneration, unconventional adsorbents like zeolites have attracted the attention of several investigation and adsorption characteristics have been widely investigated for the removal of metal ions [4].

2. MATERIALS AND METHODS

Analytical grade of Lead nitrate $Pb(NO_3)_2$ was used to prepare the standard solution of lead. Synthetic zeolite produced from an indigenous aluminosilicate at optimum conditions was used as an adsorbent. The stock solution was diluted to the required concentration and appropriate pH with drop wise addition of 0.12M NaOH and 0.12M of HNO_3 using a pH meter. Freshly diluted stock solutions were used for each experiment.

2.1 Characterization

Characterization of the samples was done at the research center of Ahmadu Bello University Zaria, Nothern province of Nigeria. Scanning Electron Microscopy was used to determine the size and morphology of the aluminosilicate and synthetic zeolite.. Atomic Absorption Spectrophotometer (model 2009 VGP Buck Scientists USA) was used to determine the oxides present in the aluminosilicate sample.

2.2 Adsorption Procedure

The stimulated wastewater used, was lead stock prepared at different concentrations, for different runs, 0.5g of synthetic zeolite was mixed with 50mil of the prepared lead stock solution at a particular concentration (20mgl-^{1} – 100mgl^{-1}), of a specific pH (5 - 8). The mixture was placed on a water bath constant temperature vibrator set at a particular temperature (20^{0}C - 45^{0}C), for a particular duration/time (20mins – 60mins). After adsorption, the solution was filtered and the filtrate's concentration was tested using Atomic Adsorption Spectrophotometer (AAS).

The amount of equilibrium adsorption, Q (mgg^{-1}) was calculated by

$$Q = \frac{(C_i - C_e)V}{m} \qquad (1)$$

Where Ci (mgl^{-1}) is the Initial lead ion Concentration in solution, Ce (mgl^{-1}) is the Equilibrium Concentration of lead ion Concentration in solution, V (liters) is volume of the solution used, and m (g) is the mass of the adsorbent.

The percentage adsorbed is given as;

$$\text{Percentage adsorbed} \quad (\%) = \frac{C_i - C_e}{C_i} \times 100 \qquad (2)$$

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3. RESULTS AND DISCUSSION

Effects of Process Variables

Batch adsorption study was carried out to determine the effects of the factors considered, which includes; pH, Concentration, Temperature and Time.

3.1 Atomic Absorption Spectrophotometer (AAS)

The AAS result is presented in table 1a, and it shows the concentration of different elements that are present in the aluminosilicate. From the result, it is observed that the major constituents are SiO_2 and Al_2O_3. The aluminosilicate had Alumina (Al_2O_3) content of 31.32wt %, and Silica (SiO_2) content of 46.01%, and silica-alumina ratio of 1.469, which is greater than 1. This is in accordance with [6] specification for clays that can be used for zeolite synthesis. The conformity of the specification invariably implies that the aluminosilicate can be used for zeolite formation.

Table 1a: Chemical composition of the aluminosilicate

Constituents	SiO_2	Al_2O_3	Cao	Fe_2O_3	MgO	Loss on Ignition
Percentage by weight	46.25	38.41	0.87	0.71	0.39	13.37

Table 1b: Chemical composition of synthetic zeolite

Chemical composition	**Percentage composition**
SiO_2	66.33
Fe_2O_3	5.91
AL_20_3	16.89
CaO	0.92
MgO	0.44
K_2O	0.04
TiO_2	3.02
CaO	0.09
MnO	0.041
V_2O_5	0.11
Cr_2O_3	0.064
ZnO	0.002
NiO	0.004
CuO	0.006
LOI	2.34
$\boldsymbol{Si}/(\boldsymbol{Al}+\boldsymbol{Fe}+\boldsymbol{Mg})$	2.786

The SEM analysis is used to show the morphology or texture, crystalline structure and surface topography of an adsorbent. The SEM analysis of some clay indicates coarse and loosely packs with some well formed flakes and irregular and hexagonal edges. Others may appear as plates of variable thickness and size with relatively well defined edges, or showing some element of curling layers in the particles.

From the SEM micrograph shown in figure 1a, it showed moderately ordered flakes, with poorly developed hexagonal outlines. Psuedohexagonal morphology is suggested, hence the edges of the particles are beveled, somewhat ragged and irregular [27].

In figure 1b, it is obvious that, larger pores of different shapes could be observed for scanning electron microscope of synthetic zeolite. This may be because the activating agents promote the contact area between the clay and the activating agent, and therefore, increases the surface area and porosity of clay.

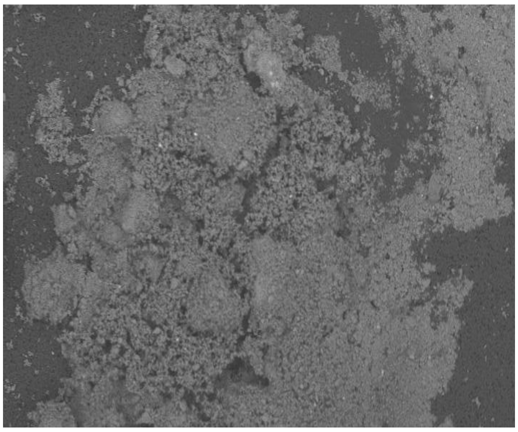

Fig 1a SEM analysis of natural clay at 200µm

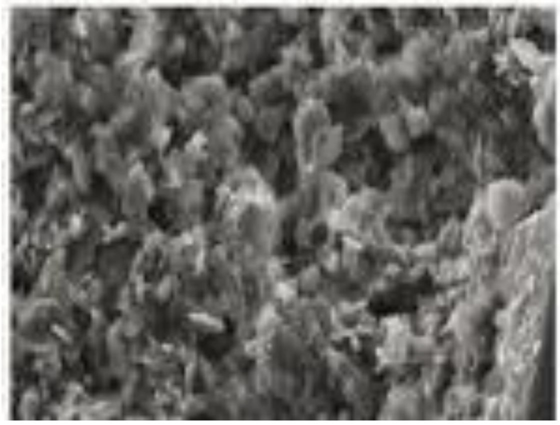

Fig 1b SEM analysis of synthetic zeolite at 200µm

3.2 Effects of pH

The effect of pH of solution on adsorption was determined because it is known to be one of the most important factors affecting sorption. The result on the effect of pH on the adsorption of lead (II) ions is shown in Figure 2. The percentage of adsorption of lead was studied over a pH range, and the result indicated that maximum removal occurred at pH of 8. It has been reported that a relationship exist between the pH point of zero percentage removal of a material, and suggested that cations adsorption on an adsorbent will be favored at pH values higher than the pH point of zero, while anions adsorption will be favored at lower pH values

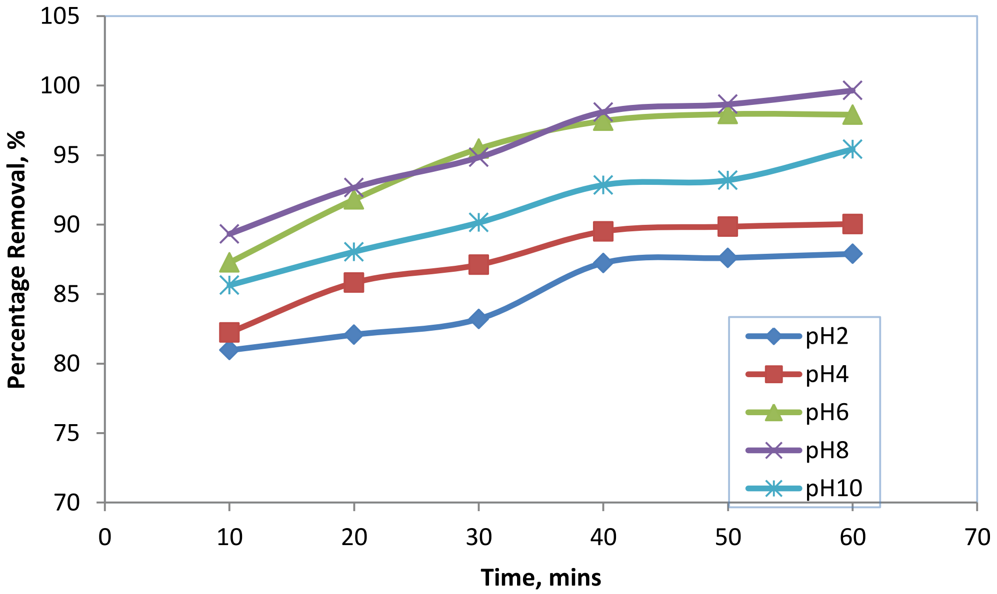

Fig 2 Effect of pH on adsorption of lead on synthetic zeolite

3.3 Effect of Concentration

The effect of initial ion concentration was studied for the initial ion concentration between 20 to 100mgl^{-1} and the results were presented in Figure 3. The result showed that a decrease in the percentage removal with increase in the concentration of lead was observed. This can be accounted for that all adsorbents have a limited or fixed number of active sites and at a certain metal ion concentration the active sites becomes used up and saturated [25][33]. This implies that further increase in the concentration of lead will only led to a decrease in the percentage removal as the active sites has been already occupied, hence more metal ions will be remaining in solution after adsorption. The increase in the concentration of metal ions led to an increase in collision between the ions and adsorbent hence increased the driving force to overcome resistance to mass transfer and thus an increase in uptake capacity [26].

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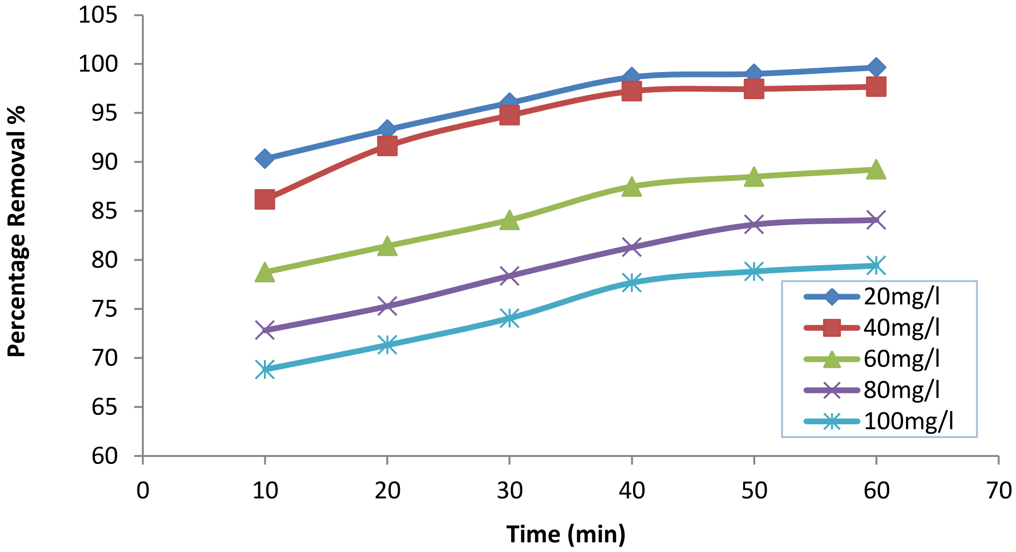

Fig 3 Effect of concentration on adsorption of lead on synthetic zeolite

3.4 Effect of temperature

Temperature values ranging from 303K to 323K were used to study the effect of temperature on adsorption. The result of the effect of temperature is shown in Figure 4. It was found out that, with increase in temperature, the time to achieve the same percentage removal was much less. This is because as the temperature increases, the rate of diffusion of adsorbate molecules across the external boundary layer and internal pores of the adsorbent particles increased.

This result indicated that as the temperature of the solution was increased from 303K to 323K, there was also a corresponding increase in the amount of lead ions adsorbed by the synthetic zeolite. This increase in adsorption capacity with increase in temperature indicated that the adsorption process is endothermic in nature, it also suggests a chemisorptions process i.e. adsorption accompanied by a chemical reaction. It is restricted to just on layer of molecules on the surface but may be followed by an additional layer of physically adsorbed metal ions [15]. Similar result has been reported [4][2]. A good literature search has revealed different types of results on the effect of temperature on the adsorption of heavy metals. A decrease in adsorption potential with increase in temperature has been observed [20].

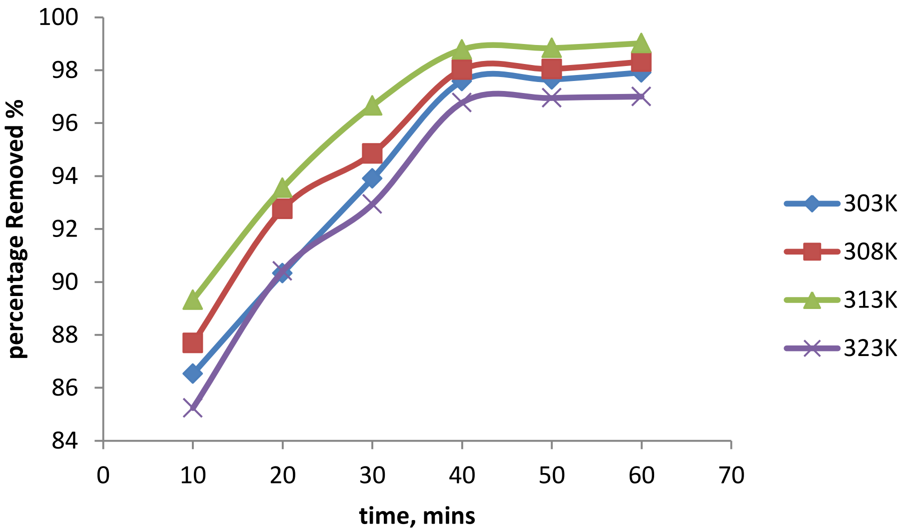

Fig. 4 Effect of temperature on adsorption of lead on synthetic zeolite

3.5 Effect of time

All the batch studies were carried out over a time. The study shows that as the time increases, the percentage adsorbed increases until equilibrium was reached at about 60mins. The initial adsorption was due to the availability of the positively charged surface of the adsorbent.

The influence of time is also an important factor to be considered on the adsorption of metal ions by an adsorbent. It has been reported that the contact time plays an important role in adsorption process. The fast rate of adsorption observed at the initial stage may be explained by an availability of abundant active sites on the adsorbent surface which gradually became occupied with time. As these sites are progressively filled the more difficult the sorption becomes as the process tends to become unfavorable [11].

3.6 Isotherm Studies

Adsorption isotherm is a relationship between the amount of a substance removed from liquid phase by unit mass of adsorbent and its concentration at constant temperature. They are useful in the evaluation of the adsorption capacity and to determine the characteristics of an adsorbent if suitable for application. Isotherm is often used as empirical models which do not make statements about the underlying mechanisms and measured variables. They are obtained from measured data by means of regression analysis. The most frequently used isotherms are the Langmuir isotherm, freundlich isotherm, Temkin isotherm, etc.

3.6.1 Langmuir isotherm model

The Langmuir isotherm is based on the theoretical principle that only a single adsorption layer (monolayer) exist on an adsorbent. It assumes that all active sites on the adsorbent are homogenous and there is no interaction between active sites [8][21].

The linear form of Langmuir equation is given as

$$\frac{C_e}{q_e} = \frac{1}{Q_o K_l} + \frac{C_e}{Q_o} \qquad (4)$$

Hence a plot of C_e/ q_e against C_e will give

Slope = $\frac{1}{q_o}$ and Intercept = $\frac{1}{Q_o K_L}$ or $\frac{1}{Q_o b}$

The Langmuir constants Q_o and b were determined from the intercept and slope, and the dimensionless factor R_L were calculated

Langmuir model assumes that uptake of lead molecules occurs on a homogeneous surface by monolayer adsorption. For Langmuir isotherm, the values of Ce/qe were plotted against Ce and is shown figure 5 below. The Langmuir constant Q and b were determined from the intercept and slope of the linear plot, and presented in table 2 The essential characteristics of langmuir equation can be expressed in terms of dimensionless separation factor, R_L defined by

$$R_L = \frac{1}{1+bC_o} \qquad (5)$$

Where C_o is the highest initial solute concentration (mgl^{-1}), b is the Langmuir constant. The value of R_L indicates whether the adsorption isotherm is unfavorable (R_L>1), Linear (R_L=1), favorable (0<R_L>), or irreversible (R_L=0). Therefore, the values of R_L were calculated and presented in table 3

For the adsorption of lead on the adsorbent, the R_L values were less than 1 showing that the adsorption was favorable. The fact that the Langmuir isotherm fits the experimental data very well may be due to homogeneous distribution of active sites on the adsorbent.

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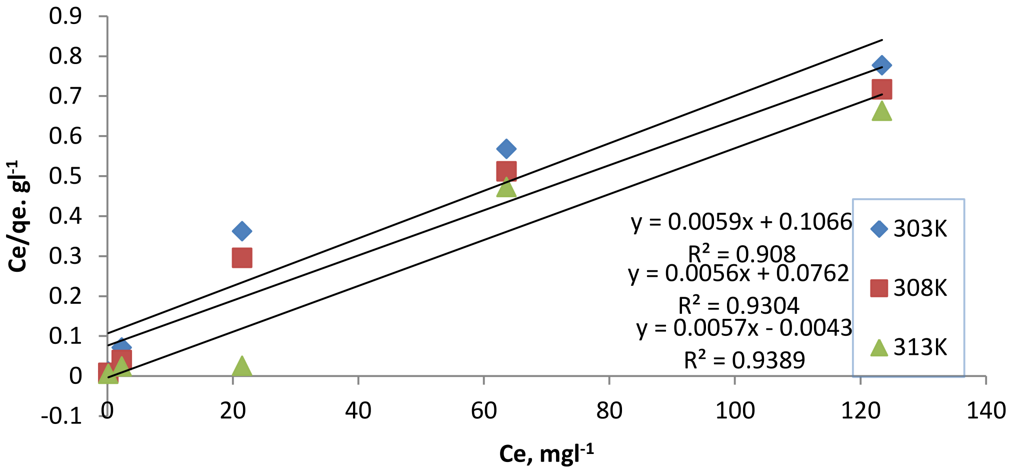

Fig 5 Langmuir isotherm for adsorption of lead on synthetic zeolite

3.6.2 Freundlich isotherm model

The Freundlich isotherm describes the extent of heterogeneity of the adsorbent surface involving a multilayer adsorption [8].Freudlich equation is given as

$$q_e = K_f C_e^{1/n} \quad (6)$$

The linear form is $$\log q_e = \log K_f + \frac{1}{n}\log C_e \quad (7)$$

Where K_f = Freundlich constant, n = Freundlich exponent

The Freundlich constant and exponent were calculated,

Freundlich isotherm involves the plot on ln q_e against C_e shown in figure 6. The values of the freundlich constants K_f and n were calculated from the intercept and the slope respectively and shown in Table 2. The constant K_f is measure of the adsorption capacity while constant n is a measure of the intensity or favorability of adsorption [1]

For beneficial adsorption, the value of n will be between 1 and 10. In this work, all the values range from 1 to 3 showing beneficial adsorption of lead on the adsorbent.

The correlation factor R^2 ranges from 0.93 to 0.99, indicating that the adsorption followed Freundlich isotherm model. Furthermore, if the value of n is below or equal to unity, the process is chemisorptions, but if greater than unity, the process is favorable physical adsorption [7].

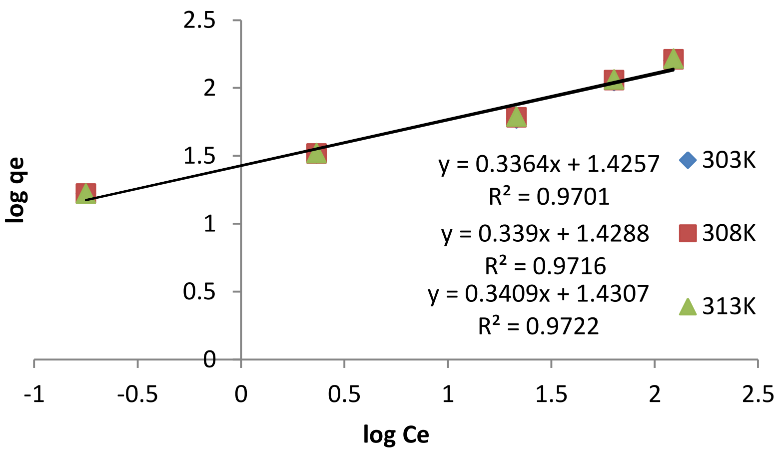

Fig 6 Freundlich isotherm for adsorption of lead on synthetic zeolite

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Temkin isotherm

The Temkin equation is given as

$$q_e = \frac{RT}{b} \ln(AC_e) \qquad (8)$$

The linear form is

$$q_e = B\ln A + B\ln C_e \qquad (9)$$

$$B = \frac{RT}{b} \qquad (10)$$

q_e = Amount adsorbed at equilibrium, C_e = Equilibrium concentration,T = Absolute Temperature, R = 8.314J/m/K

The constants A and B were calculated; hence temkin isotherm was analyzed by plotting q_e against C_e as shown in figure 7. The values of the constants A and b were calculated and presented in Table 2. The correlation coefficient R^2 of about 0.81 to 0.89 indicate that lead adsorption did not follow the temkin isotherm. Thus $R^2 > 0.91$ indicates adsorption follows temkin isotherm. From table 2, the values of b decreased with increase in temperature.

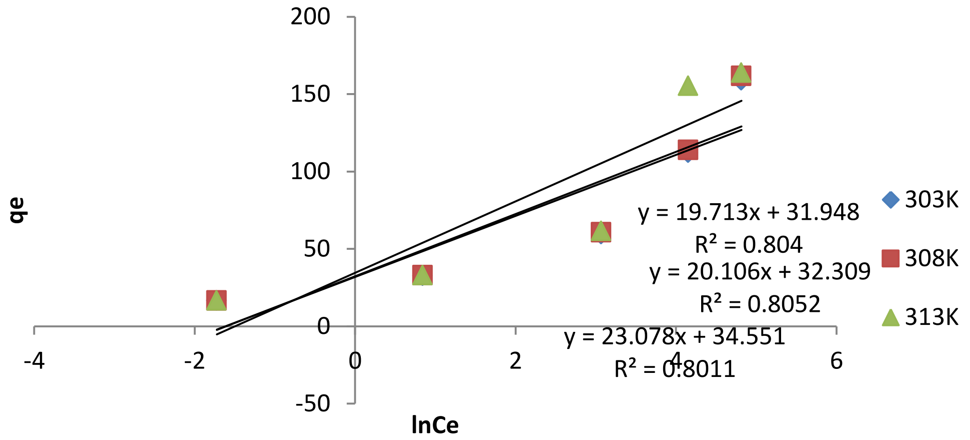

Fig 7 Temkin isotherm for adsorption of lead on synthetic zeolite

Table 2 Calculated isotherm parameters for the Adsorption of lead on synthetic zeolite

Isotherm	Temperature (K)		
Model	**303**	**308**	**313**
Langmuir			
$Q(mgg^{-1})$	200	200	200
$b(lm^{-1}g^{-1})$	0.048	0.07576	1.36
R_L	0.2753	0.1418	0.0089
R^2	0.908	0.930	0.938
Freundlich			
n	2.8767	3.848	2.941
$K_f(lg^{-1})$	26.607	26.692	26.814
R^2	0.970	0.971	0.938
Temkin			
$b(Jm^{-1}g^{-1})$	127.81	127.398	112.799
$A(lg^{-1})$	5.053	4.982	4.470
R^2	0.804	0.805	0.801

3.6.3 Kinetic Studies

The kinetic of sorption reveals the solute uptake rate of the reaction. It is one of the important characteristics in defining the efficiency of adsorption. Kinetics models are also used to investigate the mechanism of adsorption and the potential rate controlling steps such as mass transport and chemical reaction processes [12].

3.6.3.1 First-order Kinetic model

The first order kinetic equation is given as

$$-\ln\left(\frac{C_t}{C_o}\right) = K_1 t \qquad (11)$$

The first-order kinetic model was investigated by plotting $-\ln(C_t/C_o)$ against t as seen in figure 8. The rate constant k_1 gotten directly from the graph together with the values of the correlation coefficient R^2 are tabulated in table 4. From table 4 The R^2 values greater than 0.9 showing that the adsorption followed first order kinetic model.

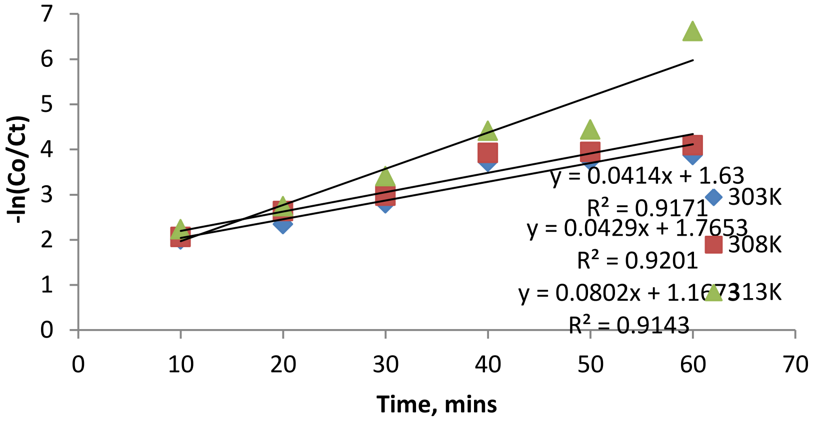

Fig 8 First-order plot for the adsorption of lead on synthetic zeolite

3.6.3.2 Pseudo first-order kinetic model

The Pseudo first order model is given as

$$\frac{dq_t}{d_t} = K_1(q_e - q_t) \qquad (12)$$

Integrating gives

$$\log(q_e - q_t) = \log q_e - \left(\frac{K_1}{2.303}\right) t \qquad (13)$$

Plot of log (q_e - q_t) against t was used to express the pseudo first-order in figure 9 at different temperatures. The values of the correlation coefficient R^2 are shown in table 3. The constant K_1 and q_e are evaluated. It can be seen that, from the table, the correlation coefficient R^2 values of all the orders are greater than 0.9 indicating that the adsorption may have followed the pseudo first-order.

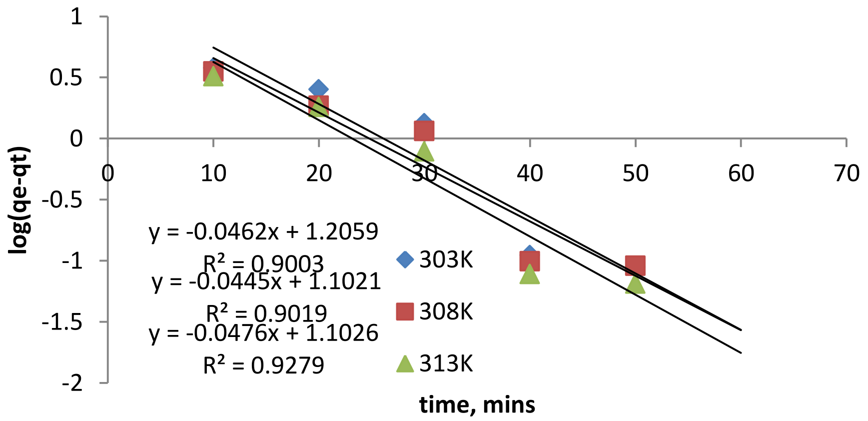

Fig 9 Pseudo First-order plots for the adsorption of lead on synthetic zeolite

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Pseudo second-order kinetic model

The equation given as

$$\frac{t}{q_t} = \frac{1}{K_2 {q_e}^2} + \frac{t}{q_e} \qquad (13)$$

At different temperatures, the plot of t/q_e versus t was used to demonstrate the pseudo-second order shown in figure 10. The values of the constants k, q_e, and R^2 calculated are shown in Table 3. The correlation coefficient R^2 has very high values between 0.99 and 1.0 for adsorption of lead on the adsorbent.

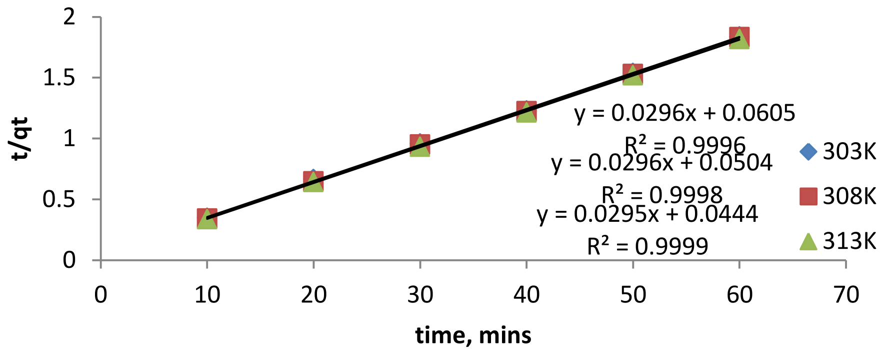

Fig 10 Pseudo second-order plot for the adsorption of lead on synthetic zeolite

3.6.3.3 Elovich kinetic model

$$q_t = \left(\frac{1}{\beta}\right)\ln(\alpha\beta) + \left(\frac{1}{\beta}\right)\ln t \qquad (14)$$

$$\text{Slope} = \left(\frac{1}{\beta}\right)$$

$$\text{Intercept} = \left(\frac{1}{\beta}\right)\ln(\alpha\beta)$$

The Elovich equation has been shown to be useful in describing chemisorptions on highly heterogeneous adsorbent. Figure 11 gives the Elovich plots of q_e against time at different temperatures. The initial adsorption rate α and the extent of coverage β were calculated from the slope and intercept respectively, and presented in Table 4 together with the correlation coefficient R^2. Hence high value of the correlation coefficient $R^2 > 0.96$ indicates that the adsorption conform to Elovich model.

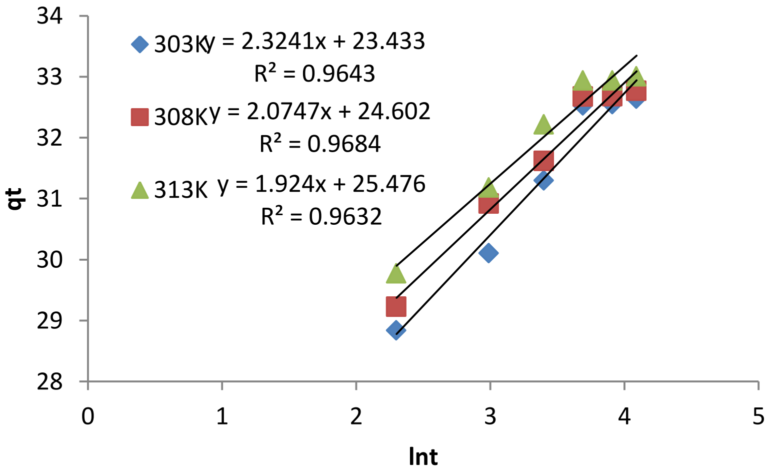

Fig 11 Elovich kinetic plot for the adsorption of lead on synthetic zeolite

Table 3 Calculated Kinetics parameters for the Adsorption of lead on synthetic zeolite

Kinetic Model	Temperature (K)		
	303	308	313
First order			
K_1 (min^{-1})	0.042	0.044	0.009
R^2	0.917	0.920	0.914
Pseudo first order			
K_1 (min^{-1})	0.1059	0.1013	0.1082
q_e (mg/g)	16.03	12.6473	12.647
R^2	0.900	0.901	0.927
Pseudo second order			
K_2 (g/mgmin)	0.0014	0.0168	0.019
q_e (mg/g)	34.482	34.482	34.482
R^2	0.999	0.999	0.999
Elovich			
α (mg/gmin)	54912.6	208844392.7	1078951.183
β (g/mg)	0.4302	0.4821	0.5197
R^2	0.963	0.968	0.964

3.7 Statistical analysis for adsorption of lead on Synthetic zeolite

A significant level of 95% was used hence all terms whose p-values are less than 0.05 are considered to be significant. The model summary and the lack of fit test for the adsorption of lead were presented below.

Table 4 Summary P-value for Lead adsorption on Synthetic zeolite

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0094	< 0.0001	0.3084	0.1613	Suggested
2FI	0.8008	< 0.0001	0.2141	-0.4747	
Quadratic	0.0230	< 0.0001	0.5119	-0.4538	Suggested
Cubic	0.3378	< 0.0001	0.5964	-13.0209	Aliased

A negative "Pred R-Squared" implies that the overall mean is a better predictor of your response than the current model. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 5.788 indicates an adequate signal. This model can be used to navigate the design space.

Std Dev = 5.25	R-Squared	0.7476
Mean = 91.23	Adj R-Square	0.5119
C.V % = 5.75	Pred R-Squared	-0.4538
PRESS = 2376.78	Adeq Precision	5.788

The adequate precision ration above 4 indicate adequate model efficacy, hence the adequate precision ratio of 5.788 indicate model efficacy.

The coefficient of regression R-Squared was used to validate the fitness of the model equation. The R-Squared has a high value of 0.7476, showing that 74.76% of the variability in the response can be explained by the model. This implies that the prediction of the experimental data is quite satisfactory.

The quadratic model equation obtained is;

$$Y (\%) = 98.39 + 2.5A + 2.17B + 1.52C + 3.77D – 0.52AC + 1.50AD + 0.57BC – 1.09BD – 2.06CD – 2.13A^2 – 3.22B^2 – 1.91C^2 – 1.7D^2 \quad (15)$$

In a regression equation, when an independent variable has a positive sign, it means that an increase in the variable will cause an increase in the response, hence an increase in temperature, time , concentration, and pH will cause an increase in the percentage adsorbed. pH will have more significant effect in the increment of the response since its coefficient is higher. Eliminating the insignificant terms, the final model equation becomes;

$$Y (\%) = 98.39 + 2.5A + 2.17B + 1.52C + 3.77D – 1.50AD – 1.09BD – 2.06CD – 2.13A^2 – 3.22B^2 – 1.91C^2 – 1.7D^2 \quad (16)$$

4. CONCLUSION

Nowadays, ecological problems initiate studies of new materials, among which can find altered zeolites with new and unique properties gained by a chemical modification. An objective of the recent works is to further extend the possibilities of the method for the preparation of other types of zeolites and/or to further optimize synthesis of the existing one **.**The equilibrium data was found to fit the Langmuir and Freundlich models. Kinetic models such as First-order, pseudo first-order, second-order and Elovich models were used to fit the experimental data. The result indicated that pseudo second-order model best describes the kinetics of the adsorption process. The maximum adsorption of lead occurred at a pH 8, Zeolite reactions were strongly spontaneous and endodermic. Increase in temperature improved adsorption performance for the zeolite, High metal uptake can be achieved with careful selection of slurry concentration to avoid masking of sorption by chemical precipitation. Synthetic zeolite showed promise as highly efficient adsorbent for lead removal.

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