Modelling the Adsorption of Lead (II) Ion from Aqueous Phase with *Carica papaya* Trunk Activated Carbon

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

Original Research Article

ABSTRACT

The study modelled the adsorption of lead (II) ion from aqueous phase using chemically activated carica papaya trunk activated carbon (CPTAC). Equilibrium and kinetics adsorption studies were conducted to investigate the adsorption capacity, adsorption isotherm and rate of adsorption respectively. The linear correlation of equilibrium data with Langmuir, Freundlich, Temkin and Hills isotherms using regression analysis showed that the adsorption of Pb fitted best into Freundlich model with R^2 values of 0.874 and K_f values of 2.64 mg/g. The adsorption kinetics/rate of adsorption could be described by Pseudo second order equation with R^2 value of 1.0 and 0.998 for the two adsorbate concentrations tested, indicating a chemisorption process onto heterogeneous surfaces forming multilayer on the surface of CPTAC. Modelling the equilibrium concentration adsorption of Pb onto CPTAC using XLSTAT 2014 gave a model equation as = 0.35230 + 0.13394 * Co − 0.05206 * CT − 0.32744 * pH + 0.01794 * Co^2 + 0.05794 * CT^2 + 0.20878 * pH^2 − 0.10567 * CT − 0.13442 * Co * pH + 0.15392 * CT * pH, with R^2 value of 0.75. Modelling the amount of Pb adsorbed gave a model equation as = 27.14770 + 22.36606 * Co + 0.05206 * CT + 0.32744 * pH − 0.01794 * Co^2 − 0.05794 * CT^2 − 0.20878 * pH^2 + 0.10567 * Co * CT + 0.13442 * Co * pH − 0.15392 * CT * pH, with R^2 value of 1.00.

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1. INTRODUCTION

Heavy metal removal from industrial wastewater is of primary importance because they not only contaminate water bodies but are also toxic to many life forms [1]. Lead poisoning can cause severe toxic damage to the human system which includes brain damage, liver cirrhosis, kidney failure and many others. Several treatment techniques such as chemical precipitation, oxidation, reduction, coagulation, solvent extraction, ion exchange, filtration, reverse osmosis, membrane technologies, adsorption and many others have been commonly employed for the removal of metal ions [2].

Adsorption is a separation process during which components of one phase of a liquid or gas is transferred onto the surface of a solid adsorbent [3]. This process is based on the capability of porous material with large surface area to selectively retain compounds on the surface of the adsorbent by a chemical bond which is established during the process. This results in relatively high concentration of molecules at the surface of the adsorbent by attractive forces and it is controlled by various parameters including temperature, contact time, adsorbate concentration and pH, nature of adsorbent and many others [4]. Adsorption is one of the many alternative methods used for heavy metal removal from industrial effluents and activated carbon is the most widely used adsorbent due to its high adsorption capacity for organic compounds [5]. Carbon has been known throughout history as an adsorbent and its usage dates back centuries.

Activated carbon can be used for the removal of organic chemicals, heavy metals, chlorine, colour, unpleasant tastes, odorous compounds and many other contaminants. Commercial activated carbon can be obtained from coal, wood, peat, lignite and many others but these materials are not renewable thus making them very expensive. Many studies have been done on the use of cheap and abundant materials for the production of activated carbon especially waste materials such as coconut shell, cassava peels, palm kennel shell, saw dust, mango seeds and many others.

This study modelled the adsorption lead from aqueous solution with activated carbon produced from carica papaya trunk. The equilibrium and kinetic studies were evaluated.

2. MATERIALS AND METHODS

2.1 Preparation of Activated Carbon

Carica papaya tree trunk was sourced from Lubara in Khana LGA of Rivers state, Nigeria. The trunk was cut into small sizes, washed, sun dried and then activated with $ZnCl_2$ at different impregnation ratios of 1:2, 1:3 and 1:4 ($ZnCl_2$/Pawpaw). The activated carica papaya trunks were carbonized at 400^oC for one hour and then cooled and crushed to powder. Thus, Carica Papaya Trunk Activated Carbon (CPTAC) was produced.

2.2 Batch Adsorption Studies

Batch adsorption studies were conducted to obtain the effect of adsorbent concentration, adsorbate pH and contact time for the removal of lead (II) ions from the simulated solution using the CPTAC. The adsorbate concentration used were 5, 27.5 and 50mg/l; the pH used were 2, 7 and 12 while the contact time used were 30, 105 and 180mins. This was in accordance with the experimental design (DoE). The stock solution was prepared by mixing lead nitrate salt (Pb $(NO_3)_2$) in distilled water to obtain the desired initial concentrations. All the chemicals used were of analytical grade. The adsorption efficiency was calculated from Equation 1, and this was used as criterion for determining optimum conditions for lead removal.

$$\% \ R = \frac{c_i - c_e}{c_i} \times 100\% \tag{1}$$

Where: R= adsorption efficiency (%)
C_e = equilibrium concentration of the adsorbate (mg/l).
C_i = the initial conc. of adsorbate (mg/l).

2.3 Batch Equilibrium Adsorption

As the adsorption process proceeds, equal amounts of solute are being adsorbed and desorbed simultaneously and the rates of adsorption and desorption would attain equilibrium [6]. The time required to attain this state of equilibrium is termed the equilibrium time, and the amount of solute adsorbed at the equilibrium time reflects the maximum adsorption capacity of the adsorbent under the operating conditions [7]. The equilibrium is described by expressing the amount of solute adsorbed per unit weight of adsorbed (q_e) as a function of concentration (C_e) of solute remaining in solution.

Thus, equilibrium adsorption studies were conducted using the optimum conditions for lead removal as reported by [8] and [9] to investigate the adsorption capacity of CPTAC. Thus, 50ml of simulated wastewater samples of 50mg/l initial Pb concentration and pH 6 were measured into different 100ml Erlenmeger flask with varying adsorbent dosage between 0.4 to 2.0g. The mixtures were stirred on magnetic stirrer for 60mins and then filtered and analyzed for residual Pb conc. using Atomic Absorption Spectrometry (AAS). The equilibrium adsorption capacity (q_e) of the adsorbent was calculated using the mass equilibrium equation given in Equation 2.

$$q_e = \frac{(C_o - C_e)}{M} V \qquad (2)$$

Where: C_o is the initial concentrations of the adsorbate (mg/l);
C_e is the equilibrium concentration of adsorbate (mg/l);
V is the experimental volume of wastewater (L),
M is mass of adsorbent (g).

2.3.1 Adsorption isotherms

Adsorption isotherms are used to describe how adsorbates would interact with adsorbents. They indicate how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state [10]. Thus, results from the equilibrium adsorption data were used to calibrate some isotherm models to describe the relationship between the amount of Pb adsorbed and its concentration in the equilibrium solution at constant temperature. Langmuir, Freundlich, Temkin and Hills isotherms models were used. These models were linearized to calculate the adsorption capacity (q_o) and the rate of adsorption (k). The linear form of Langmuir equation is given in Equation 3 as:

$$\frac{C_e}{q_e} = \frac{1}{q_o K} + \frac{C_e}{q_o} \qquad (3)$$

Where: C_e is equilibrium concentration of the adsorbate (mg/l), q_e is mass of solute adsorbed per unit mass of adsorbent, q_o is a constant related to the adsorption capacity (mg/g) and K is experimental constant.

Linear form of Freundlich equation is given in Equation 4:

$$\log(q_e) = \log k + \frac{1}{n} \log C_e \qquad (4)$$

Where: C_e is equilibrium concentration of the adsorbate, q_e is mass of solute adsorbed per unit mass of adsorbent, k and n are Freundlich constants representing the adsorption capacity of adsorbents and the intensity of the adsorption, respectively.

Linear form of Temkin equation is given in Equation 5:

$$q_e = \frac{RT}{b_T} \log A_T + \frac{RT}{b_T} \log C_e \qquad (5)$$

Where: b_T and A_T are the Temkin constants, A_T is the equilibrium binding constant corresponding to the maximum binding energy, b_T is related to the heat of adsorption (J/mol), R is the universal gas constant (8.314) J/mol K) and T is the absolute temperature in kelvin (K).

Linear Form of Hills equation is given in Equation 6:

$$Log\left(\frac{q_e}{q_{SH} - q_e}\right) = nH \log C_e - \log K_D \qquad (6)$$

Where q_{SH}, nH and K_D are Hill isotherm constants and nH is Hills coefficient describing cooperativity.

If nH >1= positive cooperative binding (the binding of one molecule onto the adsorbent, increases the affinity for other molecules to bind) while nH < 1 = negative cooperative binding (the binding of one molecule onto the adsorbent decreases the affinity for other molecules to bind). However, nH = 1 non cooperative binding (the affinity of the adsorbate to bind onto the adsorbent is not dependent on whether or not other molecules are already bound).

2.4 Batch Kinetics Studies

Kinetic models are used to investigate the rate of adsorption process and potential rate controlling steps. This is helpful for selecting optimum operating conditions for the full-scale batch process. The adsorption kinetics was conducted at varying time intervals of 10 to 90 mins using two initial concentrations 5 and 50mg/l of Pb at pH of 6. 2g of CPTAC was added to 50ml of lead (II) ions in different 100ml Erlenmeyer flasks and placed on magnetic stirrer at 500rpm. The samples were withdrawn at specific interval of time (t) and then filtered using Whatman paper and thereafter, the filtrates were analyzed for lead (II) ions residual concentration (C_t) using atomic adsorption spectrophotometer (AAS). The

adsorption capacity (q_t) of the CPTAC at specific time intervals were calculated from Equation 7.

$$q_t = \frac{(c_o - c_t)}{w} \times V \qquad (7)$$

Where: c_o = initial adsorbate conc. (mg/l).
c_o = final adsorbate conc. (mg/l).
V = volume of solution used (L)
W = weight of adsorbent used (g).

2.4.1 Kinetics of adsorption

The mechanism of adsorption for the removal of Pb (II) with CPTAC was investigated using Pseudo first order and second order equations developed by [11]. The logarithmic form of Pseudo first order equation is shown in Equation 8:

$$\log(q_e - q_t) = \log(q_e) - \frac{K_{p1}}{2.303} t \qquad (8)$$

Where: q_e is the adsorption capacity at equilibrium (mg/g), q_t is the adsorption capacity at time t (mg/g), k_{p1}, is the rate constant of first order adsorption (mg/mins).

Linear correlation of the values of **log (q_e – q_t)** against **t** gave the value of k_{p1} and q_e which were determined from the slope and intercept of the plots respectively.

Linear form of Pseudo Second order equation is given in Equation 9:

$$\frac{t}{q_t} = \frac{1}{k_{p2} q_e^2} + \frac{1}{q_e} t \qquad (9)$$

Where: k_{p2} is the rate constant of pseudo second order adsorption (mg/min).

Plots of $\frac{t}{q_t}$ against **t** of Equation (9) gave a linear relationship from which q_e and **h** values was determined from the slope and intercept respectively.

3. RESULTS AND DISCUSSION

3.1 Equilibrium Adsorption

Results from the equilibrium adsorption of Pb onto CPTAC were calibrated using Langmuir, Freundlich, Temkin and Hills isotherm models in order to obtain the best fitting isotherm. The linear correlation of equilibrium data with Freundlich, Temkin and Hills equations gave high correlation coefficient (R^2) of 0.874, 0.852 and 0.849 respectively for Pb Adsorption except for Langmuir isotherm with R^2 value of 0.58. The linear models and the corresponding isotherm parameters for all the isotherm models tested are presented in Table 1.

3.1.1 Model validation

The isotherm models were validated with another set of experimental data. The calculated isotherm parameters were fitted into the isotherm model equations to get the calculated q_e values. It was observed that, for the adsorption of Pb unto CPTAC, the Experimental q_e (Exp q_e) and Calculated q_e (Cal q_e) correlated with Langmuir and Freundlich models but did not correlate with Temkin and Hills models. The correlated models are shown in Figs. 1 to 2.

Table 1. Equilibrium Model Parameters for Adsorption of Pb onto CPTAC

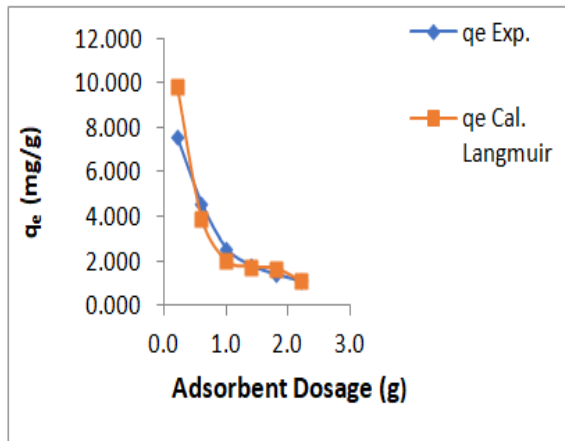| Linearized Models	Parameters	Value
Langmuir Isotherm	R^2	0.580
Y = -0.5154x + 0.9355	q_o (mg/g)	1.942
	K (L/mg)	1.816
	R_L	-0.011
Freundlich Isotherm	R^2	0.874
Y = 2.396x + 0.422	K_f (mg/g)	2.641
	1/n	0.417
Temkin Isotherm	R^2	0.852
Y = 17.23x + 3.156	A_T (L/mg)	2.156
	b_T (J/mol)	246.21
Hills Isotherm	R^2	0.849
Y = 5.196x – 0.070	q_{SH}	7
	nH	5.196
	K_D	0.070

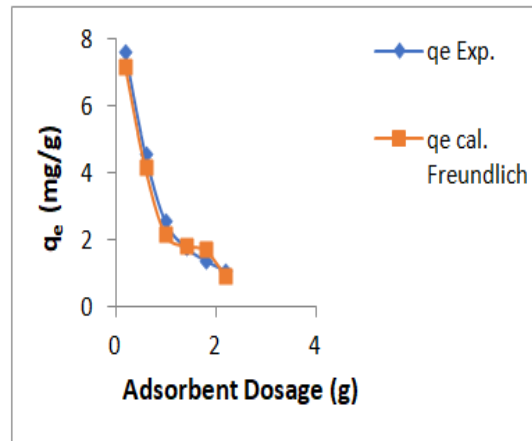**Fig. 1. Langmuir plot of q_{e-Exp}. Vs q_{e-Cal}**

Fig. 2. Freundlich plot of q_{e-Exp}. Vs q_{e-Cal}

3.1.2 Test for isotherm models

The applicability of these isotherm models was further verified using the sum of error squares (SSE, %) calculated from Equation 10 using Excel software:

$$SSE,(\%) = \sqrt{\frac{\Sigma(q_{e,exp} - q_{e,cal})^2}{N}} \qquad (10)$$

Where: N is the number of data points.

From the results of the SSE,% for each isotherm model presented in Table 1, it was observed that Freundlich isotherm model had the least error with SSE value of 0.0397% while Langmuir, Hills and Temkin had 0.496%, 7% and 12.620% respectively. Note that the higher the value of R^2, the lower the value of SSE,% and the better the goodness of fit. From these results, the adsorption of Pb onto CPTAC fitted into Freundlich and Langmuir models but could be best described by Freundlich isotherm. This indicates a chemisorption process onto heterogeneous surfaces forming multilayer on the surface of CPTAC. Similar observations were made by [12] and [13]. The Freundlich isotherm for the adsorption of Pb is shown in Fig. 3.

3.2 Results of Adsorption Kinetics

The experimental data were linearly correlated with Pseudo First order and Pseudo second order models. Correlation with Pseudo First Order equation gave very low correlation efficient (R^2) of 0.445 and 0.559 for the two adsorbate concentrations tested. Also, the Exp q_e and the Cal qe values did not correlate, thus adsorption

of Pb onto CPTAC do not follow First Order kinetics. However, the Pseudo Second Order equation gave high correlation coefficient (R^2) of 1.0 and 0.998 for the two adsorbate concentrations tested as shown in Fig. 4. A correlation was observed between the Exp q_e and the Cal q_e value, indicating chemisorptions as the leading step.

3.2.1 Test for kinetic model

The test for kinetic models was determined with Equation 10 (SSE,%) using Excel software. From the results as presented in Table 2, Pseudo second order kinetic model had the least error (0.007 and 0.009) for the two adsorbate concentrations tested respectively. Thus, the adsorption of Pb fitted well into Pseudo Second Order Kinetics. This suggests that Chemisorption may be the limiting step in the adsorption process. Similar observation was also reported by [12] and [13]. The linear models and the corresponding kinetic constants for all the kinetic models tested are presented in Table 2.

3.3 Modelling Lead Adsorption Process onto CPTAC using Response Surface Methodology

The modelling of the adsorption of lead onto CPTAC was done using XLSTAT 2014 software in which the results of the DoE of the experiments were used to model the adsorption process. The results from the experiment performed using a response surface design of experiment is presented in Table 3.
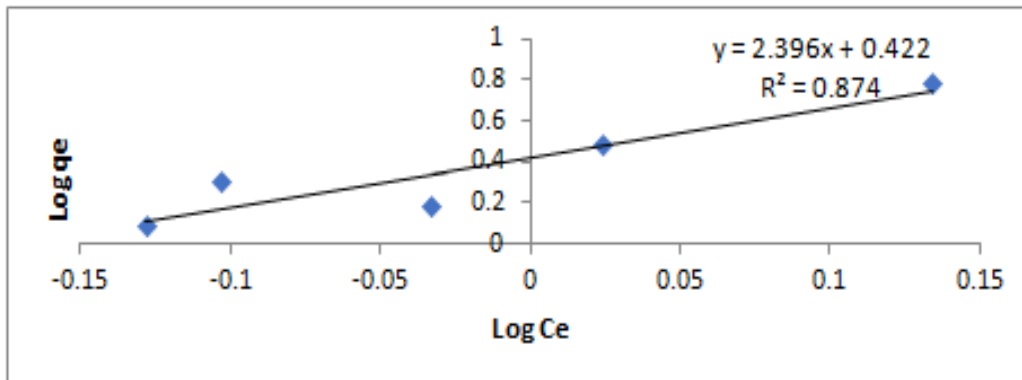
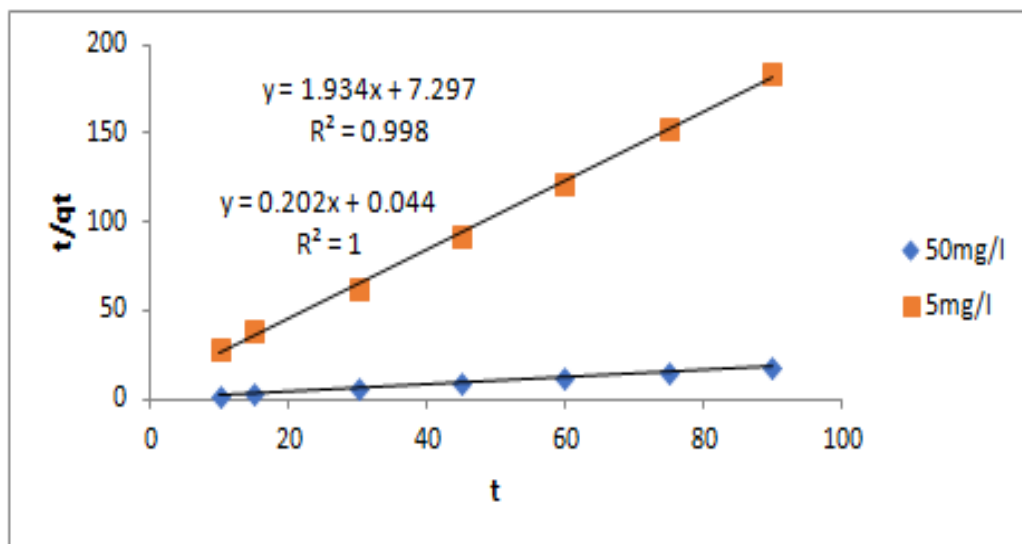**Fig. 3. Freundlich Adsorption Isotherm for Removal of lead (II) with CPTAC**

Fig. 4. Pseudo-second order kinetic Plot for Removal of lead (II) with CPTAC at varying conditions

Table 2. Adsorption Kinetic /corresponding Constants for the Adsorption of lead (II) onto CPTAC

Adsorption Kinetic Model	Parameter/Model	Adsorbate Conc. (mg/l)	
		50	**5**
Pseudo-first order	$q_{e.exp.}$ (mg/g)	4.928	0.494
	$q_{e\,cal.}$ (mg/g)	-1.216	-0.980
	kp_1	-0.016	-0.022
	R^2	0.445	0.659
	SSE%	2.32	0.557
	Linearized model	Y= -0.016x-1.246	Y= -0.022x-0.980
Pseudo-second order	$q_{e.exp.}$ (mg/g)	4.928	0.494
	$q_{e\,cal.}$ (mg/g)	4.910	0.517
	Kp_2	0.983	0.513
	R^2	1	0.998
	SSE%	0.007	0.009
	Linearized model	Y= 0.202x+0.044	Y= 1.934+7.297

Table 3. Experimental design (full factorial design with 3 Levels)

Observation	Sort order	Run Order	Repetitions	Conc.	Contact Time	pH	Equilibrium Conc.	Amount Adsorbed	Percentage removal
Obs1	1	1	1	5	30	2	0.85	4.15	83
Obs2	2	2	1	27.5	30	2	0.953	26.547	96.5
Obs3	3	3	1	50	30	2	1.565	48.435	96.9
Obs4	4	4	1	5	105	2	0.975	4.025	80.5
Obs5	5	5	1	27.5	105	2	0.786	26.714	97.1
Obs6	6	6	1	50	105	2	1.31	48.69	97.4
Obs7	7	7	1	5	180	2	0.622	4.378	87.6
Obs8	8	8	1	27.5	180	2	0.225	27.275	99.2
Obs9	9	9	1	50	180	2	1.166	48.834	97.7
Obs10	10	10	1	5	30	7	0.001	4.999	99.9
Obs11	11	11	1	27.5	30	7	0.543	26.957	98
Obs12	12	12	1	50	30	7	0.836	49.164	98.3
Obs13	13	13	1	5	105	7	0.205	4.795	95.9
Obs14	14	14	1	27.5	105	7	0.52	26.98	98.1
Obs15	15	15	1	50	105	7	0.215	49.785	99.6
Obs16	16	16	1	5	180	7	0.229	4.771	95.4
Obs17	17	17	1	27.5	180	7	0.857	26.643	96.9
Obs18	18	18	1	50	180	7	0.22	49.78	99.6
Obs19	19	19	1	5	30	12	0.249	4.751	95
Obs20	20	20	1	27.5	30	12	0.258	27.242	99.1
Obs21	21	21	1	50	30	12	0.266	49.734	99.5
Obs22	22	22	1	5	105	12	0.045	4.955	99.1
Obs23	23	23	1	27.5	105	12	0.23	27.27	99.2
Obs24	24	24	1	50	105	12	0.245	49.755	99.5
Obs25	25	25	1	5	180	12	0.551	4.449	89
Obs26	26	26	1	27.5	180	12	0.399	27.101	98.5
Obs27	27	27	1	50	180	12	0.315	49.685	99.4

3.3.1 Modelling equilibrium concentration adsorption

The adsorption of Pb onto CPTAC was modelled using XLSTAT 2014 to analyze the equilibrium concentration adsorption result and a model equation was obtained to predict the equilibrium concentration adsorption of Pb onto CPTAC. The equation of the model is presented in Equation 11.

Equilibrium Conc. = $0.35230 + 0.13394 * Co - 0.05206 * CT - 0.32744 * pH + 0.01794 * Co^2 + 0.05794 * CT^2 + 0.20878 * pH^2 - 0.10567 * CT - 0.13442 * Co * pH + 0.15392 * CT * pH$ (11)

Where Co = adsorbate concentration, CT = contact time and pH = adsorbate pH

It was observed that the coefficient of determination (R^2 value) is 0.75 which indicates

a good relationship between predicted and measured values. The relationship between equilibrium concentration and variables such as adsorbate concentration, contract time and pH are shown as trace curves in Figs. 5 to 7.
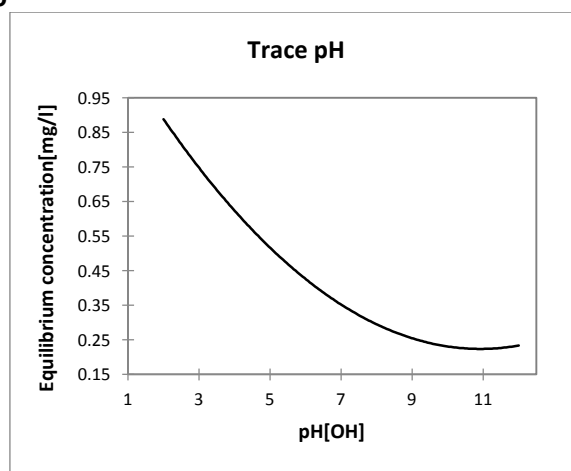
The concentration trace curve in Figure 5 shows that the equilibrium concentration increases as the adsorbate concentration increases. This may be as a result of more metal ions available for adsorption at higher concentration. Similar observation was reported by [14] and [15]. However, the trace curve for contact time as shown in Figure 6 revealed a reduction in the concentration of Pb as contact time increased and this may be due to increase in kinetic energy of the metal ions and decrease in boundary layer resistance due to mass transfer. Similar observation was made by [16]. Equilibrium was achieved around 142 mins at 0.34mg/l, after which desorption started.
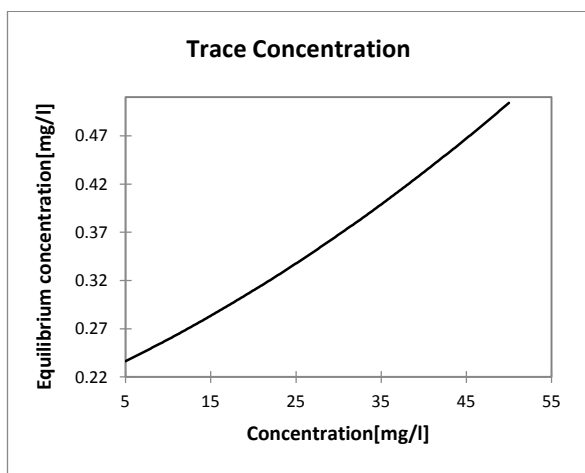
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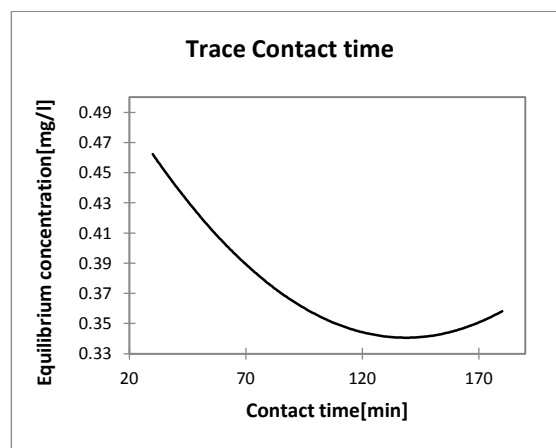
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Figs. 5, 6 and 7. Equilibrium concentration Vs Adsorbate Concentration, Contact Time and pH respectively

Furthermore, the trace curve for pH as shown in Figure 7 showed that adsorbate pH affects the solubility of the Pb ions. Note that pH of a solution is a critical parameter affecting the adsorption process because it affects the surface charge of the adsorbents as well as the degree of ionization and speciation of pollutants. The surface of the adsorbent adsorbs anions favorably at lower pH due to presence of H+ ions but adsorbs cations at higher pH due to the deposition of OH-ions [17]. Also, similar reports [18], [19] and [20] on the characterization of paw-paw peel, pawpaw trunk and pawpaw leaf activated carbon respectively, in which Fourier Transform Infrared spectroscopy (FTIR) analysis done on the activated carbon showed that the functional group indicated a broad band representing bonded OH- group. Therefore, for this study it was observed that the amount for Pb removal onto CPTAC increased as the pH tends towards alkaline and equilibrium concentration was achieved around pH 11 at 0.224mg/l. This shows that adsorbent particles may have active positive charges/sites which increased

adsorption process as it tends towards high pH environment due to deposition of OH-.

### 3.3.2 Modelling the Amount Adsorbed

The amount of Pb adsorbed onto CPTAC was modelled using XLSTAT 2014 and a model equation was obtained to predict the total amount of Pb adsorbed. The model equation is presented in Equation 12.

Amount Adsorbed = 27.14770 + 22.36606 * Co + 0.05206 * CT + 0.32744 * pH – 0.01794 * Co^2 – 0.05794 * CT^2 – 0.20878 * pH^2 + 0.10567 * Co * CT + 0.13442 * Co * pH – 0.15392 * CT * pH (12)
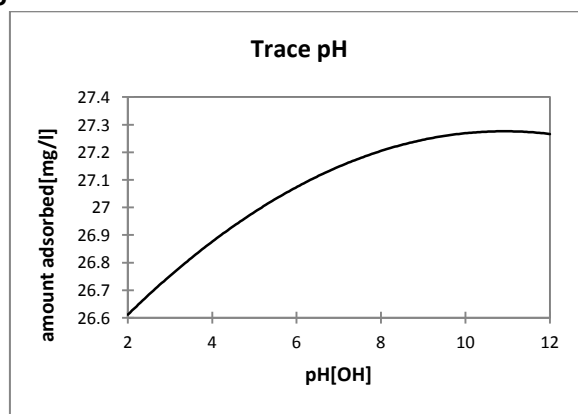
It was observed that the coefficient of determination R^2 value is 1.000. This indicates a very good relationship between predicted and measured values. The relationship between amount adsorbed and other variables such as adsorbate concentration, contract time and pH are shown as trace cures in Figs. 8 to 10.
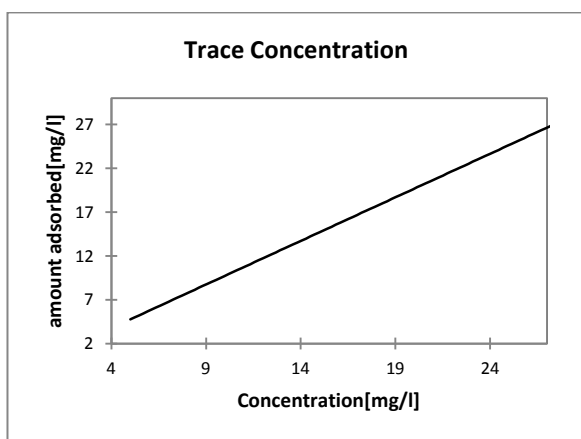
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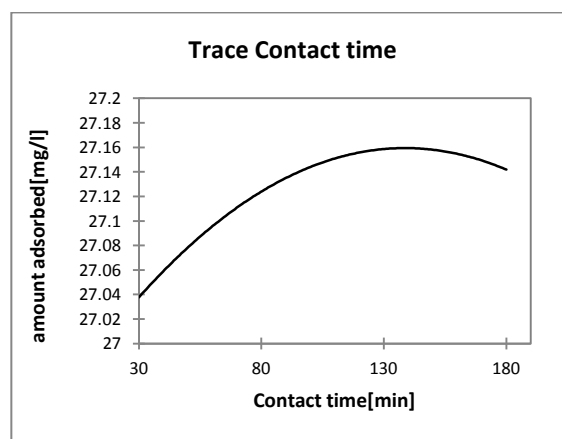
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Figs 8, 9 and 10. Amount Adsorbed Vs Adsorbate Concentration, Contact Time and pH respectively

The concentration trace curve in Figure 8 showed that amount adsorbed increased as the adsorbate concentration increases which may be as a result of more metal ions available for adsorption at higher concentration [14] and [15]. The trace curve for contact time as shown in Figure 9 revealed an increase in the amount of Pb adsorbed as contact time increased due to increase in kinetic energy of the metal ions and mass transfer [16]. The highest amount adsorbed was 27mg/l at 146 mins, after which desorption started. Furthermore, the trace curve for pH as shown in Figure 10 revealed that amount adsorbed increased as pH trends towards alkaline and the highest adsorbed at pH 11 was 27.27mg/l.

4. CONCLUSION

The study modelled the adsorption of Pb ions from a simulated aqueous phase using carica paw paw trunk activated carbon (CPTAC) produced by chemical activation with $ZnCl_2$. The batch equilibrium adsorption studies were calibrated using Langmuir, Freundlich, Temkin and Hills isotherms and results showed that adsorption process fitted best into Freundlich isotherm with R^2 value of 0.874 and adsorption capacity (K_f) of 2.64 mg/g. Model validation and test for isotherm models further confirmed that adsorption of Pb onto CPTAC could be best described by Freundlich isotherm with SSE value of 0.0397%. The kinetic data followed Pseudo second order kinetic model indicating chemisorption as the limiting step in the adsorption process. Modelling the equilibrium concentration adsorption of Pb onto CPTAC using XLSTAT 2014 resulted to Equation 11 with R^2 value of 0.75. Modelling the amount of Pb adsorbed resulted to Equation 12 with R^2 value of 1.00. This model results indicates that Equation 12 can best be used when modeling the adsorption process of Pb unto CPTAC.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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