Thermodynamic and Kinetic Behaviors of Lead (II) Adsorption on Activated Carbon Derived from Palmyra Palm Nut

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Abstract

The kinetics and thermodynamics of lead (11) uptake from aqueous using palmyra palm nut (PPN) as potential raw material for the preparation of activated carbon was studied. The effect of various factors namely pH, adsorbent dosage, and adsorbent particle size was studied to identify the adsorption capacity of palmyra palm nut. The amount of Pb^{2+} *adsorbed was found to be dependent on adsorbent dosage, pH, and particle size.. The equilibrium data conformed to Langmuir and Freundlich models while pseudo second-order best described the kinetics of the adsorption process. The thermodynamics parameters such as enthalpy, entropy and free energy were evaluated using the van't Hoff equations. The determined negative free energy changes (*ΔG*) and positive entropy changes (*ΔS*) indicate the feasibility and spontaneous nature of the adsorption process. The positive values of enthalpy change (*ΔH*) suggest that the adsorption process is endothermic. It is evident from the results that palmyra palm nut, a low-cost adsorbent, can be used for the removal of* Pb^{2+} *from aqueous solution*

Key words: Kinetics, thermodynamics, adsorption, lead, palmyra palm nut.

1. Introduction

There has been progressive increase of industrialization in Nigeria which has resulted in continuous increase of pollution. Consequently, serious effort has been devoted for minimizing these hazardous pollutants and therefore avoiding their dangerous effects on animals, plants and humans. Heavy metal ions have reported as priority pollutants, due to their mobility in natural water ecosystems and due to their toxicity (Volesky and Holan, 1995). These heavy metals are not biodegradable and their presence in streams and lakes leads to bioaccumulation in living organisms causing health problems in animals, plants, and human beings (Ong et al, 2007).

Lead is a pollutant that is present in drinking water and in air. In air, it is derived from lead emissions from automobiles because it is used as an anti knocking agent in the form of lead tetraethyl in gasoline. In water, lead is released in effluent from lead treatment and recovery industries, especially from lead battery manufacturing units. Lead is toxic to living organisms and if released into the environment can both accumulate and enter the food chain. Lead is known to cause mental retardations, reduces haemoglobin production necessary for oxygen transport and it interferes with normal cellular metabolism (Qaiser et al, 2007). Lead has damaging effects on body nervous system. It reduces 1.Q Level in children. Lead is used as industrial raw materials in the manufacture of storage batteries, pigments, leaded glass, fuel, photographic materials, matches and explosives (Raji and Anirudham, 1997). For drinking water, the maximum permissible limit of lead is 0.1mg/l (WHO, 1971). The maximum concentration allowed for discharge into inland water is less than 1mg/l (FEPA, 1991).

Several methods such as ion exchange, solvent extraction, reverse osmosis, precipitation, and adsorption have been proposed for the treatment of wastewater contaminated with heavy metals (Gupta, 2003). Among these several chemical and physical methods, the adsorption onto activated carbon has been found to be superior to other techniques because of its capability for adsorbing a broad range of different types of adsorbates efficiently, and its simplicity of design (Ahmad et al, 2006). However commercially available activated carbons are still considered expensive (Chakraborty et al, 2005). Consequently many researchers have studied cheaper substitutes, which are relatively inexpensive, and are at the same time endowed with reasonable adsorptive capacity. These studies include the use of coal (Mohan et al, 2002a), fly ash (Mohan et al, 2002b; Nollet et al, 2003; Gupta, 2003; Ricou et al, 2001; Gupta and Ali, 2004), activated clay (Wu et al, 2001), palm–fruit bunch (Nassar, 1997), Bagasse pith (Mckay, 1998), etc.

In this study, the use of Palmyra palm nut as a low-cost adsorbent for the removal of Lead (11) from aqueous solution was investigated.

2.0 Experimental

2.1 Preparation of activated carbon

Palmyra palm nut were obtained from the premises of Nnamdi Azikiwe University, Awka, Nigeria. The palm nuts were washed with deionized water, dried in the sun, ground into fine particles and sieved to a particle size of 300µm. 200g of sample was impregnated with concentrated orthophosphoric acid at the ratio of 1:2 (wt %). The impregnated sample was dried in a Memmert oven at 120^0C for 24hrs. The dried sample was carbonized in a Muffle furnace at 500^0C for 2 hours. After cooling to the ambient temperature, the sample was washed several times with de-ionized water until pH 6-7, filtered with Whatman No.1 filter paper and dried in the oven at 110^0C for 8hours. The sample was crushed and passed through different sieve sizes and then stored in a tight bottle ready for use.

2.2 Characterization of activated carbon

The pH of the carbon was determined using standard test of ASTM D 3838-80 (ASTM, 1996). Moisture content of activated carbon and raw materials was determined using ASTM D 2867-91 (1991).The bulk density of the activated carbon was determined according to the tamping procedure by Ahmedna et al (1997). The volatile content was determined by weighing 1.0g of sample and placing it in a partially closed crucible of known weight. It was then heated in a muffle furnace at 900^0C for 10mins.The percentage fixed carbon was determined as100 – (Moisture content + ash content + volatile matter). The iodine number was determined based on ASTM D 4607-86 (1986) by using the sodium thiosulphate volumetric method The specific surface area of the activated carbon was estimated using Sear's method (Al-Qadah and Shawabkah, 2009 and Alzaydien, 2009) by agitating 1.5g of the activated carbon samples in 100ml of diluted hydrochloric acid at a pH = 3. Then a 30g of sodium chloride was added while stirring the suspension and then the volume was made up to 150ml with deionized water. The solution was titrated with 0.1N NaOH to raise the pH from 4 to 9 and the volume, V recorded. The surface area according to this method was calculated as S = 32V – 25. Where, S = surface area of the activated carbon, V = volume of sodium hydroxide required to raise the pH of the sample from 4 to 9.

2.3 Adsorbate preparation and adsorption study

The reagents used were lead nitrate salt ($Pb(N0_3)_2$), and de-ionized water. The reagents were of high grade. The sample $(Pb(N0_3)_2$ was dried in an oven for 2hrs at 105^0C to remove moisture. 1.6g of $Pb(N0_3)_2$ was dissolved in 1000ml of de-ionized water to get the stock solution of 1000g/l. A known weight of activated carbon was added to 100ml of the 100mg/l of adsorbate in a conical flask and placed on a magnetic stirrer. The stirring was done at 30^0C for 3 hours. After adsorption is complete, the solution was filtered using Whatman no.1 filter paper. The residual Pb^{2+} concentrations of the effluent were determined spectrophotomically using atomic adsorption spectrophotometer at 217.0nm wavelength. The adsorption capacity, q_e (mg/g) and percentage adsorbed were calculated from equation 1 and equation 2 respectively.

$$q_e = [(C_o - C_e)\,V/m] \qquad \ldots\ldots \qquad 1$$

$$\%\ Adsorbed = [(C_o - C_e)/C_o]\ x\, 100 \qquad \ldots\ldots \qquad 2$$

Where, C_o and C_e are the metal concentrations (mg/l) at initial and any time t, respectively, V the volume of the solution (l), and m the mass of adsorbent used (g). The effects of particle size, adsorbent dosage and pH were studied.

3. Results and Discussions

3.1 Characteristics of activated carbon derived from nipa palm nut:

The physico-chemical characteristics of activated carbon are shown in Table 1.

3.2 Effect of adsorption dosage on the adsorption process

Adsorption dosage is an important parameter because this factor determines the capacity of an adsorbent for a given initial concentration of the adsorbate. The results showed that as the adsorbent dosage increased, the percentage of adsorption also increased but the amount adsorbed per unit mass of the adsorbent decreased considerably (Figure 1).

The decrease in unit adsorption with increasing dose of adsorbent is basically due to adsorption sites remaining unsaturated during the adsorption reaction (Bulut Aydin, 2005).

3.3 Effect of particle size on the adsorption process

The removal of Pb^{2+} at different particle sizes showed that the removal rate increased better with a decrease in particle size (Figure 2). This same trend was reported by Demirbas et al (2004). The relatively higher adsorption with smaller adsorbate particle may be attributed to the fact that smaller particles yield large surface area. There is a tendency that a smaller particle produces shorter time to equilibrium.

3.4 Effect of pH on the adsorption process

pH is one of the most important environmental factors influencing not only site dissociation, but also the solution chemistry of heavy metal: hydrolysis, complexation by organic and/or inorganic ligands, redox reactions, and precipitation are strongly influenced by pH and on the other hand, it strongly influences the speciation and adsorption availability of heavy metals (Sekar et al, 2004). Figure 3 presents the effect of pH on the removal of Pb^{2+}. The adsorption of the Pb^{2+} increased with increasing pH. The maximum Pb^{2+} adsorption for all the adsorbents was obtained at pH 6. Pb^{2+} adsorption decreased as pH rose beyond the optimum pH. Above pH 6, Pb(II) started precipitating as $Pb(OH)_2$. Similar result was found by Liu and coworkers (Liu et al, 2007).

3.5 Adsorption Isotherm

The adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state (Hameed et al, 2006).

3.5.1 Langmuir isotherm model

The Langmuir adsorption model is given by:

$$q_e = Q \quad \frac{b\,C_e}{1+b\,C_e} \qquad \ldots.. \qquad 3$$

The Langmuir equation can be described by the linearized form (Sivakumar and Palanisamy, 2009; Sekar et al, 2004).

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \left(\frac{1}{Q_o}\right) C_e \qquad \ldots.. \qquad 4$$

Where C_e is the equilibrium concentration of the adsorbate, q_e the amount of adsorbate per unit mass of adsorbent (mg/g), and $Q_o and\ b$ are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. When C_e / q_e is plotted against C_e, straight line with slope $1/Q_o$ indicates that adsorption follows the Langmuir isotherm. The Langmuir constants, Q and b were evaluated from the intercept and the slope of the linear plot of experimental data of $^1/q_e$ versus $^1/c_e$ and presented in Table 2. The essential characteristics of the Langmuir equation can be expressed in terms of a dimensionless separation factor, R_L, defined as (Ahmad et al, 2006)

$$R_L = \frac{1}{1+bC_O} \qquad \ldots.. \qquad 5$$

where C_o is the initial ion concentration. The R_L value implies the adsorption to be unfavourable ($R_L > 1$), Linear ($R_L = 1$), favourable ($O<R_L<1$), or irreversible (R_L=0). $R_L$values for Pb^{2+} adsorption were less than 1 and greater than zero indicating favourable adsorption under conditions used in this study (Table 2). The correlation coefficients showed that adsorption of Pb^{2+} on PPN followed Langmuir.

3.5.2 Freundlich Isotherm

The Freundlich adsorption isotherm (Ozacar, 2003) is

$$q_e = K_F C_e^{1/n} \qquad \ldots.. \qquad 6$$

A linear form of this expression is:

$$\log\ q_e = \log K_F + {}^1/_n \log C_e \qquad \ldots.. \qquad 7$$

Where K_F and n are Freundlich constants, n giving an indication of how favourable the adsorption process is and K_F is the adsorption capacity of the adsorbent. K_F and n are determined from the linear plot of $\log q_e$ versus $\log C_e$. From the linear plots of Inq$_e$ versus InC$_e$, values of Freundlich constants K$_F$ and n were calculated from the intercept and slope, respectively, and are presented in Table 2. The magnitude of the exponent, n gives an indication of the favourability and capacity of the adsorbent/adsorbate system. Karthikeyan et al (2003) has reported that n values between 1 and 10 represent favourable adsorption conditions. In this work, the exponent is $1<n<3$, showing beneficial adsorption for the system. The correlation coefficients, R^2, ranging from 0.997 to 0.999 indicate the Pb^{2+} adsorption followed Freundlich isotherm model. Similar results were obtained by Aksu and Tezer (2000).

3.5.3 Temkin isotherm

The Temkin isotherm (Choy et al, 1999) has been used in the following form:

$$q_e = \frac{RT}{b} In(AC_e) \qquad ….. \qquad 8$$

A linear form of the Temkin isotherm can be expressed as:

$$q_e = \frac{RT}{b} In\,A + \frac{RT}{b} In\,C_e \qquad ….. \qquad 9$$

Where $RT/b = B$. The values of Temkin constants A and b and correlation coefficients are listed in Table 2. The values of constants A and b obtained were similar to values obtained by Sekar et al (2004). Correlation coefficients obtained were above 0.985. The data conformed to Temkim isotherm model.

3.5.4 Dubinin-Radusherich isotherm model

The Dubinin – Radushkevich equation has the following form:

$$q_e = q_m e^{-\beta\varepsilon^2} \qquad ….. \qquad 10$$

A linear form of Dubnin – Radushkevich equation isotherm (Sivakumar and Palanisamy, 2009) is:

$$Inq_e = Inq_m - \beta\varepsilon^2 \qquad ….. \qquad 11$$

Where q_m is the Dubinin – Radushkevich monolayer capacity $(mmol/g)$, β a constant related to sorption energy, and ε is the Polanyi potential which is related to the equilibrium concentration as follows:

$$\varepsilon = RT\,In\left(1+\frac{1}{C_e}\right) \qquad ….. \qquad 12$$

Where, R is the gas constant $(8.31 J/mol\,K)$ and T is the absolute temperature. The constant β gives the mean free energy, E, of sorption per molecule of the sorbate when it is transferred to the surface of the solid from infinity in the solution and can be computed using relationship (Lin and Juang, 2002).

$$E = \frac{1}{\sqrt{2\beta}} \qquad ….. \qquad 13$$

The Dubinin-Radushkevich constants were evaluated and given in Table 2. The correlation coefficients were determined as shown in Tables 2. R^2 indicated that the adsorption of Pb^{2+} did not follow the Dubinin-Radushkevich isotherm. If the mean free energy, E is <8KJ/mol, the adsorption is physisorption and if the energy of activation is 8-16KJ/mol, the adsorption is chemisorption in nature (Sarin et al, 2006; Sivakumar and Palanisamy, 2009). Results of E obtained indicated that adsorption of Pb^{2+} on PPN is physisorption.

3.6 Adsorption Kinetics

3.6.1 Pseudo first-order kinetic model

The Pseudo first-order kinetic model was proposed by Lagergren (Maniatis and Nurmala, 1992). The equation is generally expressed as

$$\frac{dq_t}{dt} = K_I\left(q_e - q_t\right) \qquad \text{.....} \qquad 14$$

Where, q_e and q_t are the adsorption capacity at equilibrium and at time t, respectively (mgg^{-1}), K_1 is the rate constant of pseudo first -order adsorption (L min $^{-1}$).

After integration and applying boundary conditions, t = 0 to t = t and $q_t = 0$ to $q_t = q_t$, the integrated form of equation 2.16 becomes:

$$\log\left(q_e - q_t\right) = \log\left(q_e\right) - \frac{K_1}{2.303}t \qquad \text{.....} \qquad 15$$

The values of log ($q_e - q_t$) are linearly correlated with t. The slope and intercept of plots of log (q_e-q_t) versus t were used to determine the pseudo-first order rate constants, K_1 and q_e at different temperatures. The results of the q_e and K_1 were evaluated and listed in Table 3. The correlation coefficients ranged from 0.973 to 0.999 indicating that Pb^{2+} adsorption fitted well to pseudo first-order kinetic equation.

3.6.2 Pseudo second- order model

The adsorption kinetics may also be described by a pseudo second-order equation (Chiou and Li, 2002; Nwabanne and Igbokwe, 2008; Ho and Chiang, 2001).

$$\frac{dq_t}{dt} = \quad K_2(q_e - q_t)^2 \qquad \text{.....} \qquad 16$$

Integrating equation 2.19 and applying the boundary condition

$$t = 0\ to\ t = t\ and\ q_t = 0\ to\ q_t = q_t,\ gives$$

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + K_2\ t \quad 0 \qquad \text{.....} \qquad 17$$

Equation (2.20) can be rearranged to obtain a linear form:

$$\frac{t}{q_t} = \frac{1}{K_2 {q_e}^2} + \frac{1}{q_e}t \qquad \text{.....} \qquad 18$$

Where, K_2 is the rate constant, t of pseudo second-order adsorption (gmg^{-1} min^{-1}). The slope and intercept of plot of t/q $_t$ versus t were used to calculate the pseudo second-order rate constant, at different temperatures. Table 3 lists the results of the rate constant and q_e by pseudo second-order models. The correlation coefficient, R^2 for the pseudo second-order adsorption model has high value (> 0.993). Its calculated equilibrium adsorption capacity, q_e is consistent with experimental data. These facts suggest that the pseudo second-order adsorption mechanism is predominant. Similar phenomena have also been observed in the adsorption of direct dye on palm ash (Ahmad et al, 2006).

3.6.3 Elovich kinetic model

The Elovich model is generally expressed as:

$$\frac{dq_t}{dt} = \quad \alpha\ell^{-\beta q_t} \qquad \text{.....} \qquad 19$$

Integrating this equation for the boundary conditions, gives:

$$q_t = \frac{1}{\beta}\mathit{In}\left(\alpha\,\beta\right) + \frac{1}{\beta}\mathit{In}\ t \qquad \text{.....} \qquad 20$$

Where, α is the initial adsorption rate (mg/min) and β is related to the extent of surface coverage and the activated energy for chemisorption (g/mg). A plot of q_t versus lnt gives a linear trace with a slope of (1/β) and an intercept of 1/β ln (αβ).

The results of Elovich plots for the adsorption of Pb^{2+} on PPN at different temperatures are given in Table 3. The plots were linear with correlation coefficients ranging from 0.934 to 0.974. Similar results were obtained by Sivakumar and Palanisamy (2009). High values of initial adsorption rate, α were obtained for Pb^{2+} adsorption on PPN.

3.6.4 Bhattacharya – Venkobachor kinetic model

Bhattacharya – Venkobachor model (Goswami and Ghosh, 2005) is expressed as

$$Ln\left[1-U_{(t)}\right]=K_B.t \qquad \ldots.. \qquad 21$$

$$U_{(t)}=\left(C_o-C_t\right)/\left(C_o-C_e\right) \qquad \ldots.. \qquad 22$$

The effective diffusion coefficient D_2, is obtained from the equation:

$$D_2=\frac{K_B.r^2}{\pi^2} \qquad \ldots.. \qquad 23$$

Where, r is particle radius. From the plot of In [1-U (t)] vs t, the effective diffusion coefficient D_2 and rate constant, K_B were obtained and presented in Table 3. The effective diffusion coefficients determined are in the range from $3.4593\text{x}10^{-11}$ to $6.031\text{x}10^{-10}$ m^2/s.

3.7 Thermodynamics study

3.7.1 Activation Energy

The activation energy of adsorption was calculated from the linearized Arhenius equation.

$$InK_p=InK_o-E_a\,/\,RTv \qquad \ldots.. \qquad 24$$

Where K_p is the rate constant second-order of adsorption (g/mol min), K_o is the independent temperature factor (g/mol min), E_a is the activation energy of adsorption KJ/mol), R is the gas constant (J/molK) and T is the solution temperature (K). Activation energy, E_a was evaluated from the slope of the plot of In K_p versus ${}^1/T$. The E_a obtained was 10.193KJ/mol. The values were similar to the values obtained by (Bulut Aydin, 2005).

3.7.2 Thermodynamics properties

The mechanism of adsorption was determined through thermodynamic qualities such as change in free energy (ΔG), change in enthalpy (ΔH), and change in entropy (ΔS). The thermodynamic equilibrium constant K for the sorption was determined by Khan and Singh (Lyubchik et al 2004); Liu et al, 2007) from the intercept of the plots of In (q_e/C_e) versus q_e. Then, the ΔG, ΔH and ΔS are calculated from the Van't Hoff equations (Lyubchik et al 2004)

$$\Delta G=-RT\,InK \qquad \ldots.. \qquad 25$$

$$InK=\frac{\Delta S}{R}-\frac{\Delta H}{RT} \qquad \ldots.. \qquad 26$$

The thermodynamic parameters were calculated from the values of the thermodynamic equilibrium constant, K by plotting of In K versus 1/T. Then the slope and intercept were used to determine ΔH and ΔS, and the van't Hoff equation (Equation 26) was applied to calculate ΔG. The values of ΔH, ΔS and ΔG were given in Tables 4. The negative values of ΔG indicate the feasibility of the process and also the spontaneity of adsorption reaction (Goswami and Ghosh, 2005). The values of ΔH were positive and they confirm the spontaneity and endothermic character of the reaction. The positive values of ΔS show the increased randomness at the solid-solution interface during the adsorption of Pb^{2+} on the adsorbent.

4. Conclusion

The thermodynamics and kinetic study of the removal of lead (11) from aqueous solution using activated carbon prepared from palmyra palm nut has been investigated in this work. The adsorption process showed that palmyra palm nut was effective in in the uptake of lead (11) from aqueous solution. The amount of Pb^{2+} adsorbed was found to vary with pH, particle size and adsorbent dosage. The adsorption data conformed to Langmuir and Freundlich models. The adsorption data also fitted Bhattacharya-Venkobachor, Elovich, pseudo first-order and pseudo second-order models. However, pseudo second-order best described the adsorption process. The determined negative free energy changes (ΔG) and positive entropy change (ΔS) indicate the feasibility and spontaneous nature of the adsorption process. The positive value of enthalpy change (ΔH) suggests that the adsorption process is endothermic.

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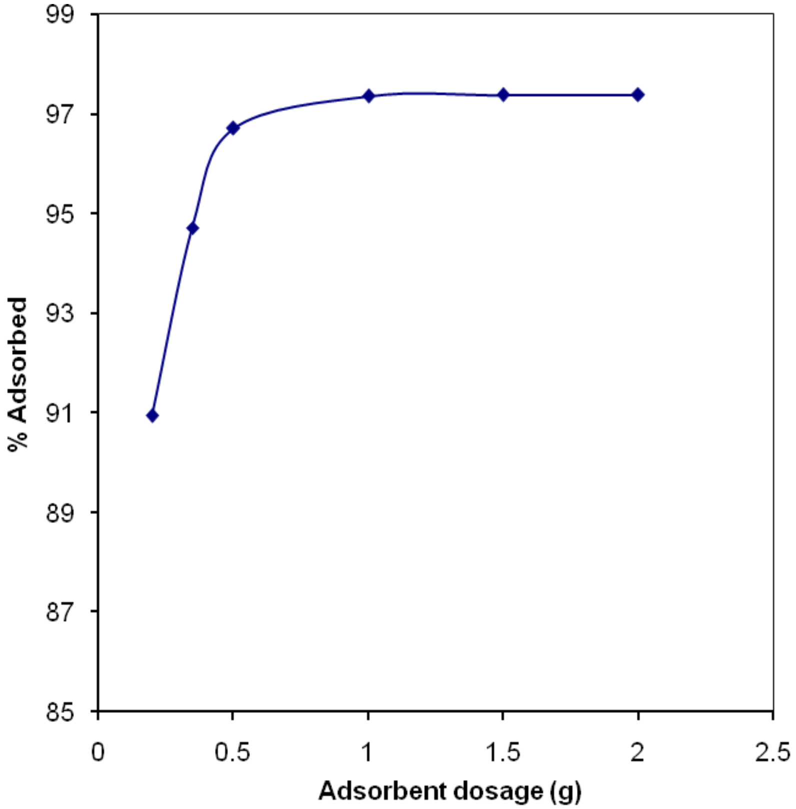

Fig. 1: Effect of adsorbent dosage on the adsorption of Pb^{2+} on PPN

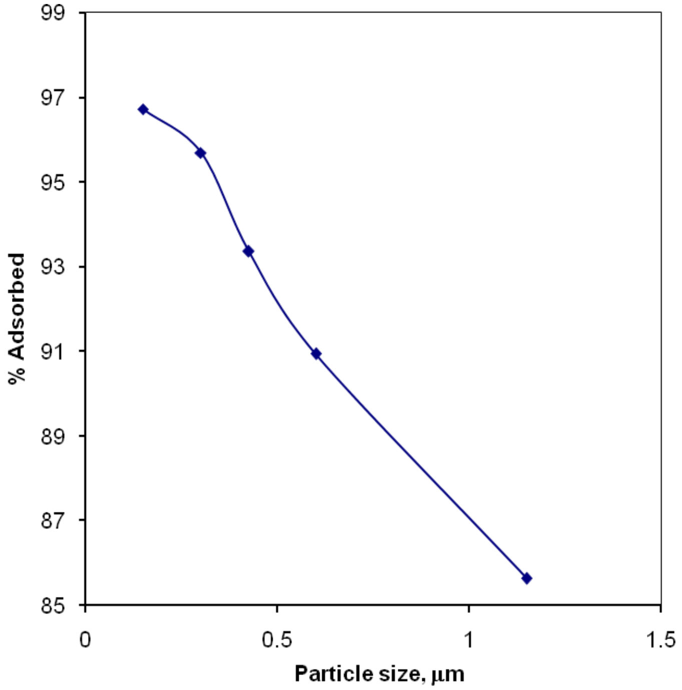

Fig. 2: Effect of particle size on the adsorption of Pb^{2+} on PPN

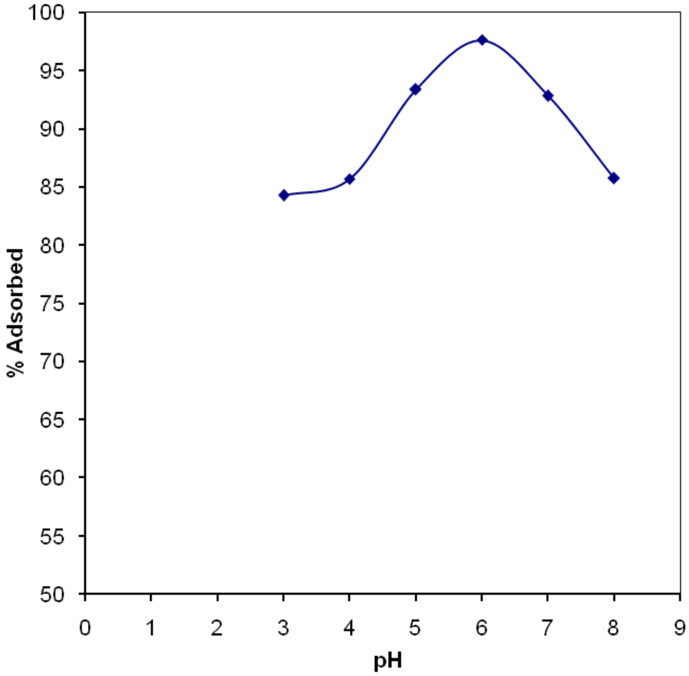

Fig. 3: Effect of pH on the adsorption of Pb^{2+} on PPN

Table 1: Physico-chemical characteristics of activated carbon derived from palmyra palm nut.

Properties	Values
pH	6.8
Bulk density , g/cm^3	0.61
Iodine number, mg/g	785.78
Moisture content, %	4.10
Volatile matter, %	18.14
Ash content, %	3.30
Fixed carbon, %	78.56
Surface Area, m^2/g	820.37

Table 2: Calculated isotherm parameters for the adsorption of Pb^{2+} on PPN

Isotherm model	Temperature, K 303	 313	 323
Langmuir			
Q (mg/g)	125	142.85	142.85
b (L/mg)	0.0620	0.0583	0.0625
R_L	0.1389	0.1464	0.1379
R^2	0.986	0.984	0.985
Freundlich			
n	2.907	2.915	2.9326
K_f (L/g)	20.464	21.086	21.627
R^2	0.999	0.998	0.997
Temkin			
b (J/mg)	135.22	139.095	143.30
A (L/g)	1.864	2.017	2.147
R^2	0.942	0.941	0.945
Dubinin-Radushevich			
q_D (mg/g)	70.246	70.598	71.307
β (mol^2/J^2)	$4x10^{-7}$	$3x10^{-7}$	$3x10^{-7}$
E (KJ/mol)	0.0001	0.0001	0.0001
R^2	0.665	0.665	0.669

Table 3: Calculated kinetic parameters for the adsorption of Pb^{2+} on PPN

Kinetic model	Temperature, K 303	 313	 323
Pseudo first-order			
K_1 (min^{-1})	0.930	0.0392	0.0392
q_e (mg/g)	1.7061	1.6634	1.5922
R^2	0.986	0.973	0.981
Pseudo second-order			
K_2 (g/mg min)	0.0595	0.0595	0.0616
q_e (mg/g)	20	20	20.4082
R^2	1	1	1
Elovich			
α (mg/g min)	$1.48x10^{16}$	$2.43x10^{16}$	$8.09x10^{16}$
β (g/mg)	2.1459	2.1692	2.2272
R^2	0.969	0.965	0.972
Bhattacharya-Venkobachor			
K_B	0.041	0.041	0.038
R^2	0.986	0.977	0.977
D_2 (m^2/s)	$3.619x10^{-11}$	$3.569x10^{-11}$	$3.493x10^{-11}$

Table 4: Thermodynamic parameters for the adsorption of Pb^{2+} on PPN

Adsorbate	T(K)	ΔG($KJmol^{-1}$)	ΔS($Jmol^{-1}K^{-1}$)	ΔH($KJmol^{-1}$)
303	-2.028			
313	-2.180	15.24		2.590
323	-2.333			