

Kinetics and thermodynamics of lead ion sorption on palm kernel fibre from aqueous solution

Yuh-Shan Ho^{a,*}, Augustine E. Ofomaja^b

^a School of Public Health, Taipei Medical University, 250 Wu-Hsing Street, Taipei 11014, Taiwan

^b Department of Chemistry, Faculty of Science, University of Benin, Ugbowo-Lagos Road, Ugbowo, PMB 1154, Benin City, Edo State, Nigeria

Accepted 8 February 2005

Abstract

The kinetics and thermodynamics of lead ions sorption from aqueous solution on palm kernel fibre have been investigated. The experimental data was analyzed by Langmuir, Freundlich and Redlich–Peterson isotherms using linear coefficient of determination and the non-linear Chi-square test. The equilibrium sorption capacity of lead ions was determined from the Langmuir equation and found to be 49.9 mg/g at 65 °C. A batch sorption model, based on the assumption of the pseudo-second-order mechanism, was applied to predict the rate constant of sorption, the equilibrium sorption capacity and the initial sorption rate with the effect of temperature. An activation energy of sorption was evaluated as 13.5 kJ/mol for the sorption of lead ions on palm kernel fibre. Various thermodynamic parameters, ΔH° , ΔS° and ΔG° , were computed from equilibrium constant values. The results show that the sorption of the lead ions on palm kernel fibre is a spontaneous and endothermic nature process.

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Keywords: Adsorption; Lead; Palm kernel fibre; Kinetic

1. Introduction

It is known that some chemical substances, if present in certain concentrations in water, may constitute danger to health. One of such chemical substance is lead. Lead is often discharged by a number of industrial processes and this can lead in turn to contamination of fresh and marine environment. Industrial emissions and mining activity contributes largely to lead in water either as direct discharge in to water bodies or as atmospheric discharge which are then deposited on water surfaces [1,2]. Other sources of lead in water includes through lead pipes [3] and deposition of air borne lead from automobile exhausts from leaded gasoline on surface water [4]. Lead is not biodegradable and tends to accumulate in living organisms causing diseases and disorders [5]. Therefore, increase in lead concentrations

above the consent limits is becoming an important subject to public health. Acute lead poisoning usually affects the gastrointestinal track, or the nervous system, and sometimes both [6].

Removal of this substance from water and wastewater becomes a challenge to researchers as new, cheaper and more effective adsorbents are required for water purification. In this study, a material relevant to the situation in Nigeria, palm kernel fibre (shaft) was chosen. The palm oil is an important cash crop in West Africa and it is a major industry whose products are palm oil, palm kernel oil, and kernel cake. The major waste material produced during palm oil production are palm kernel shell, and palm kernel fibre (shaft). In Nigeria, palm oil being a rich source of Vitamin A, is used for cooking, margarine production, soap manufacture and so on. To meet the demand of palm oil consumption, several medium and small-scale industries are operated along side the major palm oil industries producing large amounts of wastes annually. Over 15,600 tonnes of shell and fibre are generated annually and only about 5% of the waste are sometimes burnt to generate heat for the boiler [7]. Palm

* Corresponding author. Present address: Department of Environmental Sciences, College of Environmental Sciences Peking University, Beijing 100871, People's Republic of China. Tel.: +86 10 62751923.

E-mail address: dr_ysho@hotmail.com (Y.-S. Ho).

kernel fibre (shaft) is obtained after pressing out the oil from the palm nut. The fibre retains some of the oil after extraction which dries and decay on the fibre by bacteria action. This may then form humic substances already identified by researchers [8] and will increase the potential of its use as an adsorbent for metal ions.

In this study, the sorption capacity of palm kernel fibre for lead ions has been investigated by determining the equilibrium isotherm at various temperatures. In each system three isotherms, the Langmuir, Freundlich and Redlich–Peterson have been analyzed. Kinetic studies have been carried out to study the effect of temperature.

2. Materials and methods

2.1. Materials

Palm kernel fibre was obtained from the Nigerian Institute for Oil Palm Research (NIFOR) Benin City, Nigeria. Palm kernel fibre was allowed to age or decay with the residual oil, after processing for about 2 months. The raw fibre was dried in an oven at 80 °C for 6 h, grounded and screened through a set of sieves to obtain particles of size 50–60 µm. The sieved fibre was soaked in 0.02 mol/dm³ HCl overnight. The acid solution was filtered off and the fibre washed with distilled water until the pH of the wash becomes neutral. The fibre was dried at 80 °C for 24 h and stored in an air-tight container. The stock solution of lead(II) (1000 mg/dm³) was prepared in distilled water using lead nitrate salt (BDH Ltd.) all working solutions were prepared by diluting the stock solution with distilled water.

2.2. Methods

2.2.1. Equilibrium studies

A volume of 40 ml of lead(II) solution with a concentration ranging from 20 to 200 mg/dm³ was placed in a 250 ml conical flask and set at pH 5. 0.1 g of the palm kernel fibre was accurately weighed and added to the solution. The conical flask was then shaken at a constant speed of 200 rpm in a shaking water bath with temperatures 36, 45, 55 and 65 °C, respectively. After shaking the flasks for 1 h, the palm kernel fibre was separated by filtration. The filtrate was analyzed for the remaining lead concentration by atomic adsorption spectrophotometry (AAS).

2.2.2. Kinetic studies

A range of reaction temperatures (36, 45, 55 and 65 °C) were used and the flasks were agitated for 14 min. All contact investigations were performed in a 1 dm³ flask. A 1.0 g sample of palm kernel fibre was added to 400 ml volume of lead(II) solution set at pH 5 and agitated at 200 rpm for all the experiments. The experiments were carried out at initial lead concentration 120 mg/dm³ for all the studies. Samples (2.5 ml) were withdrawn at suitable

time interval, filtered and the filtrate analyzed for the remaining lead concentration with atomic absorption spectrophotometry.

3. Results and discussion

3.1. Equilibrium studies

The analysis of the isotherm data is important to develop an equation which accurately represents the results and could be used for design purposes. In order to investigate the sorption isotherm, three models were analyzed: the Langmuir, the Freundlich and the Redlich–Peterson isotherm. The Langmuir sorption isotherm [9] is perhaps the best known of all isotherms describing sorption. The theoretical Langmuir isotherm is often used to describe sorption of a solute from a liquid solution as:

$$q_e = \frac{q_m K_a C_e}{1 + K_a C_e} \quad (1)$$

The constants q_m and K_a are the characteristics of the Langmuir equation and can be determined from a linearised form of Eq. (1), represented by:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_a} + \frac{C_e}{q_m} \quad (2)$$

where C_e is the equilibrium concentration (mg/dm³); q_e the amount of lead sorbed (mg/g); q_m is q_e for a complete monolayer (mg/g); K_a is sorption equilibrium constant (dm³/mg).

The Freundlich isotherm [10] is the earliest known relationship describing the sorption equation and is expressed by the following equation:

$$q_e = K_F C_e^{1/n} \quad (3)$$

and the equation may be linearised by taking logarithms

$$\log(q_e) = \frac{1}{n} \log(C_e) + \log(K_F) \quad (4)$$

where K_F and $1/n$ are empirical constants dependent on several environmental factors.

The Redlich–Peterson isotherm [11] contains three parameters and incorporates the features of the Langmuir and the Freundlich isotherms. It can be described as follows:

$$q_e = \frac{A C_e}{1 + B C_e^g} \quad (5)$$

It has three isotherm constants, namely, A , B and g ($0 < g < 1$). These can be evaluated from the linear plot represented by Eq. (6) using a trial and error optimization method:

$$\ln\left(\frac{C_e}{q_e} - 1\right) = g \ln(C_e) + \ln(B) \quad (6)$$

3.2. Error analysis

The optimisation procedure requires an error function to be defined in order to be able to evaluate the fit of the equation to the experimental data. In this study, linear coefficient of determination and non-linear Chi-square were examined. The coefficient of determination, r^2 , represents the percentage of variability in the dependent variable that has been explained by the regression line. The value of the coefficient of determination may vary from zero to one. A coefficient of determination of one indicates that 100% of the variation of q_e has been explained by the regression equation. The linear coefficient of determination, r^2 , found from evaluation of data by linear model, was calculated with aid of the equation:

$$r^2 = \frac{S_{xy}^2}{S_{xx}S_{yy}} \quad (7)$$

where S_{xx} is the sum of squares of X

$$S_{xx} = \sum_{i=1}^n x_i^2 - \frac{\sum_{i=1}^n x_i}{n} \quad (8)$$

where S_{yy} is the sum of squares of Y

$$S_{yy} = \sum_{i=1}^n y_i^2 - \frac{\sum_{i=1}^n y_i}{n} \quad (9)$$

where S_{xy} is the sum of squares of X and Y

$$S_{xy} = \sum_{i=1}^n x_i y_i - \frac{\left(\sum_{i=1}^n x_i\right)\left(\sum_{i=1}^n y_i\right)}{n} \quad (10)$$

The Chi-square test statistic is basically the sum of the squares of the differences between the experimental data and data obtained by calculating from models, with each squared difference divided by the corresponding data obtained by calculating from models. The equivalent mathematical statement is:

$$\chi^2 = \sum \frac{(q_e - q_{e,m})^2}{q_{e,m}} \quad (11)$$

$q_{e,m}$ equilibrium capacity obtained by calculating from model (mg/g), q_e experimental data of equilibrium capacity (mg/g). If data from model are similar to the experimental data, χ^2 will be a small number and if they are different, χ^2 will be a bigger number. Therefore, it is necessary to also analyze the data set on the non-linear Chi-square test to confirm the best-fit isotherm for the sorption system.

In order to assess different isotherms and their ability to correlate with experimental results, the theoretical plots from each isotherm have been shown with the experimental data for sorption of lead ions on palm kernel fibre at the temperature of 36 °C in Fig. 1. The graph is plotted in the form of lead(II) sorbed per unit mass of fibre, q_e , against the

concentration of lead(II) remaining in solution, C_e . A comparison of coefficient of determination for three isotherms has been made and listed in Table 1. The coefficient of determination, r^2 ; are 1.000 for the Langmuir isotherms. It is clear that the Langmuir isotherms have best fitted for the sorption of lead on palm kernel fibre at various temperatures. However, the values of χ^2 of the Redlich–Peterson isotherms were smaller than that of Langmuir and Freundlich. Consequently, the Redlich–Peterson isotherm was the most suitable models for this sorption system. Unlike the linear analysis, different forms of equation would effect r^2 significantly and impact the final determination where non-linear Chi-square analysis would be a method of avoiding such errors.

The effect of temperature on the sorption isotherms is shown in Fig. 2. Results indicate that the capacity of palm kernel fibre for sorption of lead(II) increases with temperature which is typical for the biosorption of most metal ions from their solution [12,13]. When the system is in a state of equilibrium, the distribution of lead(II) between the palm kernel fibre and the lead(II) solution is of fundamental importance in determining the maximum sorption capacity of palm kernel fibre for the lead ion from the isotherm. The Langmuir isotherm is applicable to homogeneous sorption where each lead ion/fibre sorption process has equal sorption activation energy. The Langmuir constants K_a and q_m have been determined with Eq. (2) and are shown in Table 2. The equilibrium sorption capacity, q_m ; was found to increase from 40.2 to 49.9 mg/g for an increase in the solution temperatures from 36 to 65 °C. It is clear that the sorption of lead on palm kernel fibre is an endothermic process. However, the sorption constant, K_a ; increases from 0.503 to 0.514 dm³/mg, as temperatures vary from 36 to 65 °C. The Redlich–Peterson isotherm constants are also shown in Table 2. It can be seen that in most cases the values of g tend to unity (>0.984), this means that the isotherms are approaching the Langmuir form.

3.3. Thermodynamic parameters

The original concepts of thermodynamics assumed that in an isolated system, where energy cannot be gained or lost, the entropy change is the driving force. In environmental engineering practice, both energy and entropy factors must be considered in order to determine what processes will occur spontaneously. The Gibbs free energy change, ΔG° , is the fundamental criterion of spontaneity. Reactions occur spontaneously at a given temperature if ΔG° is a negative quantity. The free energy of the sorption reaction, considering the sorption equilibrium constant, K_a , is given by the following equation:

$$\Delta G^\circ = -RT \ln K_a \quad (12)$$

where ΔG° is standard free energy change (J); R is universal gas constant, 8.314 J/mol K; and T is absolute temperature (K).

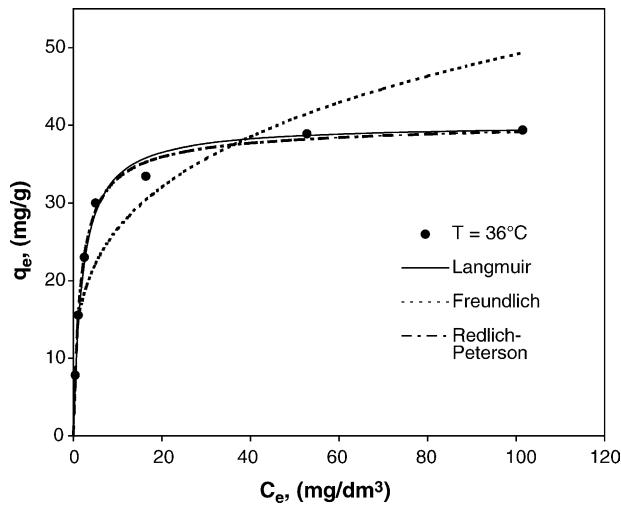


Fig. 1. Isotherms for the sorption of lead(II) using palm kernel fibre. Initial pH 5, initial concentration 120 mg/dm³ and dose 1.0 g.

Considering the relationship between free energy and the equilibrium constant, change in equilibrium constant with temperature can be obtained in the differential form as follows:

$$\frac{d \ln K_a}{dT} = \frac{\Delta H^\circ}{RT^2} \quad (13)$$

After integration, the integrated form of Eq. (13) becomes:

$$\ln K_a = -\frac{\Delta H^\circ}{RT} + Y \quad (14)$$

where *Y* is a constant. Eq. (14) can be rearranged to obtain

$$-RT \ln K_a = \Delta H^\circ - TRY \quad (15)$$

Let

$$\Delta S^\circ = RY \quad (16)$$

Substituting Eqs. (12) and (16) into Eq. (15), the Gibbs free energy change, ΔG° , can be represented as follows:

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \quad (17)$$

A plot of Gibbs free energy change, ΔG° , versus temperature, *T*; was found to be linear, Fig. 3. The values of enthalpy change, ΔH° , and the entropy change, ΔS° , were determined from the slope and intercept of the plots. The thermodynamic parameters ΔG° , are shown in Table 2. The values of ΔH° and ΔS° , for the sorption processes are calculated to be

Table 1
A comparison of Chi-square and coefficient of determination for three isotherms

<i>T</i> (°C)	Redlich–Peterson		Langmuir		Freundlich	
	χ^2	<i>r</i> ²	χ^2	<i>r</i> ²	χ^2	<i>r</i> ²
36	0.147	1.000	0.482	1.000	7.51	0.833
45	0.0347	0.999	0.0349	1.000	9.81	0.820
55	0.164	0.999	0.407	1.000	5.83	0.848
65	0.427	0.999	1.25	1.000	8.98	0.851

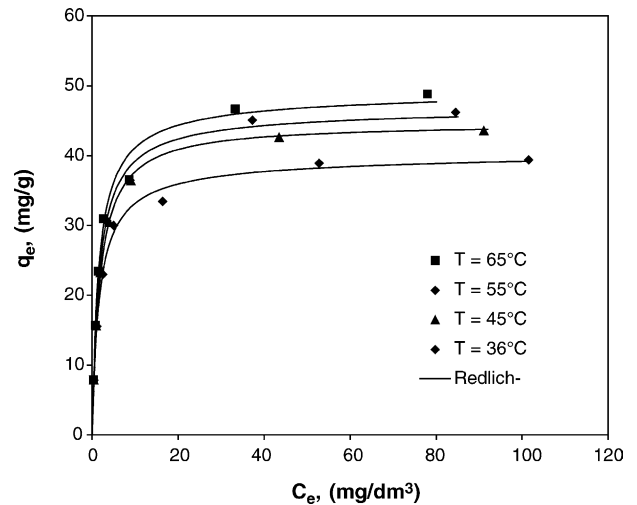


Fig. 2. Redlich–Peterson isotherms for the sorption of lead ions on palm kernel fibre.

Table 2
Isotherm constants for lead ions sorbed on palm kernel fibre

<i>T</i> (°C)	ΔG° (kJ/mol)	Langmuir		Redlich–Peterson		
		<i>q</i> _m (mg/g)	<i>K</i> _a (dm ³ /mg)	<i>A</i> (dm ³ /mg)	<i>B</i> (dm ³ /mg) ^g	<i>g</i>
36	−23.2	40.2	0.503	24.3	0.657	0.984
45	−24.1	44.4	0.542	24.2	0.543	1.000
55	−25.1	47.2	0.529	28.5	0.640	0.990
65	−26.0	49.9	0.514	31.9	0.697	0.986

0.254 kJ/mol and 0.0397 kJ/mol K, respectively. The negative values of ΔG° confirm the feasibility of the process and the spontaneous nature of sorption with a high preference of lead(II) on palm kernel fibre. The value of ΔH° is positive, indicating that the sorption reaction is endothermic. The positive value of ΔS° reflects the affinity of the palm kernel fibre for lead ions and suggests some structural changes in

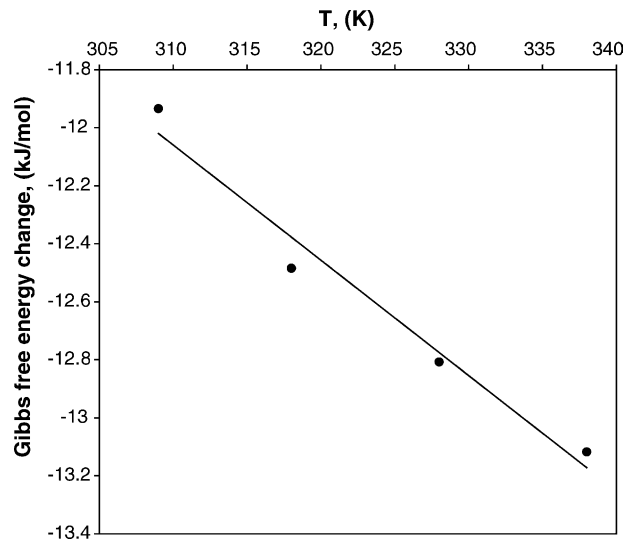


Fig. 3. Relationship between Gibbs free energy change, ΔG° , and temperature, *T*, of reaction.

lead and palm kernel fibre [14]. In addition, positive value of ΔS° shows the increasing randomness at the solid/liquid interface during the sorption of lead ions on palm kernel fibre.

3.4. Kinetic studies

Sorption in a lead/palm kernel fibre system results in the transfer of lead(II) to the surface of the palm kernel fibre, where it increases in concentration until a dynamic equilibrium is reached between palm kernel fibre and lead(II) remaining in the liquid phase. These characters of biomaterials can be involved in chemical bonding and are responsible for the cation exchange capacity. Thus, the reaction may be expressed by the following two relationships [15–17]:



and



where F^- and HF are polar sites on the palm kernel fibre surface.

The rate of pseudo-second-order reaction may be dependent on the amount of solute sorbed on the surface of palm kernel fibre and the amount sorbed at equilibrium. The rate expression for the sorption described is:

$$\frac{d(F)_t}{dt} = k[(F)_0 - (F)_t]^2 \quad (20)$$

or

$$\frac{d(HF)_t}{dt} = k[(HF)_0 - (HF)_t]^2 \quad (21)$$

where $(F)_t$ and $(HF)_t$ are the number of active sites occupied on the palm kernel fibre at time t and $(F)_0$ and $(HF)_0$ the number of equilibrium sites available on the palm kernel fibre. The kinetic rate equations can be rewritten as follows:

$$\frac{dq_t}{dt} = k(q_e - q_t)^2 \quad (22)$$

where k is the rate constant of sorption (g/mg min), q_e the amount of lead ions sorbed at equilibrium (mg/g) and q_t the amount of lead ions sorbed on the surface of the palm kernel fibre at any time t (mg/g).

Separating the variables in the equation above gives:

$$\frac{dq_t}{(q_e - q_t)^2} = k dt \quad (23)$$

integrating this for the boundary conditions $t = 0$ to t and $q_t = 0$ to q_t , gives:

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + kt \quad (24)$$

which is the integrated rate law for a pseudo-second-order reaction and Eq. (24) can be rearranged to obtain:

$$q_t = \frac{t}{(1/kq_e^2) + (t/q_e)} \quad (25)$$

which has a linear form:

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e} t \quad (26)$$

or

$$\frac{q_t}{t} = \frac{h}{1 + kq_e t} \quad (27)$$

where h can be regarded as the initial sorption rate as q_t/t when $t \rightarrow 0$, hence:

$$h = kq_e^2 \quad (28)$$

Thus, a plot of t/q_t against t of Eq. (26) should give a linear relationship with the slope of $1/q_e$ and intercept of $1/(kq_e^2)$.

A series of contact time experiments has been carried out with a constant initial lead concentration of 120 mg/dm³, palm kernel fibre dose 2.5 g/dm³ with various temperatures of reaction sorption. Fig. 4 shows the effect of agitation time on the removal of lead ions by palm kernel fibre. The results show that an increase in the reaction temperature produces an increasing in the sorption capacity of lead ions. Obviously, the endothermic nature of the reaction in the present process can be explained by this behavior of the system. The sorption of lead by palm kernel fibre may involve not only physical but also chemical adsorption.

The agreement between the sets of data reflects the extremely high coefficients of determination obtained and are shown in Table 3. The results also show the sorption rate constant, k , initial sorption rate, h , and equilibrium sorption

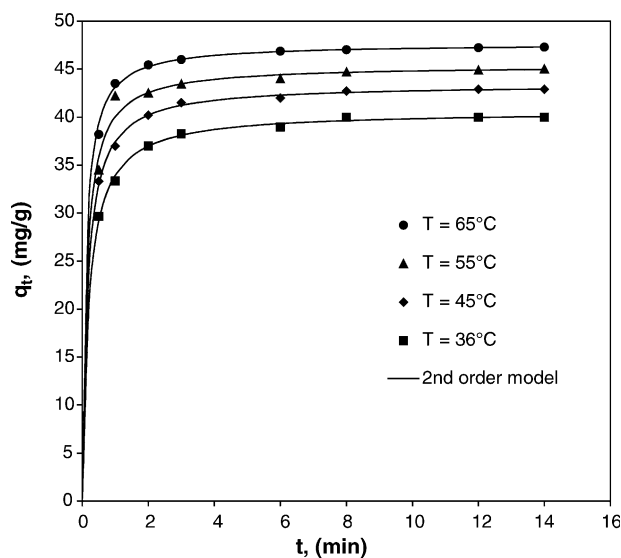


Fig. 4. Sorption kinetics for lead sorption on palm kernel fibre at different temperatures. Initial pH 5, dose = 1.0 g and initial concentration = 120 mg/dm³.

Table 3
Effect of temperature of sorption of lead ions

T ($^{\circ}\text{C}$)	q_e (mg/g)	k (g/mg min)	h (mg/g min)	r^2
36	40.6	0.125	206	1.000
45	43.4	0.144	271	1.000
55	45.4	0.164	339	1.000
65	47.7	0.198	450	1.000

capacity, q_e , as a function of solution temperature. Thus, on increasing the temperature from 36 to 65 $^{\circ}\text{C}$, the sorption capacity at equilibrium, q_e , increased from 40.6 to 47.7 mg/g. The initial sorption rate increases from 206 to 450 mg/g min, for a temperature variation from 36 to 65 $^{\circ}\text{C}$.

An examination of the effect of temperature on the equilibrium capacity, q_e , and initial sorption rate, h , produces best-fit correlation of the types shown by Eqs. (29) and (30) with high coefficient of determination.

$$q_e = 1.85 \times 10^{-3} T^{1.75} \quad (29)$$

and

$$h = 1.15 \times 10^{-19} T^{8.54} \quad (30)$$

The values of rate constant, k , were found to increase from 0.125 to 0.198 g/mg min, for an increase in the solution temperature from 309 to 338 K. There is a linear relationship between the pseudo-rate constant and temperature with coefficient of determination of 0.992 (Fig. 5). The sorption rate constant is usually expressed as a function of solution temperature by the following relationship:

$$k = k_0 \exp\left(\frac{-E}{RT}\right) \quad (31)$$

where k is the rate constant of sorption (g/mg min), k_0 the temperature independent factor (g/mg min), E the activation energy of sorption (kJ/mol), R the gas constant (8.314 J/mol K) and T the solution temperature (K).

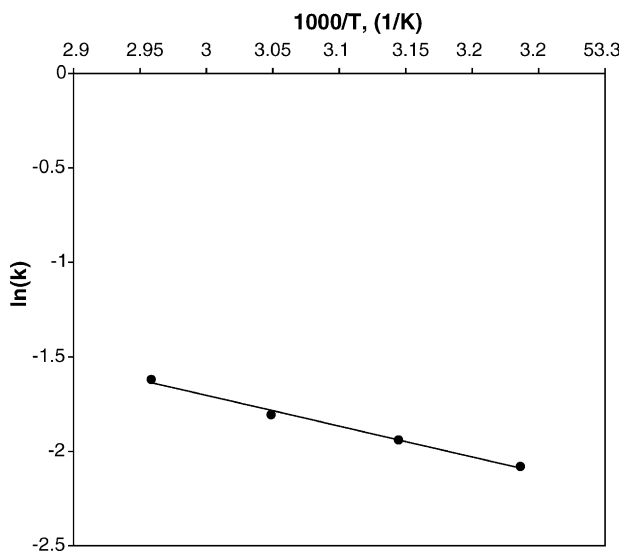


Fig. 5. Relationship between pseudo-second-order rate constant and temperature. Initial pH 5, dose = 1.0 g and initial concentration = 120 mg/dm³.

Therefore, the relationship between k and T can be represented in an Arrhenius form as:

$$k = 24.1 \exp\left(\frac{-13.5}{8.314T}\right) \quad (32)$$

From this equation, the rate constant of sorption, k_0 , is 24.1 g/mg min and activation energy of sorption, E , is 13.5 kJ/mol. This value is quite low, particularly for a reaction type process and it is more consistent of a physical sorption process. Since sorption is an endothermic process, it would be expected that an increased solution temperature would result in increased sorption capacity. The sorption of lead on cellulose-based sorbent, sphagnum moss peat [12] and mineral (pyrophyllite) of Madhya Pradesh (India) [18] have also been reported to be endothermic. Weber [19] has stated that, since diffusion is an endothermic process, the rate of sorption will increase with increased solution temperature when intraparticle transport (pore diffusion) is the rate-limiting step. Furthermore, a functional relationship common to most treatments of intraparticle diffusion is that uptake varies almost proportionately with the half-power of time, $t^{0.5}$, rather than t ; nearly linear variation of the quantity sorbed with $t^{0.5}$ is predicted for a large initial fraction of reactions controlled by rates of intraparticle diffusion [20]. Fig. 6 shows plots of the amount of lead per unit weight of fibre against the square root of t . The poor linearization of the data is observed for the initial phase of the reaction in accordance with expected behavior if intraparticle diffusion is not the rate-limiting step for the whole reaction. Straight lines could be obtained in very beginning period. A similar sorption phenomena have also been reported [21,22]. However, it did not pass close the origin that indicates intraparticle diffusion might not be the rate-limiting step.

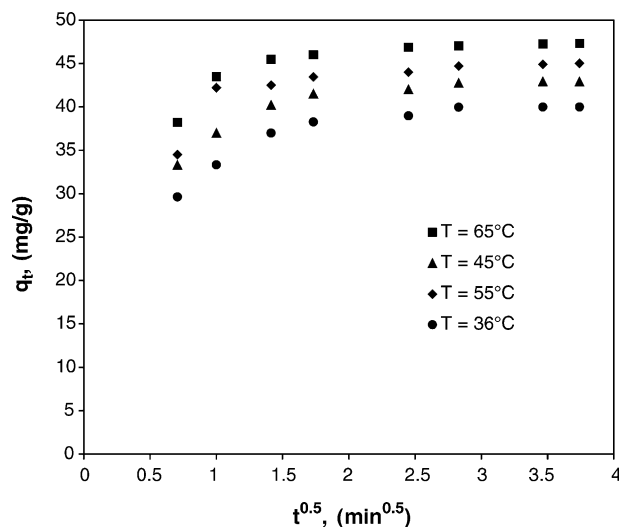


Fig. 6. Intraparticle diffusion kinetics of lead on palm kernel fibre at various temperatures.

4. Conclusion

Palm kernel fibre is able to sorb lead ions from aqueous solutions. Both linear coefficient of determination and the non-linear Chi-square test show Langmuir and Redlich–Peterson isotherms were significantly better than the coefficients for the Freundlich isotherm. The results from this study are extremely well described by the theoretical Langmuir isotherm and the three-parameter Redlich–Peterson isotherm. The equilibrium sorption of lead ions was determined from the Langmuir equation and found to be 49.9 mg/g at 65 °C. The sorption of lead ions on palm kernel fibre is of a spontaneous and endothermic nature. It was noted that an increase in the temperature resulted in a higher lead loading per unit weight of the palm kernel fibre. The kinetics of lead ion sorption on palm kernel fibre was based on the assumption of the pseudo-second-order mechanism.

Appendix A

A	Redlich–Peterson constant (dm^3/mg)
B	Redlich–Peterson constant (dm^3/mg^g)
C_0	initial metal ion concentration (mg/dm^3)
C_e	residual metal ion concentration at equilibrium (mg/dm^3)
E	activation energy (kJ/mol)
g	Redlich–Peterson constant
ΔG°	standard Gibbs free energy change (kJ/mol)
h	initial sorption rate ($\text{mg}/\text{g min}$)
ΔH°	standard enthalpy change (kJ/mol)
k	pseudo-second-order rate constant ($\text{g}/\text{mg min}$)
K_a	Langmuir sorption equilibrium constant (dm^3/mg)
q_e	metal ion sorbed per unit weight of fibre at equilibrium
q_m	Langmuir sorption constant (mg/g)
q_t	metal ion sorbed per unit weight of fibre at any time
r^2	coefficient of determination
R	gas constant (J/mol K)
ΔS°	standard entropy change (kJ/mol K)
t	time for sorption (min)
χ^2	Chi-square statistical test

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