

Adsorption characteristic studies of phosphorus onto laterite

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ABSTRACT

Soil has been widely used in many wastewater treatment systems, and proved to be an effective substrate for phosphorus removal and retention. It is significant to study its adsorption characteristics by using appropriate theoretical models. In this study, laterite (or red soil) was selected as an example to investigate the adsorption characteristic of phosphorus onto soil with the Langmuir, Freundlich, and Redlich–Peterson isotherms by both the linear and non-linear regression methods. The adsorption experiment was conducted at the temperatures of 283, 288, 298, and 308 K, respectively, to choose the appropriate method and obtain the credible adsorption parameters for soil adsorption equilibrium studies. The results showed that the non-linear regression method would be a better way to compare the better fit of isotherms for the adsorption of phosphorus onto laterite. Both the two-parameter Freundlich and the three-parameter Redlich–Peterson isotherms had higher coefficients of determination for the adsorption of phosphorus onto laterite at various temperatures. In addition, a relationship between Freundlich isotherm parameters and Redlich–Peterson isotherm parameters was presented.

Keywords: Adsorption; Isotherm; Non-linear regression; Soil; Temperature

1. Introduction

Constructed wetlands and soil infiltration systems have been widely used to treat many types of wastewaters including agricultural runoff and wastewater, urban runoff, municipal and industrial wastewaters [1,2]. Constructed wetlands and soil infiltration systems perform well in reducing chemical oxygen demand, biochemical oxygen demand and suspended solids, but its corresponding removal efficiencies for phosphorus and nitrogen are often low [1,3]. However, phosphorus and nitro-

gen are considered to be the significant nutrients causing water pollution. Unlike nitrogen removal, which can be enhanced greatly by microbial and biological processes, the removal of phosphorus in many constructed wetlands is mainly based on physical and chemical processes such as adsorption and biosorption [1,3]. Phosphorus removal and retention mechanisms during wastewater treatment in constructed wetlands and soil infiltration systems include adsorption, desorption, precipitation, leaching, uptake, fragmentation, mineralization, sedimentation (peat accretion) and burial [3].

Soil has been proved to be an effective media filled in constructed wetlands and soil infiltration systems for

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phosphorus removal and retention [2,4]. Adsorption equilibrium studies are always conducted to help researchers investigate the adsorption characteristic and capacity of pollutants onto soils. Kumar and Philip [5] studied the adsorption characteristics of endosulfan in four Indian soils, clayey soil, red soil, sandy soil, and composted soil, with the Langmuir and Freundlich isotherms by using the linear regression method. Dunne et al. [4] learnt the capacities of phosphorus adsorption by constructed wetland soils in Southeast Ireland with the Langmuir isotherm by using the linear regression method. The previous adsorption equilibrium studies on soil are usually based on the linear method; the adjustable parameters in the isotherms mentioned above are normally estimated by linear regression. The isotherms can be linearized in several different ways, and simple linear regression will result in different parameter estimates depending on which of the linearized forms is fitted, because each of these linear transformations changes the original error distribution (for better or worse) [6,7]. Using the linear regression method for comparing the best-fitting isotherms is not appropriate, the transformation is not really necessary [6]. It is significant to study the adsorption characteristics of phosphorus onto soil by using appropriate theoretical adsorption models to avoid such errors.

In this study, laterite, which is an effective substrate in many wastewater treatment systems, was selected as an example to investigate the adsorption characteristic of phosphorus onto soil. The Langmuir, Freundlich, and Redlich-Peterson isotherms were all applied by using both the linear and non-linear regression methods. The aims were to choose the appropriate method and then obtain credible adsorption parameters for soil adsorption equilibrium studies. This study could potentially promote the study of the phosphorus removal and retention mechanism for the constructed wetlands and soil infiltration systems.

2. Materials and methods

Typical laterite samples were collected from the topsoil in local Wuhan city, Hubei Province, People's Republic China. The soil samples were prescreened to remove larger gravel and wood chips, and stored in a desiccator with silica gel and dried in an oven at 170°C for 2 h before experiments. Soil samples were analyzed for particle size distribution by using laser diffraction particle size analyzer, Malvern ZetaSizer 3000, and the Surface Area and Pore Size Analyzer (Gemini V, Micromeritics) was used for estimating surface area. The X-Ray fluorescence spectrometer, Bruker AXS S4 Pioneer was used for the composition analysis. The soil samples were also analyzed as random powders on an X-ray diffractometer (XRD -6000, Shimadzu) fitted with a graphite monochromator using Cu K α radiation at 8°/min scan speed. Phase

identification was performed by comparison with the information of the JCPDS database.

A synthetic phosphorus solution comprising distilled water and potassium dihydrogen phosphate was used in this study. A stock solution of 100 mg/dm³ was prepared by dissolving a weighed amount of phosphorus in 1000 ml distilled water. The experimental solution was prepared by diluting the stock solution with distilled water when necessary.

A phosphorus solution of 50 ml with a concentration ranging from 5 to 30 mg/dm³ was placed into several 150 ml conical flasks. A weighed amount (0.3 g) of the soil samples was added to the solution. The initial pH values of the batch adsorption system ranged from 5.27 to 5.57. The conical flasks were then shaken at a constant speed of 170 rpm in a shaking water bath at temperatures of 283, 288, 298, and 308 K, respectively. After shaking the flasks for 12 h, the soil samples were separated by centrifugation. The solution was analyzed for the remaining phosphorus concentration with ammonium molybdate spectrophotometric method by a spectrophotometer. The amount of phosphorus adsorbed onto laterite was calculated by using the following expression:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where q_e is the equilibrium adsorption capacity of phosphorus adsorbed on unit mass of the laterite (mg/g); C_0 and C_e are the initial phosphorus concentration (mg/dm³) and phosphorus concentration (mg/dm³) at equilibrium respectively; V is the volume of the phosphorus solution (dm³); and m is the weight of laterite (g).

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 adsorption isotherm, three equilibrium isotherms were analyzed: the Langmuir, the Freundlich, and the Redlich-Peterson isotherms.

The Langmuir adsorption isotherm is perhaps the best known of all isotherms describing adsorption [8]. The theoretical Langmuir isotherm is often used to describe adsorption of a solute from a liquid solution as Eq. (2) [9]:

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

where q_e is the equilibrium adsorption capacity (mg/g); C_e is the equilibrium liquid phase concentration (mg/dm³); q_m is the maximum adsorption capacity (mg/g); K_a is adsorption equilibrium constant (dm³/mg).

The Freundlich isotherm is the earliest known relationship describing the adsorption isotherm [10]. This fairly satisfactory empirical isotherm can be used in adsorption from dilute solutions. The ordinary adsorption isotherm is expressed by the following equation:

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

where C_e is the equilibrium concentration in the solution (mg/dm³); q_e is the equilibrium adsorption capacity (mg/g); K_F and $1/n$ are empirical constants. K_F is the adsorption value, the amount adsorbed at unit concentration, that is, at 1 mg/dm³. It is characteristic for the adsorbent and the adsorbate adsorbed.

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

$$q_e = \frac{AC_e}{1 + BC_e^g} \quad (4)$$

where q_e is the equilibrium adsorption capacity (mg/g); and C_e is the equilibrium liquid phase concentration (mg/dm³). It has three isotherm constants, namely, A , B , and g ($0 < g < 1$).

Linear and non-linear regression methods were both used to obtain the isotherm parameters in this study [7]. Linear least-squares method and a trial-and-error non-linear regression method, which is applicable to computer operation, were used to compare the best-fitting of three isotherms using an optimization routine to maximize the coefficient of determination r^2 , between the experimental data and isotherms in the solver add-in with Microsoft's spreadsheet, Microsoft Excel [12,13]. The coefficient of determination r^2 is defined as.

$$r^2 = \frac{\sum (q_m - \bar{q}_e)^2}{\sum (q_m - \bar{q}_e)^2 + \sum (q_m - q_e)^2} \quad (5)$$

where q_m is the equilibrium capacity obtained from the isotherm model (mg/g); q_e is the equilibrium capacity obtained from experiment (mg/g); and \bar{q}_e is the average of q_e (mg/g).

3. Results and discussion

3.1. Properties of laterite

The particle size distribution of laterite was 475–590 nm (32.7%), 590–750 nm (67.3%), and the average size was 628 nm. The BET surface area of laterite is 41.90 m²/g. The result of XRF analysis is shown in Table 1, giving the main chemical composition of samples. Wide-angle X-ray diffractograms of laterite were recorded in the 10°–80° 2θ range (Fig. 1). Samples also exhibited only the small diffraction peaks characteristics of aluminum silicate, iron silicate and potassium iron silicate.

3.2. Linear regression method

The popular method used to determine the best-fitting isotherm is linear regression, and the method of least squares has been used for finding the parameters of the

Table 1
Main chemical compositions of laterite

Composition	Percent (%)
SiO ₂	53.2
Al ₂ O ₃	24.4
Fe ₂ O ₃	14.2
CO ₂	3.17
K ₂ O	1.73
TiO ₂	0.896
CaO	0.740
MgO	0.729
SO ₃	0.309
Cl	0.223
Na ₂ O	0.132
P ₂ O ₅	0.127
ZrO ₂	0.0464
Cr ₂ O ₃	0.0297
MnO	0.0210
ZnO	0.0142
CuO	0.0125
Rb ₂ O	0.0117
SrO	0.0111

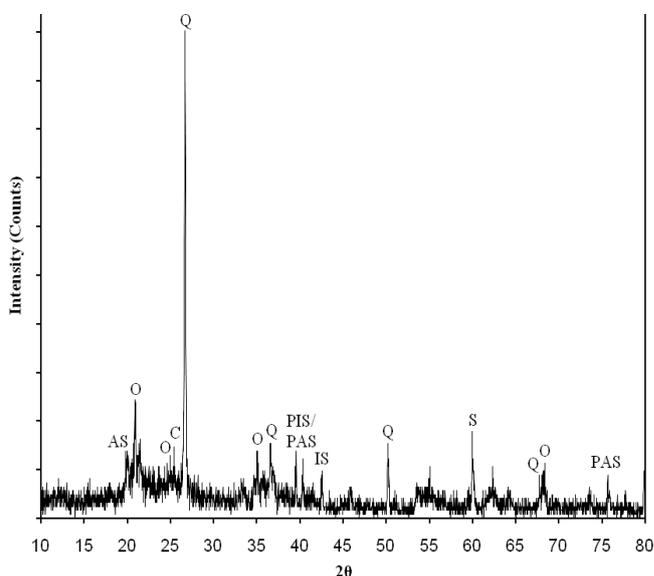


Fig. 1. XRD diffractograms of laterite. C = chlorite, O = orthoclase, Q = quartz, S = silimanite, AS = aluminum silicate, IS = iron silicate, PAS = potassium aluminum silicate, PIS = potassium iron silicate.

isotherms [13]. However, the Langmuir isotherm could be linearized as four different types, while the Freundlich isotherm had two different types usually (Table 2), and simple linear regression would result in different parameter estimates [6,13,14]. The more-popular linear

Table 2
Isotherms and their linear forms

Isotherm		Linear form	
Langmuir	$q_e = \frac{q_m K_a C_e}{1 + K_a C_e}$	Langmuir-1	$\frac{C_e}{q_e} = \frac{1}{q_m} C_e + \frac{1}{K_a q_m}$
		Langmuir-2	$\frac{1}{q_e} = \left(\frac{1}{K_a q_m} \right) \frac{1}{C_e} + \frac{1}{q_m}$
		Langmuir-3	$q_e = q_m - \left(\frac{1}{K_a} \right) \frac{q_e}{C_e}$
		Langmuir-4	$\frac{q_e}{C_e} = K_a q_m - K_a q_e$
Freundlich	$q_e = K_F C_e^{1/n}$	Freundlich-1	$\log(q_e) = \log(K_F) + \frac{1}{n} \log(C_e)$
		Freundlich-2	$\ln(q_e) = \ln(K_F) + \frac{1}{n} \ln(C_e)$
Redlich-Peterson	$q_e = \frac{AC_e}{1 + BC_e^g}$		$\ln\left(A \frac{C_e}{q_e} - 1\right) = g \ln(C_e) + \ln(B)$

forms used are Langmuir-1 and Freundlich-1. In order to assess different isotherms and their performance to correlate with experimental results, the theoretical plots from each isotherm have been shown with the experimental data for adsorption of phosphorus onto laterite at various temperatures. The graph is plotted in the form of phosphorus adsorbed per unit mass of laterite, q_e , against the concentration of phosphorus remaining in solution, C_e . Fig. 2 shows the four linear Langmuir equations with the experimental data for the adsorption of phosphorus onto laterite at a temperature of 298 K. Values of the Langmuir constants, the saturated monolayer adsorption capacity, q_m , the adsorption equilibrium constant, K_a , and the coefficient of determinations, r^2 , are presented in Table 3 for the adsorption of phosphorus onto laterite at 283, 288, 298, and 308 K, respectively. These values of the coefficient of determinations obtained from Langmuir-1 were large than 0.990 at 283, 288, 298, and 308 K. The result indicated that there was strong positive evidence that the adsorption of phosphorus onto laterite followed the Langmuir isotherm. According to Langmuir-1 isotherm, the equilibrium adsorption capacities of phosphorus onto laterite were 0.930, 0.984, 1.066, and 1.136 mg/g at 283, 288, 298, and 308 K, respectively. Dunne et al. [4] studied the adsorption capacities of phosphorus onto constructed wetland site soils in Southeast Ireland by using the Langmuir isotherm, the maximum adsorption capacities of phosphorus onto constructed wetland soils were 0.538–0.674 mg/g and 1.08–1.71 mg/g at Johnstown

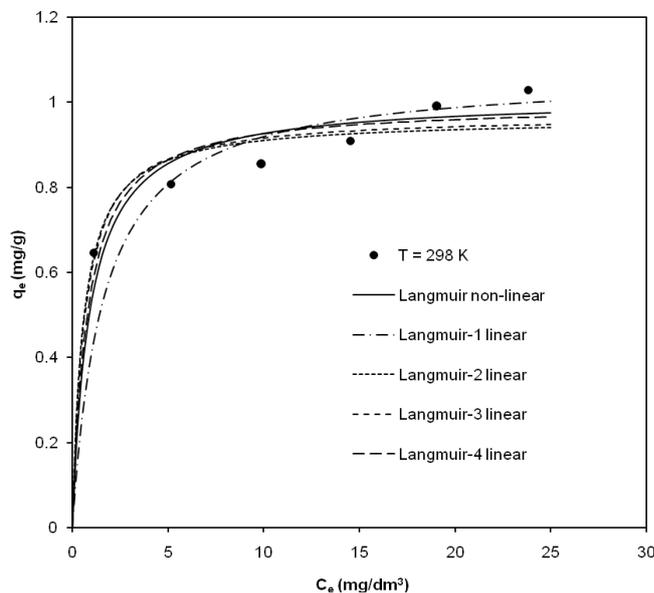


Fig. 2. Langmuir isotherms obtained using the linear and non-linear regression method for the adsorption of phosphorus onto laterite at the temperature of 298 K.

Castle and Dunhill, respectively. It can also be seen from Table 3 that the Langmuir constants obtained from various linear forms were significantly different, it indicated that transformations of the non-linear Langmuir isotherm

Table 3
Langmuir isotherm parameters obtained by using the linear regression method

Linear form	T (K)	283	288	298	308
Langmuir-1	q_m (mg/g)	0.930	0.984	1.07	1.14
	K_a (dm ³ /mg)	0.695	0.653	0.624	0.893
	r^2	0.998	0.997	0.992	0.996
Langmuir-2	q_m (mg/g)	0.872	0.922	0.962	1.05
	K_a (dm ³ /mg)	1.31	1.18	1.76	2.53
	r^2	0.935	0.955	0.885	0.877
Langmuir-3	q_m (mg/g)	0.877	0.907	0.970	1.05
	K_a (dm ³ /mg)	1.26	1.23	1.68	2.44
	r^2	0.889	0.943	0.796	0.808
Langmuir-4	q_m (mg/g)	0.889	0.939	0.995	1.08
	K_a (dm ³ /mg)	1.12	1.04	1.34	1.97
	r^2	0.889	0.909	0.796	0.808

equation to the linear forms implicitly altered the error structure and might also violate the error variance and normality assumptions of the standard least squares method [7,15]. The linear analysis using different linear forms of the Langmuir equation would significantly affect the calculation of the Langmuir parameters.

Two Freundlich linear forms (Table 2) were also analyzed, using the same set of experimental data, by plotting $\log(q_e)$ vs. $\log(C_e)$ and $\ln(q_e)$ vs. $\ln(C_e)$, respectively (Fig. 3). The Freundlich isotherm constants, K_F , $1/n$, and the coefficients of determination, r^2 , are shown in Table 4. The parameters obtained by using Freundlich-1 and Freundlich-2 were equal, that was due to the same error structures of the linear forms of the Freundlich equation. If just the linear form of Langmuir-1 was used for comparison, Langmuir-1 should be more suitable for the experimental data than the Freundlich isotherms because of the higher value of its coefficient of determinations (Tables 3 and 4). In contrast, if using the linear form of the other Langmuir linear forms, the Freundlich isotherms were more suitable for the experimental data than the Langmuir isotherms. This discrepancy was caused by the different error structures between linear forms of Langmuir and Freundlich equations. Such transformations of non-linear isotherms to linear forms implicitly altered their error structure [7,15]. It seems that using the linear regression method for comparing the best-fitting isotherms was not appropriate for the adsorption study of phosphorus onto laterite.

Fig. 4 shows the linear Redlich-Peterson isotherm with the experimental data for the adsorption of phosphorus onto laterite at various temperatures. The Redlich-Peterson isotherm constants, A , B , and g , as well as the coefficient of determination, r^2 , for the phosphorus adsorption systems using laterite were obtained using the linear form (Table 5). In all cases, the Redlich-Peterson isotherm exhibited the highest coefficient of determinations ($r^2 >$

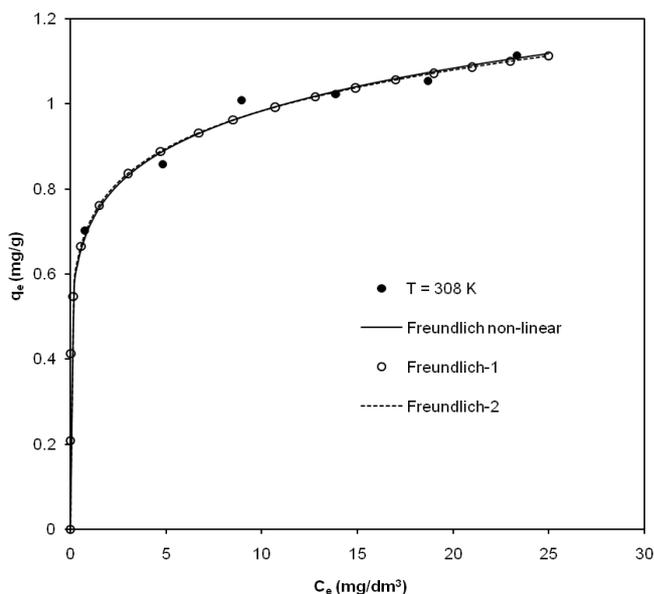


Fig. 3. Freundlich-1 and Freundlich-2 isotherms obtained by using the linear regression method for the adsorption of phosphorus onto laterite at the temperatures of 308 K.

0.999), which produced a considerably better fit compared with the Langmuir and Freundlich isotherms. It can be seen that the values of g were not close to unity, which meant the isotherms were approaching the Freundlich isotherm but not the Langmuir isotherm.

3.3. Non-linear regression method

Fig. 5 shows three widely used isotherms by applying the non-linear regression method with the experimental data at various temperatures. The related isotherm parameters obtained using the non-linear regression method is shown in Table 6. In the case of the Langmuir isotherm,

Table 4
Freundlich isotherm parameters obtained by using the linear regression method

Linear form	T (K)	283	288	298	308
Freundlich-1	$1/n$	0.152	0.164	0.147	0.135
	K_F	0.551	0.561	0.630	0.721
	r^2	0.996	0.987	0.981	0.976
Freundlich-2	$1/n$	0.152	0.164	0.147	0.135
	K_F	0.551	0.561	0.630	0.721
	r^2	0.996	0.987	0.981	0.976

Table 5
Redlich-Peterson isotherm parameters obtained by using the linear regression method

T (K)	283	288	298	308
g	0.858	0.874	0.856	0.865
B	22.8	5.80	9.35×10^7	1.71×10^9
A	13.0	3.67	5.91×10^7	1.23×10^9
r^2	1.00	1.00	1.00	0.999

the results from the four linear Langmuir equations were the same. When using the non-linear regression method, there was no problem with transformations of non-linear Langmuir equations to linear forms, because they had the same error structures [13]. The Langmuir constants obtained from the non-linear and linear regression

methods differed even when compared with the results of Langmuir-1, which had the highest coefficient of determination for any Langmuir isotherm (Tables 3 and 6).

Redlich–Peterson seemed to be the best-fitting models for the experiment results because of its coefficient of determinations were higher than those of Langmuir and Freundlich (Table 6). Consequently, the Redlich–Peterson isotherm was found to be the most-suitable model for the adsorption system of phosphorus onto laterite. Unlike the linear analysis, a different isotherm would significantly affect the coefficient of determination value and impact the final determination of parameters, and using the non-linear regression method would avoid such errors.

As shown in Eq. (4), the Redlich–Peterson isotherm has three isotherm constants, namely, A , B , and g ($0 < g < 1$), which characterize the isotherm. Its limiting behavior is summarized.

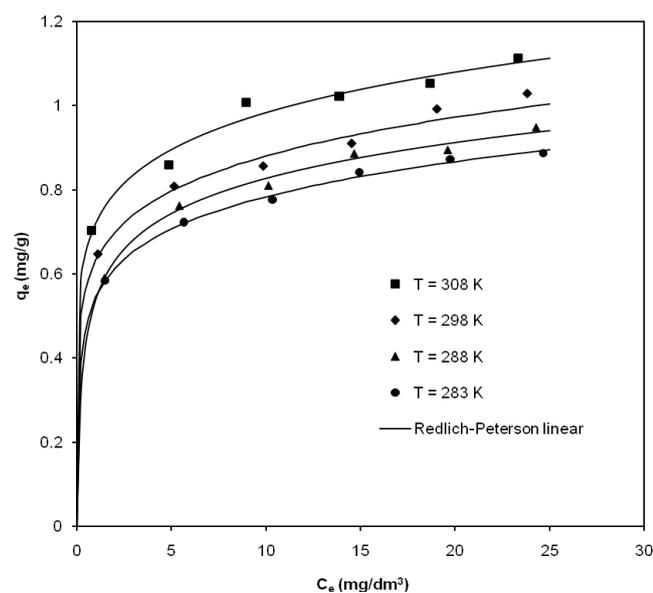


Fig. 4. Redlich-Peterson isotherms obtained by using the linear regression method for the adsorption of phosphorus onto laterite at various temperatures.

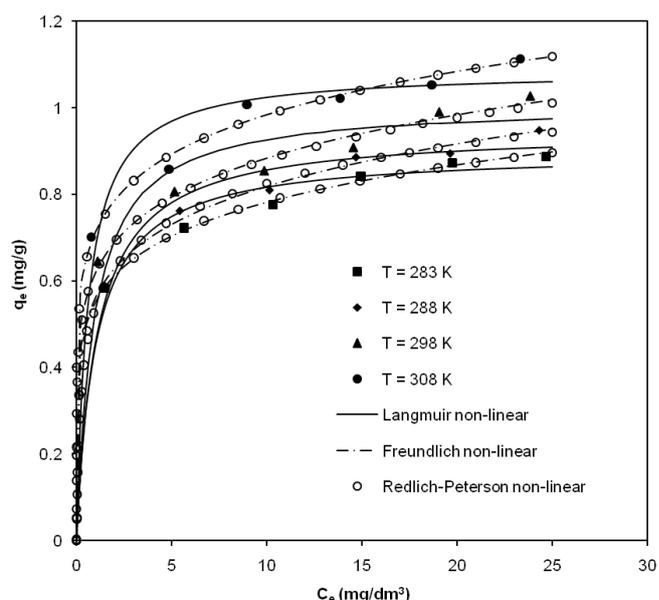


Fig. 5. Isotherms obtained by using the non-linear regression method for the adsorption of phosphorus onto laterite at various temperatures.

Table 6
Isotherms parameters obtained by using the non-linear regression method

Non-linear form	T (K)	283	288	298	308
Langmuir	q_m (mg/g)	0.898	0.948	1.01	1.09
	K_a (dm ³ /mg)	1.01	0.935	1.11	1.66
	r^2	0.922	0.935	0.859	0.866
Freundlich	$1/n$	0.152	0.164	0.155	0.140
	K_F	0.551	0.562	0.619	0.713
	r^2	0.995	0.986	0.977	0.974
Redlich–Peterson	g	0.858	0.867	0.849	0.860
	B	21.6	6.69	2.11×10^4	7.15×10^7
	A	12.3	4.15	1.31×10^4	5.09×10^7
	r^2	0.995	0.989	0.978	0.974

Where $g = 1$

$$q_e = \frac{AC_e}{1 + BC_e} \quad (6)$$

i.e. the Langmuir form results.

Where constants A and B are much greater than unity [7].

$$q_e = \frac{AC_e}{BC_e^g} \quad (7)$$

i.e. the Freundlich form results.

Where $g = 0$

$$q_e = \frac{AC_e}{1 + B} \quad (8)$$

i.e. the Henry's Law form results.

Fig. 5 shows the plots of experimental data also fitted well with the Freundlich adsorption isotherm of phosphorus onto laterite. Comparing Eq. (3) with Eq. (7), the values of parameter " K_F " correlated well with " A/B ", while " $1/n$ " correlated well with " $1 - g$ " (Tables 6 and 7). The Freundlich isotherm was also considered to be a suitable model with a high coefficient of determination. At the temperature of 308 K (Fig. 5), the Redlich–Peterson and Freundlich isotherms overlapped ($r^2 = 0.974$). It indicated that the isotherms were approaching the Freundlich isotherm form but not the Langmuir isotherm. Freundlich isotherm is a special case of Redlich–Peterson isotherm. Moreover, the values of g were less than 0.867 and not close to unity. Unlike the linear analysis, different isotherm forms would affect r^2 significantly, and impact the final determination of parameters while non-linear regression methods would prevent such errors.

3.4. Effect of temperature on equilibrium isotherm

The effect of temperature on the adsorption isotherm is shown in Fig. 5. The results showed that the capacity of

Table 7
Values of " $1 - g$ " and " A/B " at various temperatures

T (K)	$1 - g$	A/B
283	0.142	0.569
288	0.133	0.620
298	0.151	0.622
308	0.140	0.713

the laterite for phosphorus adsorption increased with the increase in temperature. Endothermic processes were also found in phosphorus adsorption onto inorganic adsorbents, for example, electrocoagulated metal hydroxides sludge [16], iron oxide coated sand [17], and kaolinite [18].

4. Conclusions

The non-linear regression method was a better way to compare the best-fitting isotherms and obtain the isotherm parameters for the adsorption of phosphorus onto laterite. Both the two-parameter Freundlich and the three-parameter Redlich–Peterson isotherms had higher values for the coefficient of determination for the adsorption of phosphorus onto laterite at various temperatures. The capacity of the laterite for phosphorus adsorption increased with the increase in temperature, higher temperature is suitable for the adsorption reaction of phosphorus onto laterite. Furthermore, a relationship between Freundlich isotherm constants " K_F " and " $1/n$ " and Redlich–Peterson isotherm constants " A/B " and " $1 - g$ " were found.

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