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Cent. Eur. J. Chem. • 7(4) • 2009 • 760–768
DOI: 10.2478/s11532-009-0098-8

Central European Journal of **Chemistry**

Equilibrium isotherm analysis of the sorption of congo red by palm kernel coat

Research Article

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Received 3 November 2008; Accepted 6 April 2009

Abstract: The ability of Palm Kernel Coat (PKC), a waste agricultural product, in the abstraction of Congo Red (CR), an anionic dye, from aqueous solution was studied. The effects of sorbent dose and temperature were studied using batch sorption system. Isotherm experiments were conducted and the data obtained were fitted with different equilibrium isotherm equations namely, Langmuir, Freundlich, Dubinin-Radushkevich (D-R), Temkin, Harkins –Jura and Halsey isotherm equations. The Langmuir isotherm equation gave the best description of the sorption process and the maximum saturated monolayer sorption capacity of the PKC for CR was 79.37 mg g^{-1}. Harkins-Jura isotherm equation gave the poorest description of the sorption process. The linear form of the Langmuir equation was used to analyze the data obtained when the sorbent dosage was optimized by method of continuous variation. The results obtained showed that the equilibrium monolayer sorption capacity, q_m, of the PKC for CR decreased (79.37-17.07 mg g^{-1}) with an increase in sorbent dosage. The relationship between the dimensionless parameter, K_R, and initial concentration, C_o, showed that the sorption of CR was favored at higher initial dye concentration and PKC dosages than the lower ones. The thermodynamic parameters, such as change in the free energy, the enthalpy and the entropy, were also evaluated. The thermodynamic analysis showed that the sorption is spontaneous and exothermic.

Keywords: *Equilibrium • Isotherm • Dye • Palm kernel coat • Sorption • Monolayer*

1. Introduction

Small and medium scale industries, *e.g.*, textile, leather and dye processing industries *etc.*, are the pivots on which the economies of emerging nations revolve. Wastewaters from such industries are relatively low in volume and spasmodically discharged, but the summation could be enormous and threatens the ecosystem. In Nigeria, there has been a leap in the activities of these industries in recent time. This is occasioned by the emerging government policies on poverty alleviation via the development of small and medium scale industries. As a consequence, large volume of untreated colored wastewater is regularly discharged into the environment.

The wastewaters, generated from these industries, are discharged untreated because of the huge cost of providing wastewater treatment facilities, which is beyond the budgetary allocations of such industries. The treatment of these wastewaters, in an economic fashion, is therefore a necessity for a continuous existence of these industries, if the government legislation on environmental protection is to be enforced.

Sorption of dye from aqueous solution is a cost effective process of cleaning dye contaminated water. Scientific reports have shown that surfeit of low cost materials have been successfully applied in the removal of dye from aqua medium. These include fuller's earth for methylene blue [1] neem leaf powder for brilliant

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green [2] Rice husk for malachite green [3] sugar cane dust for basic violet 10, basic violet 1 and basic green 4 [4] palm kernel fiber for anionic dye [5].

In the present study, Palm Kernel Coat (PKC) is used as a sorbent in the remediation of Congo red contaminated water. PKC is obtained from the oil palm Tree (*Elais guineensis*). This tree is native to the west coast of the Africa and widespread in the tropics. The *Elais guineensis* and *Elaeis Oleifera* (*i.e.*, the American oil palm) are members of the family *Arecaceae* (formerly: *palmae*). Before 1971, Nigeria was the world's largest exporter of palm oil. This tree serves as a vast resource of materials of industrial importance (*e.g.*, palm wine, palm kernel oil, palm kernel cake *etc*). Virtually all parts of the oil palm tree have been utilized in one form or the other. Palm oil used in the manufacture of soap, liniment, ointments, margarine, soap, West African and Brazilian cooking *etc*., is extracted from the fruit pulp. Palm kernel oil is obtained from the seed kernels of the fruits. Palm kernel shell and palm kernel coat (*i.e.*, the fiber) are the major waste materials produced from the extraction of palm kernel oil and palm oil respectively. Over 15,600 tonnes of shells and fibre are generated annually and only about 5% of the waste is sometimes used as biofuel [6].

The equilibrium sorption isotherm is fundamentally important in the design of sorption systems. Equilibrium studies give the capacity of the sorbent and described the sorption isotherm by constants whose values express the surface properties and affinity of the sorbent [7]. Equilibrium relationship between sorbent and sorbate are described by sorption isotherm, usually the ratio between the quantity sorbed and that remaining in the solution at a fixed temperature at equilibrium. In order to assess the ability of PKC to abstract CR from aqueous system, equilibrium analysis of the sorption system is imminent. Congo red (CR) [1-naphthalene sulfonic acid, 3,3-(4,4–biphenylenebis(azo)bis(4–amino-)disodium salt] is a benzidine - based anionic disazo dye. This dye is known to metabolize to benzidine, a known human carcinogen [8]. Effluent containing CR is generated from textiles, printing and dying, paper, rubber, plastic industries, and other industries. Owing to the structural stability, CR is difficult to biodegrade. Physiochemical or chemical treatment of such wastewater is, however possible [9,10]. The aim of the present study was to investigate the sorption of CR, an anionic dye from aqueous solution, using PKC. The process variables studied included sorbent dosage and temperature.

2. Materials and method

2.1. Sorbent Preparation

The PKC was obtained from a small scale palm oil processing factory, situated in Akungba-Akoko (the University Town), Nigeria. The raw PKC was steamed in warm deionised water, to melt the residual oil on it. The PKC was separated from the warm deionised water and 0.5 M of NaOH was added to the PKC for alkaline hydrolysis (saponification) of any residual oil on it. The saponified PKC was thoroughly washed until it stopped foaming and the pH of the washings is equal to the pH of the deionised water used in washing the PKC. The PKC was oven-dried, ground and screened through a set of sieves to obtain particle sizes in the range 53-74 μm. This fraction was then stored in an air-tight plastic container pending usage.

2.2. Sorbate Preparation

The dye used in the present studies, CR (C.I. 2212), chemical formula = $C_{32}H_{22}N_6Na_2O$ λ_{max} = 500 nm) was accurately weighed and dissolved in distilled-deionised water to prepare the stock solution (500 mg L^{-1}). Different working solutions were prepared from the stock solution by serial dilution.

2.3. Sorbate quantification

The CR concentrations in the aqueous medium were quantified by the determination of the absorbance at the characteristic wavelength using a double beam UV/Visible spectrophotometer. Standard solution of the dye was taken and the absorbance was determined at different wavelengths to obtain a plot of absorbance versus wavelength. The wavelength corresponding to the maximum absorbance (λ_{max}), as determined from this plot, was noted and the wavelength was used for the preparation of the calibration curve used in the present studies.

2.4. Isotherm studies

A volume of 50 mL of CR solution with concentration ranging from 30-150 mg L^{-1} was placed in a 250 mL conical flask. Accurately weighed PKC (0.1 g) was then shaken with the CR solution at a constant agitation speed of 200 rpm in a shaker water bath. After shaking the flask for 2 h, the PKC was separated by centrifuging and the filtrate was analyzed for the residual CR concentration using UV/visible spectrophotometer at the maximum wavelength (λ_{max}) of the dye.

Table 1a. Equilibrium Isotherm Parameters of sorption of CR by PKC.

Langmuir isotherm	Freundlich isotherm	D-R isotherm
q_m (mg g^{-1}) = 79.37	K_f (L g^{-1}) = 4.014	q_m (mol g^{-1}) = 39.678
B (L mg^{-1}) = 0.029	1/n = 0.6405	K' (mol^2 kJ^{-2}) =8.3661
r^2 = 0.9838	r^2 = 0.9698	E (kJ mol^{-1})= 0.244
		r^2 = 0.8845

Table 1b. Equilibrium Isotherm Parameters of sorption of CR by PKC.

Temkin isotherm	Harkins- Jura Isotherm	Halsey isotherm
B_1 = 16.783	A = 149.25	n = 1.560
K_T (L mg^{-1}) = 3.215	B = 1. 642	K = 2.435
r^2 = 0.9497	r^2 = 0.8147	r^2 = 0.9698

2.5. Effect of PKC dose

Equilibrium uptake of dye was studied at 309 K with sorbent dosage of 0.1, 0.2, 0.4, 0.6 and 0.8 g of PKC in contact with dye solution of concentrations ranging from 30-150 mg L^{-1}. The flasks were agitated at 200 rpm for 2 h and the equilibrium concentration of the residual dye was determined spectrophotometrically.

2.6. Effect of temperature

Batch sorption studies were performed at temperatures ranging from 309 K to 333 K, at a fixed sorbent dosage (0.1 g) and initial dye concentration from 30-150 mg L^{-1}. The flask was shaken at 200 rpm for 2 h and the equilibrium concentration of the residual dye was determined spectrophotometrically.

3. Results and Discussion

3.1. PKC composition

A proximate analysis of the saponified PKC was carried out, using the recommended standard methods of analysis [11]. The results obtained from the analysis showed that the saponified PKC contained (in % dry weight): Protein 18.72%; Lipid 3.72%; Ash 1.37%; Fibre 24.9%; Carbohydrate 51.29%; Ca 0.5%; Mg 0.14%; Na 0.21%; K 0.05%; P 0.03% and N 3.00%. (The results presented herein were obtained from the mean of triplicate determinations). The bulk density of the PKC used was 497.28 kg m $^{-3}$. The results of the particle size analysis showed that the PKC used for the present study was made up of the following particle sizes (μm) : >90 (54.18%); 90-63 (42.22%); and <63 (3.60%).

3.2 Isotherm studies

In order to understand the mechanism of the phenomenon called adsorption, different isotherm models have been employed. Langmuir and Freundlich isotherm equations have been the most widely used despite the fact that these equations have limitations on their use. Thus, these two isotherm equations may not be suitable for the elucidation of the mechanisms of the sorption of CR by PKC.

In order to understand the mechanisms of CR sorption by PKC and evaluate the relationship between the CR on the PKC and in solution, the experimental data were fitted using six different isotherm equations: the Langmuir, Freundlich, Dubinin-Radush kevich (D-R); Temkin; Harkins-Jura and Halsey isotherm. The constant parameters of the isotherm equations for this adsorption process were calculated by regression using the linear form of the isotherm equations. The different isotherm equation parameters and correlation coefficient (r^2) are presented in Table 1a.

Despite the fact that the original Langmuir equation was proposed to describe the adsorption of gas molecules onto metal surfaces [12], the Langmuir adsorption isotherm has been successfully applied to many real sorption processes. The linearized isotherm is presented as:

$$1/q_c = 1/q_m + 1/bq_m C_e \qquad (1)$$

where, q_e is the amount of CR sorbed per unit of the PKC (mg g^{-1}), C_e, is the equilibrium concentration of the adsorbate (mg L^{-1}) and q_m and, b, are the Langmuir constants related to maximum sorption capacity and energy of adsorption, respectively. When $1/q_e$ was plotted against $1/C_e$, a straight line with slope of $1/bq_m$ and intercept$1/q_m$ were obtained (Table 1a). The linearity of the plot was high (r^2 =0.9838) over the whole concentration range studied.

The Freundlich adsorption isotherm can be expressed as:

$$\log q_e = \log k_f + 1/n \log C_e \qquad (2)$$

where, q_e and C_e, are as previously defined; k_f and n are

Freundlich constants related to adsorption capacity and sorption intensity respectively. When the values of log q_e were plotted against log C_e, for the adsorption data of CR by PKC, a straight line with a high linear coefficient (r^2 =0.9698) was obtained. The Freundlich constant, K_f and 1/n were calculated from the straight line. The values of 1/n, which is less than unity, show the favorable nature of adsorption of CR on PKC.

The D-R isotherm is more general than the Langmuir isotherm because it does not assume a homogeneous surface or constant adsorption potential [13]. The D-R equation can be expressed as:

$$q_e = q_m' \exp(-K' \varepsilon^2) \tag{3}$$

where, ε (polanyi potential) is equal to RT ln (1 + 1/C_e), q_e and C_e have been defined earlier q_m is the theoretical monolayer saturation capacity, K′ is the constant of the adsorption energy (mol^2 K^{-1} J^2), R is the gas constant (KJ mol^{-1} K) and T is the temperature (K). The linear form of D-R isotherm is:

$$\ln q_e = \ln q_m - K' \varepsilon^2 \tag{4}$$

K′ is related to the mean adsorption energy E (kJ mol^{-1}) as:

$$E = 1/\sqrt{2K'} \tag{5}$$

The value of ln q_e was plotted against ε^2. A straight line graph was obtained (r^2 = 0.8845) and the D-R parameters obtained from the plot are presented in Table 1. The results obtained from the D-R plot showed that the sorption energy value (E = 0.244 kJ mol^{-1}) is very low for the adsorption of CR on PKC. Mall, et al., [8], reported similar results for the sorption of CR (E = 0.696 kJ mol^{-1}) and Orange Green (E = 0.843 kJ mol^{-1}) on mesoporous fertilizer plant waste carbon. The value of the correlation coefficient (r^2) is low, when compared with the other isotherm plots (Table 1a).

Temkin and Pyzhev [14] studied the heat of adsorption and the adsorbate–adsorbate interaction. The Temkin isotherm equation is given as:

$$q_e = RT/b \ln (K_T C_e) \tag{6}$$

The linear form of Eq. 6 is presented below:

$$q_e = B_1 \ln K_T + B_1 \ln C_e. \tag{7}$$

where, B_1 = RT/ b, T is the absolute temperature in, K, R is the universal gas constant (8.314 J mol^{-1}), K_T is the equilibrium binding constant (L mg^{-1}) and Bi is related to the heat of adsorption. The Temkin constants (Table 1b) were obtained from the plot of q_e versus lnC_e. The Temkin isotherm yields a better fit to the experimental data than the D-R isotherm.

The Harkin's-Jura adsorption isotherm can be expressed [15,16] as:

$$1/q_e^2 = (B/A) - (1/A) \log C_e \tag{8}$$

The isotherm equation accounts for multilayer adsorption and can be explained with the existence of a heterogeneous pore distribution. The Harkins- Jura isotherm parameters were obtained (Table 1b) from the plots of 1/qe^2 against log C_e . The linear correlation of this isotherm equation was the poorest of all the isotherm equation studied (r^2 = 0.8147)

Halsey adsorption isotherm can be given as [17,16] as :

$$\ln q_e = [1/n \ln k] - 1/n \ln C_e \tag{9}$$

This equation is suitable for multilayer adsorption and the fitting of the experimental data to this equation

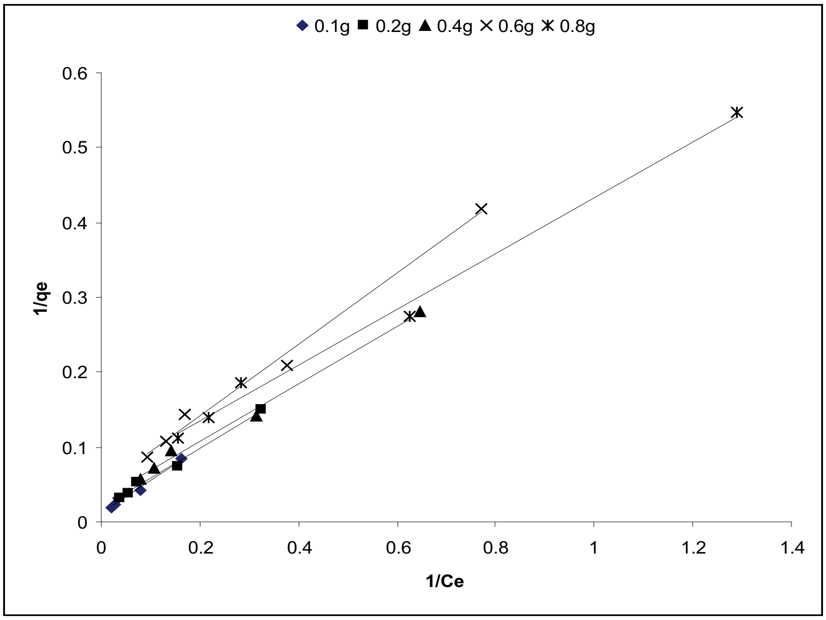

Figure 1. Langmuir isotherm plots for the sorption of CR at different PKC dosages (g).

attests to the heteroporous nature of the adsorbent [18]. The Halsey isotherm parameters were obtained from the plot of lnq_e versus ln C_e (Table 1b). The fitting of the Halsey isotherm equation was very high (r^2 = 0.9698) which is an indication of the heteroporosity (*i.e.*, macropore and micropore) of the PKC.

3.3. Effect of PKC dosage

It has been reported that Langmuir isotherm has reasonable agreement with a large number of experimental systems including those that have different interfaces between the two phases [9,19-21]. The linearity of the plot, when the data got from the isotherm experiment were analysed with the Langmuir isotherm equation, showed that this isotherm described the CR sorption by PKC better than any other isotherm equation used. Langmuir isotherm model has also been successfully used to describe the sorption of other anionic dye –sorbent system such as Acid Red 57 onto sepiolite [22]; Procion crimson H-EXL (PC) onto pyrophyllite [23]; acid yellow 36 on carbon from saw dust [24] and acid blue 113 on activated carbon [25]. Sequel to this, isotherm experiments was conducted and the data were analysed using the linear forms of the Langmuir equation, to determine the effect of PKC dosage on the CR removal capacity of the different CR /PKC systems (Fig. 1). Isotherms were determined for five different PKC dosages ranges from 0.1 – 0.8g/50mL and the results obtained are presented in Table 2.

The results presented in Table 2 show that as the PKC dose was increased from 0.1 to 0.8g/50mL, a reduction was observed in the values of the monolayer sorption capacities, q_m , from 79.37 to 17.07 mg g $^{-1}$. The reduction in the sorbent capacity, *i.e.*, the amount of dye sorbed per unit weight of sorbent with increase in PKC dose could be ascribed to two reasons: the increase in sorbent dose at constant dye concentration and volume will lead to unsaturation of sorption sites through the sorption process [26,27] and secondly may be due to particulates interactions. Such aggregation would lead to a decrease in total surface area of the sorbent and an increase in diffusional path length [26]. The value of the sorption equilibrium constant, b, increased with increase in the sorbent dose.

The corresponding linear plots of the value of q_m and b against m_s (PKC dose) were regressed to derive

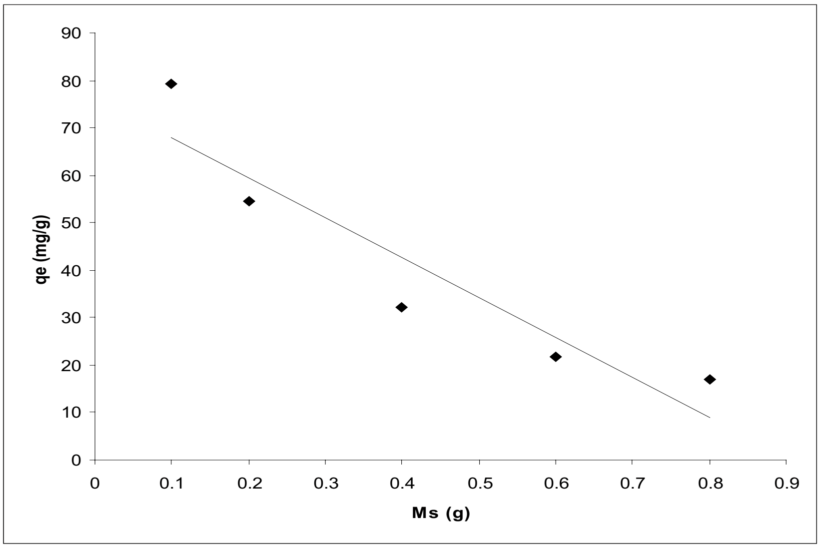

Figure 2. Linear plot of the value of q_e (mg g^{-1}) against m_s (g) for the sorption of CR by PKC.

Table 2. Langmuir isotherm constants for CR at various PKC dosages

m_s (g/50mL)	q_m (mg g^{-1})	b (L mg^{-1})	r^2
0.1	79.37	0.029	0.9838
0.2	54.64	0.046	0.9910
0.4	32.15	0.081	0.9927
0.6	21.88	0.096	0.9922
0.8	17.07	0.157	0.9951

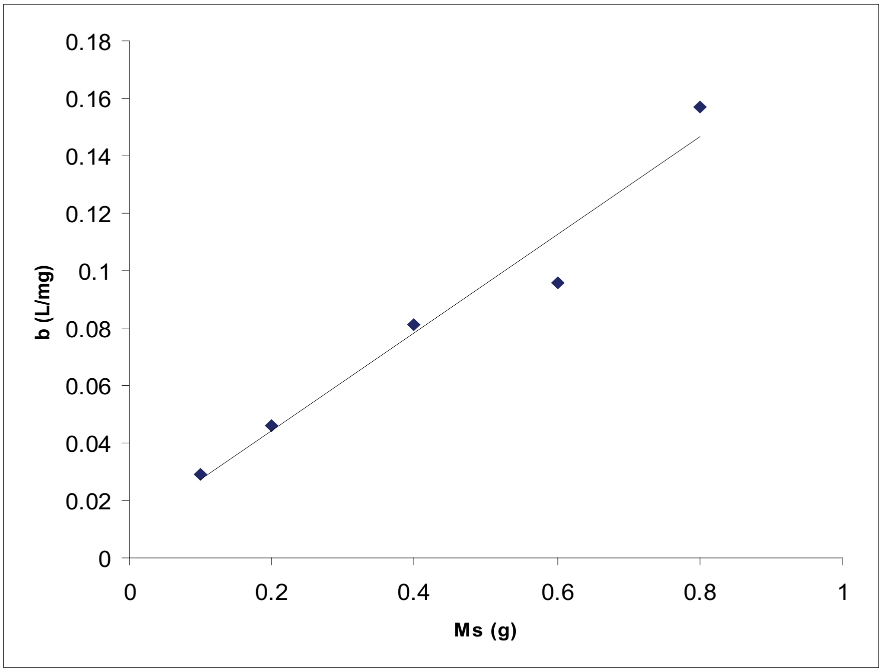

Figure 3. Linear plot of the value of b (L mg^{-1}) against m_s (g) for the sorption of CR by PKC

a mathematical relationship with high coefficients of determinations ($r^2 = 0.8707$ and 0.9604, respectively), Figs. 2 and 3. Sequel to this q_m and, b, can be expressed as a function of m_s thus:

$$q_{m} = -84.26 m_s^{76.41}$$

and

$$b = 0.1705 m_s^{0.0102}$$

According to [28] the essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameters K_R that can be defined by the following relationship:

$$K_R = \frac{1}{1 + bC_o} \tag{10}$$

where, K_R is a dimensionless separation factor, C_o, is the initial concentration (mg L^{-1}) and, b, is the Langmuir constant (L mg^{-1}). The parameter KR indicates the shape of the isotherm accordingly:

Values of K_R	Types of Isotherm
$K_R > 1$	Unfavorable
$K_R = 1$	Linear
$0 < K_R < 1$	Favorable
$K_R = 0$	Irreversible

The relationship between K_R and C_o at different PKC dosages is presented in Fig. 4. This is to show the essential features of Langmuir isotherm for the sorption of CR by PKC. The results in Fig. 4 show that sorption was favored at higher initial dye concentration and PKC dosages than the lower ones.

3.4. Thermodynamic Parameters

In environmental engineering practice, both energy and entropy factors must be considered in order to determine which process will occur spontaneously [7]. 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, b, is given by the equation presented below:

$$\Delta G^{o} = -RT \ln b \tag{11}$$

where, ΔG_o is the standard free energy change (J), R the Universal gas constant, 8.314 J mol^{-1} K, and T is the absolute temperature (K).

Considering the relationship between free energy and equilibrium constant, change in equilibrium constant with temperature can be obtained in the differential form as follows [5]:

$$\frac{d\ln K_L}{dT} = \frac{\Delta H^{o}}{RT^2} \tag{12}$$

After integration, the integrated form of Eq. 12 becomes:

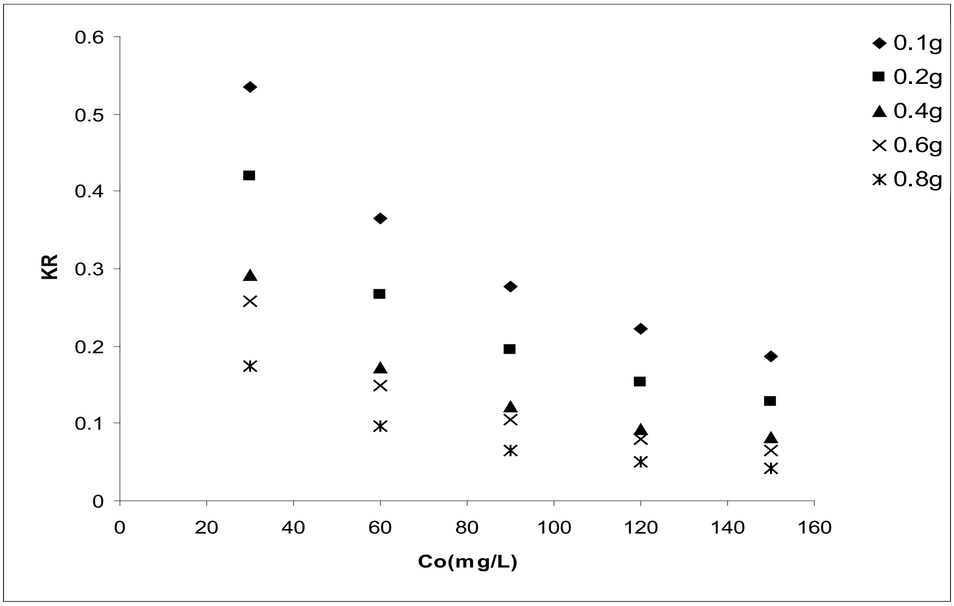

Figure 4. Relationship between the separation factor, K_R, and initial CR for the sorption of CR by PKC at varying sorbent dosages (g).

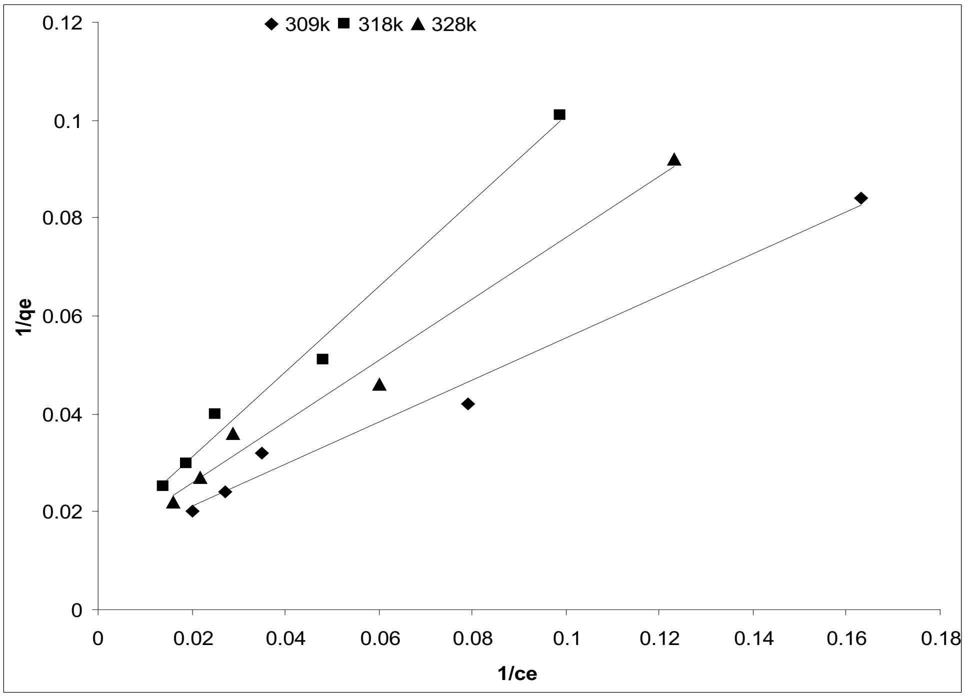

Figure 5. Langmuir isotherm plots for the sorption of CR by PKC at different temperatures (K).

Table 3. Langmuir isotherm constants for CR at various temperatures.

T(K)	q_m (mg g^{-1})	b (L mg^{-1})	ΔG^0 (J)	r^2
309	79.365	0.029	-90.96	0.9838
318	76.34	0.021	-102.14	0.9845
328	72.46	0.016	-112.77	0.9885

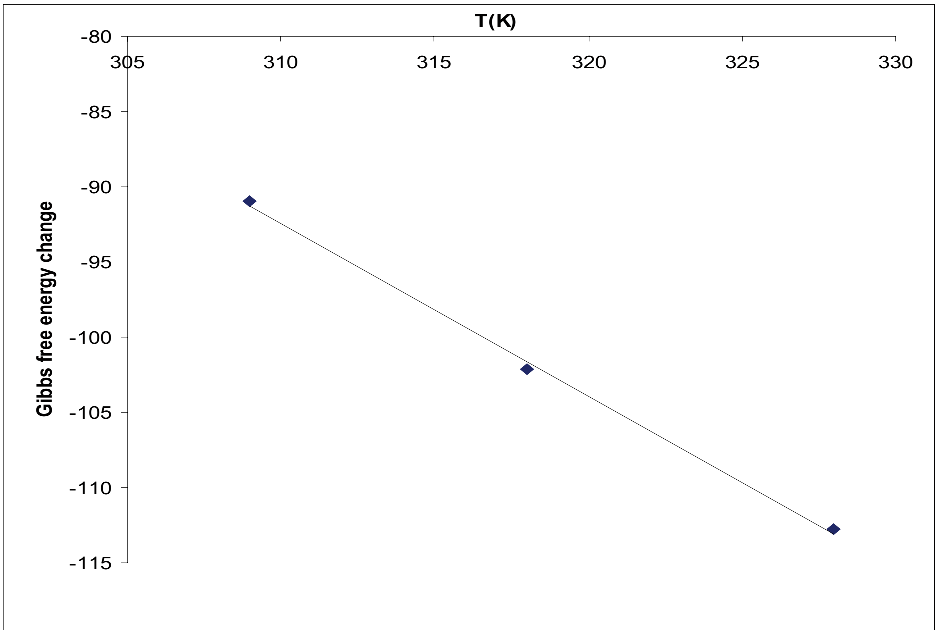

Figure 6. Relationship between Gibbs free energy change (ΔG^0) and temperature for the sorption of CR by PKC

$$\ln K_L = \frac{\Delta H^o + Y}{RT^2} \qquad (13)$$

where, Y is a constant. Eq. 13 can be rearranged to obtain

$$-RT \ln K_L = \Delta H^o \ - TRY \qquad (14)$$

Let

$$\Delta S^o = RY \qquad (15)$$

Substituting Eqs. 14 and 15, the Gibbs free energy change, ΔG°, can be represented as follows:

$$\Delta G^o = \Delta H^o - T\Delta S^o \qquad (16)$$

The values for the sorption equilibrium constant, b, obtained from the Langmuir plots (Fig. 5) decreased with increasing temperature. The values of the Gibbs free energy change ΔG° for the sorption of CR by PKC are presented in Table 3. The negative values of ΔG° confirm the feasibility of the process and the spontaneous nature of the sorption process. The values of ΔG° increased from -90.96 kJ mol^{-1} at 309 K to -112.77 kJ mol^{-1} at 328 K. The negative values of ΔG° confirmed the feasibility of the process and the spontaneous nature of sorption with a high affinity of the PKC for CR. The values of ΔH° and ΔS° calculated from the plot of Gibbs free energy change, ΔG°, versus absolute temperature (Fig. 6) are -1.1463 and 262.96, respectively. The enthalpy change, ΔH°, is negative indicating that the biosorption reaction is exothermic. The positive values of ΔS° reflects the affinity of the PKC for CR and suggest some structural changes in CR and PKC interaction [29]. In addition, positive value of ΔS° shows increasing randomness at the solid/liquid interface during the biosorption of CR onto PKC.

4. Conclusion

The ability of PKC to abstract CR from aqueous solution was studied using equilibrium isotherm analysis. Langmuir, Freundlich, Dubinin-Radushkevich (D-R), Temkin, Harkins-Jura and Halsey isotherm equations were used to describe the sorption process. The Langmuir isotherm equation had better correlation than the other isotherm equations. This shows that a monolayer sorption of the CR on PKC is the favoured mechanism of sorption. The monolayer sorption capacity of PKC for this dye was 79.37 mg g^{-1}. The linear form of the Langmuir equation was used to analyse the data obtained when the effect of PKC dosage on the CR removal was optimized. The monolayer sorption capacity, $q_{m,}$ decreased with the increase in sorbent dosages and sorption was favored at higher initial dye concentration and PKC dosages than the lower ones. The thermodynamic analysis of the sorption process indicates that the system is spontaneous and exothermic.

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