



Adsorption of 4-chlorophenol from wastewater-based activated carbon prepared from *Jatropha* seed husks

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ABSTRACT

The removal of 4-chlorophenol (4-CP) from aqueous solution using activated carbon (AC8) prepared from *Jatropha* seed husks by potassium hydroxide as activating agent at 800°C was investigated. The AC8 was characterized by Fourier transform infrared spectroscopy and energy dispersive X-ray spectroscopy. Batch adsorption was carried out systematically. Experimental results showed that the removal of 4-CP increased with increase in the contact time, adsorbent dosage, and initial concentration of the 4-CP. The Freundlich model was a more suitable model for description of the adsorption isotherm than the Langmuir model. The pseudo-second-order model was better used to describe the adsorption kinetics than the pseudo-first-order model. The thermodynamic parameters such as ΔG° , ΔH° , and ΔS° were determined to describe the adsorption process of the system.

Keywords: 4-Chlorophenol; Activated carbon; *Jatropha* seed husks; Adsorption; Isotherm; Kinetics; Thermodynamic

1. Introduction

The toxicity and readily biodegradable chlorophenols make them among the most important groups of priority pollutants. There are many uses of chlorophenols such as herbicides and fungicides [1], Kraft bleaching of paper pulp [2], and wood protection [3]. It is also used for the production of fungicides, dyes, and drugs [4], as well as an antiseptic in the treatment of root canal [5]. However, these compounds affect the environment as they may remain in the environment

because of its chlorinated nature [6]. As such, 4-chlorophenol (4-CP) is a good model compound to study many cases such as wastewater treatment efficiency [7].

Activated carbon (AC8) has special surface reactivity, high adsorption capacity, and microporous structure effective for the use as adsorbents of organic compounds [8]. However, the high cost of AC8 has encouraged researchers to produce AC8 from cheaper raw materials [9,10]. Recently, there have been attempts to produce AC8 from agricultural resources with high quality and at a cheaper cost [11]. In Malaysia, large amounts of *Jatropha* seed husks are

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produced as waste annually. The conversion of these materials into useful materials can contribute to the increase in its economic usefulness and helps to reduce environmental wastage [12]. In this study, *Jatropha* seed husks were used to produce AC8 for the removal of 4-CP from wastewater using an activating agent, potassium hydroxide (KOH). The isotherms and adsorption kinetics together with thermodynamics of 4-CP adsorption onto AC8 prepared from *Jatropha* seed husks were studied.

2. Materials and methods

2.1. Adsorption studies

The 4-CP stock solution was prepared by dissolving accurately weighted 4-CP (about 1 g) in 1,000 ml of distilled water. Synthetic wastewater solutions with various concentrations were prepared by diluting the prepared stock solution with distilled water.

2.2. Adsorption experiment

The batch model was used for adsorption measurement capabilities. 0.01 g of dried AC8 with 25 ml of synthetic wastewater containing 4-CP was put in a stoppered brown bottle 30 ml capacity. The bottles were shaken at room temperature and at 150 rpm. After the desired contact time, the adsorbent and the solution were separated by filtration. Ultraviolet–visible spectroscopy was used to determine the amount of remaining 4-CP. The adsorbed amount of the 4-CP and removal percentage of 4-CP by the AC8 were obtained by following Eqs. (1) and (2), respectively:

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

$$\% \text{ Removal} = \frac{(C_0 - C_e)}{C_0} 100 \quad (2)$$

where q_e is the adsorbed amount of adsorbate ion by the adsorbent (mg/g); C_0 is the initial concentration of the adsorbate in the solution (mg/L), C_e is the remaining concentration in the solution (mg/L) after contact time, V is the aqueous solution volume (L), and W is the adsorbent dosage (g).

2.3. Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) was used to investigate the characteristics of AC8 in

relation to its specific functional groups. Samples were analyzed with a Perkin–Elmer spectrum 400 FTIR spectrometer by the ATR technique from wavenumber ranging from 4,000 to 650 cm^{-1} and a resolution of 4 cm^{-1} .

2.4. Energy dispersive X-ray spectroscopy

Energy dispersive X-ray (EDX) spectroscopy was used to determine the elemental composition of the AC8. From the intensities of X-ray energy, it is possible to know the amount of each element and the kind of element which is present on the surface of the sample. Measurements were made using Oxford XMAX 50.

3. Results and discussion

3.1. FTIR and EDX of AC8 adsorbent

3.1.1. FTIR spectra

The FTIR spectra were used to determine the characteristic vibrational frequencies of the functional groups present. Fig. 1 shows the spectra of the raw material and that of the AC8. Before activation there is a presence of C=O bonds with peaks at 1,637, 1,711, and 1,739 cm^{-1} as well as a strong broad O–H band in the region of 3,100–3,600 cm^{-1} . Peaks showing the presence of C–O–C linkages occur at 1,053, 1,094, and 1,162 cm^{-1} . The aliphatic C–H stretches occur at 2,857 and 2,926 cm^{-1} . There is a presence of new peaks in the AC8 in the region of 1,900–2,700 cm^{-1} . These are attributed to C≡C stretchings at 1,908, 2,105, 2,325, and 2,653 cm^{-1} . The peak at around 3,784 cm^{-1} belongs to O–H groups that are located near C≡C linkages. There is also a C≡C–C–H bending at 633 cm^{-1} .

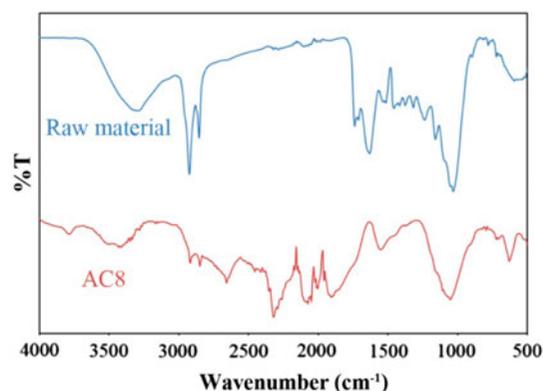


Fig. 1. FTIR spectrum of AC8 adsorbent.

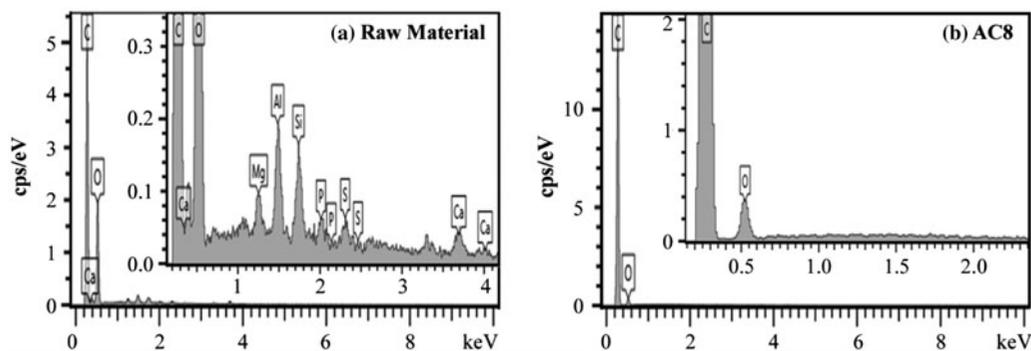


Fig. 2. EDX spectrum of the (a) raw material and (b) AC8. Inset figures show enlarged view of the spectrum for the trace elements.

Fig. 2 shows that the peaks of the raw material has changed after being subjected to heat and chemical agent treatment where the carbon and oxygen elements were the dominantly remaining elements that were present in the AC8. As seen in Table 1, the percentage of elemental composition of the samples as measured by EDX confirms that carbon and oxygen are the main constituents as well as traces of other elements. High carbon content is favorable, as it is expected that the phenolic compounds will be taken or adsorbed mainly by the carbon element.

3.2. Effect of contact time on the 4-chlorophenol adsorption

Fig. 3 shows the relationship between the adsorption capacity of the adsorbent and contact time at different initial concentrations of 4-CP. It is clear that the adsorption capacity increased with the increase of contact time and increased with decrease in initial concentration. The removal of 4-CP was rapid in the first 90 min of contact time, after that the removal became less rapid until a plateau value was achieved in spite of increase of contact time. The maximum value of adsorption capacity was at around 150 min for almost all initial concentrations. Therefore, 150 min can be regarded as the best contact time for the removal of 4-CP by the AC8.

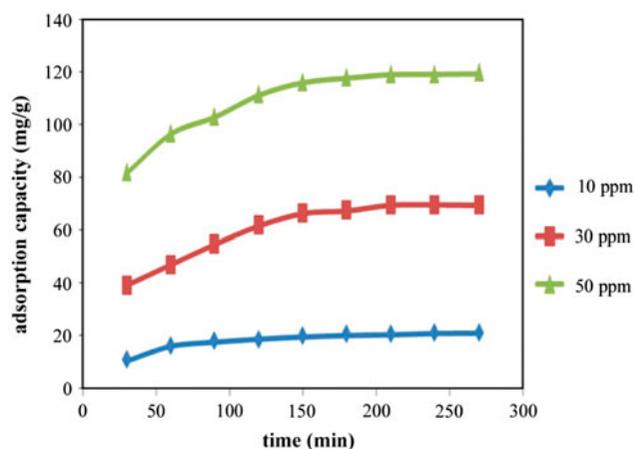


Fig. 3. Effect of contact time on 4-CP adsorption by AC8 prepared at the following conditions: temperature = 298 K; 150 rpm; and sample dose = 0.01 g/25 mL.

3.3. Effect of solution pH on the adsorption process of 4-chlorophenol

The impact of solution pH on the adsorption of 4-CP onto AC8 was studied for the initial concentration of 50 mg/L at room temperature and 150 rpm. The pH values of the 4-CP solutions were ranged from 2 to 10. As seen in Fig. 4, the equilibrium adsorption capacities decrease with an increase of pH value of

Table 1

The percentages of carbon and oxygen compositions of the raw material and AC8 sample

Sample	C Wt.%	O Wt.%	Others Wt.%	Total 100%	C Atomic%	O Atomic%	Others Atomic%	Total 100%
Raw	66.64	30.38	2.53	100.00	73.48	25.52	1.00	100.00
AC8	94.16	5.84	0	100.00	95.55	4.45	0	100.00

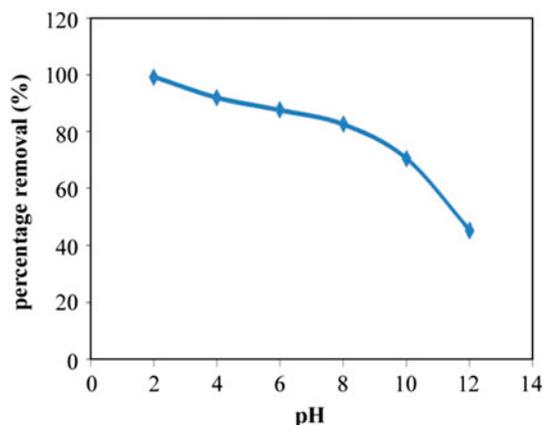


Fig. 4. Effect of solution pH values on the adsorption capacity by AC8 prepared from *Jatropha* seed husks for 50 ppm 4-CP concentration at the following conditions: contact time = 150 min; temperature = 298 K; 150 rpm; and sample dose = 0.01 g/25 mL.

4-CP solutions from 2 to 10. The highest equilibrium adsorption capacity and lowest equilibrium adsorption capacity occur at pH 2 and 10, respectively. At high pH values, the gradient of decrease of adsorption was slightly steeper due to the pH of the solution becoming greater than pK_a of 4-CP (pK_a of 4-CP = 9.41). This is because of the characteristic property of 4-CP which is anionic and is a proton donor [13]. When the solution pH becomes greater than pK_a of 4-CP, this leads to the creation of a repulsive force between the negative group on the AC8 surface and 4-CP anions [14,15]. Because of this, the 4-CP adsorption will continually decrease as the pH increases.

3.4. Effect of adsorbent dosage

The removal percentage of 4-CP by different weights of the same sample of AC8 was investigated and shown in Fig. 5. The dosage of AC8 was varied between 0.01 and 0.10 g/L. It was found that the percentage removal of 4-CP increased with an increase of AC8 dosage until it reached a plateau after 0.06 g/L. Therefore, 0.06 g/L of AC8 is the minimum amount required to adsorb entire molecules of 4-CP from the aqueous solution.

3.5. Effect of initial concentration on adsorption process

As seen in Fig. 6(a), the adsorption capacity of adsorbent increased with the increase of the initial concentrations. Previous researchers have explained that this is due to the driving force of mass transfer which increases with the increase of initial concentration of the 4-CP when the 4-CP molecules passes to the

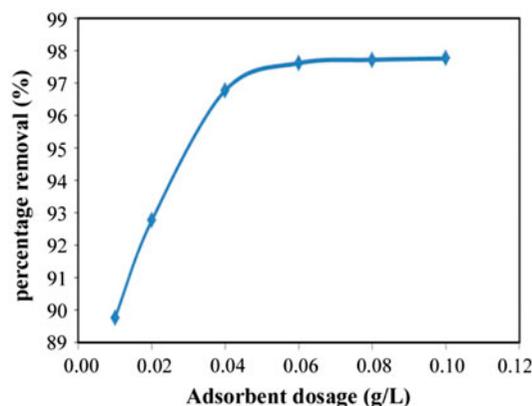


Fig. 5. Effect of adsorbent dosage on the removal percentage of AC8 prepared from *Jatropha* seed husks for 50 ppm 4-CP concentration at the following conditions: contact time = 150 min; temperature = 298 K; 150 rpm; and normal solution pH.

particle surface from the solution [16]. Also, the improvement of the adsorption capacity of the adsorbents may be explained by the fact that more molecules of adsorbate are available at high concentrations. On the other hand, the percentage removal of 4-CP decreased with increase of initial concentration above 50 mg/L at all adsorbents as shown in Fig. 6(b). This can be interpreted that either a decrease of the ratio between adsorbate molecules and adsorption sites or an accumulation of adsorbate molecules in the opening of pores leading to blockage of the pores.

3.6. Adsorption isotherm

The relationship between adsorbate concentration on the adsorbent surface and the adsorbate concentration in the solution at equilibrium can be described by adsorption isotherm models. The Langmuir and Freundlich adsorption isotherm models have been used by many authors to describe their experimental data [17–19].

3.6.1. Langmuir isotherm

According to the Langmuir isotherm model, when the molecules of adsorbate are attached to a site, no other molecule can take the same site [20] and the adsorption forms a monolayer at homogenous sites on the adsorbent surface. The equation for the Langmuir isotherm is given as [21,22]:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 K_L} + \frac{C_e}{Q_0} \quad (3)$$

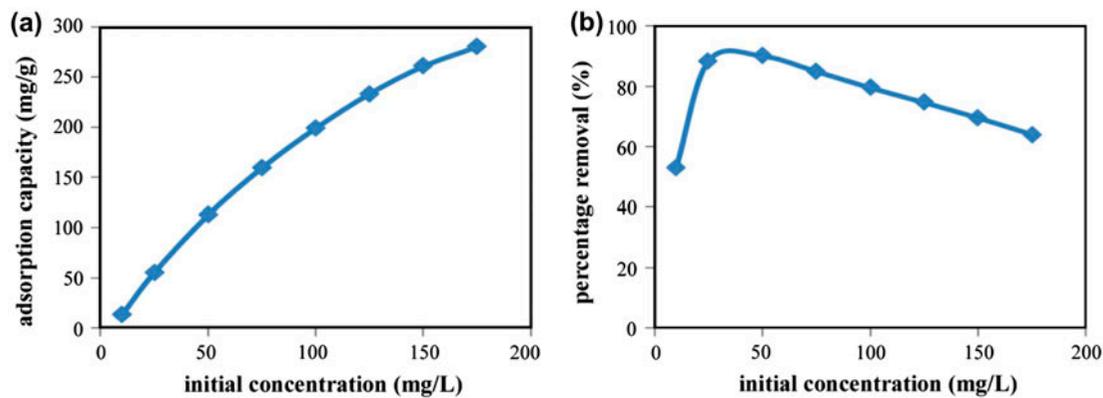


Fig. 6. (a) The effect of initial concentration on 4-CP adsorption by AC8 and (b) the percentage removal of 4-CP initial concentration by AC8.

where C_e is the equilibrium concentration in the solution (mg/g), q_e is the quantity of adsorbate adsorbed per unit mass of adsorbent. K_L and Q_0 are the Langmuir constants for adsorption energy and adsorption capacity, respectively, and can be calculated from the intercept and slope of the equation in the linear plot of C_e/q_e vs. C_e , as shown in Fig. 7.

Table 2 shows the values of the Q_0 and the coefficient correlation (R^2) obtained by mathematical calculations from the plot in Fig. 7. The R^2 value for the 4-CP adsorption is 0.924, indicating that the 4-CP adsorption on AC8 favors following the Langmuir isotherm. The values of Q_0 and K_L were 333.33 mg/g and 0.04, indicating that the 4-CP adsorption is monolayer on the homogenous sites.

The R_L is another parameter which can be calculated from the K_L value by the following equation:

$$R_L = \frac{1}{1 + K_L C_0} \quad (4)$$

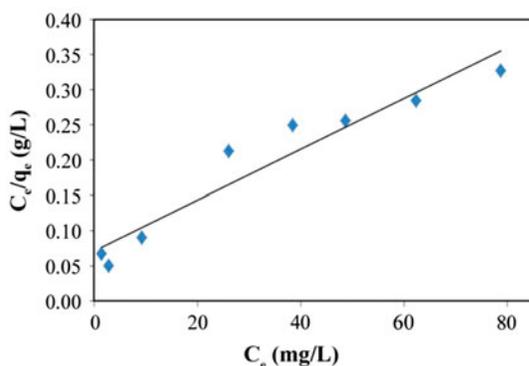


Fig. 7. Langmuir plot for the adsorption isotherm of 4-CP onto activated AC8 as adsorbent.

The values of R_L shows the nature of the adsorption process to be irreversible if ($R_L = 0$), favorable if ($0 < R_L < 1$), linear if ($R_L = 1$), or unfavorable if ($R_L > 1$) [23]. Fig. 8 shows the dimensionless equilibrium term (separation factors) R_L for the equation of Langmuir isotherm model at normal pH and room temperature where all the R_L values obtained were less than one and greater than zero indicative of that the Langmuir isotherm model was suitable for describing the dynamics of 4-CP adsorption by AC8, and the experimental conditions of adsorption process in this research were favorable.

3.6.2. Freundlich isotherm

The Freundlich isotherm is an empirical model and suitable for a highly heterogeneous adsorption form by sites on the adsorbent surface.

The logarithmic form of the Freundlich equation is expressed as follows [24]:

$$\log q_e = \log K_F + 1/n \log C_e \quad (5)$$

where q_e , K_F , n , and C_e are the equilibrium concentration of 4-CP per unit mass, adsorption capacity, adsorption intensity, and equilibrium concentration in solution, respectively. The K_F and n are the Freundlich constants indicating adsorption capacity and intensity of adsorption. Fig. 9 shows the plot of $\log q_e$ vs. $\log C_e$ whereby the slope and intercept were used to calculate the Freundlich constants.

From Table 2, the Freundlich constant values of K_F and n are 24.43 and 1.89, respectively. The value of K_F is high which means the conditions of 4-CP adsorption is favorable in liquid phase. The 4-CP adsorption value of R^2 was 0.94, slightly higher than the value of

Table 2
Parameters of Langmuir and Freundlich models for the adsorption isotherm of 4-CP on AC8 at room temperature

Adsorbent	Langmuir			Freundlich			
	Q_0	K_L	R^2	K_F	$1/n$	n	R^2
AC8	333.3333	0.042254	0.924	24.434	0.528	1.89	0.942

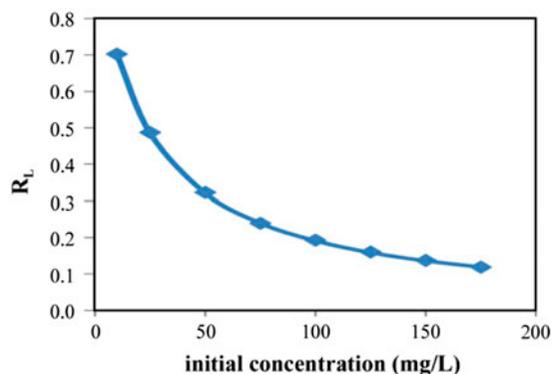


Fig. 8. The calculated separation factor (R_L) against 4-CP ions at various concentrations (mg/L) for AC8 at the following conditions: adsorbent dose = 0.01 g/25 mL; contact time = 150 min; temperature = 298 K; and normal solution pH.

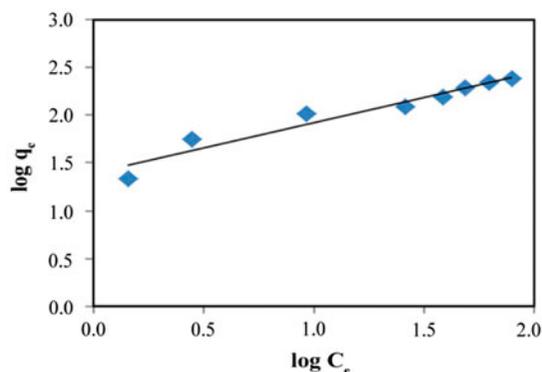


Fig. 9. Freundlich plot for the adsorption isotherm of 4-CP onto AC8 material as an adsorbent.

Langmuir isotherm which means a highly favorable adsorption.

3.7. Adsorption kinetics models

The most important part of kinetic studies of adsorption is to get information about the mechanism of interaction between a solute molecule and a solid through adsorption process. There are several kinetic

models to describe that interaction mechanism. Among them, the pseudo-first-order and pseudo-second-order models were studied to determine which is the better model to describe the adsorption kinetics of 4-CP at concentration of 50 mg/L.

The equation of pseudo-first order is given as follows [25,26]:

$$\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \tag{6}$$

where q_e and q_t are the adsorbed amount at time and equilibrium time in minutes, k_1 is the pseudo-first-order adsorption rate constant in min^{-1} . The value of q_e and k_1 can be calculated from the intercept and slope of plotting $\log (q_e - q_t)$ vs. t , as shown in Fig. 10.

The values of rate constant and the correlation coefficient (R^2) for the kinetic model of pseudo-first order are shown in Table 3. At all adsorbate concentrations, the values of R^2 were not high for the first-order kinetic model.

It is possible that the pseudo-second-order model would better describe the adsorption kinetics. The pseudo-second-order equation is given as [25,26]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{7}$$

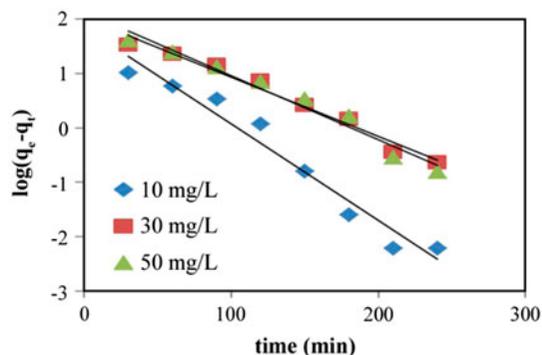


Fig. 10. Pseudo-first-order kinetics of 4-CP adsorption onto AC8 at different adsorbate concentrations and normal condition.

Table 3
Adsorption kinetic model parameters of phenol onto AC8

C_0 (mg/L)	$q_{e, \text{exp}}$ (mg/g)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
		$q_{e, \text{cal}}$ (mg/g)	k_1 (1/h)	R^2	$q_{e, \text{cal}}$ (mg/g)	k_2 (g/mgh)	R^2
10	20.79383	70.22633	0.040993	0.959	20.79002	0.0481	1
30	72.76463	106.4388	0.025103	0.976	72.9927	0.0137	1
50	112.243	137.2461	0.027175	0.968	112.3596	0.0089	1

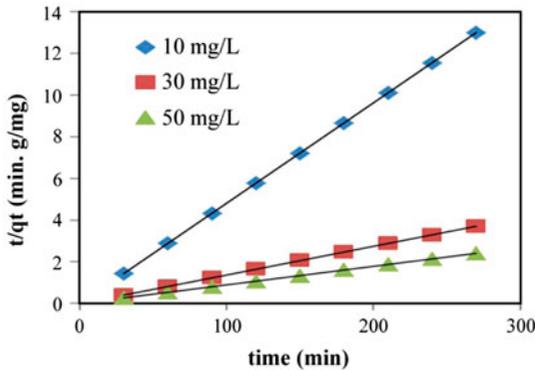


Fig. 11. Pseudo-second-order kinetic of 4-CP adsorption onto AC8 at different adsorbate concentrations and normal condition.

where the values of q_e and k_2 are from the slope and intercept of the linear plot of t/q_t vs. t [27] as seen in Fig. 11. As seen in Table 3, the k_2 values decreased as the 4-CP concentrations increase. The 4-CP adsorption

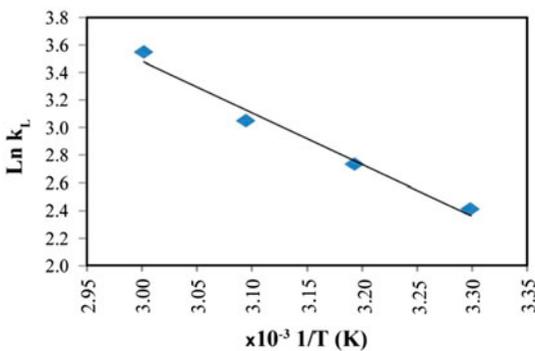


Fig. 12. Plot of $\ln K_d$ vs. $1/T$.

Table 4
Thermodynamic parameters for 4-CP adsorption on AC8

Adsorbent	K_d				ΔG°				ΔH°	ΔS°	R^2
	30°	40°	50°	60°	30°	40°	50°	60°			
AC8	11.1	15.3	21.1	34.53	-6.07	-6.88	-7.69	-8.93	31.14	0.12	0.98

onto AC8 could be sensibly described by the pseudo-second-order model, because values of the R^2 for all the 4-CP concentrations were calculated to be 1 and larger than the R^2 values of the pseudo-first order. Additionally, the calculated q_e values of the pseudo-second order are closer to the experimental q_e values than calculated q_e values of the pseudo-first order.

3.8. Adsorption thermodynamics

The thermodynamic parameters can be obtained by the following equations [28,29]:

$$K_d = \frac{q_e}{C_e} \quad (8)$$

$$\Delta G^\circ = -RT \ln K_d \quad (9)$$

$$\ln K_d = -\Delta H^\circ / RT + \Delta S^\circ / R \quad (10)$$

where K_d , T (K), and R are the adsorbent distribution coefficient, the absolute temperature of the solution, and the universal gas constant (8.314 J/mol K), respectively. The values of ΔH° and ΔS° can be calculated from the slope and intercept of the linear plot of $\ln K_d$ vs. $1/T$ as shown in Fig. 12.

Table 4 shows that the thermodynamic constants, ΔG° , values are negative, indicating that the adsorption is spontaneous in nature mainly physical. Since the values of ΔG° were between -8.927 and -6.069 [30], it has a feasible and spontaneous nature. The ΔH° value is positive, indicating that the adsorption is endothermic. The ΔS° value is also positive, indicating that the adsorbate organization became more random at the interface of solid and solution [31].

4. Conclusions

KOH was employed as an activation agent in the modification of *Jatropha* seed husks into AC8. The AC8 was characterized by FTIR and EDX spectroscopy. The AC8 was employed as an adsorbent for removal of 4-CP from synthetic wastewater. Solutions with low pH, high adsorbent dosage, high contact time, and low 4-CP adsorption were the best combination to obtain highest 4-CP removal. The model that better describes the equilibrium adsorption was the Freundlich isotherm and the adsorption kinetics follows a pseudo-second-order model. A negative value for ΔG° , a positive value for ΔH° , and a positive value for ΔS° indicated that adsorption occurs in a spontaneous, endothermic behavior and the adsorbate organization became more random at the interface of solid and solution.

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