



Removal of phenol from aqueous solutions by adsorption onto activated carbon prepared from biomass material

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ABSTRACT

Activated carbon derived from rattan sawdust (ACR) was evaluated for its ability to remove phenol from an aqueous solution in a batch process. Equilibrium studies were conducted in the range of 25–200 mg/L initial phenol concentrations, 3–10 solution pH and at temperature of 30 °C. The experimental data were analyzed by the Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherm models. Equilibrium data fitted well to the Langmuir model with a maximum adsorption capacity of 149.25 mg/g. The dimensionless separation factor R_L revealed the favorable nature of the isotherm of the phenol-activated carbon system. The pseudo-second-order kinetic model best described the adsorption process. The results proved that the prepared activated carbon was an effective adsorbent for removal of phenol from aqueous solution.

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1. Introduction

Phenols are widely used for the commercial production of a wide variety of resins including phenolic resins, which are used as construction materials for automobiles and appliances, epoxy resins and adhesives, and polyamide for various applications [1]. Phenols are considered as priority pollutants since they are harmful to organisms at low concentrations and many of them have been classified as hazardous pollutants because of their potential harm to human health. The US Environmental Protection Agency (EPA) regulations call for lowering phenol content in the wastewater to less than 1 mg/L [2]. The ingestion of such contaminated water in the human body causes protein degeneration, tissue erosion and paralysis of the central nervous system and also damages the kidney, liver and pancreas [3]. Therefore, it is considered necessary to remove the phenol from industrial effluents before discharging into the water stream.

There are many methods such as oxidation, precipitation, ion change, solvent extraction and adsorption for removing phenols and its derivatives from aqueous solutions [4,5]. The treatment of wastewater with activated carbon is considered to be an effective method for the removal of phenol from waste solution because of its large surface area, micro-porous nature, high adsorption capacity, high purity and easy availability. However, the use of effective commercial activated carbons based on relatively expensive start-

ing materials (wood or coal) is unjustified for most pollution control applications. It is, therefore, very important to examine the feasibility of using cheaper raw materials to prepare activated carbon. Many reports have appeared on the development of activated carbon from cheaper and readily available materials. Activated carbons have been prepared from corncob [6], sugar beet bagasse [7], apricot shell [8], sunflower seed hull [9], agricultural waste material [10], bagasses [11], rubber seed coat [12], coconut shell [13], apricot stone shells [14], oil palm fibre [15], bamboo [16], coconut husk [17] and *Hevea brasiliensis* seed coat [18].

To produce a value added product from rattan sawdust, it is proposed to convert it to activated carbon. In our previous study, activated carbon derived from rattan sawdust showed remarkable efficiency for the removal of basic dye from aqueous solution [19]. In this work, the suitability of the activated carbon prepared from rattan sawdust for phenol adsorption was assessed. The equilibrium and kinetic data of the adsorption were then studied to understand the adsorption process.

2. Experimental

2.1. Adsorbate and solution

Analytical-reagent grade phenol supplied by Sigma–Aldrich (M) Sdn Bhd, Malaysia was used as the adsorbate in adsorption. A stock solution was prepared by dissolving the required amount of phenol in double distilled water without pH adjustment. Working solutions of the desired concentrations were obtained by successive dilutions.

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2.2. Preparation of activated carbon

The rattan sawdust was collected from a local furniture factory. It was washed with hot distilled water to remove impurities like dust, and the material was then dried and finally sieved to discrete sizes. The apparatus and experimental methods employed in preparation of activated carbon in the present work were similar to those of our work as reported previously [19]. Initially the raw material was carbonized at 700 °C under nitrogen atmosphere for 1 h. A certain amount of produced char was then soaked with potassium hydroxide (KOH) at impregnation ratio of 1:1 (KOH pellets (g):char (g)). The mixture was dehydrated in an oven overnight at 105 ± 1 °C; then pyrolysed in a stainless steel vertical tubular reactor placed in a tube furnace under high purity nitrogen (99.995%) flow of $150 \text{ cm}^3/\text{min}$ to a final temperature of 850 °C and activated for 2 h. Once the final temperature was reached, the nitrogen gas flow was switched to carbon dioxide and activation was continued for 2 h. The activated product was then cooled to room temperature under nitrogen flow and washed with deionized water to remove remaining chemical. Subsequently the sample was transferred to a beaker containing a 250 mL solution of 0.10 M hydrochloric acid, stirred for 1 h, and then washed with hot deionized water until the pH of the washing solution reached 6.5–7.

2.3. Batch equilibrium and kinetic studies

In adsorption equilibrium, experiments were conducted in a set of 250 mL Erlenmeyer flasks, where solutions of phenol (200 mL) with different initial concentrations (25–200 mg/L) were added in these flasks. Equal masses of 0.20 g of ACR of particle size $150 \mu\text{m}$ were added to phenol solutions and each sample was kept in an isothermal shaker of 120 rpm at 30 ± 1 °C for 24 h to reach equilibrium of the solid–solution mixture. The pH of the solutions was not adjusted. A similar procedure was followed for another set of Erlenmeyer flask containing the same phenol concentration without activated carbon to be used as a blank. The flasks were then removed from the shaker and the final concentration of phenol in the solution was analyzed using a double beam UV–vis spectrophotometer (Shimadzu, Japan) at 270 nm wavelength. The samples were filtered prior to analysis in order to minimize interference of the carbon fines with the analysis. Each experiment was duplicated under identical conditions. The amount of adsorption at equilibrium, q_e (mg/g), was calculated by

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

where C_0 and C_e (mg/L) are the liquid-phase concentrations of phenol at initial and equilibrium, respectively. V (L) is the volume of the solution, and W (g) is the mass of dry adsorbent used.

The procedures of kinetic experiments were basically identical to those of equilibrium tests. The aqueous samples were taken at preset time intervals and the concentrations of phenol were similarly measured.

2.4. Effect of initial pH

The adsorption of phenol by the ACR was studied over a pH range of 3–10 at 30 °C and the studies were carried out for 24 h. Initial concentration of phenol was 200 mg/L and the adsorbent dose was kept at 0.20 g. Agitation was provided with a constant agitation speed of 130 rpm. At equilibrium, the phenol concentrations were measured. The pH was adjusted by adding a few drops of diluted 0.1N NaOH or 0.1N HCl. The pH was measured by using a pH meter (Ecoscan, EUTECH Instruments, Singapore).

3. Results and discussion

3.1. Effect of contact time and initial concentration on phenol adsorption

Fig. 1 depicts the effect of contact time on the removal of phenol at various initial concentrations (25–200 mg/L). The saturation curves rise sharply in the initial stages, indicating that there are plenty of readily accessible sites. Eventually, a plateau is reached in all curves indicating that the adsorbent is saturated at this level. It can be seen from Fig. 1 that the contact time needed for phenol solutions with initial concentrations of 25–150 mg/L to reach equilibrium was 4 h. However, for phenol solutions with higher initial concentrations, longer equilibrium times were required. It was also seen that an increase in initial phenol concentration resulted in increased phenol uptake. The removal curves are single, smooth and continuous, indicating the formation of monolayer coverage of the phenol molecules onto the outer surface of the adsorbent.

The adsorption capacity at equilibrium (q_e) increased from 23.44 to 140.68 mg/g with an increase in the initial phenol concentrations from 25 to 200 mg/L. Three consecutive mass transport steps are associated with the adsorption of solute from solution by porous adsorbent [20]. First, the adsorbate migrates through the solution, i.e., film diffusion, followed by solute movement from particle surface into interior site by pore diffusion and finally the adsorbate is adsorbed into the active sites at the interior of the adsorbent particle. This phenomenon takes relatively long contact time.

3.2. Effect of solution pH on phenol adsorption

To study the influence of pH on the adsorption capacity of ACR for phenol, experiments were performed at temperature of 30 °C and phenol initial concentration of 200 mg/L using different initial solution pH values, changing from 3 to 10. Fig. 2 shows that

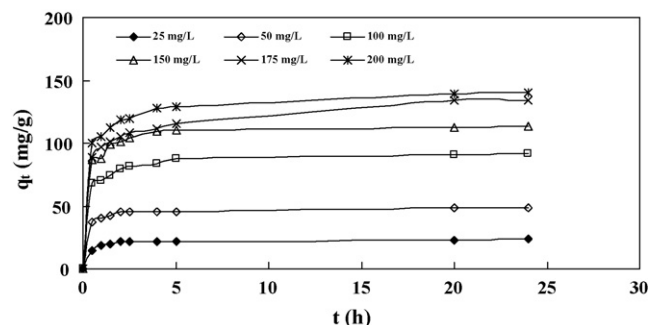


Fig. 1. The variation of adsorption uptake with time at various initial phenol concentrations.

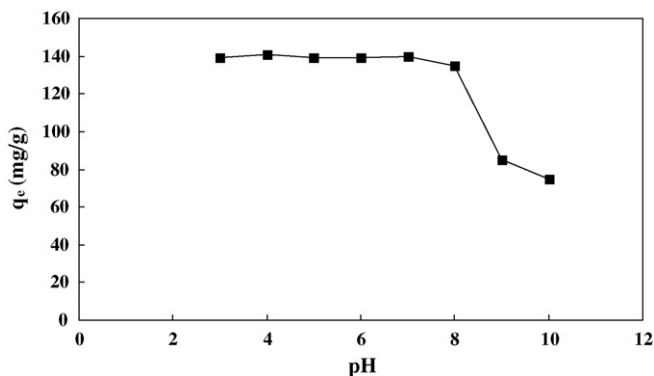


Fig. 2. Effect of solution pH on adsorption of phenol onto ACR.

the phenol removal was maximum and unaffected when the initial pH of the phenol solution was in the range of 3–8. A similar trend of pH effect was observed for the adsorption of phenol on activated carbon-commercial grade and laboratory grade [21]. Generally, solution pH affects the surface charge of adsorbent and degree of ionization of the adsorbate [22]. Phenol as a weak acid compound with $pK_a \approx 9.89$ [9] is dissociated at $pH > pK_a$. Therefore, the adsorption decrease at high pH values due to ionization of adsorbate molecules. The reason could be also due to the electrostatic repulsions between the negative surface charge and the phenolate–phenolate anions in solution [23]. While at acidic pH, the percentage removal was higher because phenol was undissociated and the dispersion interaction predominated.

3.3. Adsorption isotherms

Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. The Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherm models were used to describe the relationship between the amount of phenol adsorbed and its equilibrium concentration in solutions.

The Langmuir isotherm is valid for monolayer adsorption on a surface containing a finite number of identical sites. The model assumes uniform adsorption on the surface and no transmigration in the plane of the surface [24]. The linear form of the Langmuir isotherm can be represented by the following equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \tag{2}$$

where C_e (mg/L) is the equilibrium concentration of the adsorbate, q_e (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbent, Q_0 and b are Langmuir constants related to adsorption capacity and rate of adsorption, respectively.

Fig. 3 shows a linear relationship of C_e/q_e versus C_e using experimental data obtained, suggesting the applicability of the Langmuir model ($R^2 = 0.98$). The applicability of the model suggests monolayer coverage of the adsorbate at the outer surface of the adsorbent is significant. Values of Q_0 and b calculated from the plot shown in Fig. 3 using the least square methods are listed in Table 1.

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless separation factor (R_L) [24] which is defined by

$$R_L = \frac{1}{1 + bC_0} \tag{3}$$

where b is the Langmuir constant and C_0 is the highest phenol concentration (mg/L). The value of R_L indicates the type of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). The value of R_L was found to be 0.0303 and this again confirmed that the Langmuir isotherm was favorable for

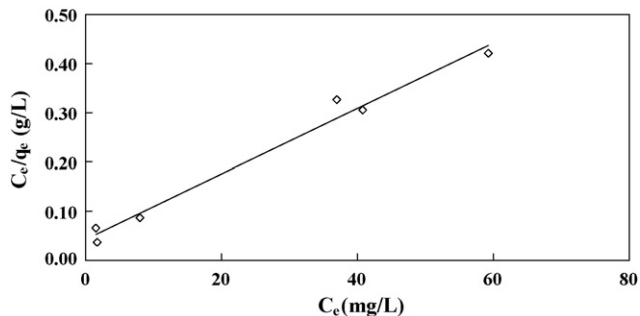


Fig. 3. Langmuir adsorption isotherm of phenol onto ACR at 30 °C.

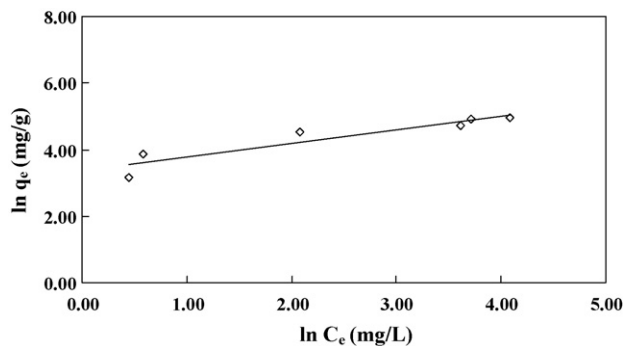


Fig. 4. Freundlich adsorption isotherm of phenol onto ACR at 30 °C.

adsorption of phenol onto the activated carbon under the conditions used in this study.

The Freundlich isotherm is given as [25]:

$$q_e = K_F C_e^{1/n} \tag{4}$$

where K_F ((mg/g)(L/mg) $^{1/n}$) is roughly an indicator of the adsorption capacity and $(1/n)$ of the adsorption intensity. Value of $n > 1$ represents a favorable adsorption condition [26]. The linear form of Eq. (4) is

$$\ln q_e = \ln K_F + (1/n) \ln C_e \tag{5}$$

which will have a straight line with a slope of $1/n$ and an intercept of $\ln(K_F)$ when $\ln(q_e)$ is plotted against $\ln(C_e)$ (Fig. 4). The value of n was found to be 2.48, indicating that the adsorption condition was favorable.

Temkin and Pyzhev [27] considered the effects of indirect adsorbate/adsorbate interactions on adsorption isotherms. The heat of adsorption of all the molecules in the layer would decrease linearly with coverage due to adsorbate/adsorbate interactions. The linear form of Temkin isotherm is

$$q_e = \left(\frac{RT}{b}\right) \ln(A) + \left(\frac{RT}{b}\right) \ln C_e \tag{6}$$

where $B = RT/b$. This isotherm assumes that (i) the heat of adsorption of all the molecules in the layer decreases linearly with coverage due to adsorbent–adsorbate interactions, and that (ii) the adsorption is characterized by a uniform distribution of binding energies, up to some maximum binding energy [27]. Fig. 5 shows a plot of q_e versus $\ln C_e$. The constant A and B together with the R^2 values are listed in Table 1. The correlation coefficient of Temkin isotherm equation ($R^2 = 0.95$) is lower than that obtained for the Langmuir isotherm.

Another model for the analysis of isotherms of a high degree of rectangularity is Dubinin–Radushkevich isotherm [28] which is as follows:

$$q_e = q_s \exp(-B\epsilon^2) \tag{7}$$

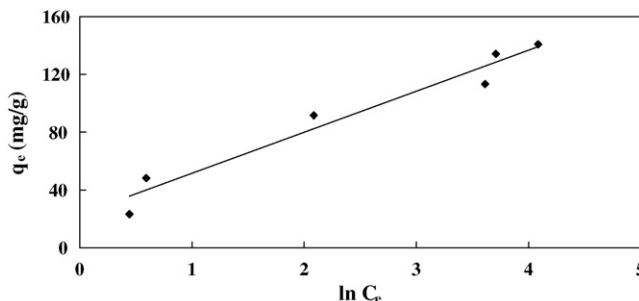


Fig. 5. Temkin adsorption isotherm of phenol onto ACR at 30 °C.

Table 1
Isotherms constants for adsorption of phenol onto ACR at 30 °C

Isotherms	Parameters
Langmuir	
Q_0 (mg/g)	149.25
b (L/mg)	0.16
R^2	0.98
Freundlich	
K_F (mg/g (L/mg) ^{1/n})	29.37
n	2.48
R^2	0.86
Temkin	
A (L/g)	2.252
B	28.451
R^2	0.95
Dubinin–Radushkevich	
q_s (mg/g)	122.99
E	707.107
R^2	0.93

where ε can be correlated as

$$\varepsilon = RT \ln \left[1 + \frac{1}{C_e} \right] \tag{8}$$

The constant B gives the mean free energy E of adsorption per molecule of the adsorbate when it is transferred to the surface of the solid from infinity in the solution and can be computed by using the relationship:

$$E = \frac{1}{\sqrt{2B}} \tag{9}$$

where R is the gas constant (8.31 J/mol K) and T is the absolute temperature. A plot of $\ln(q_e)$ versus ε^2 (Fig. 6) enables the constants q_s and E to be determined (Table 1). The parameters of the four isotherms were computed and listed in Table 1. The Langmuir isotherm fitted quite well with the experimental data (correlation coefficient $R^2 = 0.98$), compared to the other three isotherm models which had lower correlation coefficients. A similar result was reported for the adsorption of phenol on coconut shell based activated carbon and commercial Filtrasorb 100 activated carbon [13].

Table 1 indicates that the computed maximum monolayer adsorption capacity Q_0 of phenol on the ACR was relatively large, which was 149.25 mg/g. The large adsorption capacity could be attributed to its large surface area (1083 m²/g). Table 2 lists a comparison of adsorption capacity of the prepared activated carbon with those obtained in the literature for the sorption of phenol. It can be seen that the prepared ACR is more effective for this purpose.

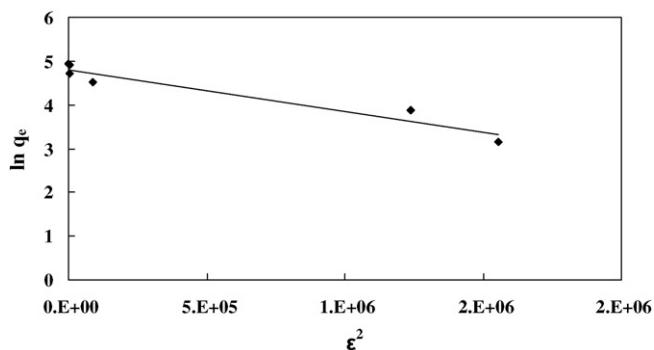


Fig. 6. D–R adsorption isotherm of phenol onto ACR at 30 °C.

Table 2
Comparison of adsorption capacities of various activated carbons for phenol

Adsorbent	Q_0 (mg/g)	Reference
Rattan sawdust based activated carbon	149.25	This work
Activated coal	1.481	[29]
Granular activated carbon	165.80	[30]
Commercial activated carbon	49.72	[31]
Sugarcane bagasse fly ash	23.832	[32]
Activated carbon-commercial grade (ACC)	30.2187	[32]
Activated carbon-laboratory grade (ACL)	24.6458	[32]

3.4. Adsorption kinetics

Kinetic adsorption data were treated with pseudo-first-order kinetic model [33]:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{10}$$

where q_e and q_t refer to the amount of phenol adsorbed (mg/g) at equilibrium and at any time, t (h), respectively, and k_1 is the equilibrium rate constant of pseudo-first-order sorption (1/h). Integration of Eq. (10) for the boundary conditions $t = 0$ to t and $q_t = 0$ to q_t , gives

$$\log \frac{q_e}{(q_e - q_t)} = \frac{k_1}{2.303} t \tag{11}$$

Eq. (11) can be rearranged to

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{12}$$

The slope and intercept of plot of $\log(q_e - q_t)$ versus t (Fig. 7) were used to determine the first-order rate constant k_1 . In many cases, the first-order equation of Lagergren does not fit well with the whole range of contact time and is generally applicable over the initial stage of the adsorption processes [34].

Kinetic data were further treated with the pseudo-second-order kinetic model [35]. The pseudo-second-order equation is also based on the sorption capacity of the solid phase. Contrary to the other model, it predicts the behaviour over the whole range of adsorption and is in agreement with an adsorption mechanism being the rate-controlling step. The differential equation is the following:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{13}$$

where k_2 is the equilibrium rate constant of pseudo-second-order adsorption (g/mg h). Integrating Eq. (13) for the boundary condition $t = 0$ to t and $q_t = 0$ to q_t , gives

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + k_2 t \tag{14}$$

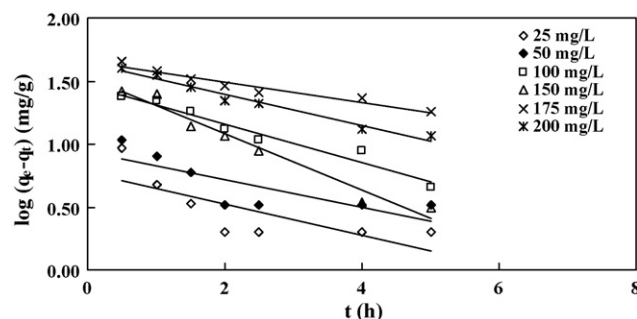


Fig. 7. Pseudo-first-order kinetics for adsorption of phenol onto ACR at 30 °C.

Table 3
Comparison of the pseudo-first-order and pseudo-second-order adsorption rate constants, and calculated and experimental q_e values for different initial phenol concentrations

C_0 (mg/L)	$q_{e,exp}$ (mg/g)	Pseudo-first-order kinetic model				Pseudo-second-order kinetic model			
		$q_{e,cal}$ (mg/g)	k_1 (1/h)	R^2	Δq (%)	$q_{e,cal}$ (mg/g)	k_2 (g/mg h)	R^2	Δq (%)
25	23.44	5.88	0.284	0.587	89.90	23.26	0.149	0.999	3.27
50	48.20	8.66	0.251	0.633	95.35	48.78	0.086	0.999	4.64
100	91.99	29.22	0.354	0.959	83.69	92.59	0.032	0.999	6.90
150	113.02	34.25	0.520	0.966	82.08	113.64	0.041	1.000	3.77
175	134.2	44.95	0.185	0.939	87.59	136.99	0.012	0.999	11.77
200	140.68	43.92	0.285	0.971	85.76	142.86	0.018	0.999	7.59

which is the integrated rate law for a pseudo-second-order reaction. Eq. (14) can be rearranged to obtain a linear form:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (15)$$

The slope and intercept of plot of t/q_t versus t (Fig. 8) were used to calculate the second-order rate constant k_2 . It is more likely to predict the behaviour over the whole range of adsorption and is in agreement with the chemisorption mechanism being the rate-controlling step [35].

Table 3 lists the results of the rate constant studies for different initial phenol concentrations by the pseudo-first-order and second-order models. For the pseudo-second-order model in Table 3, the rate constant generally decreased with the increase of the initial phenol concentration. The correlation coefficients obtained for the second-order kinetic model were greater than 0.99 at all the concentrations studied.

Besides the value of R^2 , the applicability of both kinetic models were further verified through normalized standard deviation Δq (%) defined as

$$\Delta q(\%) = 100 \times \sqrt{\sum \frac{[(q_{exp} - q_{cal})/q_{exp}]^2}{(n-1)}} \quad (16)$$

where the subscripts 'exp' and 'cal' refer to the experimental and calculated values, respectively and n is the number of data points. The higher the value of R^2 and the lower the value of Δq (%) indicate the better a fit is. Table 3 lists the Δq (%) values obtained. The results suggested that the pseudo-second-order adsorption mechanism was predominant, and that the overall rate of the phenol

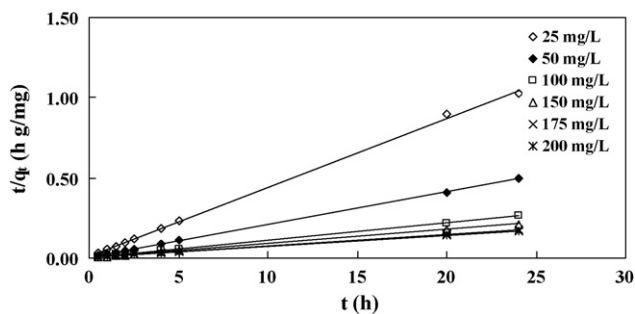


Fig. 8. Pseudo-second-order kinetics for adsorption of phenol onto ACR at 30 °C.

Table 4
Intraparticle diffusion constants for different initial phenol concentrations

C_0 (mg/L)	k_{i1} (mg/g h ^{0.5})	c_1	R^2	k_{i2} (mg/g h ^{0.5})	c_2	R^2
25	10.041	7.7054	0.958	0.495	20.57	0.850
50	10.406	29.874	0.988	1.116	42.99	0.945
100	15.998	55.467	0.956	2.913	78.13	0.872
150	19.474	72.356	0.928	1.159	107.27	0.998
175	23.045	72.726	0.995	8.048	96.36	0.985
200	24.137	82.649	0.975	5.572	114.39	0.928

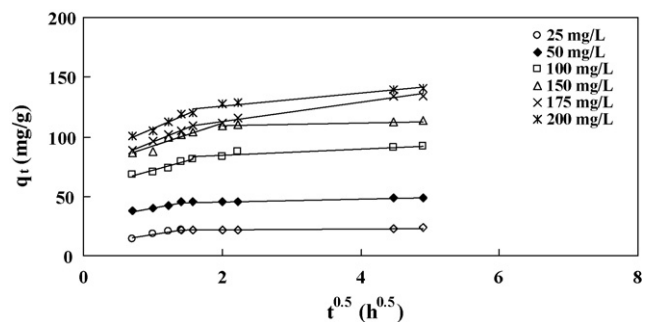


Fig. 9. Intraparticle diffusion plot for adsorption of phenol onto ACR at 30 °C for different initial phenol concentrations.

adsorption process appeared to be controlled by the chemisorption process [34,35]. The similar phenomena have also been observed in the adsorption of phenol on activated carbons prepared from beet pulp [36] and plum kernels [37].

3.5. Intraparticle diffusion modeling

Since neither the pseudo-first-order nor the second-order model can identify the diffusion mechanism, the kinetic results were analyzed by the intraparticle diffusion model to elucidate the diffusion mechanism, which is expressed as [38]:

$$q = k_i t^{0.5} + c \quad (17)$$

where q is the amount of phenol adsorbed (mg/g) at time t , k_i is intraparticle diffusion constant (mg/g min^{0.5}), and c is the intercept [38]. The constants were calculated and listed in Table 4. If the value of c is zero, then the rate of adsorption is controlled by intraparticle diffusion for the entire adsorption period. However, the plot of q against $t^{0.5}$ usually shows more than one linear portion, and if the slope of the first portion is not zero, then film (boundary layer) diffusion controls the adsorption rate at the beginning. As seen from Fig. 9, the plots were not linear over the whole time range, implying that more than one process affected the adsorption. Ofomaja [39] noted the similar trend for dye adsorption on palm kernel fibre at various initial concentrations. It was reported that the multiple nature observed in the intraparticle diffusion plot suggested that intraparticle diffusion was not solely rate-controlling. Besides, external mass transfer of dye molecules onto sorbent particles was also significant in the sorption process, especially at the initial reaction period [39].

4. Conclusions

The activated carbon prepared from biomass can be effectively used as adsorbent for the removal of phenol from aqueous solutions. The solution pH played a significant role in influencing the capacity of an adsorbent towards phenol molecules. A decrease in the pH of solutions led to a significant increase in the adsorp-

tion capacity of activated carbon. The obtained results showed that the ACR possessed a high adsorption capacity to remove phenol. The Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherm models were used to express the sorption phenomena of phenol to the prepared activated carbon. Consequently, linear regression of the experimental data showed that the Langmuir equation best represented phenol adsorption data. The maximum adsorption capacity of ACR was 149.25 mg/g. The pseudo-first-order and pseudo-second-order kinetic models were used to analyze the data obtained for phenol adsorption onto the prepared activated carbon. The results indicated that the pseudo-second-order equation provided the better correlation for the adsorption data. The experimental results indicated that the prepared ACR was economically promising twofold. The non-wood forest product waste would be utilized and the production of activated carbon for the treatment of wastewaters in Malaysia would be achieved at low cost.

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