

# PHENOL ADSORPTION BY ACTIVATED CARBON OF DIFFERENT FIBRE SIZE DERIVED FROM EMPTY FRUIT BUNCHES

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## ABSTRACT

The capacity of empty fruit bunch-based activated carbon for phenol adsorption was studied. Four different particle sizes of activated carbon (AC) (unsieved, >2, 0.355-1.0 and <0.15 mm mesh number) were produced by the activation of prepared charcoal with CO<sub>2</sub>. Activated carbon with particle size greater than 2 mm demonstrated the highest percentage of phenol adsorption. The performance of this activated carbon in phenol removal was better than commercially available activated carbon with an adsorption capacity of the AC sample and the commercial AC at equilibrium time amounting to 73% and 68%, respectively. The experimental data were analysed using the Langmuir and Freundlich adsorption models. The kinetics of adsorption were well described by a pseudo-second order model, whilst the adsorption equilibrium was best represented by the Langmuir isotherm model.

**Keywords:** activated carbon, adsorption, empty fruit bunches, phenol.

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## INTRODUCTION

Phenolics are a pollutant that are often found in effluents from the palm oil mill, petrochemical, pharmaceutical, paint, pulp and paper, and other industries (Shaarani and Hameed, 2010). These compounds are normally used as intermediates in the synthesis of pesticides, colours, plastics, insecticides, and many more substances. Improper treatment of these polluting compounds may produce a negative impact on human life and the environment, by contaminating the soil and polluting the ground water. Furthermore, their toxicity may seriously affect microorganisms. The permissible level of phenolic compounds before discharge into surface water and municipal sewers

set by the Environmental Quality Act of Malaysia is 0.001 mg litre<sup>-1</sup>.

Currently, phenols are abated in wastewater by a separation process: through liquid-liquid extraction, distillation, adsorption, membrane-solvent extraction, electrochemical treatments, or microwave-induced heating (Bi *et al.*, 2007; Busca *et al.*, 2008). It appears that the adsorption of phenols by activated carbon is the best option over the other methods due to zero-sludge production.

Activated carbon (AC) is most widely used in the adsorption process due to its chemical and mechanical stability, high degree of surface reactivity and high adsorption capacity (Demirbas, 2004; Azargohar and Dalai, 2005). In fact, the United States Environmental Protection Agency (USEPA) has recommended AC as one of the best available technologies in removing organic pollutants. However, commercially available AC is expensive, and thus researchers have been conducting a lot of studies on the feasibility of using less expensive raw materials, such as from rice husk (Yalcin and Sevinc, 2000), sawdust (Malik, 2004), hard wood (Arriagada *et al.*, 2005), coconut shell (Hu and Srinivasan, 1999; Yang *et al.*, 2010), coffee grounds (Reffas *et al.*, 2010), and sugar-cane (Liou, 2010).

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The Malaysian palm oil industry generates a total of 15 million tonnes of empty fruit bunches (EFB) annually (as reported in 2008), representing one of the most abundant agricultural residues in the country. About 9.69 million and 6.36 million tonnes of EFB per year were produced in Peninsular Malaysia and in Sabah and Sarawak, respectively (Anis *et al.*, 2008). With this availability, there is a huge potential in utilising EFB as a raw material for creating high value-added products. One of the potential products is AC, and much interest has been shown in this area. Several studies have reported on the application of AC made from EFB in phenol adsorption (Alam *et al.*, 2007; 2009; Tan *et al.*, 2009).

The mechanism of phenol adsorption onto AC can be evaluated by processing the adsorption data using equilibrium and kinetic modelling. Langmuir and Freundlich isotherms are among the models that are commonly utilised to understand the adsorption process in liquid phases. The current study was carried out by fitting the experimental data with the isotherm's parameter. Thus, the objective of this study was to determine the phenol adsorption capacity of AC prepared from EFB with different particle sizes. The equilibrium and kinetic data of phenol adsorption were analysed to compare the performance of the prepared AC with that of a commercially available one in removing phenol.

## MATERIALS AND METHODS

### Raw Material and Preparation of Activated Carbon

The oil palm EFB were obtained from the Malaysian Palm Oil Board (MPOB). The length and width of the raw EFB particles were first measured by fibre analysis, and their characteristics were determined. EFB were then air-dried before they were ground and sieved to different mesh sizes: > 2, 0.355-1.0, and < 0.15 mm. These sizes were selected based on the largest, intermediate and the smallest mesh sizes of the sieve. It was expected that the trend of phenol adsorption would be the same between the largest and smallest fibre sizes that were obtained by sieving the fibres. EFB were then carbonised at 700°C, 8°C min<sup>-1</sup> and 1.5 litres min<sup>-1</sup> N<sub>2</sub> flow. The EFB samples were placed in the middle of a ceramic tube set inside a horizontal tube furnace (Carbolite, UK). Metal enclosures were used to seal the inlet and outlet at both ends of the tube furnace. Purified nitrogen gas was flushed into the furnace for 30 min before heating in order to displace O<sub>2</sub> gas. The samples were left for 30 min until the furnace reached a temperature of 700°C when it was switched off, and left to cool down to

room temperature. The N<sub>2</sub> gas was allowed to flow through during the cooling process. The charcoal obtained was weighed to determine the yield percentage. A set of charcoal from different fibre sizes – unsieved (BC1), > 2 mm (BC2), between 0.355 and 1.0 mm (BC3), and < 0.15 mm (BC4) – were stored at 30% relative humidity for comparison with the AC subsequently produced.

Charcoal obtained above was activated at 900°C at a 0.1 litre min<sup>-1</sup> CO<sub>2</sub> flow rate and activation time of 15 min (Alam *et al.*, 2009). Four sets of AC of different fibre sizes were obtained: unsieved (AC1), > 2 mm (AC2), between 0.355 and 1.0 mm (AC3), and < 0.15 mm (AC4). The AC samples produced were also stored at 30% relative humidity, and were heated overnight in the oven at 105°C before use in phenol adsorption analysis. Commercially available AC from Sigma Aldrich with a 20-60 mesh size was used as a standard for comparison in the efficacy tests.

### Batch Adsorption Test

The AC and charcoal samples produced were studied for their efficiency in phenol removal. The commercial AC from Sigma Aldrich was used for comparison. A set of AC (0.05 g of each sample) were added into phenol solutions (100 ml, 0.4 mM) in separate 250-ml conical flasks. The solutions were agitated with a rotary shaker at 165 rpm for 6 hr. The AC and phenol solution mixes were then separated by filtration. The final concentration of the phenol solutions was determined by a UV spectrophotometer according to a standard calibration curve. The amount of phenol adsorbed onto the adsorbents,  $q_t$  (mg g<sup>-1</sup>), was calculated by the mass balance relationship:

$$q_t = (C_o - C_t) \frac{V}{w} \quad (1)$$

where  $V$  is the volume of the solution (l),  $C_o$  is the initial-phase phenol concentration while  $C_t$  is final liquid-phase phenol concentration (mg litre<sup>-1</sup>) at time  $t$ , and  $w$  is the weight of adsorbent (g).

The percentage of phenol removal is determined by the equation:

$$q_t = \frac{C_o - C_t}{C_o} \times 100\% \quad (2)$$

where  $C_o$  and  $C_t$  are the initial and final phenol concentrations (mM) at time  $t$ , respectively.

## RESULTS AND DISCUSSION

### Sample Fibre Size

The particle sizes of the raw material from the different sets of AC and BC are shown in *Table 1*. The

TABLE 1. PARTICLE SIZE OF EMPTY FRUIT BUNCHES (EFB) FIBRE

Sample	AC1/BC1 (unsieved)	AC2/BC2 (> 2 mm)	AC3/BC3 (0.355-1.0 mm)	AC4/BC4 (< 0.15 mm)
Range of fibre length (mm)	1.3-30.1	9.6-30.1	3.3-11.3	1.3-3.8
Average fibre length (mm)	8.18	14.6	8.5	2.7
Range of fibre width (mm)	0.03-1.4	0.19-1.40	0.05-0.61	0.03-0.18
Average fibre width (mm)	0.32	0.54	0.25	0.09

length and width of the original unsieved EFB fibre prepared for this study were between 1.3 and 30.1 mm and from 0.03-1.4 mm, respectively. AC1 and BC1 had average fibre length and fibre width of 14.6 and 0.54 mm, respectively, while the fibre of AC3 and BC3 that passed through a sieve with 0.355-1.00 mm mesh number had average length and width of 8.5 and 0.25 mm, respectively. The samples with the smallest fibre size prepared for this study were AC4 and BC4, which passed through a sieve of less than 0.15 mm mesh number, having an average length and width of 2.7 and 0.09 mm, respectively.

**Adsorption Kinetics**

The equilibrium time and rate of phenol adsorption onto the surface of the AC and charcoal samples with different particle sizes were obtained from an adsorption kinetics study. The changes in phenol concentration with adsorption contact time for the different adsorbents are illustrated in Figure 1. Adsorption reached equilibrium within 2 hr for all the phenol adsorbents. Additionally, the highest rate occurred within the first hour of contact time. This finding is quite consistent with other findings that had tested phenol removal by other carbon materials. Mukherjee *et al.* (2007) found that most of the phenol removal took place within the first hour of phenol-activated carbon contact time. Another study which used maize cob-activated carbon found that a contact time of less than 2 hr

was needed to reach equilibrium (Shaarani and Hameed, 2010).

In the total 7 hr of contact time, the AC of different sizes (AC1-AC4) removed phenol in the range of 55% to 73%, while biochar (BC1-BC4) adsorbed only 14% to 23% of the phenol. This trend is to be expected because AC has a greater pore distribution and greater surface area following activation compared with biochar (Jia and Lua, 2008), as is clearly shown in Figure 2.

On the effect of particle size, AC2 with an average fibre length 14.6 mm and average fibre width of 0.54 mm recorded the highest phenol adsorption (73%) which was slightly higher than that of the commercial AC (68%). In general, the adsorption capacity of phenol decreased in the order of AC2 > AC4 > AC1 > AC3 > BC4 > BC1 > BC2 > BC3. Additionally, phenol concentration reduced substantially within 1 hr of the adsorption process. Phenol concentration decreased from 0.41 to 0.14 mM and from 0.41 to 0.21 mM during the first hour of contact for AC2 and commercial AC, respectively, as shown in Table 2.

The quality and competitiveness between the AC samples produced and the commercial AC on the total phenol adsorbed were also analysed. The percentages of phenol removal are listed in Table 2. It is noteworthy that 68.8% of phenol was removed by AC2 after 2 hr while commercial AC removed 55.43% phenol and took about 7 hr to achieve 68% phenol removal. Unsieved AC and <0.15 mm sized

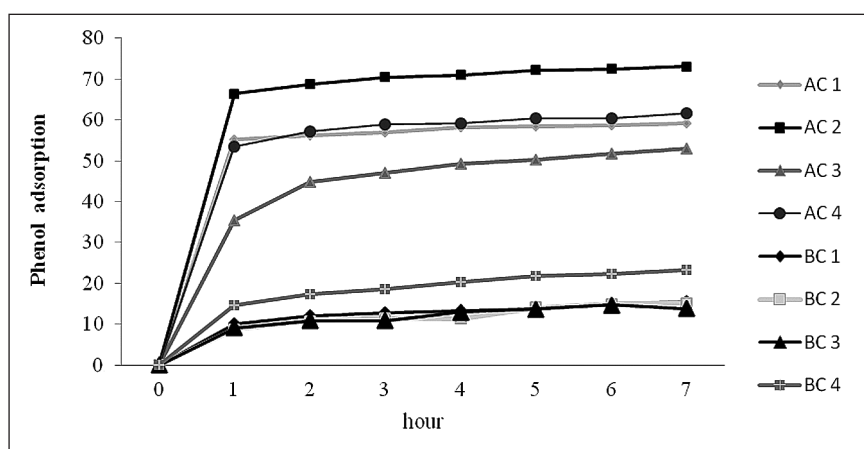


Figure 1. Phenol adsorption by different adsorbents.

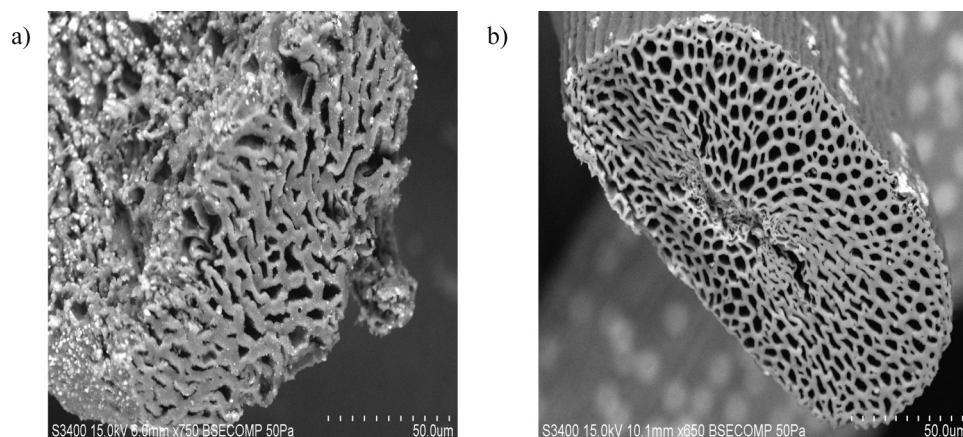


Figure 2. Pore distribution images from a scanning electron microscope. a) Selected biochar sample (B2) and (b) selected AC sample (AC2).

TABLE 2. PHENOL CONCENTRATION AND PHENOL REMOVAL BY ACTIVATED CARBON SAMPLES IN COMPARISON WITH COMMERCIAL ACTIVATED CARBON

Sample/ time (hr)	Concentration (mM)					Removal (%)				
	AC1	AC2	AC3	AC4	CAC	AC1	AC2	AC3	AC4	CAC
0	0.407	0.407	0.407	0.407	0.407	0	0	0	0	0
1	0.182	0.137	0.263	0.189	0.214	55.19	66.44	35.32	53.58	47.38
2	0.178	0.127	0.225	0.174	0.181	56.19	68.80	44.73	57.27	55.43
3	0.175	0.120	0.215	0.168	0.164	56.99	70.56	47.10	58.83	59.63
4	0.170	0.118	0.207	0.166	0.151	58.27	71.05	49.26	59.19	62.84
5	0.169	0.113	0.202	0.161	0.143	58.43	72.29	50.26	60.43	64.96
6	0.168	0.112	0.196	0.161	0.136	58.79	72.53	51.78	60.51	66.56
7	0.166	0.109	0.191	0.156	0.130	59.15	73.17	52.98	61.67	68.00

AC also had an adsorption capacity slightly greater than commercial AC, at the equilibrium time of 2 hr. This trend shows that the AC prepared in this study were better in removing phenol than the commercial AC. This finding indicates that oil palm biomass-based AC is a good choice to replace the expensive commercial one.

Pseudo-first order and pseudo-second order kinetic models were used to analyse the phenol adsorption mechanism. Both kinetic models were used to investigate the adsorption rate mechanism. The pseudo-first order kinetics differential equation is:

$$\frac{dq_t}{dt} = k_1 (q_e - q_t) \tag{3}$$

Integrating the above equation for the boundary conditions  $t = 0$  to  $t$  and  $q_t = 0$  to  $q_t$  will give:

$$\lg(q_e - q_t) = \lg q_e - \frac{k_1 t}{2.303} \tag{4}$$

where  $q_e$  is the amount of phenol adsorbed ( $\text{mg g}^{-1}$ ) at equilibrium,  $q_t$  is the amount of phenol adsorbed ( $\text{mg g}^{-1}$ ) at time  $t$ , while  $k_1$  is the equilibrium rate constant ( $\text{min}^{-1}$ ). A straight line of  $\lg(q_e - q_t)$  against  $t$  was plotted to get the rate constant.

The kinetic data were further analysed with the pseudo-second order kinetic model, using the differential equation:

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \tag{5}$$

Integrating the above equation for the boundary range  $t = 0$  to  $t$  and  $q_t = 0$  to  $q_t$ , and rearranging to get a linear equation will give:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{6}$$

where  $k$  is the equilibrium rate constant [ $\text{g}/(\text{mg}\cdot\text{hr})$ ]. The rate parameters were obtained by plotting a straight line of  $t/q_t$  against  $t$ . The graphs of pseudo-first order and pseudo-second order equations for the AC and biochar samples are shown in Figures 3 and 4, respectively.

The correlation coefficient (R<sup>2</sup>) and linear equations of each adsorbent are shown in Table 3.

The correlation coefficient (R<sup>2</sup>) of the second-order kinetic model were all higher than 0.97. This shows that the experiment results generally fitted the pseudo-second order equations better than the pseudo-first order equations. Furthermore, the values of the calculated equilibrium constant of

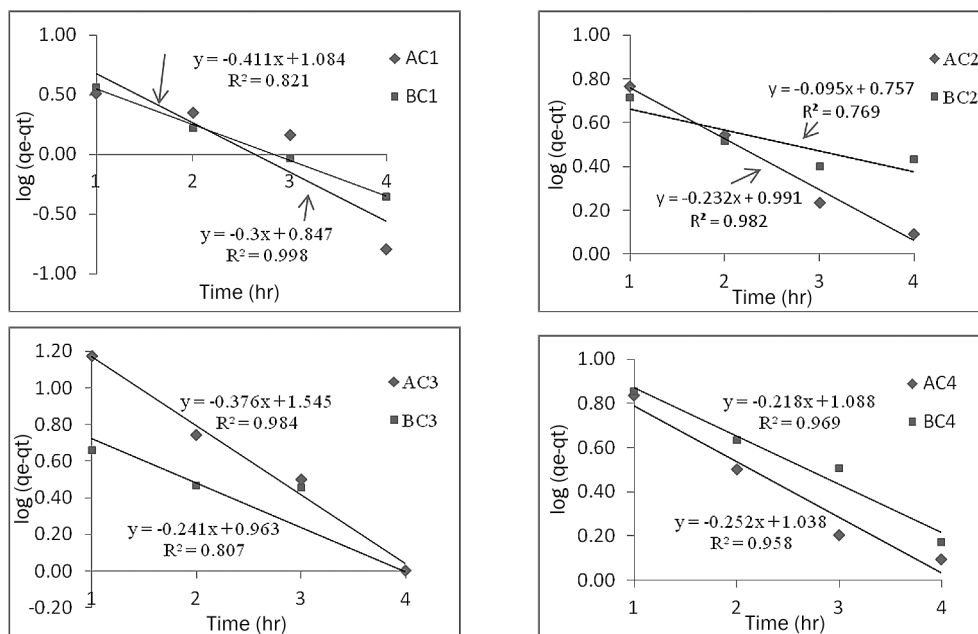


Figure 3. Pseudo-first order kinetics for adsorption of phenol onto empty fruit bunches (EFB)-activated carbon (AC) and charcoal samples.

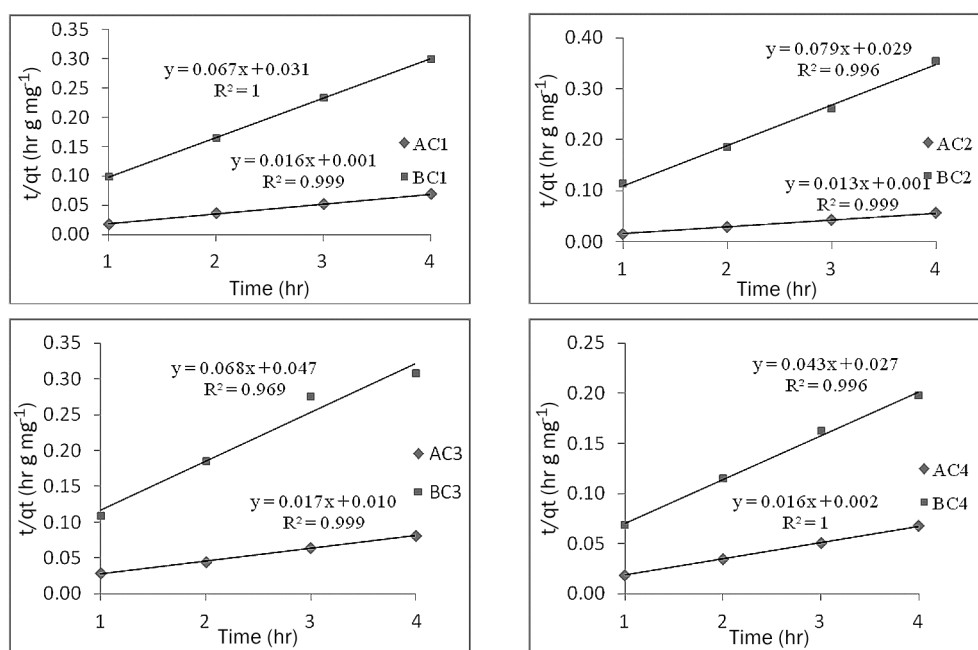


Figure 4. Pseudo-second order kinetics for adsorption of phenol onto empty fruit bunches (EFB)-activated carbon (AC) and charcoal samples.

TABLE 3. PSEUDO-FIRST AND PSEUDO-SECOND ORDER ADSORPTION RATE CONSTANT VALUES FOR PHENOL ADSORPTION ONTO EMPTY FRUIT BUNCHED (EFB)-BASED ACTIVATED CARBON AND BIOCHAR SAMPLES

Sample	Pseudo-first order equation				Pseudo-second order equation		
	$q_e(\text{exp})(\text{mg g}^{-1})$	$k_1$ (hr <sup>-1</sup> )	$q_e(\text{calc})(\text{mg g}^{-1})$	$R_1^2$	$k_2$ (g mg <sup>-1</sup> hr)	$q_e(\text{calc})(\text{mg g}^{-1})$	$R_1^2$
AC1	44.76	0.94	12.13	0.821	0.29	62.50	0.999
AC2	55.38	0.53	9.79	0.982	0.19	76.92	0.999
AC3	38.50	0.87	35.08	0.984	0.04	58.82	0.999
AC4	46.30	0.58	10.91	0.958	0.14	62.50	1.000
BC1	10.55	0.69	7.03	0.998	0.01	14.93	1.000
BC2	10.74	0.22	5.71	0.769	0.18	12.66	0.996
BC3	10.52	0.56	9.18	0.807	0.11	14.71	0.969
BC4	16.63	0.69	12.25	0.958	0.08	23.26	0.996

the pseudo-first order did not agree as closely with the experimental values as they did with those of the pseudo-second order equations. These results indicate that the second-order kinetic model is applicable to the adsorption process of phenol on the prepared AC with the rate of adsorption depending on the square of the phenol concentration. The results obtained are consistent with the findings of others (Fierro *et al.*, 2008; Hameed and Rahman, 2008; Shaarani *et al.*, 2010).

**Adsorption Isotherms**

Adsorption isotherms play an important role in identifying the adsorption capacity of different adsorbents, predicting the model as well as the analysis and design of the adsorption systems. The most frequent models applied in studying the relationship between amounts of adsorbent adsorbed onto the adsorbate are the Freundlich and Langmuir. The correlation coefficient (R<sup>2</sup>) value is used to describe the adsorption process (Alam *et al.*, 2007).

The Freundlich is an isotherm that is used for non-ideal sorption which occurs on the heterogeneous surface of energy systems. It is assumed that the adsorbate will occupy the strong binding sites first and that the strength decreases as the degree of site occupation increases. The general form of the Freundlich isotherm is:

$$q_e = K_F C_e^{1/n} \tag{7}$$

The above equation can be modified to:

$$q_e = \frac{C_o - C_e}{M} = K_F C_e^{1/n} \tag{8}$$

where M is the adsorbent dose (g); K<sub>F</sub> is adsorption capacity and 1/n is the intensity of adsorption. Generally, the value of K<sub>F</sub> is proportional to the adsorption capacity of an adsorbate. Additionally, representation of adsorption favourability is shown by the value of 1/n where n > 1 indicates favourable adsorption conditions. The values of 1/n and K<sub>F</sub> are determined from the slope and intercept, respectively, of the logarithm plot of Equation (7).

$$\ln q_e = \ln K_F + 1/n \ln C_e \tag{9}$$

The values of K<sub>F</sub> and n are listed in Table 4. All the carbon samples gave n values greater than 0. Values of n > 0 for all the experimental data represented the favourable adsorption conditions for this test.

Another isotherm that is generally applied in determining the adsorption isotherm is the Langmuir isotherm. This isotherm is a model for monolayer adsorption and is applied for various adsorption conditions. The isotherm is based on certain assumptions, namely, that there is no transmigration of adsorbate molecules in a plane of the adsorbate surface and that the energy on the adsorbate surface is constant. The linear model is represented as:

$$\frac{C_e}{q_e} = \frac{1}{k_L q_m} + \frac{1}{q_m} C_e \tag{10}$$

where q<sub>e</sub> is the amount of phenol adsorbed at equilibrium (mg g<sup>-1</sup>); C<sub>e</sub> is the phenol equilibrium concentration (mg litre<sup>-1</sup>); k<sub>L</sub> is the Langmuir constant (litre mg<sup>-1</sup>), while q<sub>m</sub> is the maximum adsorption capacity (mg g<sup>-1</sup>). These two values can be obtained from the interception and slope, respectively, of the C<sub>e</sub>/q<sub>e</sub> vs. C<sub>e</sub> graph that is obtained from the linear plot of the experimental data.

The equilibrium parameter or separation factor (R<sub>L</sub>) is an important feature, as this value helps to characterise the Langmuir isotherm. The equilibrium parameter is expressed by the following relationship:

$$R_L = \frac{1}{1 + k_L C_o} \tag{11}$$

The adsorption is characterised as favourable when 0 < R<sub>L</sub> < 1, unfavourable when R<sub>L</sub> > 1, linear when R<sub>L</sub> = 1, and irreversible when R<sub>L</sub> = 0 (Liu *et al.*, 2010). As shown in Table 4, the values of R<sub>L</sub> for AC1 to AC4 were between 0 and 1. This result indicates that the formation process of the carbon material was favourable for phenol removal treatment under the conditions selected in this study.

The values of the correlation coefficient, R<sup>2</sup>, were compared with those of the Freundlich isotherm in order to test the applicability of the Langmuir isotherm. Plots of the linearised Freundlich and Langmuir isotherms for samples AC2 and AC4 are shown in Figures 5 and 6.

TABLE 4. LANGMUIR AND FREUNDLICH CONSTANT VALUES FOR PHENOL ADSORPTION ON EMPTY FRUIT BUNCHES (EFB)-BASED ACTIVATED CARBON

Sample	Langmuir isotherm				Freundlich isotherm		
	q <sub>e</sub> (mg g <sup>-1</sup> )	K <sub>L</sub> (litre mg <sup>-1</sup> )	R <sub>L</sub>	R <sup>2</sup>	K <sub>F</sub>	n	R <sup>2</sup>
AC1	66.67	0.224	0.104	0.951	16.14	2.48139	0.980
AC2	62.50	0.271	0.088	0.985	17.38	2.69542	0.955
AC3	41.67	0.131	0.166	0.966	12.11	3.40136	0.931
AC4	41.67	0.364	0.067	0.992	15.67	3.46021	0.983

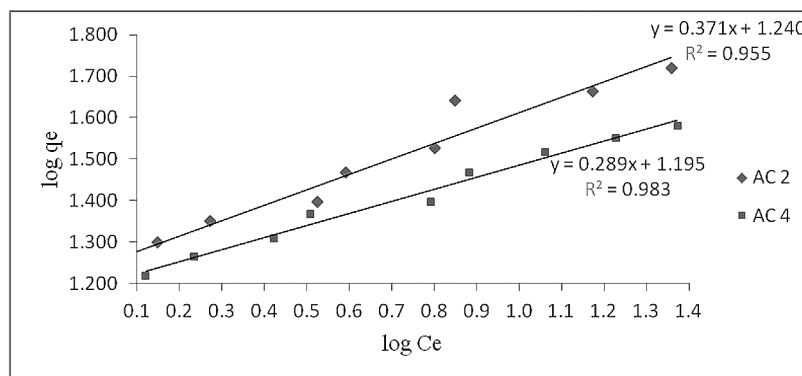


Figure 5. Linearised Freundlich isotherm plots for phenol adsorption by AC2 and AC4.

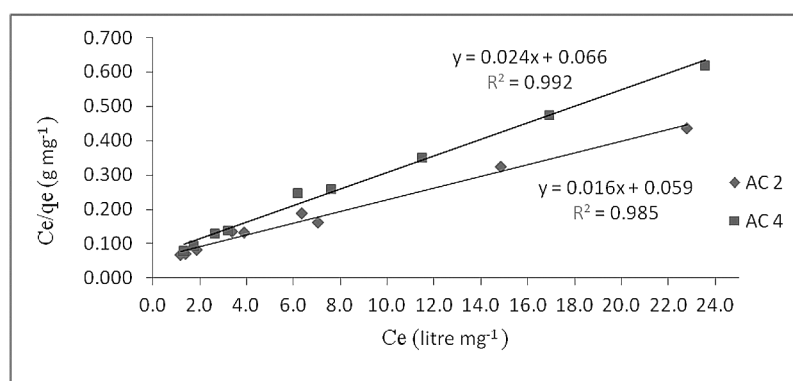


Figure 6. Linearised Langmuir isotherm plots for phenol adsorption by AC2 and AC4.

TABLE 5. COMPARISON OF THE MAXIMUM MONOLAYER ADSORPTION CAPACITY OF PHENOL ONTO VARIOUS ADSORBENTS

Adsorbent	q <sub>o</sub> (mg g <sup>-1</sup> )	Reference
EFB AC	66.67	This work
EFB AC	13.11	Alam <i>et al.</i> (2007)
AC fibre	110.20	Liu <i>et al.</i> (2010)
Hydroxyapatite (HAp) nanopowders	2.9841	Lin <i>et al.</i> (2009)
Beet pulp carbon	90.61	Dursun <i>et al.</i> (2005)
Date-pit activated carbon (DP-AC)	262.3	El-Naas <i>et al.</i> (2010)
Rattan sawdust-based activated carbon	149.25	Hameed and Rahman (2008)
Commercial activated carbon	49.72	Özkaya (2006)

The values of  $R^2$  show that the experimental data better fitted the Langmuir isotherm than the Freundlich isotherm. This indicates the homogenous nature of the AC surface, where AC adsorption had an equal adsorption activation energy for each phenol molecule. Moreover, this result also implies the presence of a monolayer coverage of the phenol molecules on the outer surface of the AC. A similar phenomenon was observed in the adsorption of dye from wastewater using sawdust-based AC (Malik, 2004), and also in the removal of lead by biochar that was produced by the hydrothermal liquefaction method (Liu and Zhang, 2009).

Table 4 shows the Langmuir and Freundlich isotherm parameters. The difference in value of the adsorption coefficients,  $q_e$ , between the isotherm and kinetic values in Table 3 is quite significant. This is due to the different assumptions made between the two models, and this finding is in line with the research on AC-phenol adsorption by Srivastava *et al.* (2006). They found that the value of  $q_e$  for the pseudo-second order equation was 7.0689 while for that for the Langmuir isotherm was 30.2187.

Table 5 shows the comparison of the maximum monolayer adsorption capacity of phenol onto various adsorbents. The best AC prepared in this

study had relatively low yet quite comparable phenol adsorption compared to several other adsorbents reported in the literature.

### CONCLUSION

The research confirmed that AC samples prepared from EFB fibre are more efficient alternatives to commercially available AC for phenol removal. AC with a mesh size greater than 2 mm is the most ideal adsorbent amongst all the AC samples produced as well as the commercial AC. The kinetics of direct phenol adsorption on EFB-based AC follow the pseudo-second order model. The equilibrium data fit well with the Langmuir isotherm which indicates there is monolayer phenol molecule coverage at the outer surface of the activated carbon. A comparison of AC samples produced with commercial AC showed that the AC produced are comparable with the commercial one. Future studies will focus on determining the effects of other process conditions, such as temperature, heating rate and residential time, on the production of EFB-based AC for phenol removal and also for other applications.

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