



Activated carbons derived from oil palm empty-fruit bunches: Application to environmental problems

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Abstract

Activated carbons derived from oil palm empty fruit bunches (EFB) were investigated to find the suitability of its application for removal of phenol in aqueous solution through adsorption process. Two types of activation namely; thermal activation at 300, 500 and 800°C and physical activation at 150°C (boiling treatment) were used for the production of the activated carbons. A control (untreated EFB) was used to compare the adsorption capacity of the activated carbons produced from these processes. The results indicated that the activated carbon derived at the temperature of 800°C showed maximum absorption capacity in the aqueous solution of phenol. Batch adsorption studies showed an equilibrium time of 6 h for the activated carbon at 800°C. It was observed that the adsorption capacity was higher at lower values of pH (2–3) and higher value of initial concentration of phenol (200–300 mg/L). The equilibrium data fitted better with the Freundlich adsorption isotherm compared to the Langmuir. Kinetic studies of phenol adsorption onto activated carbons were also studied to evaluate the adsorption rate. The estimated cost for production of activated carbon from EFB was shown in lower price (USD 0.50/kg of activated carbon) compared the activated carbon from other sources and processes.

Key words: activated carbon; adsorption; lignocellulosic empty fruit bunches; phenolic compound; solid waste management

Introduction

Phenols are a common pollutant found in the effluents from pesticides, pharmaceuticals, petrochemicals, pulp and paper, paint and other industries (Kim *et al.*, 2002; Rao and Viraraghavan, 2002; Aleksieva *et al.*, 2002; Jung *et al.*, 2001; Diez *et al.*, 1999). Because of improper treatment of these toxic materials from such industrial effluents they have widely contaminated soil, ground water and receiving water bodies, and their toxicity seriously affects living microorganisms.

Treatment of phenolic compounds from industrial effluents by adsorption technique using activated carbon is an alternative process compared to other conventional processes (Tancredi *et al.*, 2004; Nakagawa *et al.*, 2004; Kim *et al.*, 2001; Aksu and Yener, 1998; Gupta *et al.*, 2000; Chen *et al.*, 2002). Production of activated carbon however increases the cost which is a drawback of the process. Therefore methods are needed to minimize the cost of production through utilizing cheap and abundant local raw materials which may reduce solid waste pollution as well as decrease the production cost of activated carbon.

In Malaysia oil palm (OP) industries are producing huge quantities of non-oil palm biomass of about 90×10^6 t of lignocellulosic biomass each year, of which about 40×10^6 t are the form of empty fruit bunches (EFB), oil palm trunks (OPT) and oil palm fronds (OPF) (MPOB, 2003). The EFB

represents about 9% of this total solid waste production. At present, the biomass is either left at the plantation to provide organic nutrients to the oil palm trees or burned illegally or used as solid fuel in the boiler to generate steam or electricity at the mills (Jaafar and Sukaimi, 2001).

No research to our knowledge has been conducted to investigate the production of activated carbon from EFB for the adsorption of phenol from aqueous solution. However, there have been a considerable number of studies during the last decade on the production of activated carbons from agricultural by-products (coconut tree sawdust, corn cob, banana pith, rice husk, rice straw, sugarcane bagasse), oil palm stone, olive waste cake, sewage sludge, paper mill sludge and municipal waste for different applications especially in industrial wastewater treatment (Bacacoui *et al.*, 2001; Gua and Lua, 2000; Kadirvelu *et al.*, 2003; Rozana *et al.*, 2003; Khalili *et al.*, 2002; Ahmedna *et al.*, 2000; Nagano *et al.*, 2000). Currently OP industries are also generating palm oil mill effluent (POME) of about 10×10^6 t which contain phenolic compounds (MPOB, 2003). The activated carbon derived from EFB could be a potential application for the removal and/or recovery of phenolic compounds in POME that would be useful for manufacturing industries especially nylon and epoxy-adhesives. Therefore the present study was undertaken to produce activated carbons by thermal and physical activation process utilizing EFB as abundant local raw material for application in efficient phenol removal.

1 Materials and methods

1.1 Sample collection

The EFB sample used was collected from a palm oil industry (Seri Ulu Langat Palm Oil Mill Sdn. Bhd.) in Dengkil, Selangor, Malaysia, in plastic bags and stored in the laboratory cold room at 4°C for use.

1.2 Pre-treatment of sample and production of activated carbon

The EFB samples were dried at 105°C for 24 h in the oven to remove the moisture content until constant weight. Two types of activation: physical and thermal activation were conducted to produce activated carbon. For thermal activation, the processed samples were activated in the high temperature chamber furnace (Carbolite, UK) at 300, 500 and 800°C for 30 min followed by crushing and sieving to size fraction of less than 150µm. For physical activation, the raw material was boiled in distilled water at 150°C for 2 h in an oven and the treated sample was dried at 105°C until constant weight. The dried samples of untreated EFB and activated carbon by physical treatment were crushed and sieved to the same size fractions of the thermal activated carbons. The activated carbons were identified as the EFB C for inactivated (control, untreated EFB), EFB B for boiling treatment, and EFB 300, EFB 500 and EFB 800 for the thermal treatment at 300, 500 and 800°C, respectively. All activated carbons were stored at 4°C until further use for adsorption tests.

1.3 Batch adsorption test

The activated carbons prepared from EFB were studied for removal efficiency of phenol from aqueous solution under different experimental conditions. 0.25 g of each activated carbon produced was added into 100 ml conical flasks containing 25 ml aqueous solution of phenol at pH 3 with concentration of 50 mg/L. The samples were agitated in a rotary shaker at temperature of 30°C for 24 h at 150 r/min. At 1, 2, 3, 4, 5, 6, 12, 18 and 24 h interval of time, samples (five different activated samples) were withdrawn to determine the residual concentration of phenol and its equilibrium time. The activated carbon and treated solutions were then separated by filtration. The total phenol content in solution was determined according to the method suggested by Box (1983). The absorbance was measured at 725 nm against reagent blank using a UV spectrophotometer (Anthelie Junior, SECOMAM). The final concentration was measured from the standard calibration curve.

The same experimental conditions (contact time 6 h, adsorbent dose 0.25 g, and agitation 150 r/min) were studied for optimum pH and concentration varying with the pH values of 2, 3, 5 and 8, and concentration of 25, 50, 100, and 200 mg/L respectively. All the data presented are the average of three replicates.

The adsorption capacity, q , was calculated from the difference between the initial and equilibrium adsorbate (phenol) concentration using the relationship:

$$q = \frac{(C_0 - C_e)}{M} V \quad (1)$$

where q is the adsorption capacity (mg/g); C_0 and C_e are the initial and equilibrium concentration (mg/L) respectively; M is the adsorbent dosage (g) and V is the solution volume (L).

2 Results and discussion

2.1 Activated carbons and its phenol released in aqueous solution

Four types of activated carbons were produced from EFB with three from thermal activation at 300, 500 and 800°C, and one from boiling treatment at 150°C. Activated carbon produced from boiling treatment, and control were similar in color (brown) while produced from the heat treatment, 300, 500 and 800°C were black in color.

Since the activated carbons also contained phenol from the EFB used in aqueous solution, the phenol released rate was determined to evaluate the actual adsorption capacity of each adsorbent. The results showed that only activated carbons produced from EFB at temperatures of 500°C (EFB 500) and 800°C (EFB 800) was releasing very low concentrations (1–2 mg/L) of phenol into distilled water (Table 1). The concentration of phenol released were observed to be in the order of EFB C>EFB 300>EFB B>EFB 500>EFB 800. The releasing rate was increased for all samples except for EFB C where the concentration of phenol released decreased after 12 h. The releasing rate was higher during the first hour of operation after that the rate was slow until 24 h of treatment for most of the cases.

Table 1 Concentration of phenol released by various activated carbons produced from EFB in aqueous solution

Time (h)	Concentration of phenol released (mg/L)				
	EFB C	EFB B	EFB 300	EFB 500	EFB 800
1	24.64	7.44	10.82	0.91	0.52
2	24.96	7.50	10.99	0.99	0.54
3	25.28	7.56	11.16	1.11	0.60
4	25.61	7.62	11.33	1.29	0.64
5	25.93	7.68	11.50	1.46	0.66
6	26.25	7.74	11.66	1.54	0.69
12	28.19	8.11	12.67	1.62	0.73
18	27.46	8.47	13.68	1.71	0.79
24	26.69	8.84	14.69	1.87	0.83

2.2 Adsorption test

2.2.1 Effect of contact time

The adsorption capacity of phenol were observed to increase in the order of EFB 800>EFB 500>EFB 300> EFB C>EFB B as shown Fig.1. The concentration of phenol treated by activated carbon at temperature 800°C (EFB 800) was decreased substantially from initial concentration of 50 to 15.2 mg/L within 1 h of operation.

The percentage removal during the first hour was around 69.6% at the adsorption capacity of about 3.5 mg/g. For EFB 500, the initial concentration of 50 mg/L was

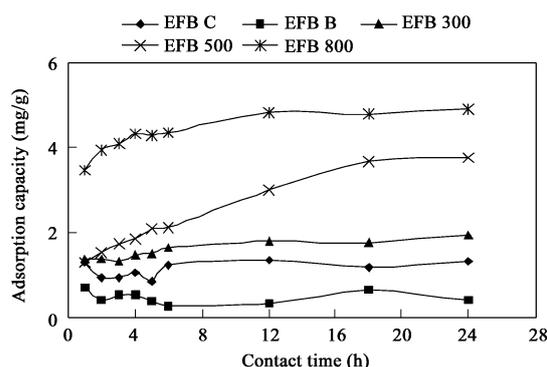


Fig. 1 Variation of contact time on the adsorption capacity of activated carbons produced from EFB in aqueous solution of phenol. Adsorbent dosage: 1% w/v; initial concentration: 50 mg/L; temperature: 30°C; pH 3; agitation: 150 r/min.

decreased to 37.2 mg/L during the first hour, while the adsorption capacity and percentage removal were 1.3 mg/g and 25.7% respectively. The final concentration of phenol adsorbed by both samples reached equilibrium point within 6 h of operation. The activated carbons EFB C, EFB B and EFB 300 have reduced the phenol concentrations of 25.5%, 14.1% and 27.8% respectively from an initial concentration of 50 mg/L during the first hour of operation. Thermal activated EFB carbons showed better adsorption capacity compared to the untreated EFB (EFB C) and boiling treatment (EFB B). Only two activated carbons, EFB 500 and EFB 800 were selected for further optimization of pH and concentration of phenol solution with the equilibrium time of 6 h since the two samples released very small phenol concentrations in aqueous solution.

2.2.2 Effect of initial pH

The variation of pH on adsorption capacity of phenol by activated carbons is shown Fig.2. The adsorption capacity for EFB 500 and EFB 800 was decreased from 2.7 to 1.8 mg/g and 4.5 to 4.2 mg/g respectively when the initial pH of the aqueous solution was increased from pH 2 to 5. The phenol content remained constant in the pH range of 2 to 3. However, adsorption capacity for both carbons was increased to 2.1 mg/g for EFB 500 and 4.4 mg/g for EFB 800 when the pH was further increased from pH 5 to 8. It may be related to the presence of excess H^+

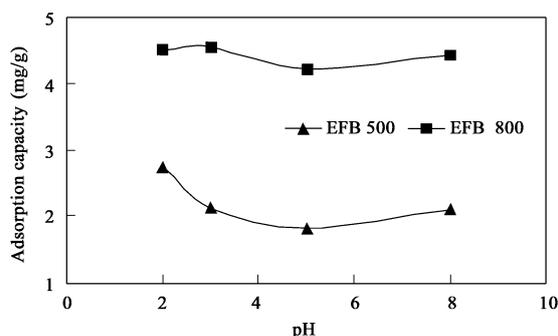


Fig. 2 Removal of phenol by activated carbons (EFB 500 and EFB 800) with the variation of pH. Adsorbent dosage: 1% w/v; concentration: 50 mg/L; temperature: 30°C; agitation: 150 r/min; contact time: 6 h.

ions accelerating the removal of the phenol with the anion OH^- in the aqueous solution. It is also possible that the surface properties of the activated carbons are also depend on the pH of the solution. Similar trend was observed in the sorption of phenol, *o*-chlorophenol and *p*-chlorophenol (pH 1–3) by dried activated sludge in studies by Aksu and Yener (1998).

2.2.3 Effect of initial phenol concentrations

The effect of initial phenol concentrations (25–200 mg/L) on adsorption capacity by the activated carbons, EFB 500 and EFB 800 is shown in Fig.3. Activated carbon EFB 800 had a higher adsorption capacity which varied between 2.7 and 10.8 mg/g at initial phenol concentrations of 25 to 200 mg/L. EFB 500 had an adsorption capacity between 1.4 and 6.6 mg/g for the same conditions. The increase in adsorption capacity of adsorbent may be due to the higher probability of collision between phenol and adsorbent.

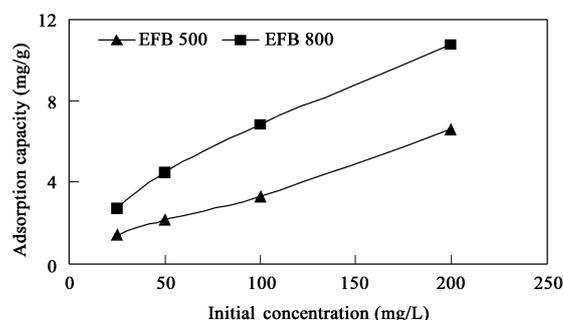


Fig. 3 Effect of initial phenol concentration on adsorption by activated carbons. Adsorbent dosage: 1% w/v; temperature: 30°C; pH 3; agitation: 150 r/min; contact time: 6 h.

Increasing the adsorbate concentration generally caused a decrease in adsorption capacity. Lower initial concentration also favored higher adsorption yield which was the limited to 200 mg/L in this study. Further higher concentration (>200 mg/L) may reduce the adsorption capacity with the activated carbon (Aksu and Yener 1998).

2.3 Adsorption isotherms

The Freundlich isotherm has been widely adopted to characterize the adsorption capacity of organic pollutants using different adsorbents by fitting the adsorption data. The Freundlich isotherm has the general form such as:

$$q_e = K_F C_e^{1/n} \quad (2)$$

This equation can be modified as:

$$q_e = \frac{C_0 - C_e}{M} = K_F C_e^{1/n} \quad (3)$$

where q_e is the adsorption capacity (mg/g); C_0 and C_e are the initial and equilibrium concentration (mg/L) respectively; M is the adsorbent dose (g); K_F and $1/n$ are the adsorption capacity and intensity of adsorption respectively. The values of K_F and $1/n$ can be respectively determined from the intercept and slope of the logarithmic

plot in Equation (3).

$$\ln q_e = \ln K_F + 1/n \ln C_e \quad (4)$$

In order to facilitate the estimation of the adsorption capacities at various conditions, the Langmuir adsorption isotherm, a typical model for monolayer adsorption was applied. The linearized Langmuir model can be written as

$$C_e/q_e = \frac{1}{K_L q_m} + \frac{1}{q_m} C_e \quad (5)$$

where q_e is the amount of phenol adsorbed at equilibrium (mg/g); C_e is the equilibrium concentration of phenol (mg/L); K_L (L/mg) and q_m (mg/g) are the Langmuir constant and maximum adsorption capacity. The constants q_m and K_L can be determined from the intercept and slope of the linear plot of the experimental data of C_e/q_e vs. C_e .

The applicability of the isotherm equations is compared by judging the correlation coefficients, R^2 . These adsorption isotherms were made based on the equilibrium data obtained from phenol adsorption starting at different initial adsorbate concentrations (25–200 mg/L).

In Freundlich, a plot of $\ln q_e$ vs. $\ln C_e$ from Fig.4 enables the empirical constant K_F and $1/n$ to be determined from the intercept and slope of the linear regression is shown in Table 2. For Langmuir, a plot of C_e/q_e vs. C_e from Fig.5, q_m and K_L can be determined from its slope and intercept as shown in Table 2.

Table 2 Langmuir and Freundlich isotherms parameters (EFB 500 and EFB 800) for phenol adsorption by activated carbon

Parameter	Langmuir isotherms	Parameter	Freundlich isotherms
Activated carbon (EFB 500)			
q_m (mg/g)	13.11	$1/n$	0.69
K_L (L/mg)	0.007	K_F (mg/g)(mg/L) ⁿ	0.22
R^2	0.7958	R^2	0.9874
Activated carbon (EFB 800)			
q_m (mg/g)	11.61	$1/n$	0.30
K_L (L/mg)	0.122	K_F (mg/g)(mg/L) ⁿ	2.79
R^2	0.9799	R^2	0.9988

The correlation coefficient showed that the experimental data were better fitted in the Freundlich isotherm ($R^2=0.9988$ for EFB 800 and 0.9874 for EFB 500) than the Langmuir isotherm ($R^2=0.9799$ for EFB 800 and 0.7958 for EFB 500). The Freundlich equation was more suitable when a change in the mechanism of adsorption with concentration was considered. The Langmuir model equation is used for homogenous surfaces while the Freundlich isotherm model assumes neither homogeneous site energies nor limited levels of adsorption and can result from the overlapping patterns of several Langmuir-type adsorption phenomena occurring at different sites on complex adsorbents (Calace *et al.*, 2002).

2.4 Adsorption kinetics

In order to investigate the mechanism of adsorption rate for the adsorption of phenol by activated carbons derived from EFB, the rate constants were determined

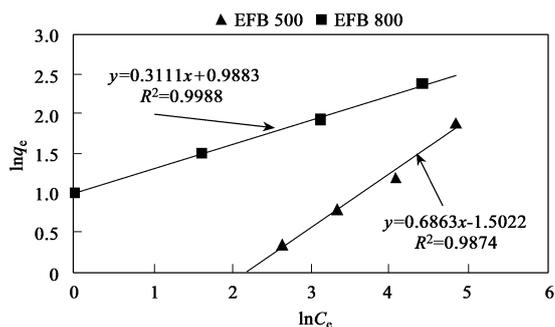


Fig. 4 Linearized Freundlich adsorption isotherm of phenol. Adsorbent dosage: 1% w/v; temperature: 30°C; pH 3; agitation: 150 r/min.

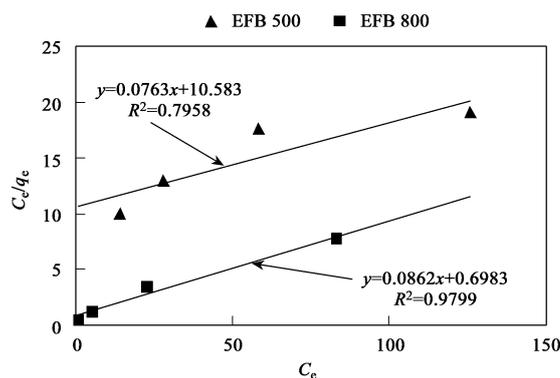


Fig. 5 Linearized Langmuir adsorption isotherm of phenol. Adsorbent dosage: 1% w/v; temperature: 30°C; pH 3; agitation: 150 r/min.

by applying the equations of Lagergren (1989), a pseudo first order mechanism and Ho (1995), a pseudo second order mechanism respectively. The differential equation of a pseudo first order kinetic is as follows:

$$\frac{dq_t}{dt} = K_1(q_e - q_t) \quad (6)$$

Integrating Equation (6) for the boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_t$, gives:

$$\lg(q_e - q_t) = \lg q_e - \frac{K_1 t}{2.303} \quad (7)$$

where q_e is the amount of phenol adsorbed at equilibrium (mg/g); q_t is the amount of phenol adsorbed at time t (mg/g) and K_1 is the equilibrium rate constant h^{-1} . In order to obtain the rate constant the straight line plot of $\lg(q_e - q_t)$ versus t for different treatment conditions was analyzed. The differential equation of a pseudo-second order kinetic is as follows:

$$\frac{dq_t}{dt} = K(q_e - q_t)^2 \quad (8)$$

Integrating Equation (8) for the boundary conditions, $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_t$, rearranging the final equation as:

$$\frac{t}{q_t} = \frac{1}{K q_e^2} + \frac{t}{q_e} \quad (9)$$

where K is the equilibrium rate constant ($g/(mg \cdot h)$). The straight line plot of t/q_t versus t was drawn to obtain rate parameter.

The constant adsorption parameters K_1 and K for pseudo first and second order kinetics were determined from the plots of $\lg(q_e - q_t)$ against time t , and the plot of t/q_t against time t are presented in Fig.6 and Fig.7. The constant parameters and regression coefficients (R^2) are shown in Table 3. The results indicated the straight lines which showed the applicability of first- and second-order rate expression. The correlation coefficient showed that the second order equation ($R^2=0.9997$) was better fitted by the experimental results compared to the first order equation ($R^2=0.8883$) for activated carbon produced from POME at activation temperature of 800°C.

The constant parameter K_1 in first order kinetics indicates the phenol adsorbed per gram of adsorbent for every hour of operation. The results indicated that 0.827 mg of phenol was adsorbed by each gram of activated carbon EFB 800 for every hour of operations compared to the activated carbon EFB 500 (0.71 mg). While the constant parameter K for second order kinetics equation indicates the amount of adsorbent needed in gram per unit of phenol in each hour of operations. 3.66 g of EFB 500 was needed

Table 3 Pseudo first- and second-order equation parameters (EFB 500 and EFB 800) for adsorption of activated carbon in aqueous solution of phenol

Parameter	Pseudo-first order equation	Parameter	Pseudo-second order equation
Activated carbon (EFB 500)			
K_1 (h ⁻¹)	0.71	K (g/(mg·h))	3.664
R^2	0.8153	R^2	0.9829
Activated carbon (EFB 800)			
K_1 (h ⁻¹)	0.827	K (g/(mg·h))	0.652
R^2	0.8883	R^2	0.9997

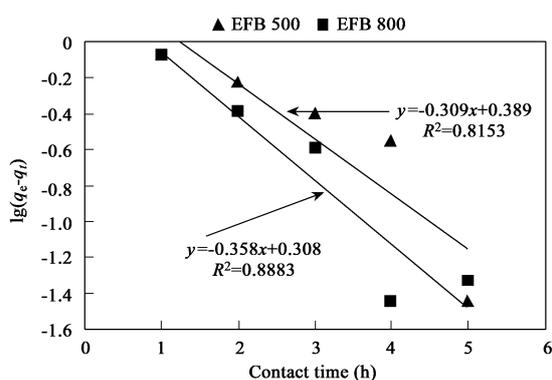


Fig. 6 Pseudo first order kinetic and second order kinetic of phenol onto activated carbons EFB 500 and EFB 800. Adsorbent dosage: 1% w/v; temperature: 30°C; pH 3; agitation: 150 r/min.

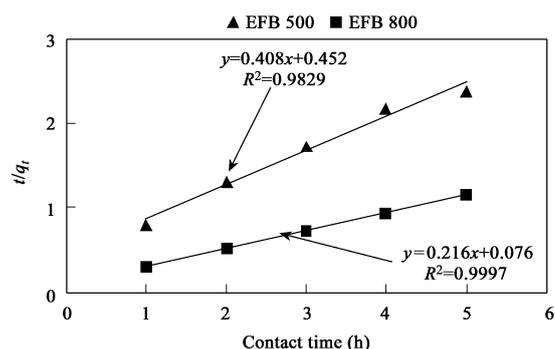


Fig. 7 Pseudo second order kinetic and second order kinetic of phenol onto activated carbons EFB 500 and EFB 800. Adsorbent dosage: 1% w/v; temperature: 30°C; pH 3; agitation: 150 r/min.

to adsorb per mg of phenol in 1 h of operation while only 0.65 g was required for EFB 800.

Bharvava and Sheldarkar (1993) produced unripped tamarind nut shell activated carbon (TNSAC) and rinsed TNSAC for adsorption studies and the experimental data was observed to fit Lagergren equation with the regression coefficient of 0.957 and 0.967 respectively. First order equation of Lagergren did not fit well for the whole range of contact time and was generally applicable over the initial 20–30 min of sorption process. The second order equation is based on sorption capacity on the solid phase. It predicts the behavior over the whole range of studies supporting a second order kinetics and is in agreement with chemisorptions being the rate controlling step (Ho and Makay, 1998; Ho and Chiang, 2001).

2.5 Estimated production cost of powdered activated carbon from EFB

Costs were developed based on 15% yield or 1500 kg/d output of powdered activated carbon, 320 d per year of production, 6 men per two shifts of 6 h. Production of 480000 kg/a of powdered activated carbon by thermal activation requires a fixed capital investment of USD 635000 and an annual operating cost of USD 175200 are shown in Table 4. The estimated cost for powdered activated carbon from EFB is USD 0.50/kg. The estimated cost for several activated carbons from different treatment processes has been determined by the Ng *et al.* (2003). The study has identified the estimated costs of USD 2.72/kg and USD 3.12/kg for pecan shell-based activated carbon and sugar cane-based granular activated carbon by steam activation. The activated carbon from EFB by thermal activation was cheaper due to the reason of lower cost of labor and utilities

Table 4 Estimated capital costs for thermal activation of empty fruit bunches (EFB)

Item	Estimate capital (USD)	Item	Annual operating cost (USD)
Plant and machinery	550000	Raw materials	Free (on site)
Buildings	75000	Utilities (steam water, natural gas and electricity)	80000
Contingencies	10000	Labor (operating and maintenance)	60000
Total capital costs	635000	Supplies	20000
		General works	15000
		Depreciation ^a	63500
		Total annual operating costs	238500

^aCapital costs (Table 1) divided by an economic life of 10 years, rounded off.

in Malaysia compared to USA. In addition the raw material (EFB) was free of cost even though negative cost since it was considered as waste.

3 Conclusions

Three activated carbons were derived from thermal activation at 300, 500 and 800°C and one from physical activation (boiling treatment at 150°C). The results revealed that the activated carbons at thermal activation of 500 and 800°C showed good adsorption capacity with very low content of phenol released in aqueous solution compared to others. It was observed that the adsorption capacity was higher at lower values of pH (2–3) and higher value of initial concentration of phenol (200–300 mg/L). The correlation coefficient showed that the Freundlich isotherm ($R^2 = 0.9988$ and 0.9874 for EFB 800 and EFB 500, respectively) and pseudo second order kinetic ($R^2 = 0.9997$ and 0.9829 for EFB 800 and EFB 500, respectively) were better fitted than the Langmuir isotherm ($R^2 = 0.9799$ and 0.7958 for EFB 800 and EFB 500, respectively) and pseudo first order kinetic ($R^2 = 0.8883$ and 0.8153 for EFB 800 and EFB 500, respectively). Overall, EFB 800 was found to be more efficient in removal of phenol compared to EFB 500. The activated carbon EFB 800 was identified to be promising for the application of phenol removal from industrial wastewaters that could be encouraged for use in waste management for resource recovery and reuse.

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